REVISED FINAL
Remedial Action Work Plan

Former Charlotte Naval Ammunition Depot
Charlotte, North Carolina

December 2011

Revision 1 – February 2012

Revision 2 – May 2012

DERP-FUDS Project No. I04NC080301
Contract No.:W912DY-10-D0025
Delivery Order No.: 0007

PREPARED FOR:

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REVISED FINAL
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Former Charlotte Naval
Ammunition Depot
Charlotte, North Carolina

Prepared for:
U.S. Army Corps of Engineers,
Huntsville Center

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Our Reference:
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Acronyms and Abbreviations

ASIP	Arrowood Southern Industrial Park
APP	Accident Prevention Plan
ARAR	Applicable or Relevant and Appropriate Requirement
bgs	Below Ground Surface
BRA	Baseline Risk Assessment
CBP	Commerce Business Park
CERCLA	Comprehensive Environmental Response, Compensation, & Liability Act
CFR	Code of Federal Regulations
cis-DCE	Cis-1,2-dichloroethene
CNAD	Charlotte Naval Ammunition Depot
COD	Chemical Oxygen Demand
CQCP	Construction Quality Control Plan
CSM	Conceptual Site Model
CVOC	Chlorinated Volatile Organic Compound
DID	Data Item Description
DMP	Data Management Plan
DO	Dissolved Oxygen
EQ	Equalization Tank
ERD	Enhanced Reductive Dechlorination
ESA	Environmental Site Assessment
FFS	Focused Feasibility Study
FSP	Field Sampling Plan
GPM	Gallons per Minute
HSU	Hydrostratigraphic Unit
IDW	Investigation-Derived Waste

IRZ	In-situ Reactive Zone
JV	Joint Venture
M&E	Metcalf and Eddy, Inc.
MCL	Maximum Contaminant Level
mm	Millimeter
MNA	Monitored Natural Attenuation
NCAC	North Carolina Administrative Code
NCDENR	North Carolina Department of Environment and Natural Resources
NCP	National Oil and Hazardous Substances Pollution Contingency Plan
O&M	Operation and Maintenance
ORP	Oxidation-Reduction Potential
OSWER	Office of Solid Waste and Emergency Response (USEPA)
PE	Professional Engineer
PIKA	PIKA International, Inc.
PIKA-PIRNIE JV Team	PIKA International, Inc. (PIKA)/Malcolm Pirnie, Inc. (Pirnie) Joint Venture (JV), LLC
Pirnie	Malcolm Pirnie, Inc.
PMP	Project Management Plan
PVC	Polyvinyl Chloride
PWR	Partially Weathered Rock
PWS	Performance Work Statement
QAPP	Quality Assurance Project Plan
RAO	Remedial Action Objective
RAR	Remedial Action Report
RAWP	Remedial Action Work Plan
RI	Remedial Investigation

ROI	Radius of Influence
SAIC	Science Applications International Corporation
SAP	Sampling and Analysis Plan
SARA	Superfund Amendments and Reauthorization Act
SSHP	Site Safety and Health Plan
TCE	Trichloroethylene
TOC	Total Organic Carbon
µg/L	Micrograms per Liter
UIC	Underground Injection Control
U.S.	United States
USACE	United States Army Corps of Engineers, Huntsville Center
USEPA	United States Environmental Protection Agency
VC	Vinyl Chloride
VOC	Volatile Organic Compound
WERS	Worldwide Environmental Remediation Services

1. Introduction

In accordance with the Performance Work Statement (PWS) dated June 7, 2011, revised July 20, 2011 (USACE, 2011b), the PIKA International, Inc. (PIKA)/Malcolm Pirnie, Inc. (Pirnie) Joint Venture (JV), LLC (the PIKA-PIRNIE JV Team) has developed this Remedial Action Work Plan (RAWP) for the Remedial Action at the Former Charlotte Naval Ammunition Depot (CNAD) in Charlotte, North Carolina. This document was prepared under the Worldwide Environmental Remediation Services (WERS) Contract Number W912DY-10-D0025, Delivery Order Number 0007, administered by the United States (U.S.) Army Corps of Engineers, Huntsville Center (USACE) in accordance with the PWS and all applicable USACE guidance and Data Item Descriptions (DIDs), Federal, North Carolina Department of Environment and Natural Resources (NCDENR), and local regulations.

The PIKA-PIRNIE JV Team has been tasked under the PWS to implement the selected remedy, as presented in the Decision Document (USACE, 2011a) approved on April 18, 2011, for the cleanup of contaminated groundwater at the Former CNAD complex. This RAWP provides a detailed description of the remedial action and technical approach. Specifically, in-situ enhanced reductive dechlorination (ERD) will be used to stimulate biological degradation of chlorinated volatile organic compounds (CVOCs) in groundwater within the impacted water bearing units that underlie the Former CNAD complex.

1.1 Site Location

The Former CNAD complex is located in Charlotte, Mecklenburg County, North Carolina (see **Figure 1-1**). At the time of operation, the entire CNAD complex encompassed approximately 2,266 acres of land. This area, now known as the Arrowood Southern Industrial Park (ASIP), is currently occupied by light industrial and commercial businesses.

The Former CNAD complex is bound by Cordage Street to the north, ASIP Building III to the east, Frito-Lay, Inc. to the south, and Nevada Boulevard to the west.

1.2 Basis of Design

As detailed in the Decision Document, the selected remedy for the Former CNAD complex includes active ERD treatment followed by passive monitored natural attenuation (MNA), and post-remedial monitoring to ensure long-term compliance with Remedial Action Objectives (RAOs) for select trichloroethene (TCE) in groundwater.

Consistent with the fate and transport modeling presented as part of the *Focused Feasibility Study* (FFS) (Science Applications International Corporation [SAIC], 2009), the projected clean-up duration to achieve RAOs following implementation of the selected remedy is 14 years.

The remedial technical approach involves implementing active ERD treatment in areas where TCE concentrations are present at concentrations greater than 500 micrograms per liter (µg/L), followed by MNA to achieve the long-term remedial goals. The active ERD treatment period will include periodic injections of a carbohydrate solution to provide an organic carbon source to increase the rate at which naturally occurring bacteria breakdown TCE under anaerobic conditions. The carbohydrate solution injected in the treatment area will consist of dilute molasses. The remedial approach includes subsurface injections to address TCE impacts in the transition zone and bedrock hydrostratigraphic units (HSUs). The hydrogeology and nature and extent of TCE impacts in each of these HSUs are further detailed in Section 2 of this RAWP.

1.3 RAOs

RAOs are the desired outcome of the groundwater clean-up action. The following RAOs for the Former CNAD complex, as specified in the PWS, include:

- Address TCE at concentrations exceeding 500 µg/L in groundwater through enhanced bioremediation using a dilute molasses solution injections in both the transition and bedrock zones.

- MNA to reduce volatile organic compound (VOC) concentrations to below the North Carolina Administrative Code (NCAC) 2L standard 2.8 µg/L;

The RAOs described in this RAWP focus on the design, construction, implementation, and operation of the in-situ ERD remedial system.

1.4 Work Plan Organization

This RAWP consists of the following sections and guidance documents, provided as appendices:

1. **Introduction** – Section 1 presents the purpose of the report, basis of design, RAOs, and the report organization.

2. **Conceptual Site Model (CSM)** – Section 2 presents background information, summarizes previous investigations, and provides site-specific characteristics for the Former CNAD complex.

3. **Remedial Design/Remedial Action –** Section 3 defines the remedial design and construction activities necessary for the implementation of the selected remedy.

4. **Remedial Action Monitoring** – Section 4 presents the monitoring well installation details and monitoring programs for the active, passive, and post-remedial groundwater monitoring phases.

5. **Reporting –** Section 5 presents a summary of the reports that will be submitted during the remedial action.

6. **Schedule** – Section 6 presents the proposed schedule for implementation of the remedial action.

7. **References** – Section 7 lists the references used in this document.

Appendices:

A. Design Drawings

B. Accident Prevention Plan (APP) and Site Safety and Health Plan (SSHP)

C. Sampling and Analysis Plan (SAP) (including Field Sampling Plan [FSP], Quality Assurance Project Plan [QAPP] , and Data Management Plan [DMP])

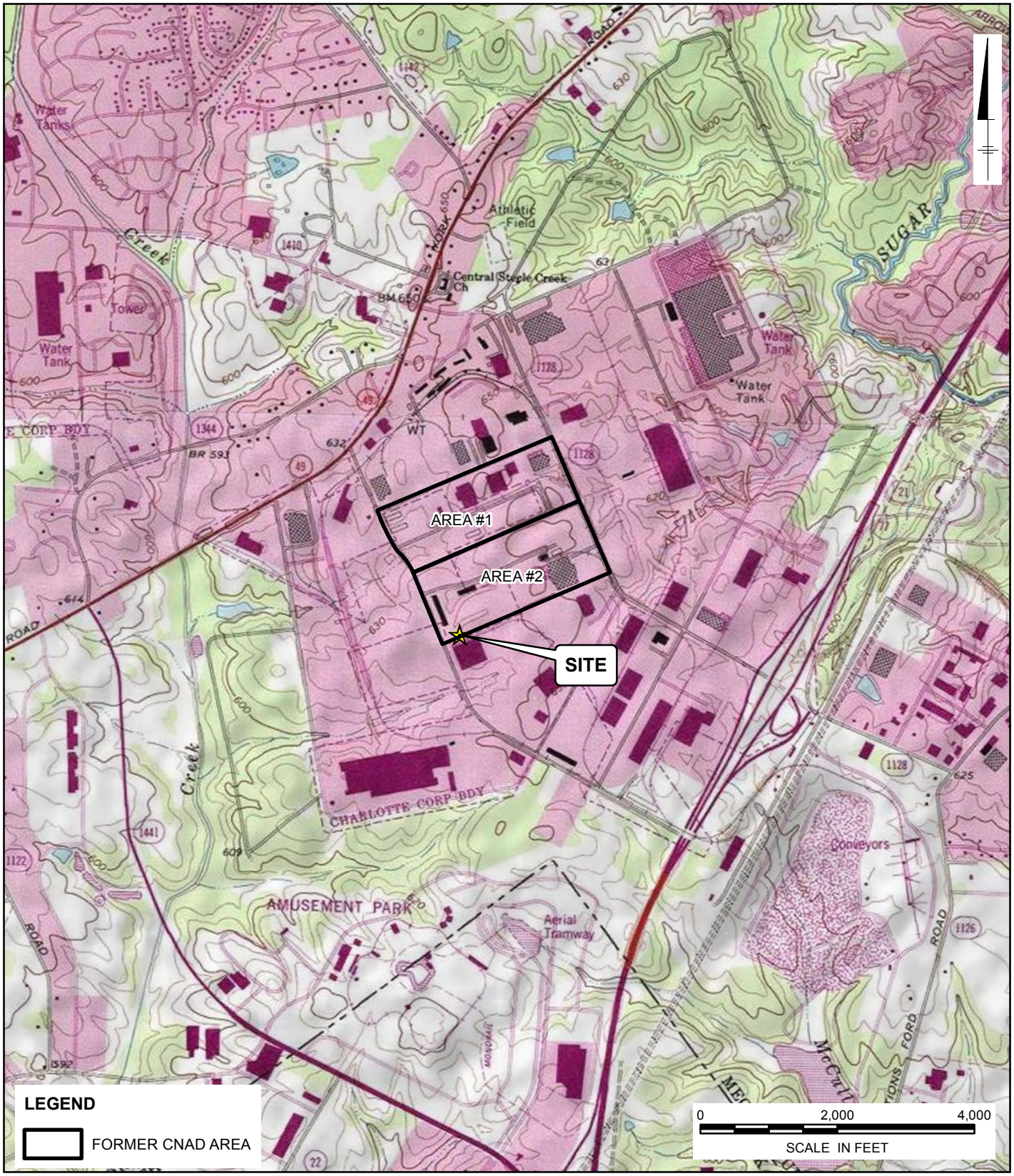
D. Project Management Plan (PMP)

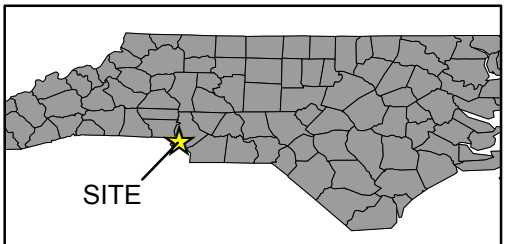
E. Operation and Maintenance (O&M) Plan

F. Investigation-Derived Waste (IDW) Plan

G. Construction Quality Control Plan (CQCP)

H. Groundwater Monitoring Plan





MECKLENBURG COUNTY, NORTH CAROLINA

CHARLOTTE NAVAL AMMUNITION DEPOT
2820 NEVADA BLVD, CHARLOTTE, NC

REMEDIAL ACTION WORK PLAN

SITE LOCATION MAP

PIKA INTERNATIONAL, INC. — A JOINT VENTURE — MALCOLM PIRNIE

FIGURE
1-1

2. Conceptual Site Model

Understanding the CSM of a site is an important component in optimizing the remedial system design and implementing the appropriate selected remedy. The development of the CSM incorporates the history of the site, the hydrostratigraphic framework, the lateral and vertical extent of the site contaminants, and the processes that control the fate and transport of the contaminants within the system. The components of the CSM are summarized in the following sections.

2.1 Site History

In June 1942, the Department of Navy signed a contract with the U.S. Rubber Company for the construction of 40-millimeter (mm) anti-aircraft ammunition shell loading and assembly plant in Charlotte, North Carolina known as the CNAD. In 1945, planned production was cut and operation of the facility was transferred to the U.S. Navy. In 1956, the Naval Depot status was changed from Maintenance to Inactive. At the time of operation, the entire CNAD complex occupied approximately 2,266 acres of land. In 1959, the Former CNAD complex was sold to a local partnership and is currently occupied by light industrial and commercial businesses.

Two areas (1 and 2) of the site were used for the production of 40-mm anti-aircraft munitions. Area 1 consisted of anti-aircraft ammunition loading lines. This area was dedicated to the assembly of final rounds and was composed of 22 buildings (See Figure 2-1). The largest of the buildings in Area 1 (1-60 and 1-70) were used for final assembly, packaging, and shipping of munitions.

The operations carried out in Area 2 were reportedly identical to those conducted in Area 1. Area 2 was also used to process returned ammunition after World War II. Only Area 2 was used after 1945 for reconditioning of returned munitions. A TCE vapor-degreasing operation was located on the southeast corner of building 2-30. The unit was used to remove cutting oil and preservatives from the exteriors of returned shells.

2.2 Previous Investigations

Environmental remediation activities at the Former CNAD are performed in accordance with the provisions of the Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA), 42 USC §9601 *et seq*., as amended by the Superfund Amendments and Reauthorization Act (SARA) of 1986, and to the extent practicable, the National Oil and Hazardous Substances Pollution Contingency Plan (NCP), 40 Code of

Federal Regulations (CFR) Part 300 *et seq*., as amended. The NCDENR concurs with the selected remedy, as outlined in the Decision Document (USACE, 2011a).

Investigations were conducted at the Former CNAD beginning in the late 1980's. From 1989 to 1992 a series of Environmental Site Assessments (ESAs) were conducted in this area. These ESAs included an initial Phase I ESA, which was completed in February 1990 and recommended a Phase II study be conducted to evaluate the potential groundwater contamination. Stage 1 of the Phase II was completed in July 1990 and Stage 2 of the Phase II was completed in October 1990. The ESAs identified low levels of TCE and toluene present in soil and TCE and 1,2-dichloroethane present in groundwater at concentrations exceeding the Maximum Contaminant Levels (MCL). As part of these ESAs, a baseline risk/health assessment (BRA) was completed. The assessment only evaluated groundwater as a potentially complete pathway. Hypothetical future groundwater ingestion by an industrial worker was considered as part of the BRA, but was determined to be very unlikely given public water supply in the area. The assessment determined that the hypothetical risk for groundwater ingestion was 4E-04. This value exceeded the most commonly used target of 1E-06 but only marginally exceeded the acceptable range for remediation of Superfund sites (1E-06 to 1E-04). The assessment also concluded that the HI exceeded a target of 1 (at 2.6) but was below 10. Based on the calculated risk value above the acceptable range for remediation of Superfund sites, an applicable or relevant and appropriate requirement (ARAR) analysis was conducted. The ARAR analysis determined the TCE concentrations in the groundwater exceeded the State ARAR for groundwater quality standards. This is the basis for conducting remedial action at the Former CNAD.

Impacts to environmental receptors were also determined to be minimal. In 1992, Stage 3 of the Phase II Assessment was completed to address areas in the Commerce Business Park (CBP) located northeast of the Former CNAD and ASIP areas. This phase of investigation identified TCE in the soil as well as groundwater and identified it as the primary contaminant in groundwater where former degreasing activities were performed (Building 2-30).

In 1994, a Phase I Remedial Investigation (RI) (Metcalf and Eddy, Inc. [M&E], 1995) was conducted that focused efforts in the Former CNAD areas. The RI concluded that soil was not significantly impacted; however, VOCs were identified in groundwater, specifically TCE and associated degradation products. Concentrations of TCE were generally observed to be higher in bedrock samples and in samples collected in Area 2. A qualitative risk evaluation concluded that groundwater was the most significant exposure pathway, but was incomplete given the public water supply in use in the area. These

Phase I RI investigations were not sufficient to complete delineation of contaminants in groundwater, and a Phase II investigation was recommended.

In 1999, a Phase II Remedial Investigation (M&E, 2000) was completed in an effort to delineate the horizontal and vertical extent of contamination as well as to determine the geologic and hydrogeologic framework of the shallow and bedrock aquifers. Investigation activities included the completion of three geophysical surveys (seismic refraction survey, shear-wave, and electromagnetic survey), borehole geophysical logging, aquifer testing, a baseline risk assessment, and monitor well installation and sampling. The Phase II RI concluded that TCE was the most widespread constituent with the largest contaminant mass present in the Transition Zone groundwater. Delineation of the horizontal extent of contamination was also completed with the exception of the southwestern portion of the plume, in the vicinity of Nevada Boulevard. Contamination was identified to extend vertically to a depth of approximately 70 feet below ground surface (bgs). The human health baseline risk assessment concluded that the hypothetical risk for groundwater ingestion and Health Index exceeded commonly used target values.

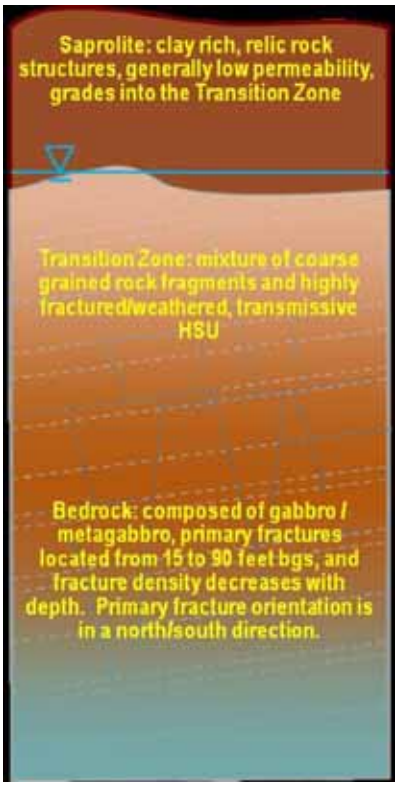
In November 2000, recommendations of the Phase II RI were implemented including the collection of additional information to support development of feasibility and pilot studies. Activities included completion of TCE delineation in groundwater and collection of natural attenuation data. These investigations resulted in the conclusion that the extent of TCE to the north was overestimated during previous evaluations. This evaluation also identified a significant drop in groundwater elevations, particularly in bedrock. This water level drop was product of both drought conditions in the region as well as the newly installed production wells, west of the Former CNAD, which were producing approximately 500,000 gallons per day of water to the neighboring facility. Use of the production wells was discontinued in May 2001 with water levels recovering almost immediately. Investigations continued including the installation of additional bedrock coreholes, geophysical surveys, collection of groundwater samples for chemical analyses, additional soil analysis, and a dense nonaqueous-phase liquid (DNAPL) evaluation. These investigations concluded that operation of the off-site well field influenced the TCE plume geometry which was exhibiting concentrations of greater than 2,500 µg/L at depths greater than 100 feet bgs, with designated areas extending greater than 200 feet bgs. Geophysics concluded that bedrock was very competent with a small volume of fractures controlling contaminant migration.

Per the Phase II RI, a pilot study using a combination bromide tracer and sodium lactate injection, followed by a monitoring period of 8 months was completed. Results of the study and subsequent monitoring events were documented in the *Site-Wide Groundwater*

Sampling Report for the Future Remedial Design (SAIC, 2008) and the *Focused Feasibility Study for the Former Naval Ammunition Depot, Mecklenburg County, Charlotte, North Carolina* (SAIC, 2009).

2.3 Hydrostratigraphic Framework

The Former CNAD site is located in Charlotte, North Carolina in the Piedmont physiographic region. The hydrostratigraphic framework beneath the site is composed of three units, which include the saprolite (shallow), transition zone and bedrock. This hydrostratigraphic framework is typical of the conditions encountered in the Piedmont and represents a highly heterogeneous and interconnected series of aquifer units. The saprolite HSU is characterized by fine grained unconsolidated residuum and saprolitic soils. The composition of the saprolite HSU is characterized as a low-plasticity clay near ground surface (M&E, 2000), which grades to an interbedded composition of silty sand, clay-rich silts, and silty clays (SAIC, 2008). Previous investigation activities at the site indicate that the saprolite is thin across the site (1 to 20 feet). The fine grained nature of the saprolite HSU presents a low conductivity unit that primarily functions as a storage unit, draining in to the underlying transition zone.



At the base of the saprolite, the soils become more coarsely grained with larger fragments of partially weathered rock (PWR) present in a saprolitic matrix. This zone of partially weathered rock and coarse grained sand becomes more competent with depth and is characteristic of highly fracture bedrock with some weathered zone. The combination of the coarse gained sands and PWR immediately overlying the bedrock and the upper portion of the fractured bedrock are defined as the transition zone. The thickness of the transition zone is proportionate to the thickness of the overlying saprolite and varies as a function of the shallow fractures present in the bedrock, the differential weathering patterns along the top of bedrock, and around the shallow fractures. In general, the demarcation between the saprolite and the transition zone is approximately 5 to 8 feet above auger refusal. The thickness of the transition zone

related to injection well placement will be further discussed as part of the ERD system design (Section 3.2.1).

Based on the available site data, the transition zone was identified as serving two roles in the groundwater fate and transport model. First, the presence fine grained saprolitic soils intermixed with the coarse sand and PWR provides a reservoir for contaminant mass storage within this HSU. The second role this unit serves is to provide a pathway lateral contaminant migration which in conjunction with the interconnection with underlying bedrock, provides a continuing source of contaminants to the bedrock HSU.

The third HSU located at the site is the fractured metamorphic bedrock, which has been primarily characterized as a massive medium grained gabbro/metagabbro. The fracture density in the bedrock decreases with depth, with primary fracture sets observed at depths ranging from 15 to 90 feet bgs; however, significant water bearing fractures have been observed in the vicinity of the site at depths up to 600 feet bgs (M&E, 2000). The primary fracture orientation is in a north/south direction, which was observed from outcrops near the Site, lineament traces, and bedrock cores (SAIC, 2008).

Groundwater flow directions, and subsequently contaminant migration directions, are controlled by the characteristics of the bedrock surface and the orientation of the fractures. Potentiometric maps for the transition zone HSU vary over time, but in general, indicate a groundwater flow direction to the west. Based on the distribution and orientation of the TCE plumes, the groundwater flow direction is not controlled solely by the hydraulic gradient, but is also influenced by the orientation of the fractures and undulations in the bedrock surface. In addition, groundwater extraction at the well field across Nevada Boulevard was observed to influence the transition zone wells and plume migration, further confirming the connection between the transition zone and bedrock HSUs. A potentiometric map of the transition zone is included as **Figure 2-2**.

Groundwater flow in the bedrock zone is dominated by the orientation of the north/south trending fracture sets. The groundwater flow direction based on the potentiometric maps is to the south along the central portion of the site, but transitions to a western flow to the west of the site; however, the western portion of the site is largely inferred due to the resolution of the monitoring well network. This southern component of groundwater flow was confirmed with the chemical oxygen demand (COD) distribution observed during the pilot study (SAIC, 2009). The interpretation of the pilot study data along with the evaluation of groundwater data indicates that the flow at the site is not entirely driven by the groundwater gradients and is structurally controlled by the fracture orientation. The most recent potentiometric map of the bedrock surface is included as **Figure 2-3**.

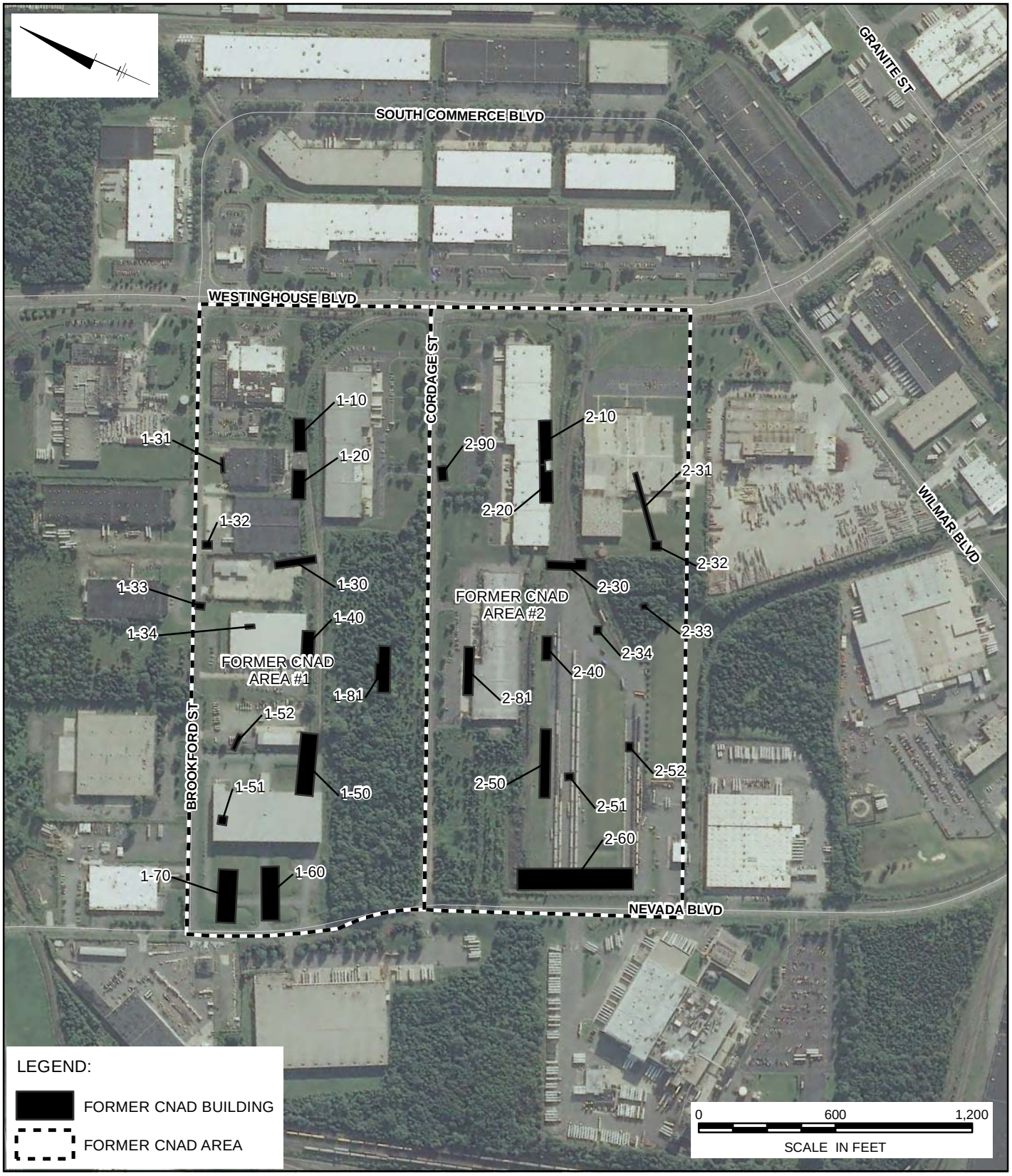
The groundwater impacts around the Former CNAD site are in the vicinity of a bedrock topographic high, which represents an area of groundwater recharge. The vertical hydraulic gradients in this area are in a downward direction from the transition zone to the bedrock under ambient conditions. In addition, the vertical gradients in the bedrock HSU indicate an overall downward hydraulic gradient; however based on the available data for the bedrock HSU, the magnitude and direction can vary based on location. The natural flow path of the groundwater from the transition zone to the bedrock is an important consideration for developing the remedial approach for the site. Reagent injected in the transition zone will provide indirect treatment of the underlying bedrock as the reagent naturally distributes post-injection. A similar reagent distribution is anticipated in the bedrock. The deeper groundwater impacts, although not directly targeted by the injection, can be indirectly treated by natural groundwater flow in the fracture network which will enhance reagent distribution and facilitate mixing between treated groundwater and groundwater within the dilute bedrock plume.

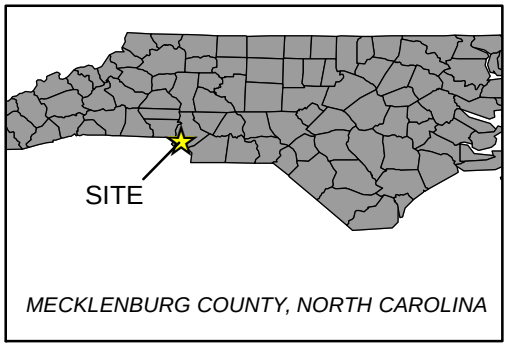
2.4 Nature and Extent of Groundwater Impacts

Historical investigations indicate that the primary constituent present in groundwater is TCE in the transition zone and bedrock HSUs. Plume maps for both the transition zone and bedrock HSUs were adapted from the *Focused Feasibility Study* (SAIC, 2009) and have been included as **Figures 2-4** and **2-5**, respectively. The plumes illustrated on these figures define the areas where concentrations of TCE in groundwater are greater than 500 µg/L based on the most recent, available laboratory analytical data.

The lateral extent of TCE in the transition zone is characterized by five distinct hot spots (**Figure 2-4**). The maximum observed concentration in the transition zone is 6,200 µg/L in NADMW-58 (SAIC, 2009). The vertical extent of TCE observed to a depth of 42 feet bgs, but in general, the impacts are at more shallow depths ranging from 10 to 25 feet bgs.

The groundwater impacts in the bedrock are characterized by one large area of impact centered near SAIC-14 (**Figure 2-5**). The concentrations of TCE in groundwater in the bedrock zone were reported at a maximum concentration of 40,000 µg/L and to a vertical extent of 305 feet bgs (SAIC, 2009).





MECKLENBURG COUNTY, NORTH CAROLINA

CHAF ON DEPOT
282 OTTE, NC

REMEDIAL ACTION WORK PLAN

FORMER CNAD LAYOUT MAP

PIKA INTERNATIONAL, INC. A JOINT VENTURE MALCOLM PIRNIE

FIGURE **2-1**

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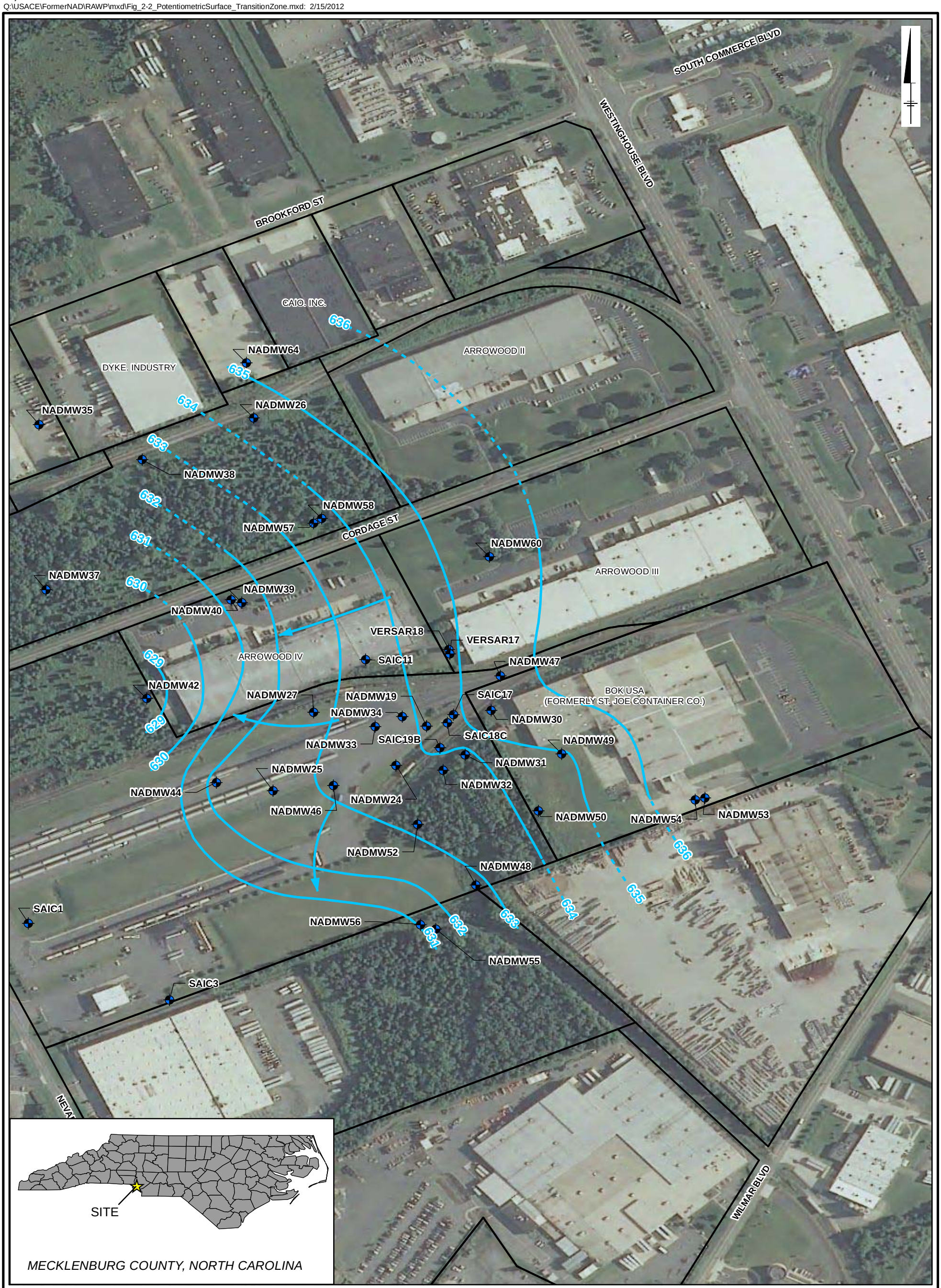

LEGEND:

- TRANSITION ZONE WELL
- POTENTIOMETRIC SURFACE (DASHED WHEN INFERRED)
- GROUNDWATER FLOW DIRECTION
- SURROUNDING PROPERTY BOUNDARY

NOTE:

1. THE LOCATIONS SHOWN ON THIS FIGURE ARE APPROXIMATE AND BASED ON GEOREFERENCED DATA FROM HISTORIC SITE FIGURES.
2. POTENTIOMETRIC SURFACE ADAPTED FROM THE FOCUSED FEASIBILITY STUDY (SAIC, 2009)

CHARLOTTE NAVAL AMMUNITION DEPOT
2820 NEVADA BLVD, CHARLOTTE, NC

REMEDIAL ACTION WORK PLAN

POTENTIOMETRIC SURFACE IN TRANSITION ZONE - SEPTEMBER 2006

PIKA INTERNATIONAL, INC. A JOINT VENTURE MALCOLM PIRNIE

FIGURE **2-2**

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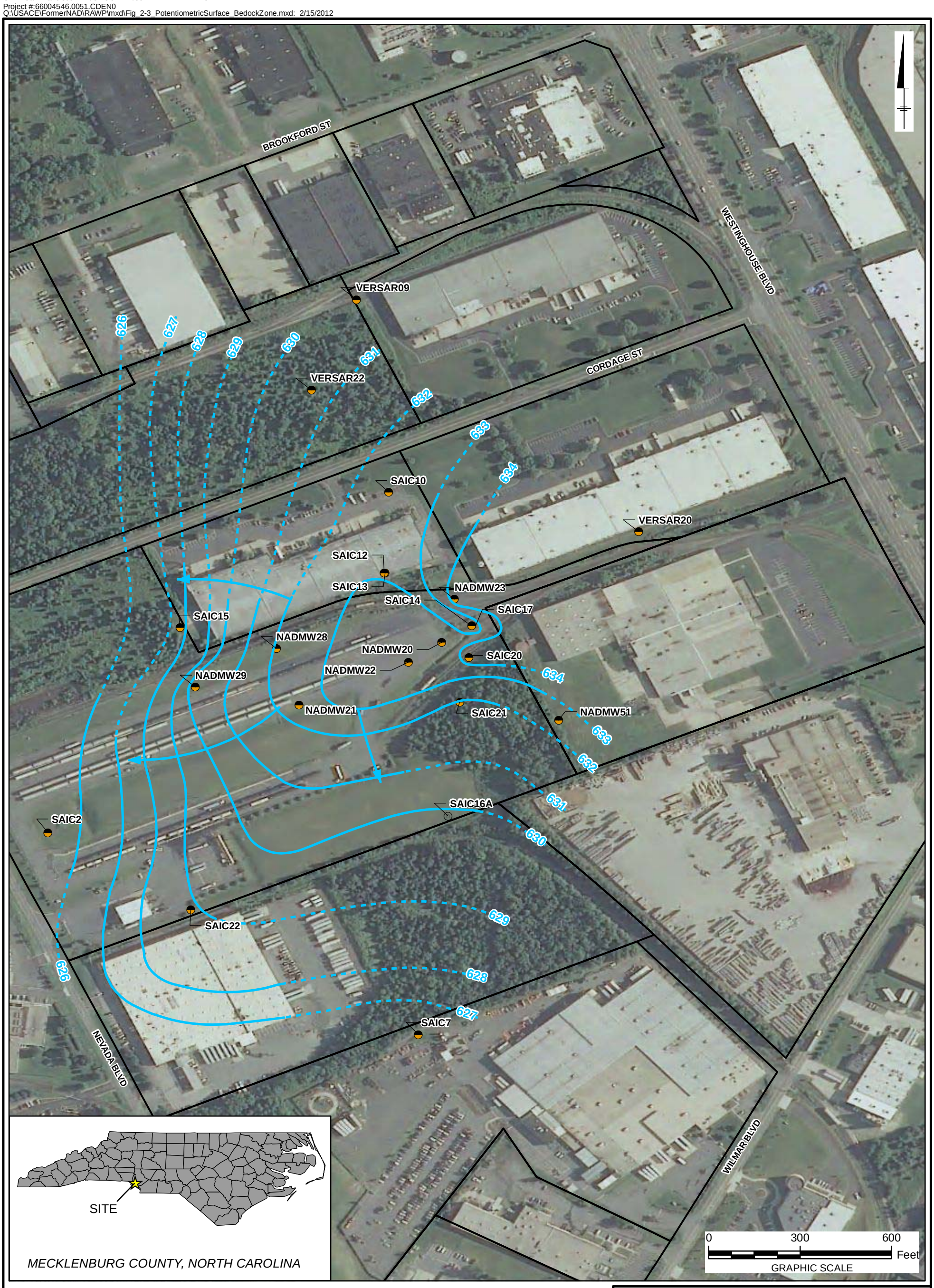

LEGEND:

- BEDROCK WELL
- MULTIPORT DEEP BEDROCK WELL
- POTENTIOMETRIC SURFACE (DASHED WHEN INFERRED)
- GROUNDWATER FLOW DIRECTION
- SURROUNDING PROPERTY BOUNDARY

NOTE:

1. THE LOCATIONS SHOWN ON THIS FIGURE ARE APPROXIMATE AND BASED ON GEOREFERENCED DATA FROM HISTORIC SITE FIGURES.
2. POTENTIOMETRIC SURFACE ADAPTED FROM THE FOCUSED FEASIBILITY STUDY (SAIC, 2009)

CHARLOTTE NAVAL AMMUNITION DEPOT
2820 NEVADA BLVD, CHARLOTTE, NC

REMEDIAL ACTION WORK PLAN

POTENTIOMETRIC SURFACE IN BEDROCK ZONE - SEPTEMBER 2006

PIKA INTERNATIONAL, INC. A JOINT VENTURE MALCOLM PIRNIE

FIGURE 2-3

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Project #:66004546.0051.CDEN0
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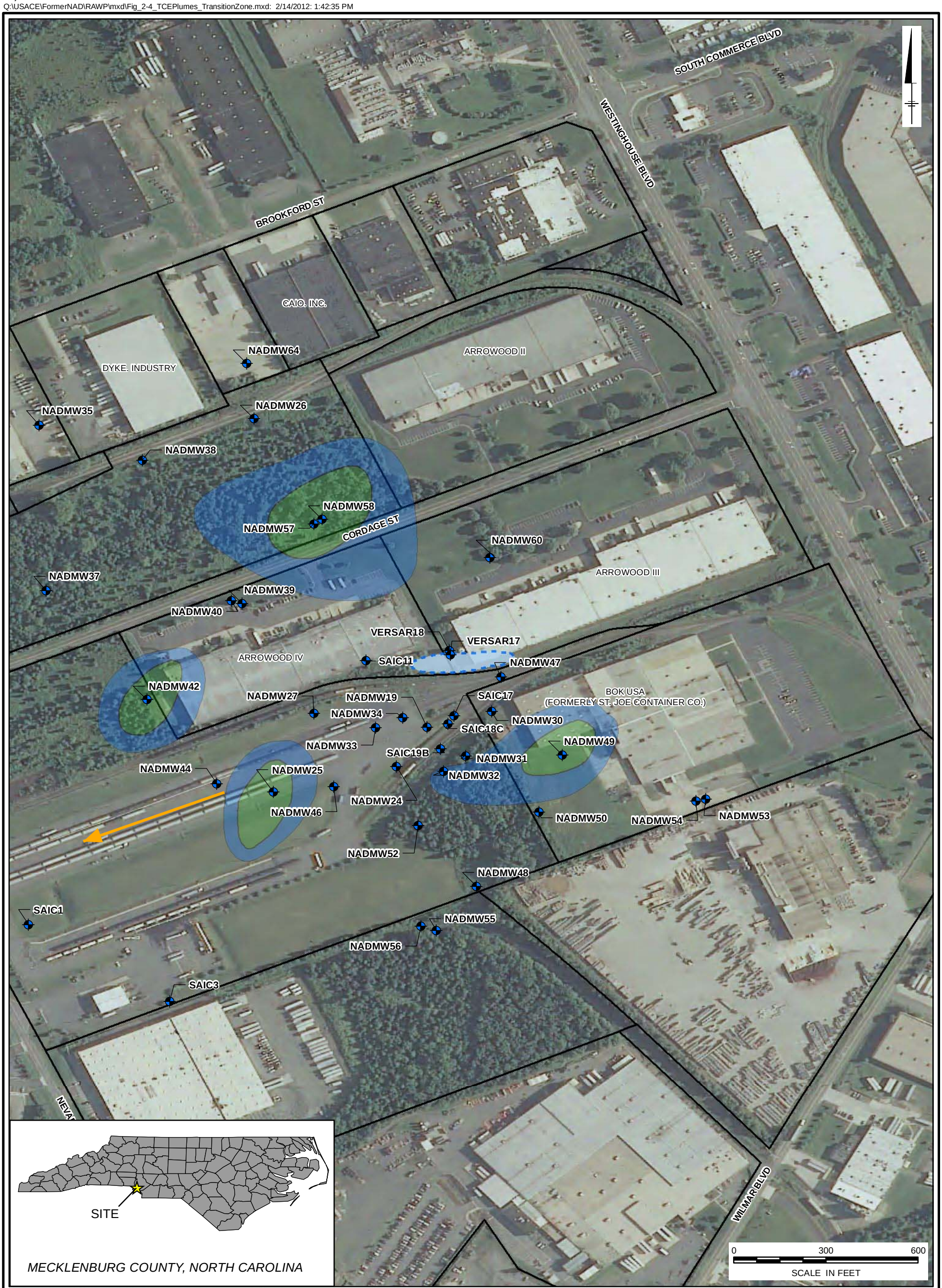

LEGEND:

- TRANSITION ZONE WELL
- APPROXIMATE DIRECTION OF GROUNDWATER FLOW IN THE TRANSITION ZONE
- SURROUNDING PROPERTY BOUNDARY

TCE PLUME: TRANSITION ZONE

- INFERRED TCE CONTOUR > 500 ug/L
- TCE CONTOUR > 500 ug/L
- TCE CONTOUR > 1,000 ug/L

NOTE:

1. THE LOCATIONS SHOWN ON THIS FIGURE ARE APPROXIMATE AND BASED ON GEOREFERENCED DATA FROM HISTORIC SITE FIGURES.
2. PLUMES ADAPTED FROM THE FOCUSED FEASIBILITY STUDY (SAIC, 2009)

CHARLOTTE NAVAL AMMUNITION DEPOT
2820 NEVADA BLVD, CHARLOTTE, NC

REMEDIAL ACTION WORK PLAN

CONCENTRATION OF TCE IN TRANSITION ZONE WELLS

PIKA INTERNATIONAL, INC. — A JOINT VENTURE — MALCOLM PIRNIE

FIGURE 2-4

CITY: San Fransisco DIV/GROUP: IM/85 DB: ME LD: PIC: PM: TM: TR:
Project #:66004546.0051.CDEN0
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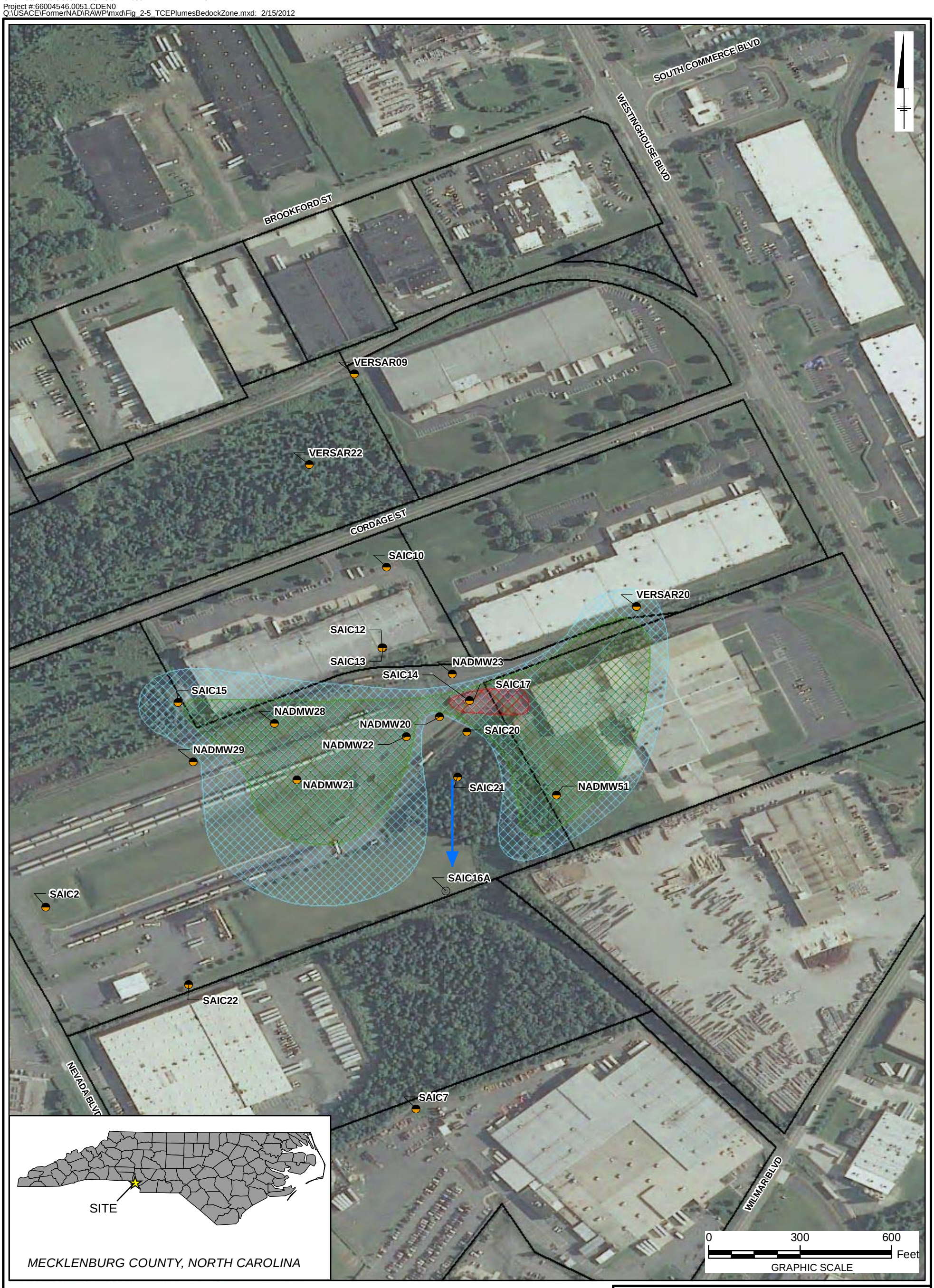

LEGEND:

- BEDROCK WELL
- MULTIPORT DEEP BEDROCK WELL
- APPROXIMATE DIRECTION OF GROUNDWATER FLOW IN THE BEDROCK ZONE
- SURROUNDING PROPERTY BOUNDARY

TCE PLUME - BEDROCK ZONE

- TCE CONTOUR > 500 ug/L
- TCE CONTOUR > 1,000 ug/L
- TCE CONTOUR > 10,000 ug/L

NOTE:

1. THE LOCATIONS SHOWN ON THIS FIGURE ARE APPROXIMATE AND BASED ON GEOREFERENCED DATA FROM HISTORIC SITE FIGURES.
2. PLUMES ADAPTED FROM THE FOCUSED FEASIBILITY STUDY (SAIC, 2009)

CHARLOTTE NAVAL AMMUNITION DEPOT
2820 NEVADA BLVD, CHARLOTTE, NC

REMEDIAL ACTION WORK PLAN

CONCENTRATION OF TCE IN BEDROCK ZONE WELLS

PIKA INTERNATIONAL, INC. A JOINT VENTURE MALCOLM PIRNIE

FIGURE 2-5

3. Remedial System Design

To address the RAOs detailed in Section 1.3, active ERD consisting of two years of active injection will be conducted following installation of the required system delivery infrastructure. During this period, performance monitoring will be conducted to collect the data necessary for optimization of the injection program, to evaluate the performance of the remedy and transition to the MNA phase of the remedial action. Following cessation of the active ERD injection program, the remedial action will transition to a MNA approach to achieve the desired objectives. The following sections discuss the remedial action program and the details associated with the implementation and operation of the remedy.

3.1 Selected Remedial Action

The selected remedial action for the Site is a phased approach that utilizes both active ERD and MNA technologies to meet the RAOs. ERD will be implemented at the Site in both the transition zone and bedrock HSUs to reduce TCE concentrations to levels that are amenable to MNA (less than 500 µg/L), following which remedial activities will be transitioned to MNA to reduce TCE concentrations to less than the NCAC 2L standard. Design details pertaining to implementation of the remedial action technologies are presented in the following sections.

3.1.1 ERD

ERD is an engineered bioremediation technique that falls into a class of remedial actions known as in-situ reactive zone (IRZ) technologies. The ERD technology involves the periodic delivery of sufficient organic carbon into the target treatment zone to support the development of reducing conditions within the IRZ. The injected carbon reagent provides an electron donor to reduce the CVOC constituents to inert end products (ethene and ethane), thereby achieving the remedial goals. Organic carbon injections are conducted to achieve four basic goals:

1. *Overcome the continuous electron acceptor supply:* This includes oxygen, nitrate, and other electron acceptors that tend to support a more aerobic microbial community.

2. *Produce molecular hydrogen (H_2) through fermentation*: Molecular H_2 is a product of fermentation and is used as an electron donor by dechlorinating bacteria.

3. *Achieve complete dechlorination of the target compounds*: Dechlorinating bacteria use the hydrogen produced through fermentation as an electron donor and VOCs as electron acceptors. Hydrogen atoms are substituted for chlorine atoms in the dehalorespiration process resulting in a sequential chemical reduction of VOC molecules, which for TCE follows the pathway:

   TCE → cis-DCE → VC → Ethene

4. *Achieve dissolution of nonaqueous phase contaminant mass*: Under natural conditions, the dissolution of hydrophobic organic compounds is very slow, allowing groundwater plumes to persist for many decades if the dissolution rate cannot be enhanced. ERD enhances the dissolution rate of this contaminant mass and thus making it available for treatment in the dissolved phase.

The key to successful implementation of ERD is delivering, distributing, and sustaining an adequate supply of organic carbon donor to create strongly-reducing conditions, while maintaining the pH above 5. As demonstrated by the ERD pilot test (SAIC, 2009), sufficient volumes of carbohydrate solution can be delivered to the subsurface to reduce TCE concentrations to below 500 µg/L and achieve complete dechlorination. Based on the results from the successful pilot test, the PIKA-PIRNIE JV Team has designed a full-scale ERD remedy for treatment of CVOCs within the transition zone and bedrock HSUs at the Site. Design details pertaining to system installation and operation are presented later in this section, and performance monitoring associated with ERD operations are presented in greater detail in Section 4.

3.1.2 MNA

As defined by the United States Environmental Protection Agency (USEPA) in Office of Solid Waste and Emergency Response (OSWER) Directive 9200.4-17P (USEPA, 1999), MNA refers to the reliance on natural attenuation processes to achieve site-specific remedial objectives within a timeframe that is reasonable compared to other methods. These natural attenuation processes include: physical (dilution, dispersion, volatilization), chemical (hydrolysis, precipitation), and biological processes that will reduce the mass, toxicity, mobility, volume, and concentration of compounds in soil and groundwater. The time required for these processes to lower concentrations to levels that are protective of human health and the environment varies widely based on the hydrogeologic system, chemical compounds present, and the applicable remedial goals. As MNA utilizes naturally occurring processes for site remediation, the USEPA

requires that MNA strategies are implemented at sites with a low potential for contaminant migration (USEPA, 1999).

Natural attenuation can be monitored and documented by evaluating trends in VOCs and by evaluating the geochemical indicators present during biodegradation over a historical monitoring time period. As stated above, natural attenuation includes multiple mechanisms that work to reduce contaminants in groundwater systems. Reductive dechlorination is the primary biological degradation process for CVOCs under anaerobic conditions, which will be enhanced over the course of the active ERD program. Following completion of the active organic carbon injection remedy, anaerobic conditions within the target injection intervals and within the IRZ will sustain residual biodegradation during the MNA period. In addition, reduction in CVOCs during the active ERD program will allow MNA mechanisms to facilitate VOC treatment and accomplish remedial objectives. While advanced dechlorination to ethene requires relatively strong reducing conditions (sulfate reducing to methanogenic), continued dechlorination of TCE can occur under less reducing conditions. The MNA component of the remedial action for the Site is discussed in detail later in this section.

3.2 ERD Groundwater Remedy

Full-scale ERD has been selected to address areas of the Site where TCE concentrations are present at concentrations exceeding 500 µg/L. The ERD treatment approach will be accomplished via the installation of the full-scale delivery system including injection wells, conveyance piping and instrumentation, mixing and delivery systems and associated telemetry monitoring equipment. Following installation, full-scale injection activities will be conducted on a quarterly basis for a period of two years to deliver the organic carbon reagent into the subsurface to promote treatment. This section presents the design of these system components in addition to injection start up and operation, and permitting activities.

3.2.1 System Design

The ERD system is designed to provide treatment of both the transition zone and bedrock HSUs and achieve RAOs. Design parameters necessary for the ERD system are based on the CSM as discussed in Section 2 and IRZ design concepts. The details of the different design components are discussed in this section.

3.2.1.1 Transition Zone

To address the highest TCE impacts (>500 µg/L) in the transition zone HSU, fifty-three (53) injection wells will be installed in the transition zone (**Figure 3-1**). Injection well locations are proposed to address the four identified plume areas where TCE concentrations greater than 500 µg/L have been observed. Well locations will be installed in nine transect lines oriented approximately north-south or perpendicular to groundwater flow direction in the transition zone. Injection wells in each transect will be spaced approximately 35-feet apart in order to achieve complete lateral distribution and coverage based on the expected organic carbon ROI. Prior to installation of the proposed injection lines, the baseline groundwater sampling event will be completed to confirm the orientation of the injection well network. In addition to this, an iterative well installation process will be applied in which select wells from each injection line will be sampled during the drilling period to confirm transect position and orientation. The groundwater samples will be analyzed for VOCs only to provide data to support the adaptive design of the injection well transects. The sample methodology will be the same as the methodologies utilized in the baseline, performance monitoring events (Appendix C-1).

Each injection well will be installed to an approximate total depth of 25 feet bgs with a 17 foot screened interval from 8 to 25 feet bgs. Each well will be installed via hollow stem auger and air rotary drilling methods and constructed out of 2-inch diameter Schedule 80 polyvinyl chloride (PVC) casing with stainless steel wire-wrapped well screens. A coarse sand pack will be installed to 2 feet above the top of screen; 2 feet of fine sand will be placed on top of the coarse sand pack to separate the sand pack from the well seal. The remainder of the well annulus will be sealed with a neat cement grout. Grain size analysis will be performed on the designated transition zone monitor wells installed and used to confirm design of the appropriate filter pack of the transition zone injection wells. All final well construction details will be determined in the field.

As detailed above, the actual locations of the injection wells will be revised as needed based on results of the baseline sampling event and following review of results collected during the iterative well installation sampling activities. The current orientation of the treatment areas is based on data collected during 2006 or earlier, and the iterative program will be designed to increase delineation within these areas and ensure that the injection wells are positioned to achieve maximum remedial benefit. Therefore, the well locations presented on **Figure 3-1** are subject to change based on the results of the baseline monitoring event, the well installation sampling program, and

revisions to the CSM. This baseline monitoring event will be conducted prior to the installation of the injection wells in the transition zone. In addition to the above, subsurface conditions and existing Site constraints may require adjustment of the proposed injection well locations. The proposed layout for the transition zone injection wells and distribution system is included in Appendix A – Drawing C1.

3.2.1.2 Bedrock

To address the greatest TCE impacts (>500 µg/L) in the bedrock HSU, twenty-five (25) injection wells will be completed in bedrock (**Figure 3-2**). Injection wells will be installed along three transect lines oriented from east to west and perpendicular to the bedrock fracture planes and understood groundwater flow direction in bedrock. Based on results of the pilot test, injection wells in each transect will be spaced approximately 80-feet apart in order to achieve complete lateral distribution and coverage based on the expected organic carbon radius of influence (ROI).

Each injection well will be installed to an approximate total depth of 100 feet bgs with a screened interval from 25 to 100 feet bgs. Each well will be installed via hollow stem auger and air rotary drilling methods. A 6-inch PVC casing will be installed from ground surface and through the transition zone to avoid interconnection of the HSUs. The borehole will then be advanced to the terminal depth and the well will be constructed out of two-inch diameter Schedule 80 PVC casing with stainless steel wire-wrapped well screen. A coarse sand pack will be installed to 2 feet above the top of screen; 2 feet of fine sand will be placed on top of the coarse sand pack to separate the sand pack from the well seal. Similar to the transition zone injection wells, the remaining well annulus will be sealed with a neat cement grout. All final well construction details will be determined in the field.

As detailed above, the location of the injection well transects are subject to change based on subsurface conditions or other unanticipated field conditions, drilling or property access or CSM updates based on results of the baseline monitoring event. The layout of the bedrock injection well locations and distribution lines are presented on Appendix A – Drawing C1.

3.2.1.3 Piping Layout

The conceptual locations of the injection wells, manifolds, and conveyance piping are presented on **Figures 3-1** and **3-2** for the transition zone and bedrock injection well networks, respectively. Approximately 6,500 linear feet of individual 1-inch diameter

subsurface conveyance piping will be installed from the wells to one of the seven remote distribution manifolds located across the Site (Appendix A, Drawings C1 and PID2). Each of the seven remote distribution manifolds will be installed in a subsurface, traffic-rated concrete vault, and will contain individual totalizing flow meters and pressure gauges required to monitor injection flow rates and wellhead pressures during injection events. Each manifold will be connected to one of the two treatment systems (*i.e.,* north and south of the railroad tracks) via 2-inch diameter conveyance lines that will transport the injection solution to the injection wells. All conveyance piping will be installed in trenches and backfilled with native soil removed from the Site. Where necessary, sections of the delivery piping will be installed via horizontal boring underneath railroad spurs or roadways to connect the reagent delivery lines to injection wells located between the two rail spurs (injection wells IW-44 through IW-53) or across Cordage Street, (IW-1 through IW-11, IW-14).

The locations of the conveyance piping depicted on the design drawings is based on the current injection transect locations and current understanding of site constraints. During system installation, the selected locations of the conveyance piping may be revised as needed based on subsurface conditions encountered, existing Site constraints or based on revisions to the injection transects identified during baseline groundwater or well installation sampling activities.

3.2.1.4 Injection System

Two injection systems will be used to facilitate mixing and delivery of the organic carbon solution during injection events. A unique injection system will be constructed and positioned on each side of the railroad tracks to minimize the required extent of horizontal boring beneath the railroad during conveyance piping installation. Each injection system will consist of injection equipment including injection and mixing pumps, tanks to store dilution water and the bulk organic carbon solution, and electrical control and remote telemetry equipment. Detailed engineering drawings for the ERD injection system are included in Appendix A; the different components of the injection system are presented below.

3.2.1.4.1 Injection Systems

Two injection systems will be utilized at the Site. One of the systems will be positioned north of the project site and in between the Arrowood III and Arrowood IV buildings. The other injection system will be positioned on the south side of the Site, in a large grassed area. Each system will be maintained in a secure condition using chain link

fence or other approved methods. The approximate location of these two systems during the active ERD injection program is presented on **Figures 3-1** and **3-2**.

The northern injection system will be used to deliver the organic carbon solution to all bedrock injection wells (BIW-1 through BIW-25) as well as the northern four transition zone injection well transects (IW-1 through IW-30). Connection to an existing 8-inch water main located along Cordage Street will be established and will be connected to the northern system via subsurface piping. This water supply will provide the necessary dilution water to facilitate mixing and delivery of the organic carbon solution during the injection program.

The southern injection system will be used to deliver organic carbon solution to all transition zone injection well transects south of the railroad (IW-31 through IW-53). Conveyance piping from this system will be constructed to support injection solution delivery to the dedicated distribution manifolds. Piping will be trenched into the subsurface and will include horizontal boring underneath the railroad in the vicinity of well NADMW46 (**Figure 3-1**). Water supply for this mixing trailer will be supplied by an existing 8-inch water main south of the Site along Nevada Boulevard.

Solution delivery from each injection system will be conducted independently and semi-automated mixing will be facilitated by operators. Each semi-automated injection system will consist of a substrate storage tank, dilution water equalization (EQ) tank, pumps, in-line mixer, filtration system, flow meters, pressure gauges, and valving (Appendix A, Drawing PID1). Water connections will be made to each of the injection systems from the two existing municipal water supplies available in the vicinity of the Site. Potable water from these water connections will be directed to an EQ tank staged within each of the injection systems conex boxes to ensure a constant water supply during injection events.

Water pumped from the EQ tank will be pumped to an in-line static mixer to achieve an approximate 1 percent dilute molasses by volume, organic carbon injection solution (Appendix A, Drawing PID1). Molasses will be pumped concurrently to the in-line mixer via a positive displacement molasses transfer pump. Once mixed, the dilute molasses solution will be pumped through two bag filters arranged in parallel to remove any potential solids or particulate material and to prevent fouling of the injection wells. The injection solution will then be routed to a manifold that controls delivery to each dedicated headers for the individual distribution manifolds vaults.

3.2.1.4.2 Molasses Handling and Storage

During each injection event, it is anticipated that approximately 21,000 gallons of 100 percent feed-grade molasses will be delivered to the Site via tanker and offloaded to an 11,000 gallon external vented and jacketed carbon storage tanks staged adjacent to each injection system. Each tank will be connected via heat traced pipe to the associated conex box that houses the injection system equipment and controls. Additional deliveries of molasses will take place, as necessary, over the course of each injection event. Based on performance observations (*e.g.,* required injection volumes to achieve complete distribution within the transition zone and bedrock geologies, required injection wells included in the program to achieve RAOs), the total volume of molasses may be adjusted over time. These adjustments will be conducted to optimize remedial performance and ensure focused operation of the injection system.

3.2.1.4.3 Molasses Concentration

A 1 percent molasses solution (by volume) will be used for injections at the Site. This concentration may be adjusted based on the operational parameters (total organic carbon [TOC] concentration and pH) and performance monitoring data (VOC concentration and treatment extent) observed within the treatment area (detailed in Section 4). During each injection event, the pump supplying the bulk molasses solution and the flow rate of the water supply line will be adjusted as necessary to facilitate mixing of the required injection solution. Flow totalizers on each of these supply lines will be monitored to ensure the design mixing strength is achieved and grab samples will be collected during each injection event to confirm the actual injected TOC strength.

3.2.1.4.4 Injection Area Logistics

During each injection event, approximately 2,086,000 total gallons of 1 percent (by volume) molasses solution will be delivered to the 53 transition zone and 25 bedrock injection wells. Based on a review of the pilot test data, it is anticipated that approximately 12,000 gallons and 58,000 gallons will be injected into the transition zone and bedrock zone wells, respectively. Over the course of remedial operations, data collected from the performance monitoring well network will be used to confirm the required injection volumes and ensure that sufficient TOC distribution is being achieved to promote treatment. Based on these results, the target injection volumes will be adjusted, as needed. The injection solution will be delivered to each well via the two injection systems, conveyance lines, and distribution manifolds (Appendix A – Drawings PFD1 and PID1).

Injection events will be conducted over the course of active remedial operations within the limits of the available water supply. Using the design injection flow rates provided in the FFS (1.5 gallons per minute [GPM] for transition wells and 6 gpm for bedrock wells), anticipated water supplies of 195 GPM and 35 GPM would be required for the northern and southern injection areas, respectively. Based on previous discussion with the Charlotte Mecklenberg Utilities Department, the Cordage Street water supply line (north injection system) has an available flow of 70 GPM and the Nevada Boulevard water supply line has an available flow of 100 GPM. Based on the dilution water required for continuous operation of the northern and southern injection systems, it is anticipated that injections within the northern area will be separated into three different groups of injection wells. Under this arrangement, injections would proceed within the first set of wells until the target injection volume is achieved, following which flow controls would be reconfigured by operators to facilitate injection into the subsequent set of wells. Within the southern area, based on the estimated dilution water supply required (35 GPM) and the available water source (100 GPM), injection into all southern injection wells will be conducted simultaneously. Specific groupings of wells may be adjusted over the course of the program to optimize overall efficiency.

It is anticipated that injection events will be conducted on a quarterly basis, but this frequency may be adjusted based on the observed longevity of TOC within the subsurface, washout of the TOC from the injection area, and the required dosing frequency to achieve the remedial objectives. TOC samples collected during the performance monitoring events (detailed in Section 4) will be used to evaluate the required injection frequency and adapt the injection program, as necessary. Operation of typical IRZ systems relies on sustaining TOC concentrations above baseline levels to promote development of reducing conditions and support dechlorination. Therefore, performance monitoring data will be used to adapt the injection frequency and total injection volumes over the course of the active injection program to ensure optimal remedial performance.

3.2.2 Injection Startup and Operation

Active injections will begin once the full-scale system mixing and delivery infrastructure has been installed and connected to the potable water supply lines. As part of startup/shakedown procedures, system piping, valves, and conveyance equipment will be checked for leaks and proper operation. System controls required for semi-automated mixing will be tested under the range of expected operating scenarios and trouble-shooting will be conducted when necessary.

Once the mixing and injection systems are determined to be operating properly, concentrated organic carbon solution will be delivered to the Site and offloaded to the holding tanks staged next to the two injection trailers. The following chronology details the activities that will be performed prior to and during each of the injection events:

- The concentrated organic carbon solution will be delivered before each quarterly injection event and offloaded to the external vented and jacketed carbon tanks. Additional molasses deliveries will be conducted as necessary over the course of each injection event.

- Molasses will be pumped from the external storage tank via a positive displacement molasses transfer pump and dilution water will be pumped from the EQ tank. Automated flow control will ensure that pumping rates achieve the desired TOC injection concentrations via in-line mixing. The 1 percent dilute molasses solution will then be routed through the bag filters and delivered to the injection manifolds (Appendix A, Drawing PID1).

- During injection, periodic flow readings will be collected from both the conveyance headers leaving the injection systems and from the individual injection lines. Flow meters on each injection line will be used to monitor the flow rate to each injection well and determine when the target volume has been achieved. The total cumulative volume of injection solution delivered to each distribution manifold will be determined using totalizers installed on each distribution line (Appendix A, Drawing PID1). The total volume and flow rates for each individual injection well will also be verified within the distribution manifold (Appendix A, Drawing PID2). The flow rates for each of the injection wells will be compared to the total volume pumped from the batch mixing tanks to confirm accuracy.

- Once the target volume of solution has been delivered to all injection wells, potable water from the water supply will be routed through the conveyance lines to rinse residual molasses solution from the mixing system and flush both the delivery piping and injection wells. These activities will be conducted to ensure the long-term integrity of the delivery system and ensure that residual organic carbon solution is not present within the mixing components or individual delivery lines. It is estimated that approximately 50 to 100 gallons will be required for per well for complete system flushing, but these volumes may vary based on visual observations during operation. The system is designed so that the clean water flush will include both concentrated organic

carbon solution holding tanks and the batch tank. A sufficient volume of water will be rinsed through the molasses storage tanks, batch mixing tanks and conveyance infrastructure such that clean water arrives at the injection wells at the end of the rinsing cycle. The clean water flush will remove residual molasses solution from the system and thereby ensure the longevity of the distribution infrastructure and prevent molasses fermentation within system components.

The relevant injection logs, component specifications, and operational procedures for injection and operational monitoring are included in greater detail in the O&M Plan/O&M Manual (Appendix E).

3.2.3 Permitting

3.2.3.1 Underground Injection Control (UIC) Permitting

While an UIC Permit for the proposed ERD injections is not required for Superfund Projects under the North Carolina Administrative Code, it is required that the RAWP be technically reviewed and approved by the NCDENR UIC Program prior to any activities at the Site. To meet these requirements, a copy of this RAWP, a draft UIC Permit and supporting documentation has been prepared and forwarded to the NCDENR UIC Program for review. No injection activities will be initiated without prior approval from the NCDENR UIC Program.

3.2.3.2 Access Agreements

Prior to implementation of any remedial activities, access agreements will be obtained for the project Site and all properties where remedial activities are proposed. Agreements will be obtained by the USACE. These agreements will be maintained as necessary throughout the duration of activities associated with the groundwater remedy.

CITY: San Fransisco DIV/GROUP: IM/85 DB: ME LD: PIC: PM: TM: TR:
Project #:66004546.0051.CDEN0
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MECKLENBURG COUNTY, NORTH CAROLINA

0 300 600 Feet
GRAPHIC SCALE

NOTE:

1. THE LOCATIONS SHOWN ON THIS FIGURE ARE APPROXIMATE AND BASED ON GEOREFERENCED DATA FROM HISTORIC SITE FIGURES.
2. PLUMES ADAPTED FROM THE FOCUSED FEASIBILITY STUDY (SAIC, 2009)

LEGEND:

- PROPOSED INJECTION WELL
- PROPOSED MONITORING WELL
- PROPOSED MANIFOLD VAULT
- PROPOSED HEADER VAULT
- EXISTING MONITORING WELL
- SURROUNDING PROPERTY BOUNDARY
- INJECTION WELL TRANSECT
- DELIVERY PIPING
- PROPOSED MIXING TRAILER STAGING AREA
- APPROXIMATE DIRECTION OF GROUNDWATER FLOW IN THE TRANSITION ZONE

TCE PLUME: TRANSITION ZONE

- INFERRED TCE CONTOUR > 500 ug/L
- TCE CONTOUR > 500 ug/L
- TCE CONTOUR > 1,000 ug/L

CHARLOTTE NAVAL AMMUNITION DEPOT
2820 NEVADA BLVD, CHARLOTTE, NC

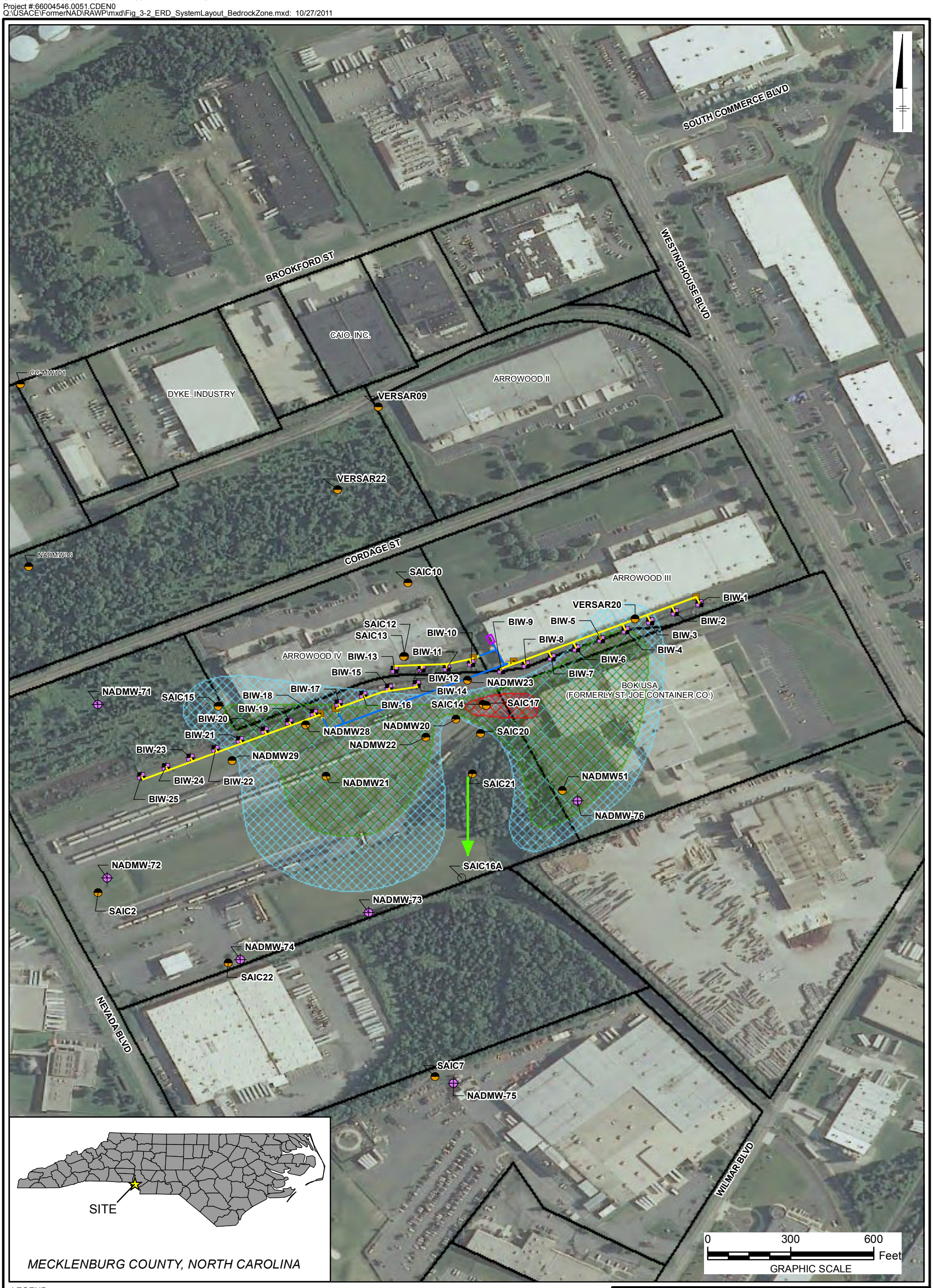
REMEDIAL ACTION WORK PLAN

ERD SYSTEM LAYOUT - TRANSITION ZONE HSU

PIKA INTERNATIONAL, INC. A JOINT VENTURE MALCOLM PIRNIE

FIGURE 3-1

CITY: San Fransisco DIV/GROUP: IM/85 DB: ME LD: PIC: PM: TM: TR:
Project #:66004546.0051.CDEN0
Q:\USACE\FormerNAD\RAWP\mxd\Fig_3-2_ERD_SystemLayout_BedrockZone.mxd: 10/27/2011



LEGEND:

- PROPOSED INJECTION WELL
- PROPOSED MONITORING WELL
- PROPOSED MANIFOLD VAULT
- EXISTING MONITORING WELL
- MULTIPORT DEEP BEDROCK WELL
- SURROUNDING PROPERTY BOUNDARY
- INJECTION WELL TRANSECT
- DELIVERY PIPING
- PROPOSED MIXING TRAILER STAGING AREA
- APPROXIMATE DIRECTION OF GROUNDWATER FLOW IN THE BEDROCK ZONE

TCE PLUME - BEDROCK ZONE

- TCE CONTOUR > 500 ug/L
- TCE CONTOUR > 1,000 ug/L
- TCE CONTOUR > 10,000 ug/L

NOTE:

1. THE LOCATIONS SHOWN ON THIS FIGURE ARE APPROXIMATE AND BASED ON GOREFERENCED DATA FROM HISTORIC SITE FIGURES.
2. PLUMES ADAPTED FROM THE FOCUSED FEASIBILITY STUDY (SAIC, 2009)

CHARLOTTE NAVAL AMMUNITION DEPOT
2820 NEVADA BLVD, CHARLOTTE, NC

REMEDIAL ACTION WORK PLAN

ERD SYSTEM LAYOUT - BEDROCK ZONE HSU

PIKA INTERNATIONAL, INC. A JOINT VENTURE — MALCOLM PIRNIE

FIGURE 3-2

4. Remedial Action Monitoring

The scope of work detailed in the PWS provides an organized structure for the completion of remedial action monitoring associated with ERD activities at the CNAD site. Monitoring activities have been broken down into a baseline groundwater monitoring event, eight performance monitoring events, and eight MNA monitoring events. Details of each type of monitoring event are provided in the sections below.

The monitoring program will focus on sample collection from existing and proposed monitoring well locations. With the exception of groundwater samples collected during the iterative well installation process, samples will not be regularly collected from the injection well network. The routine addition of the carbon substrate solution into the injection wells will result in dilution of any samples collected. For performance monitoring purposes, well locations positioned at variable distances from the injection well network will be used to evaluate treatment extent and to collect operational parameters to optimize ERD operations.

4.1 Monitoring Well Installation

A total of 11 new monitoring wells will be installed at the Former CNAD facility to expand the existing monitoring well network and provide sufficient well locations to monitor ERD progress. Six monitoring wells will be installed within the bedrock zone and five monitoring wells will be installed in the transition zone, as defined in the CSM. Monitoring wells will be completed similar to the construction detail outlined in the FFS (SAIC, 2009):

- Bedrock monitoring wells – 250 foot total depth with a screened interval from 230 to 250 feet bgs.
- Transition zone monitoring wells – 25 foot total depth with a screened interval from 15 to 25 feet bgs.
- Each well will be constructed with a 2-inch diameter stainless steel screen with Schedule 80 PVC risers and will be completed with a flush mount well vault.

It should be noted that the construction of each well may vary from the specification listed above in order to provide monitoring wells that are located entirely in the intended HSU. Total depth, screened interval, and final well construction will be based on field conditions and be determined by the site Geologist. Adjustments will be made based on the expected variations in depth and thickness of the partially weathered rock layers

encountered within the Transition Zone. The locations of the proposed monitoring wells are shown on **Figure 4-1**.

4.2 Baseline Monitoring

The baseline groundwater monitoring event will be conducted after completing installation of the 11 new monitoring wells. The baseline monitoring event includes the sampling and analyses of 32 monitoring wells for field parameters (pH, specific conductance, temperature, dissolved oxygen [DO], oxidation-reduction potential [ORP], and turbidity), VOCs, light gases (methane, ethane, ethene), TOC, and biogeochemical parameters (nitrate, total and dissolved iron, sulfate, alkalinity). Samples will be collected in accordance with the procedures outlined in the SAP, included as Appendix C to this Work Plan. A summary of the baseline groundwater monitoring event is included as Appendix H.

4.3 Performance Monitoring (Events #1 through #8)

4.3.1 Performance Monitoring

Each performance monitoring event will be initiated approximately 60 days after its associated ERD injection event. Laboratory analytical data will be used to gauge the effectiveness of the ERD injections and to provide a constant feedback loop between the injection program and operational data. Data collected during each performance monitoring event will support decisions pertaining to potential modifications to the reagent strength, injected volumes, or injection frequency over the duration of the active ERD period. The SOW for each performance monitoring event will be consistent and includes:

- Groundwater sample collection from a network of 20 monitoring wells in accordance with the SAP, provided as Appendix C to this Work Plan;
- Submittal of samples for laboratory analysis including VOCs, light gases (methane, ethane, ethene), and TOC; and
- Field monitoring with a hand-held device for field parameters including pH, ORP, specific conductance, temperature, and DO.

Over the duration of the injection program, additional monitoring wells not included in Appendix H may replace or be used to supplement the proposed monitoring program, as necessary. Based on the results of the performance monitoring, modifications may

be made to both the ERD injection (volumes, locations) and the Performance Monitoring (number of wells, locations monitored, parameters monitored). Note, that significant deviations from the proposed Performance Monitoring will be submitted to the USACE and NCDENR with justification for approval prior to implementation.

4.3.2 Methane Monitoring

Methane monitoring will be conducted to evaluate the potential risk associated with vadose zone methane concentrations. The development of reducing conditions within the injection areas and the subsequent increase in dissolved-phase methane concentrations may potentially result in increased methane at the groundwater interface. While vapor phase methane attenuates readily under aerobic conditions, the proposed monitoring program will be used to confirm that methane present in the vadose zone does not represent a vapor intrusion risk. Six soil gas probes will be installed in the vicinity of occupied buildings (See **Figure 4-1**) where the potential for methane generation is the greatest as a result of ERD activities. Sampling activities will initially be conducted during each injection and monitoring event using a landfill gas meter over the 2-year time frame in which injection and monitoring events are ongoing. After completion of the eighth ERD injection event, the frequency of methane monitoring may be reduced, based on the results of previous monitoring activities. During the MNA program, it is anticipated that methane monitoring would be reduced to coincide with groundwater monitoring activities on a quarterly basis. Should monitoring results indicate that vadose zone methane concentrations are not increasing as a result ERD activities, the methane monitoring program may be reduced in frequency or discontinued.

4.4 MNA Monitoring

MNA monitoring will be completed on a quarterly basis for a period of 2 years (8 groundwater monitoring events) following the completion of ERD injection events and performance monitoring activities.

The monitoring well network will consist of 32 monitor wells, similar to the well network utilized during the Baseline Groundwater Monitoring Program (Appendix H). Samples will be collected and analyzed for VOCs, light gases, and TOC. While the anticipated monitoring network is presented in Appendix H, the well network will potentially be refined following completion of the ERD injection activities. This will ensure selection of the most efficient monitoring well network to provide supporting data to validate the effectiveness of both the ERD and MNA technologies.

CITY: San Fransisco DIV/GROUP: IM/85 DB: ME LD: PIC: PM: TM: TR:
Project #:66004546.0051.CDEN0
Q:\USACE\FormerNAD\RAWP\mxd\Fig_4-1_MonitoringNetwork_TransitionZone.mxd: 12/8/2011

LEGEND:

- PROPOSED INJECTION WELL
- PROPOSED MONITORING WELL
- PROPOSED SOIL VAPOR MONITORING LOCATION
- EXISTING MONITORING WELL
- SAMPLED DURING BASELINE AND MNA EVENTS
- SAMPLED DURING ALL SAMPLING EVENTS
- SURROUNDING PROPERTY BOUNDARY
- APPROXIMATE DIRECTION OF GROUNDWATER FLOW IN THE TRANSITION ZONE

TCE PLUME: TRANSITION ZONE

- INFERRED TCE CONTOUR > 500 ug/L
- TCE CONTOUR > 500 ug/L
- TCE CONTOUR > 1,000 ug/L

NOTE:

1. THE LOCATIONS SHOWN ON THIS FIGURE ARE APPROXIMATE AND BASED ON GEOREFERENCEDDATA FROM HISTORIC SITE FIGURES.
2. PLUMES ADAPTED FROM THE FOCUSED FEASIBILITY STUDY (SAIC, 2009)

CHARLOTTE NAVAL AMMUNITION DEPOT
2820 NEVADA BLVD, CHARLOTTE, NC

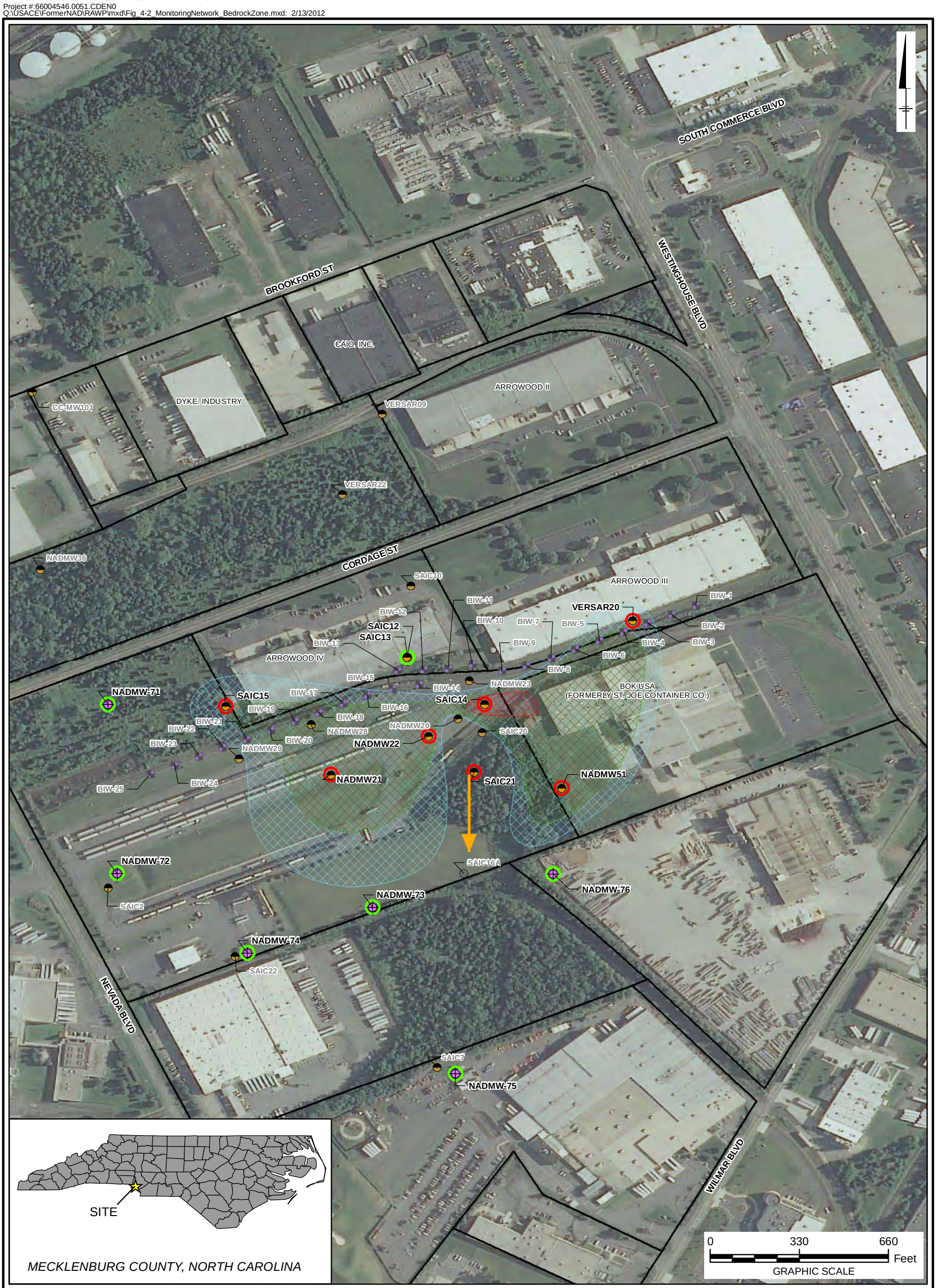
REMEDIAL ACTION WORK PLAN

TRANSITION ZONE HSU - MONITORING PROGRAM

PIKA INTERNATIONAL, INC. A JOINT VENTURE — MALCOLM PIRNIE

FIGURE 4-1

CITY: San Fransisco DIV/GROUP: IM/85 DB: ME LD: PIC: PM: TM: TR:
Project #:66004546.0051.CDEN0
Q:\USACE\FormerNAD\RAWP\mxd\Fig_4-2_MonitoringNetwork_BedrockZone.mxd: 2/13/2012



LEGEND:

- PROPOSED INJECTION WELL
- PROPOSED MONITORING WELL
- EXISTING MONITORING WELL
- MULTIPORT DEEP BEDROCK WELL
- SAMPLED DURING BASELINE AND MNA EVENT
- SAMPLED DURING ALL SAMPLING EVENT
- APPROXIMATE DIRECTION OF GROUNDWATER FLOW IN THE BEDROCK ZONE

TCE PLUME - BEDROCK ZONE

- TCE CONTOUR > 500 ug/L
- TCE CONTOUR > 1,000 ug/L
- TCE CONTOUR > 10,000 ug/L
- SURROUNDING PROPERTY BOUNDARY

NOTE:

1. THE LOCATIONS SHOWN ON THIS FIGURE ARE APPROXIMATE AND BASED ON GEOREFERENCED DATA FROM HISTORIC SITE FIGURES.
2. PLUMES ADAPTED FROM THE FOCUSED FEASIBILITY STUDY (SAIC, 2009)

CHARLOTTE NAVAL AMMUNITION DEPOT
2820 NEVADA BLVD, CHARLOTTE, NC

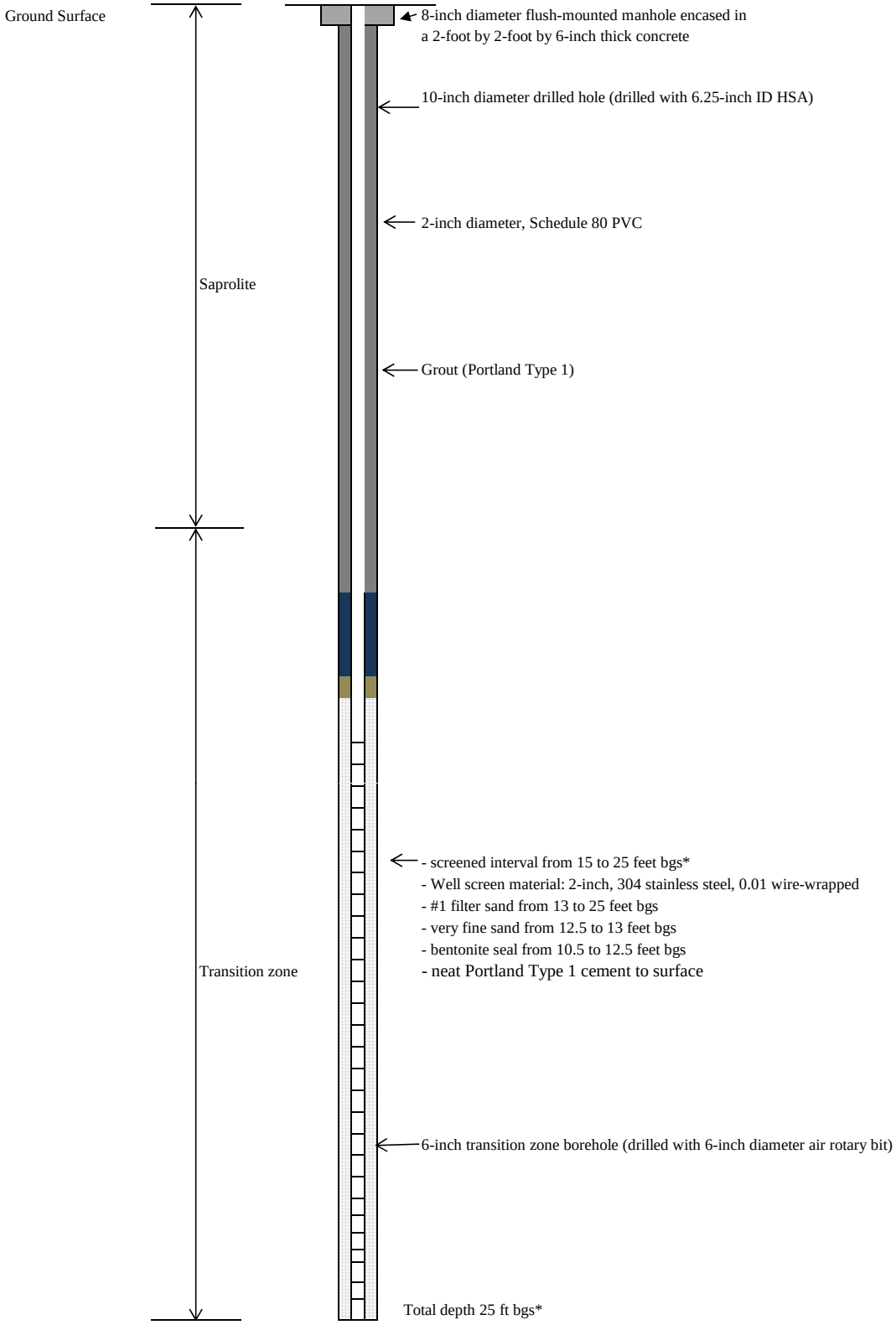
REMEDIAL ACTION WORK PLAN

BEDROCK ZONE HSU MONITORING PROGRAM

PIKA INTERNATIONAL, INC. — A JOINT VENTURE — MALCOLM PIRNIE

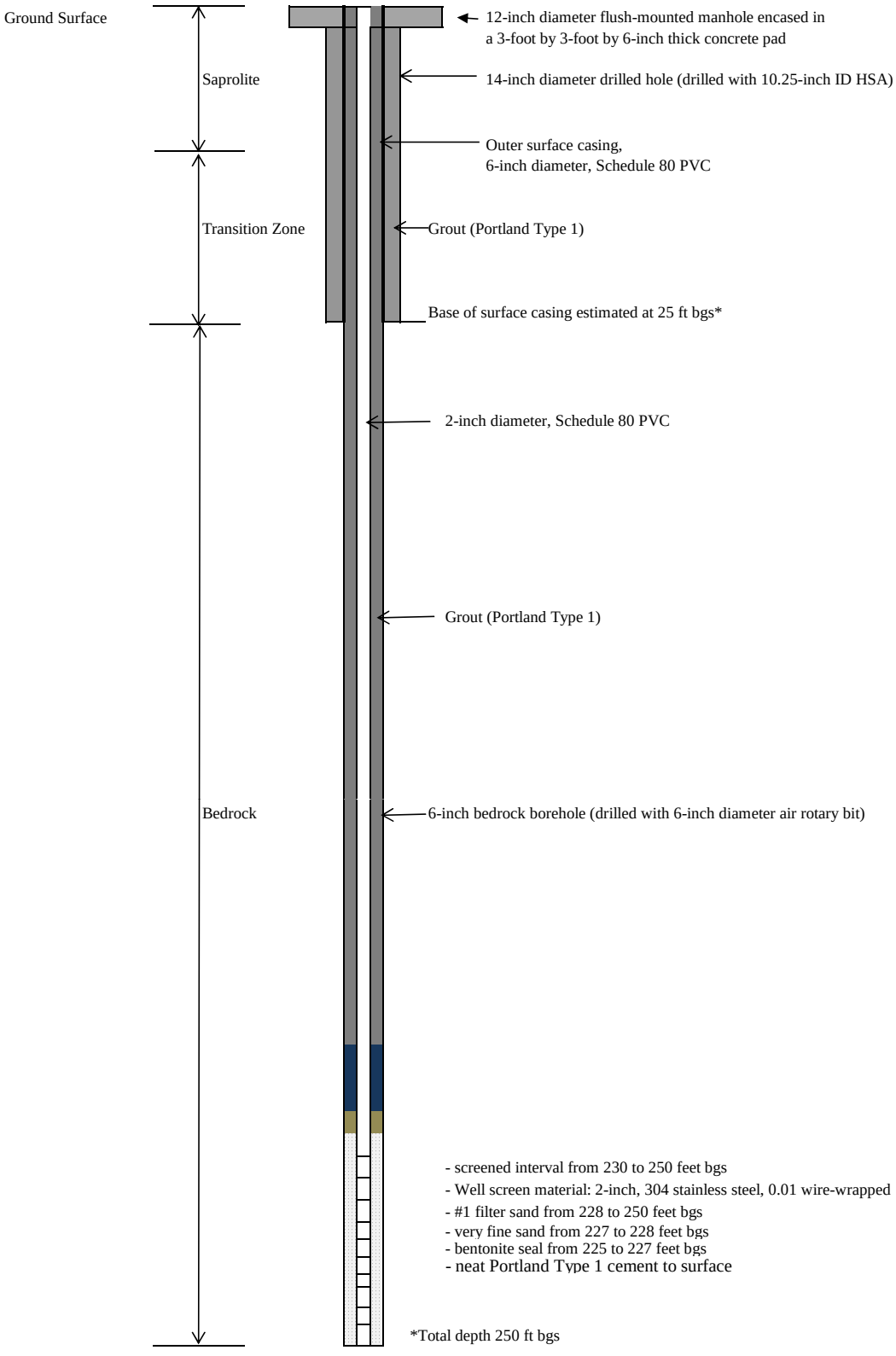
FIGURE 4-2

Figure 4-3
Proposed Transition Zone Well Construction Detail
Former CNAD - Charlotte, North Carolina



* All depth measurements are subject to change based on field conditions.
Not to scale

Figure 4-4
Proposed Bedrock Monitoring Well Construction Detail
Former CNAD - Charlotte, North Carolina



* All depth measurments are subject to change based on field conditions.
Not to scale.

5. Reporting

After each monitoring event (baseline and performance monitoring), a report will be prepared to document the activities completed, to provide a summary of the most recent analytical data and to provide a summary of subsequent phases of work.

5.1 Baseline Monitoring Report

Following completion of monitoring well installation and the baseline groundwater sampling, a Baseline Monitoring Report will summarize activities completed at the CNAD site up to and including the baseline groundwater monitoring event. The Baseline Monitoring Report will summarize the following activities:

- Installation of 11 new monitoring wells in the transition zone and bedrock zone. Information provided will include soil boring logs, well construction details, site maps, and well development data;
- IDW disposal documentation including field notes, waste profiles, and disposal manifests;
- Installation of six methane monitoring points and baseline methane monitoring results;
- Summary of site-wide groundwater elevations and potentiometric maps developed for the transition zone and bedrock zone;
- Summary of baseline groundwater monitoring field activities completed, including field notes, results of field parameter monitoring and sample collection;
- Summary of laboratory analytical results. Particular focus will be placed on TCE and establishing plume delineation prior to implementation of the injection system; and
- Conclusions including any potential modifications necessary to the proposed ERD system layout that may be necessary as a result of new information obtained.

5.2 Remedial Action Report

The Remedial Action Report (RAR) will be prepared and submitted following completion of the remedial system construction and initiation of the first ERD injection event. The report will be prepared in order to certify that all elements in the RAWP have been completed. Elements of the report are as follows:

- The RAR will provide a project summary including the monitor and injection well installation activities, ERD injection system construction activities, and the initial ERD injection event. The report will include field notes, documentation of construction, and any deviation from the proposed design;
- Updated schedule of completion detailing the ERD injection schedule, monitoring schedule, and reporting schedule;
- As built drawings, stamped by a Professional Engineer, (PE), showing all pertinent items associated with the remedial system at the site.

5.3 Performance Monitoring Report

Each Performance Monitoring Report will document the performance monitoring activities detailed in Section 4.3. Performance Monitoring Reports will be prepared and submitted on a quarterly basis, following receipt, review, and evaluation of groundwater laboratory analytical results, for a period of 2 years (8 reports total). Reports will include the following:

- Summary of field activities completed, including an ERD injection summary table, completed field forms, IDW disposal manifests, and discussion regarding significant O&M activities;
- Evaluation of groundwater flow including potentiometric surface maps and a tabular summary of water levels collected during the performance monitoring events;
- Key observations pertaining to operational ERD parameters including TOC and methane;
- Evaluation of TCE concentrations including isocontour figures, tabular comparison of TCE concentrations to historical values;

- Evaluation of ERD processes including graphical summary of CVOC parent and daughter product concentrations, TOC, methane, and pH trend plots over time;

- Results of methane monitoring;

- Laboratory analytical data, corresponding chains of custody, and data validation reports; and

- Recommendations for modifications to the remedial action tasks including the ERD injection program and the groundwater monitoring program.

5.4 Annual MNA Monitoring Report

After completion of ERD injections, a MNA program will be implemented to track continued TCE reduction in groundwater. The MNA program will include quarterly groundwater monitoring, as detailed in Section 4.4. The first Annual MNA Monitoring Report will be prepared and submitted following the first four quarters of MNA monitoring. The report will document the following:

- Summary of field activities completed, including completed field forms, IDW disposal manifests, and discussion regarding significant O&M activities;

- Evaluation of groundwater flow including potentiometric surface maps and tabular summary of quarterly water levels collected during the monitoring events;

- Key observations pertaining to operational ERD parameters including TOC and methane;

- Evaluation of TCE concentrations including isocontour figures, tabular comparison of TCE concentrations to historical values, and graphical summary of TCE trend plots over time;

- Evaluation of ERD processes including graphical summary of CVOC parent and daughter product concentrations, TOC, methane, and pH trend plots over time;

- Results of methane monitoring;
- Laboratory analytical data, corresponding chains of custody, and data validation reports; and
- Recommendations for modifications to the remedial action tasks associated with the MNA groundwater monitoring program.

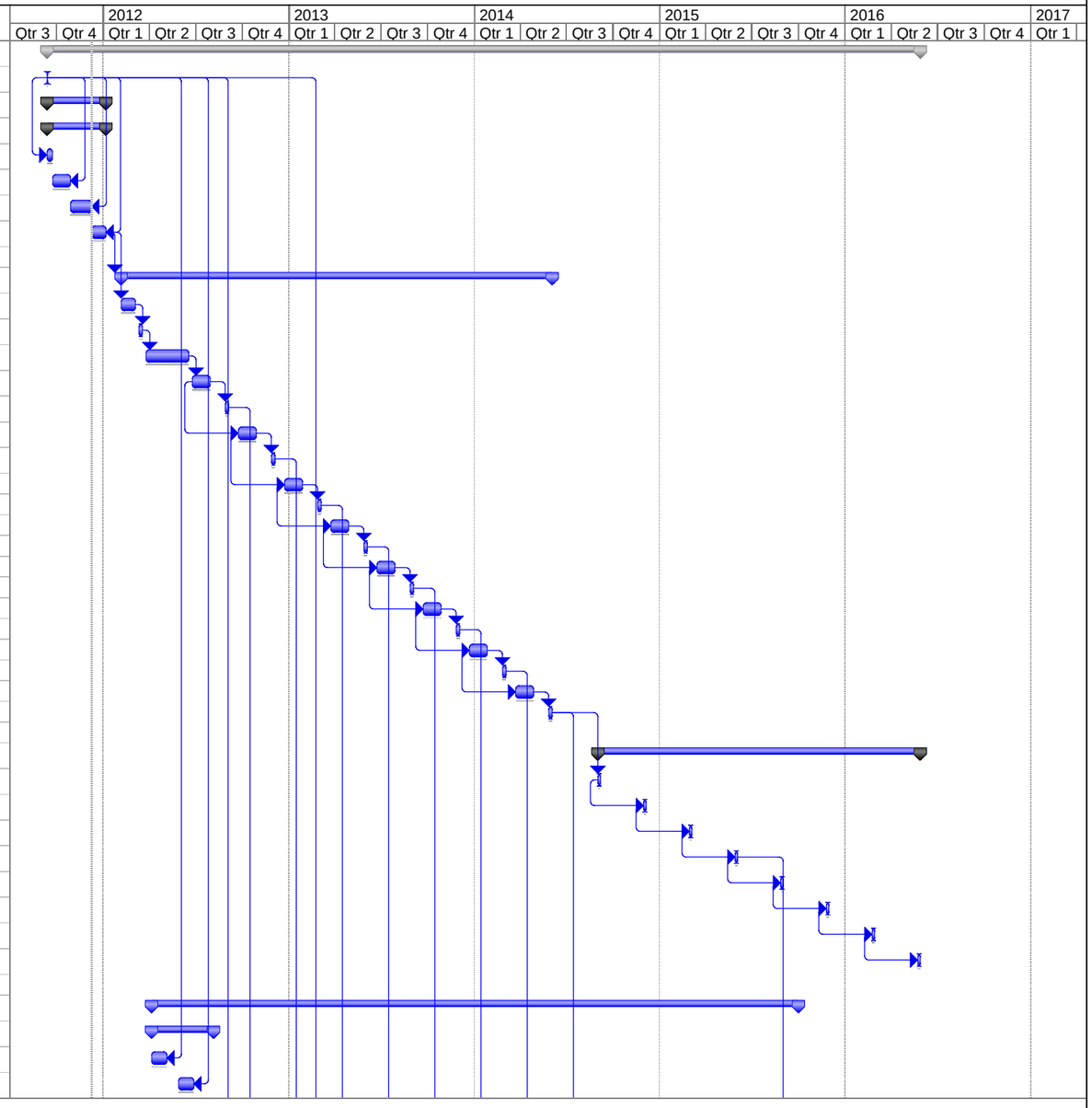
6. Schedule

A schedule has been prepared to show the anticipated timeframe necessary to complete tasks associated with the Former CNAD project. Specific tasks include RAWP preparation, well installation, baseline sampling, remedial system construction, ERD implementation, monitoring, and reporting activities. The schedule has been included as **Figure 6-1**.

It should be noted that the schedule was developed using several assumptions including designated report review times, accepted site access agreements, and the ability to implement the approved remedial system, as designed. If any of these assumptions are proven inaccurate, the proposed schedule may shift, extending the project duration. Updates of the schedule will be provided as part of Monitoring Reports to document the current status of the project and to identify the current dates for completion of remaining tasks.

Charlotte Naval Ammunition Depot
Project Schedule

ID	Task Name	Duration	Start	Finish	Predecessors
0	Charlotte Naval Ammunition Depot	1720 days	Tue 9/13/11	Sat 5/28/16	
1	Notice to Proceed	1 day	Tue 9/13/11	Tue 9/13/11	
2	Task 1	116 days	Tue 9/13/11	Fri 1/6/12	
3	Remedial Action Work Plan	116 days	Tue 9/13/11	Fri 1/6/12	
4	Progress Chart	10 days	Tue 9/13/11	Fri 9/23/11	1SS
5	Draft	35 days	Sat 9/24/11	Fri 10/28/11	1FF+45 days
6	Final	42 days	Sat 10/29/11	Fri 12/9/11	1FF+87 days
7	Revised Final	28 days	Sat 12/10/11	Fri 1/6/12	1FF+115 days
8					
9	Task 2: Remedial Action Implementation	849 days	Mon 2/6/12	Tue 6/3/14	7
10	Installation of transition zone and bedrock MWs	28 days	Mon 2/6/12	Sun 3/4/12	7FS+30 days
11	Baseline sampling event	7 days	Mon 3/12/12	Sun 3/18/12	10FS+7 days
12	Installation of Injection wells (bedrock / transition)	84 days	Mon 3/26/12	Sun 6/17/12	11FS+7 days
13	Injection Event #1	35 days	Mon 6/25/12	Sun 7/29/12	12FS+7 days
14	Performance Monitoring Event #1	7 days	Wed 8/29/12	Tue 9/4/12	13FS+30 days
15	Injection Event #2	35 days	Mon 9/24/12	Sun 10/28/12	13SS+91 days
16	Performance Monitoring Event #2	7 days	Wed 11/28/12	Tue 12/4/12	15FS+30 days
17	Injection Event #3	35 days	Mon 12/24/12	Sun 1/27/13	15SS+91 days
18	Performance Monitoring Event #3	7 days	Wed 2/27/13	Tue 3/5/13	17FS+30 days
19	Injection Event #4	35 days	Mon 3/25/13	Sun 4/28/13	17SS+91 days
20	Performance Monitoring Event #4	7 days	Wed 5/29/13	Tue 6/4/13	19FS+30 days
21	Injection Event #5	35 days	Mon 6/24/13	Sun 7/28/13	19SS+91 days
22	Performance Monitoring Event #5	7 days	Wed 8/28/13	Tue 9/3/13	21FS+30 days
23	Injection Event #6	35 days	Mon 9/23/13	Sun 10/27/13	21SS+91 days
24	Performance Monitoring Event #6	7 days	Wed 11/27/13	Tue 12/3/13	23FS+30 days
25	Injection Event #7	35 days	Mon 12/23/13	Sun 1/26/14	23SS+91 days
26	Performance Monitoring Event #7	7 days	Wed 2/26/14	Tue 3/4/14	25FS+30 days
27	Injection Event #8	35 days	Mon 3/24/14	Sun 4/27/14	25SS+91 days
28	Performance Monitoring Event #8	7 days	Wed 5/28/14	Tue 6/3/14	27FS+30 days
29					
30	MNA Events for Transition and Bedrock wells	635 days	Tue 9/2/14	Sat 5/28/16	
31	Quarter #1	5 days	Tue 9/2/14	Sat 9/6/14	28FS+90 days
32	Quarter #2	5 days	Mon 12/1/14	Fri 12/5/14	31SS+90 days
33	Quarter #3	5 days	Sun 3/1/15	Thu 3/5/15	32SS+90 days
34	Quarter #4	5 days	Sat 5/30/15	Wed 6/3/15	33SS+90 days
35	Quarter #5	5 days	Fri 8/28/15	Tue 9/1/15	34SS+90 days
36	Quarter #6	5 days	Thu 11/26/15	Mon 11/30/15	35SS+90 days
37	Quarter #7	5 days	Wed 2/24/16	Sun 2/28/16	36SS+90 days
38	Quarter #8	5 days	Tue 5/24/16	Sat 5/28/16	37SS+90 days
39					
40	Task 3: RA Report / Monitoring Report	1274 days	Fri 4/6/12	Thu 10/1/15	
41	Baseline Sampling Event Report	122 days	Fri 4/6/12	Sun 8/5/12	
42	Draft	30 days	Fri 4/6/12	Sat 5/5/12	1FF+235 days
43	Final	30 days	Tue 5/29/12	Wed 6/27/12	1FF+288 days



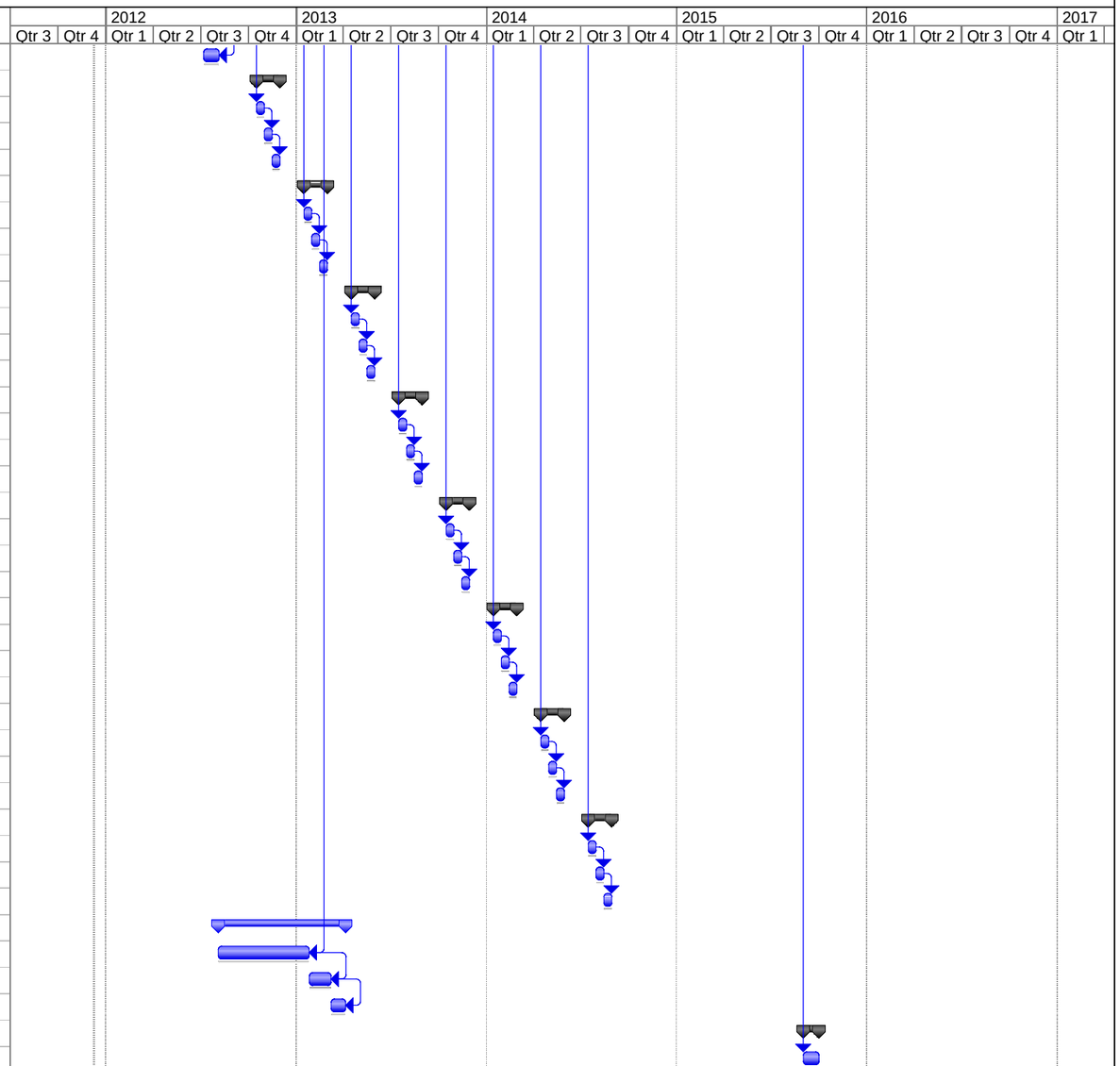
Project: Charlotte Naval Ammunition D
Date: Thu 12/8/11

Task | Progress | Summary | External Tasks | Deadline
Split | Milestone | Project Summary | External Milestone

Contract No. W912DY-10-D0025, DO 0007
December 2011

Charlotte Naval Ammunition Depot
Project Schedule

ID	Task Name	Duration	Start	Finish	Predecessors
44	Revised Final	30 days	Sat 7/7/12	Sun 8/5/12	1FF+327 days
45	1st Performance Monitoring Report	45 days	Wed 10/17/12	Fri 11/30/12	
46	Draft	15 days	Wed 10/17/12	Wed 10/31/12	14FS+42 days
47	Final	15 days	Thu 11/1/12	Thu 11/15/12	46
48	Revised Final	15 days	Fri 11/16/12	Fri 11/30/12	47
49	2nd Performance Monitoring Report	45 days	Wed 1/16/13	Fri 3/1/13	
50	Draft	15 days	Wed 1/16/13	Wed 1/30/13	16FS+42 days
51	Final	15 days	Thu 1/31/13	Thu 2/14/13	50
52	Revised Final	15 days	Fri 2/15/13	Fri 3/1/13	51
53	3rd Performance Monitoring Report	45 days	Wed 4/17/13	Fri 5/31/13	
54	Draft	15 days	Wed 4/17/13	Wed 5/1/13	18FS+42 days
55	Final	15 days	Thu 5/2/13	Thu 5/16/13	54
56	Revised Final	15 days	Fri 5/17/13	Fri 5/31/13	55
57	4th Performance Monitoring Report	45 days	Wed 7/17/13	Fri 8/30/13	
58	Draft	15 days	Wed 7/17/13	Wed 7/31/13	20FS+42 days
59	Final	15 days	Thu 8/1/13	Thu 8/15/13	58
60	Revised Final	15 days	Fri 8/16/13	Fri 8/30/13	59
61	5th Performance Monitoring Report	45 days	Wed 10/16/13	Fri 11/29/13	
62	Draft	15 days	Wed 10/16/13	Wed 10/30/13	22FS+42 days
63	Final	15 days	Thu 10/31/13	Thu 11/14/13	62
64	Revised Final	15 days	Fri 11/15/13	Fri 11/29/13	63
65	6th Performance Monitoring Report	45 days	Wed 1/15/14	Fri 2/28/14	
66	Draft	15 days	Wed 1/15/14	Wed 1/29/14	24FS+42 days
67	Final	15 days	Thu 1/30/14	Thu 2/13/14	66
68	Revised Final	15 days	Fri 2/14/14	Fri 2/28/14	67
69	7th Performance Monitoring Report	45 days	Wed 4/16/14	Fri 5/30/14	
70	Draft	15 days	Wed 4/16/14	Wed 4/30/14	26FS+42 days
71	Final	15 days	Thu 5/1/14	Thu 5/15/14	70
72	Revised Final	15 days	Fri 5/16/14	Fri 5/30/14	71
73	8th Performance Monitoring Report	45 days	Wed 7/16/14	Fri 8/29/14	
74	Draft	15 days	Wed 7/16/14	Wed 7/30/14	28FS+42 days
75	Final	15 days	Thu 7/31/14	Thu 8/14/14	74
76	Revised Final	15 days	Fri 8/15/14	Fri 8/29/14	75
77	Remedial Action Report	245 days	Sat 8/4/12	Fri 4/5/13	
78	Draft	175 days	Sat 8/4/12	Fri 1/25/13	1FF+500 days
79	Final	42 days	Sat 1/26/13	Fri 3/8/13	78FF+42 days
80	Revised Final	28 days	Sat 3/9/13	Fri 4/5/13	79FF+28 days
81	MNA / Reporting	30 days	Wed 9/2/15	Thu 10/1/15	
82	1st Annual MNA Monitoring Report	30 days	Wed 9/2/15	Thu 10/1/15	34FS+90 days



Project: Charlotte Naval Ammunition D
Date: Thu 12/8/11

Task | Progress | Summary | External Tasks | Deadline
Split | Milestone | Project Summary | External Milestone

Contract No. W912DY-10-D0025, DO 0007
December 2011

7. References

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Appendix A

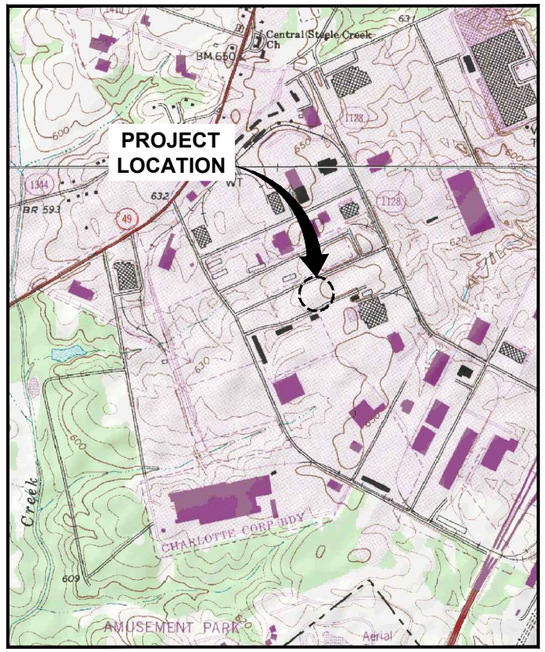
Design Drawings

CONCEPT DESIGN DRAWINGS

FORMER CHARLOTTE NAVAL AMMUNITION DEPOT

CHARLOTTE, NORTH CAROLINA

ENHANCED REDUCTIVE DECHLORINATION DESIGN



REFERENCE: BASE MAP USGS 7.5 MINUTE QUADRANGLE., (QUADNAME), (ST)., (DATE)

LOCATION MAP

0 2000' 4000'
GRAPHIC SCALE

NORTH CAROLINA

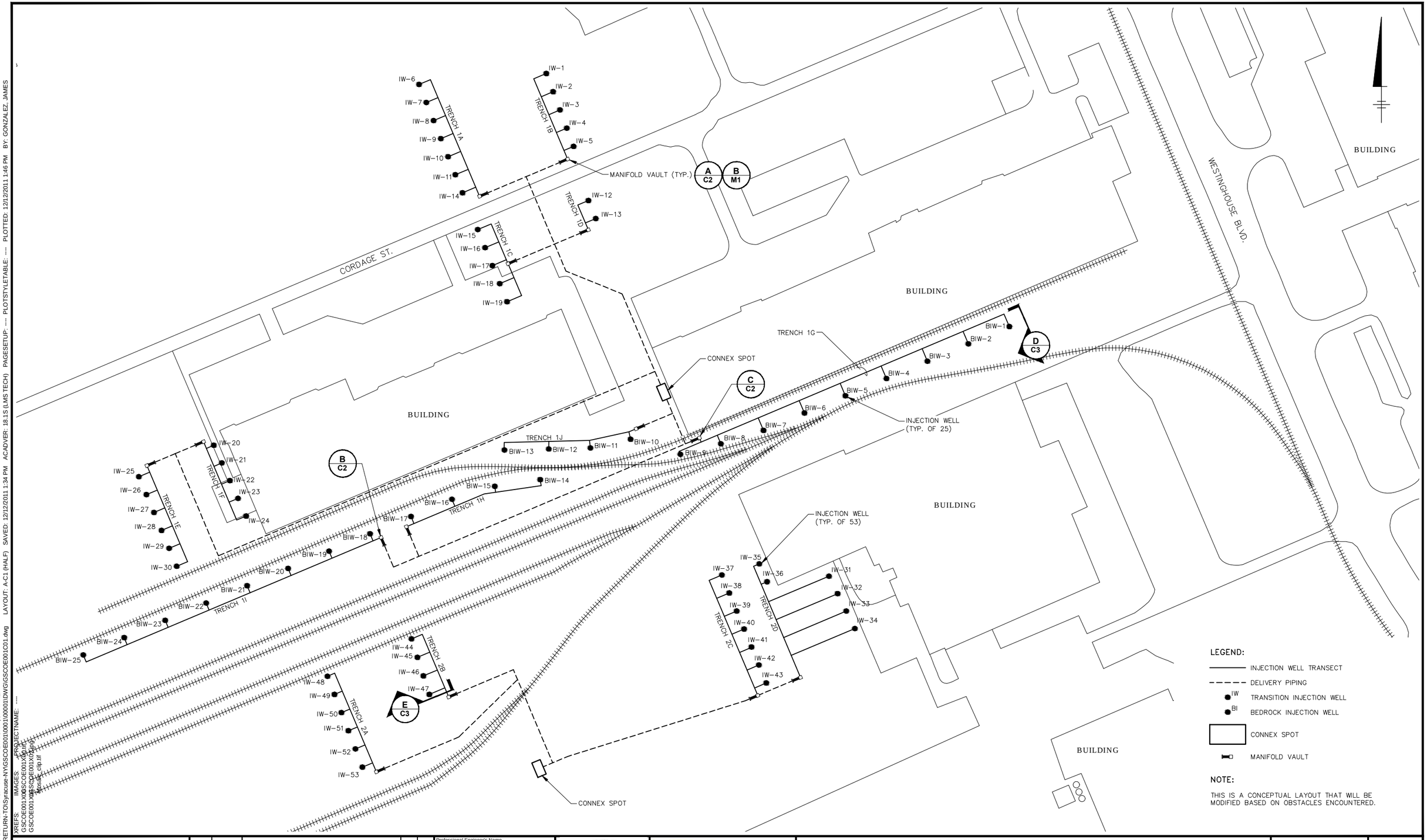
DECEMBER 2011

UNITED STATES ARMY CORPS OF ENGINEERS
CHARLOTTE, NORTH CAROLINA

PIKA INTERNATIONAL, INC. A JOINT VENTURE — MALCOLM PIRNIE

MALCOLM PIRNIE, INC.
ENGINEERING LICENSE # F-0299

INDEX TO DRAWINGS



80' 0 80' 160'
1"=80'

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No.	Date	Revisions	By	Ckd

Professional Engineer's Name: **ANDREW DAVIS**
Professional Engineer's No.: NC#027773

State	Date Signed	Project Mgr.
NC		AD
Designed by	Drawn by	Checked by
SS	SS	MS

NORTH CAROLINA PROFESSIONAL SEAL 027773 ENGINEER ANDREW A. DAVIS 12/9/11

PIKA INTERNATIONAL, INC. A JOINT VENTURE MALCOLM PIRNIE
MALCOLM PIRNIE, INC.
ENGINEERING LICENSE # F-0299

UNITED STATES ARMY CORPS OF ENGINEERS • CHARLOTTE, NORTH CAROLINA
CONCEPT DESIGN PHASE

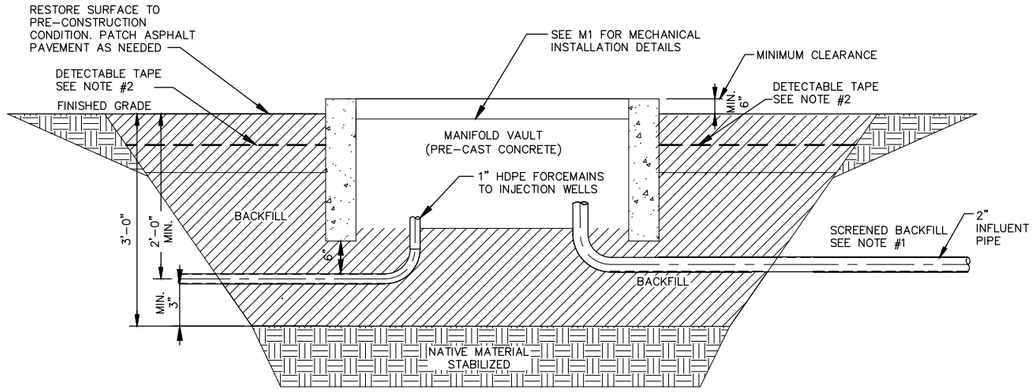
INJECTION LINE LAYOUT PLAN

CIVIL

Project No. GSCOE001.0001.00001
Date DECEMBER 2011

30 PATEWOOD DR.
SUITE 155
GREENVILLE, SC 29615
TEL: 864-987-3900

A-1 C1



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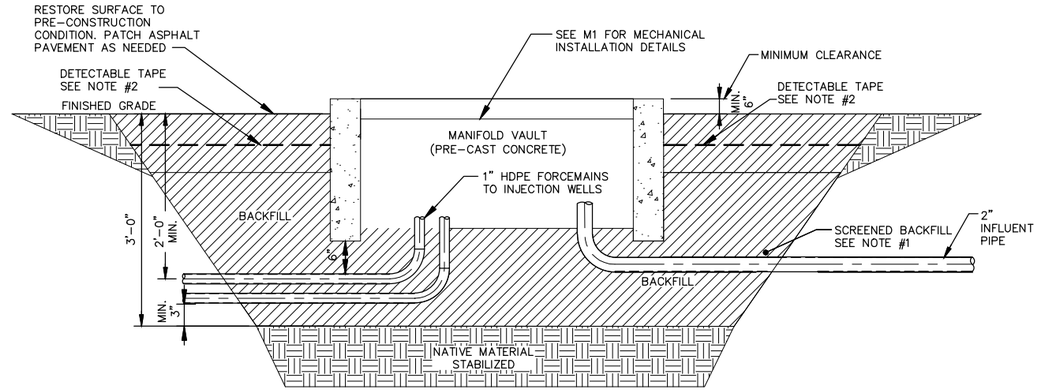

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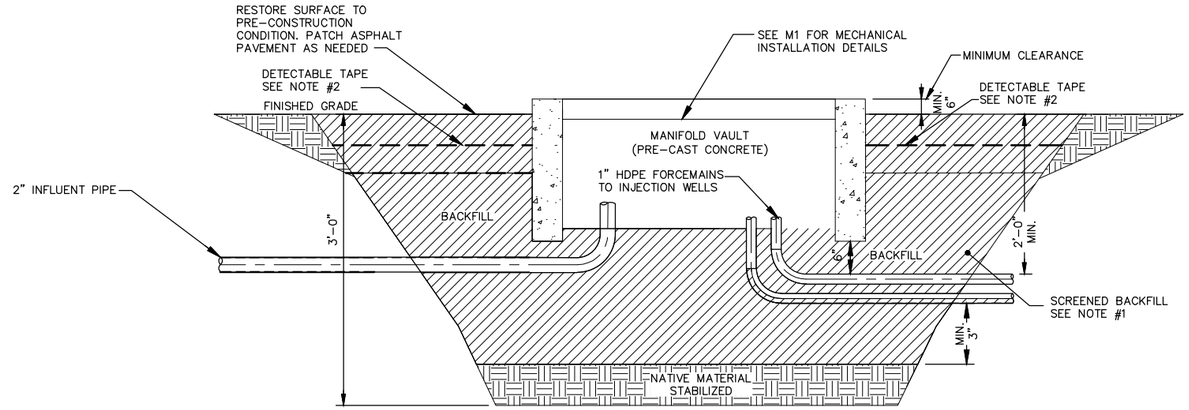

MANIFOLD VAULT DETAIL (1 DIRECTION)

NOT TO SCALE

C

NOTES:
1. ALL BACKFILL MATERIAL SURROUNDING PIPE WILL BE NATIVE, MOISTURE CONDITIONED SOIL MATERIALS THAT PASS 1" SCREEN.
2. A 3" DETECTABLE TAPE WILL BE INSTALLED ABOVE THE PROCESS PIPELINES. BLUE TAPE WILL BE CENTERED ABOVE THE , 1 FOOT BELOW FINISHED GRADE AND LABELED "CAUTION BURIED PIPELINE BELOW".

CITY: DV/GROUP: DB: LD: PIC: PM: TM: LYR:ON=*;OFF=*REF*
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SCALE(S) AS INDICATED

THIS BAR REPRESENTS ONE INCH ON THE ORIGINAL DRAWING: USE TO VERIFY FIGURE REPRODUCTION SCALE

No.	Date	Revisions	By	Ckd

Professional Engineer's Name: ANDREW DAVIS
Professional Engineer's No.: NC#027773

State	Date Signed	Project Mgr.
NC		AD

Designed by	Drawn by	Checked by
SS	SS	MS

NORTH CAROLINA PROFESSIONAL SEAL 027773 ENGINEER ANDREW A. DAVIS 12/9/11

PIKA INTERNATIONAL, INC. A JOINT VENTURE MALCOLM PIRNIE

MALCOLM PIRNIE, INC.
ENGINEERING LICENSE # F-0299

UNITED STATES ARMY CORPS OF ENGINEERS • CHARLOTTE, NORTH CAROLINA
CONCEPT DESIGN PHASE

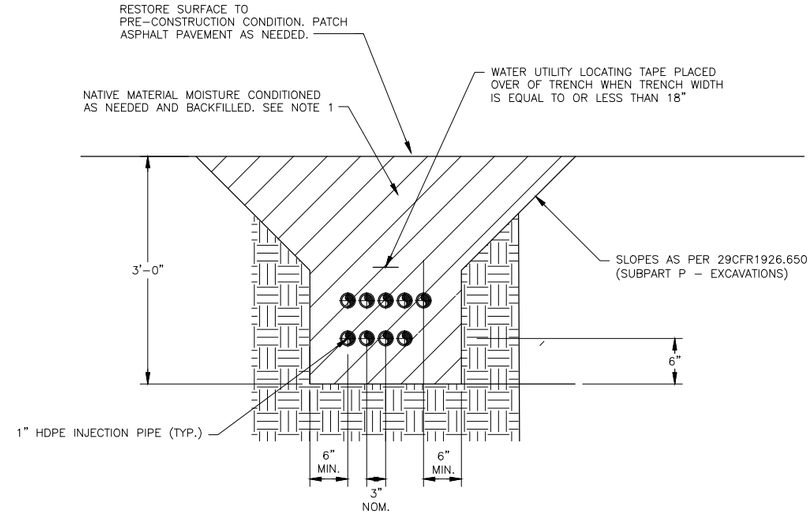
CIVIL DETAILS

CIVIL

Project No. GSCOE001.0001.00001
Date DECEMBER 2011

30 PATEWOOD DR.
SUITE 155
GREENVILLE, SC 29615
TEL: 864-987-3900

A-2
C2

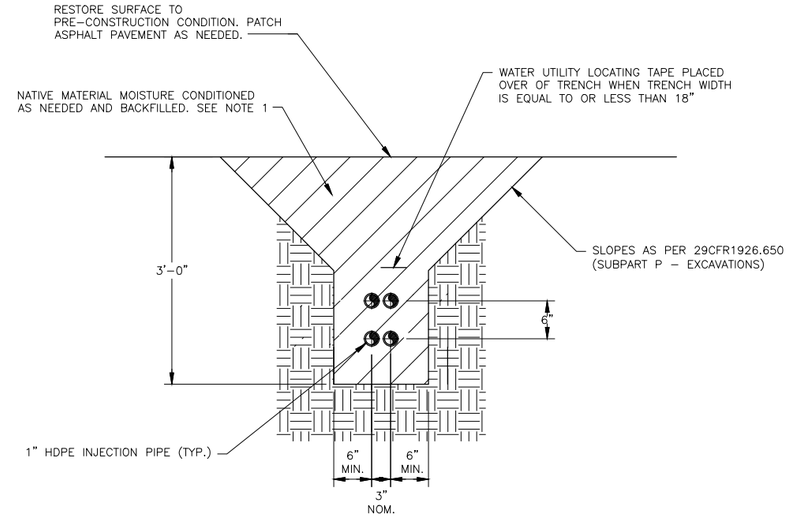


D C1 C2 TRENCHING SECTION 1G (9 PIPES)

NOT TO SCALE

TRENCHING SCHEDULE		
TRENCH ID	# OF HDPE INJECTION PIPELINES	MINIMUM WIDTH OF TRENCH
1A	6	1'-6"
1B	5	1'-6"
1C	6	1'-6"
1D	2	1'-3"
1E	6	1'-6"
1F	5	1'-6"
1G	9	2'-0"
1H	4	1'-3"
1I	4	1'-3"
1J	8	1'-9"
2A	6	1'-6"
2B	4	1'-3"
2C	7	1'-9"
2D	6	1'-6"

NOTES:
1. ALL BACKFILL MATERIAL SURROUNDING PIPE WILL BE NATIVE, MOISTURE CONDITIONED SOIL MATERIALS THAT PASS 1" SCREEN.
2. A 3" DETECTABLE TAPE WILL BE INSTALLED ABOVE THE PROCESS PIPELINES. BLUE TAPE WILL BE CENTERED ABOVE THE FORCEMAINS, 1 FOOT BELOW FINISHED GRADE AND LABELED "CAUTION BURIED PIPLINE BELOW".
3. ALL TRENCHES SHALL BE CONSTRUCTED USING THIS DETAIL AS A REFERENCE. DIMENSIONS BETWEEN PIPELINES WILL REMAIN THE SAME, OVERALL TRENCH DIMENSIONS WILL VARY BASED ON THE NUMBER OF PIPES. SEE TABLE.



E C1 C2 TRENCHING SECTION 2B (4 PIPES)

NOT TO SCALE

CITY: DIV/GROUP: DB: LD: PIC: PM: TM: LYR:ON=*;OFF=*REF*
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XREFS: IMAGES: PROJECTNAME: ----
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GSCOE001X00.tif
GSCOE001X02.jpg

SCALE(S) AS INDICATED

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USE TO VERIFY FIGURE REPRODUCTION SCALE

No.	Date	Revisions	By	Ckd

Professional Engineer's Name: ANDREW DAVIS
Professional Engineer's No.: NC#027773
State: NC | Date Signed: 12/9/11 | Project Mgr.: AD
Designed by: SS | Drawn by: SS | Checked by: MS

NORTH CAROLINA PROFESSIONAL SEAL 027773 ENGINEER ANDREW A. DAVIS

PIKA INTERNATIONAL, INC. A JOINT VENTURE MALCOLM PIRNIE

MALCOLM PIRNIE, INC.
ENGINEERING LICENSE # F-0299

UNITED STATES ARMY CORPS OF ENGINEERS • CHARLOTTE, NORTH CAROLINA
CONCEPT DESIGN PHASE

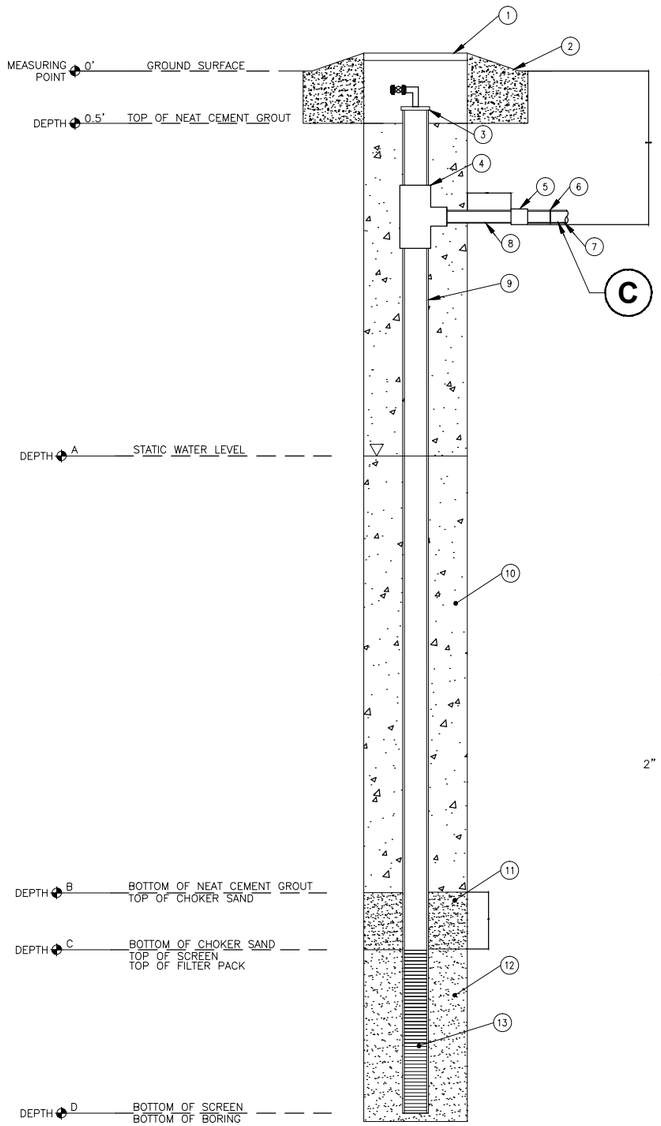
INJECTION WELL DETAILS

CIVIL

Project No. GSCOE001.0001.00001
Date DECEMBER 2011

30 PATEWOOD DR.
SUITE 155
GREENVILLE, SC 29615
TEL: 864-987-3900

A-3
C3

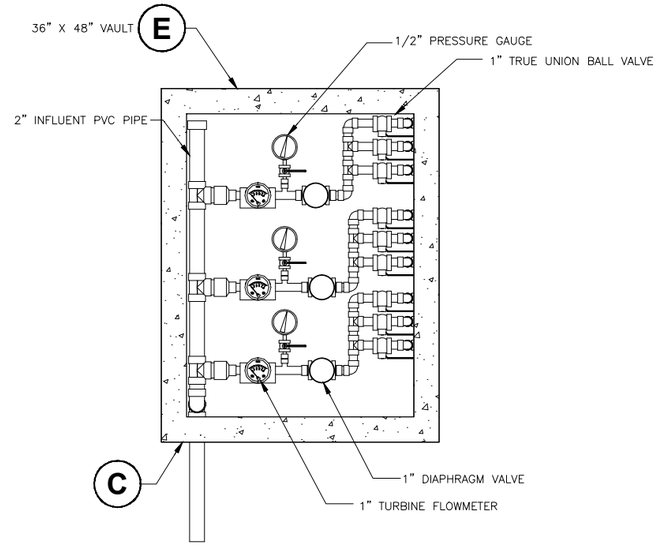


INJECTION WELL DETAIL (TYP.) A

NOT TO SCALE

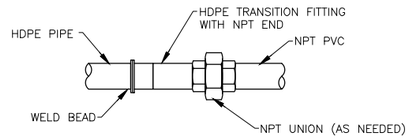
1	COVER FLUSH MOUNT MONUMENT
2	CONCRETE PAD
3	2" DIAMETER WELL COMPRESSION CAP WITH 1/4" MANUAL PRESSURE BLEED VALVE
4	2"x2"x1" SCHEDULE 80 PVC REDUCING TEE
5	1" SCHEDULE 80 PVC COUPLING
6	1" HDPE TO PVC TRANSITION FITTING
7	1" HDPE FORCEMAIN
8	1" SCHEDULE 80 PVC PIPE
9	2" SCHEDULE 80 PVC WELL CASING
10	ANNULAR SPACE, 100% NEAT CEMENT GROUT
11	ANNULAR SPACE, CHOKER SAND
12	ANNULAR SPACE, FILTER PACK, EXTRA FINE SAND (DSI FILTER SAND)
13	2" DIAMETER STAINLESS STEEL WIRE WRAPPED 0.01" SLOT WELL SCREEN

INJECTION WELL KEY (TYP.)



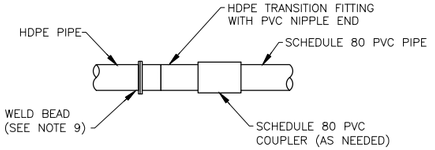
MANIFOLD VAULT DETAIL (TYP.) B

NOT TO SCALE



THREADED HDPE TRANSITION (TYP.) C

NOT TO SCALE

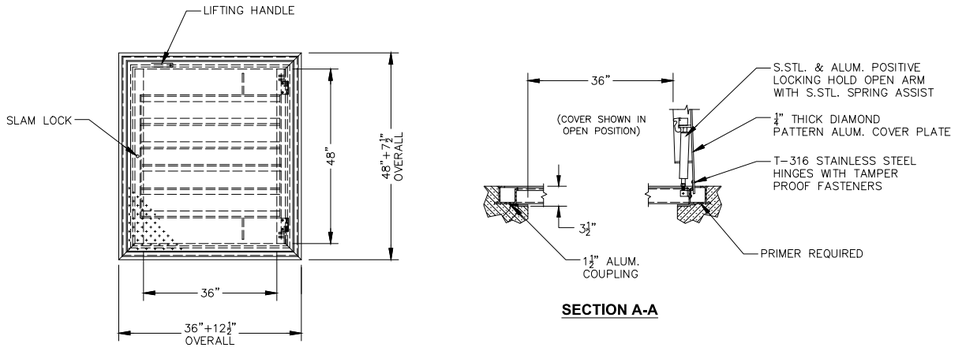


PVC PIPE TRANSITION (TYP.) D

NOT TO SCALE

NOTES:

1. ALL PIPING, INSTRUMENTATION, AND OTHER COMPONENTS SHALL BE INSTALLED AS PER THE MANUFACTURER'S GUIDELINES.
2. ALL PIPING AND INSTRUMENTATION SHALL BE INSTALLED PER PID1, PID2, AND PID3.
3. INSTALLATION OF ELECTRIC COMPONENTS SHALL COMPLY WITH ALL APPLICABLE NEC AND LOCAL CODES.
4. CROSS SECTIONS SHOW ONLY THOSE COMPONENTS NECESSARY TO INDICATE ELEVATIONS AND SPACE REQUIREMENTS FOR PIPING AND MAJOR COMPONENTS.
5. ALL VALVE HANDLES SHALL BE INSTALLED SUCH THAT THEY ARE ACCESSABLE AND OPERABLE THROUGH ENTIRE STROKE.
6. PIPE SIZES AND MATERIALS ARE SHOWN ON THE P&ID.
7. ROUTING OF PIPING SHOWN AS PROPOSED AND APPROXIMATE. ACTUAL ROUTING TO BE FIELD DETERMINED.
8. WELD BEAD SHALL COMPLY WITH MANUFACTURER'S RECOMMENDATIONS.



SECTION A-A

NOTE:

1. ACCESS FRAMES AND COVERS SHALL HAVE A 1/4" ONE-PIECE, MILL FINISH, EXTRUDED ALUMINUM FRAME, INCORPORATING A CONTINUOUS CONCRETE ANCHOR. A 1 1/2" DRAINAGE COUPLING SHALL BE LOCATED IN THE FRONT LEFT CORNER OF THE CHANNEL FRAME. THE INSIDE OF THE FRAME SHALL HAVE A DOOR-SUPPORT LEDGE ON TWO (2) SIDES. BOTH FRAME AND LEDGE MUST BE SUPPORTED BY A FULL BED OF CLASS A CONCRETE. THE DOOR PANEL SHALL BE 1/4" ALUMINUM DIAMOND PLATE, REINFORCED TO WITHSTAND A LIVE LOAD OF H-20 DESIGNATION. DOOR SHALL OPEN TO 90 DEGREES AND AUTOMATICALLY LOCK WITH A T-316 STAINLESS STEEL HOLD OPEN ARM WITH ALUMINUM RELEASE HANDLE. DOOR SHALL CLOSE FLUSH WITH THE FRAME. HINGES AND ALL FASTENING HARDWARE SHALL BE T-316 STAINLESS STEEL. UNIT SHALL LOCK WITH A NON-CORROSIVE LOCKING BAR AND HAVE A NON-CORROSIVE HANDLE. UNIT SHALL CARRY A LIFETIME GUARANTEE AGAINST DEFECTS IN MATERIAL AND/OR WORKMANSHIP.

ACCESS HATCH DETAIL E

NOT TO SCALE

CITY: DIV/GROUP: DB: LD: PIC: PM: TM: LYR:ON=*;OFF=*REF*
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SCALE(S) AS INDICATED

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No.	Date	Revisions	By	Ckd

THIS DRAWING IS THE PROPERTY OF THE ARCADIS ENTITY IDENTIFIED IN THE TITLE BLOCK AND MAY NOT BE REPRODUCED OR ALTERED IN WHOLE OR IN PART WITHOUT THE EXPRESS WRITTEN PERMISSION OF SAME.

Professional Engineer's Name: ANDREW DAVIS
Professional Engineer's No.: NC#027773
State: NC | Date Signed: SEPT 12 9 11 | Project Mgr.: AD
Designed by: SS | Drawn by: SS | Checked by: MS

NORTH CAROLINA PROFESSIONAL ENGINEER SEAL 027773 ANDREW A. DAVIS

PIKA INTERNATIONAL, INC. A JOINT VENTURE MALCOLM PIRNIE
MALCOLM PIRNIE, INC.
ENGINEERING LICENSE # F-0299

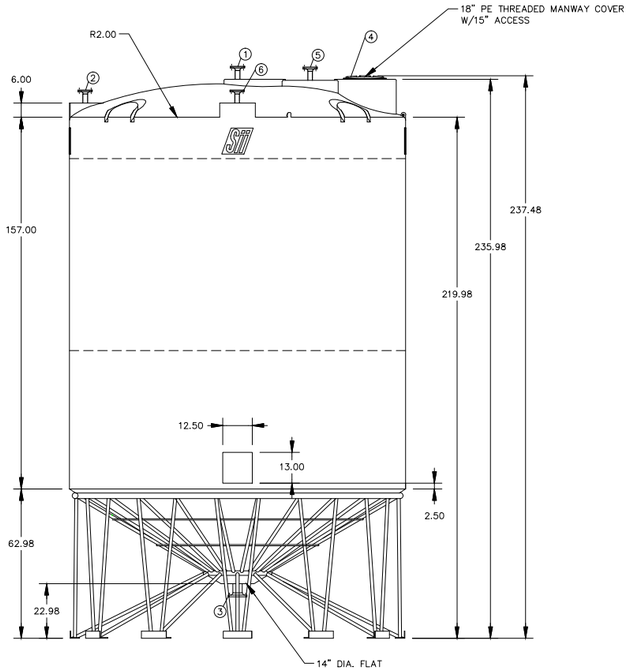
UNITED STATES ARMY CORPS OF ENGINEERS • CHARLOTTE, NORTH CAROLINA
CONCEPT DESIGN PHASE

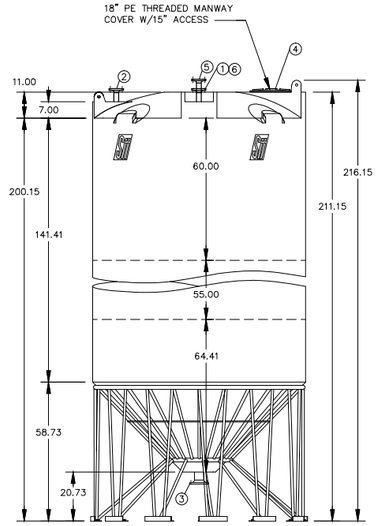
MECHANICAL LAYOUTS

MECHANICAL

Project No. GSCOE001.0001.00001
Date DECEMBER 2011
30 PATEWOOD DR. SUITE 155 GREENVILLE, SC 29615 TEL: 864-987-3900

A-4 M1



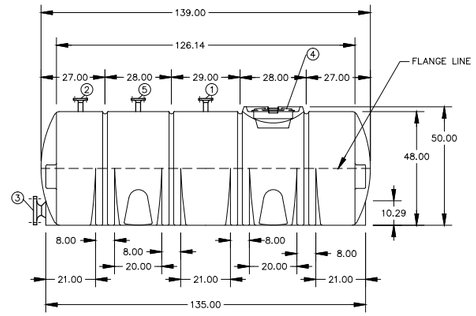


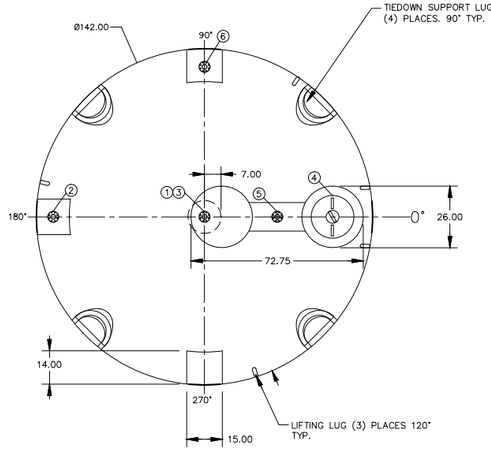
SECTION VIEWS REPRESENTATIVE OF ELEVATIONS ONLY. PLAN VIEW REPRESENTS ORIENTATION OF THE NOZZLES ON THE TANK.

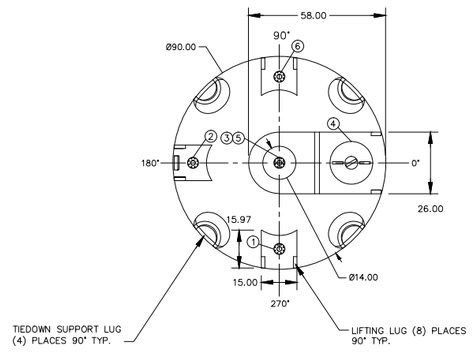
HDLPE = HIGH-DENSITY LINEAR POLYETHYLENE

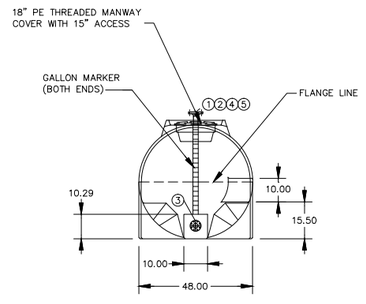
F.R.P. = FIBERGLASS REINFORCED PLASTIC

XLPE = CROSS-LINKED HIGH DENSITY POLYETHYLENE









T-101: MOLASSES STORAGE TANK

LEVEL CONTROL	1	1	2" PVC FLANGE ADAPTER AND 150# PVC BULKHEAD FITTING	PE/PVC	236.00"
VENT	2	1	2" PVC FLANGE ADAPTER AND PVC/EPDM BULKHEAD FITTING	PE/PVC	226.00"
DISCHARGE	3	1	2" PVC FLANGE ADAPTER AND 150# PVC BULKHEAD FITTING	PE/PVC	23.00"
ACCESS	4	1	18" DIA. TOP MANHOLE (15" ACCESS)	PE	237.48"
INLET	5	1	2" PVC FLANGE ADAPTER AND 150# PVC BULKHEAD FITTING	PE/PVC	236.00"
WASH	6	1	1 1/2" PVC FLANGE ADAPTER AND 150# PVC BULKHEAD FITTING	PE/PVC	226"
SERVICE	TAG	QTY.	DESCRIPTION	MATERIAL	ELEVATION (1)

(1) ELEVATIONS ARE RELATIVE TO THE CONCRETE PAD

T-102: MOLASSES STORAGE TANK

LEVEL CONTROL	1	1	2" 150# FLANGE ADAPTER AND 150# PVC SIDEWALL FITTING	PE/PVC	207.15"
VENT	2	1	2" PVC FLANGE ADAPTER AND PVC/EPDM BULKHEAD FITTING	PE/PVC	207.15"
DISCHARGE	3	1	2" PVC FLANGE ADAPTER AND 150# PVC BULKHEAD FITTING	PE/PVC	20.73"
ACCESS	4	1	18" DIA. TOP MANHOLE (15" ACCESS)	PE	211.15"
INLET	5	1	2" PVC FLANGE ADAPTER AND 150# PVC BULKHEAD FITTING	PE/PVC	211.15"
WASH	6	1	1 1/2" PVC FLANGE ADAPTER AND 150# PVC BULKHEAD FITTING	PE/PVC	207.15"
SERVICE	TAG	QTY.	DESCRIPTION	MATERIAL	ELEVATION (1)

(1) ELEVATIONS ARE RELATIVE TO THE CONCRETE PAD

T-103: DILUTION WATER STORAGE TANK

LEVEL CONTROL	1	1	2" 150# FLANGE ADAPTER AND 150# PVC SIDEWALL FITTING	PE/PVC	48.00"
VENT	2	1	2" PVC FLANGE ADAPTER AND PVC/EPDM BULKHEAD FITTING	PE/PVC	48.00"
DISCHARGE	3	1	2 PVC FLANGE ADAPTER AND 150# PVC BULKHEAD FITTING	PE/PVC	5.25"
ACCESS	4	1	18" DIA. TOP MANHOLE (15" ACCESS)	PE	48.00"
INLET	5	1	2" PVC FLANGE ADAPTER AND 150# PVC BULKHEAD FITTING	PE/PVC	48.00"
SERVICE	TAG	QTY.	DESCRIPTION	MATERIAL	ELEVATION (1)

(1) ELEVATIONS ARE RELATIVE TO THE CONCRETE PAD

SCALE(S) AS INDICATED

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No.	Date	Revisions	By	Ckd

Professional Engineer's Name: ANDREW DAVIS
Professional Engineer's No.: NC#027773

State	Date Signed	Project Mgr.
NC		AD

Designed by	Drawn by	Checked by
SS	SS	MS

NORTH CAROLINA PROFESSIONAL SEAL 027773 ENGINEER ANDREW A. DAVIS 12/9/11

PIKA INTERNATIONAL, INC. A JOINT VENTURE MALCOLM PIRNIE
MALCOLM PIRNIE, INC.
ENGINEERING LICENSE # F-0299

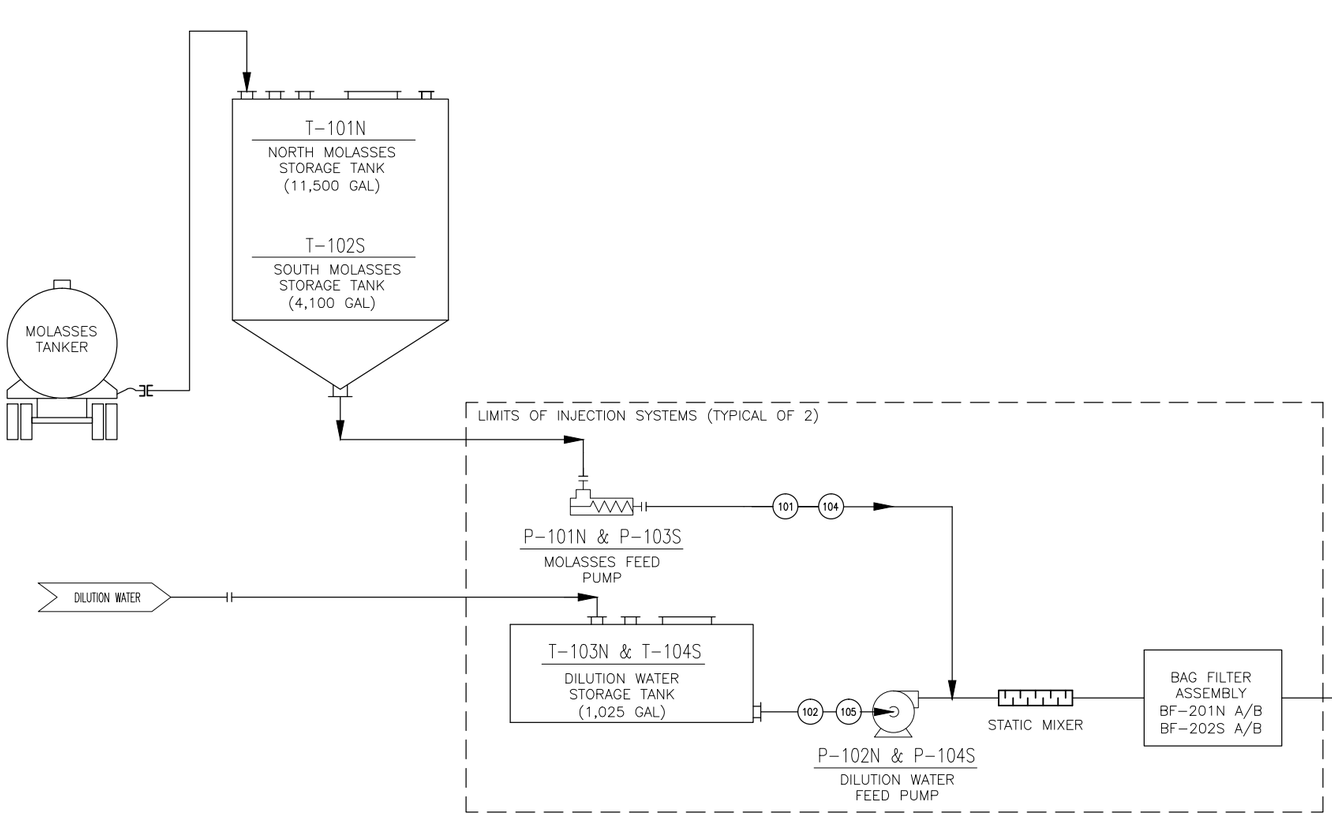
UNITED STATES ARMY CORPS OF ENGINEERS ● CHARLOTTE, NORTH CAROLINA
CONCEPT DESIGN PHASE

STORAGE TANK NOZZLE SCHEDULE

PROCESS

Project No. GSCOE001.0001.00001
Date DECEMBER 2011
30 PATEWOOD DR. SUITE 155 GREENVILLE, SC 29615 TEL: 864-987-3900

A-5 M2



	NORTH INJECTION AREA			SOUTH INJECTION AREA			INJECTION MANIFOLD		
STREAM ID	101	102	103	104	105	106	X01	X02	X03
DESIGN FLOW (GPM)	0.7	65.3	66.0	0.1	11.9	12.0	1.5	1.5	6
VOLUME (GALLONS)	--	--	--	--	--	--	12,000	12,000	58,000
COMMENTS	NOTE 2,3	NOTE 2,3	NOTE 2,3	NOTE 2,3	NOTE 2,3	NOTE 2,3	NOTE 1,5	NOTE 1,5	NOTE 1,4

NOTES:

1. FLOW ESTIMATED BASED ON RESULTS OF PILOT TESTING
2. FLOW RATES VARY. DESIGN FLOW RATES SHOWN FOR SIMPLICITY.
3. FLOW RATES ARE BASED ON (1) INJECTION WELL PER BRANCH
4. BIW MANIFOLD WILL BE IN A SEPARATE MANIFOLD FROM IW. BIW REQUIRES APPROX. 6 GPM.
5. IW REQUIRES APPROX. 1.5 GPM.

GPM – GALLONS PER MINUTE
GAL – GALLONS

INJECTION WELL MANIFOLD				INJECTION WELL MANIFOLD				INJECTION WELL MANIFOLD			
WELL NAME	UNIQUE ID NO. (XXX)	UNIQUE ID NO. (XXX)	DROP PIPE SIZE	WELL NAME	UNIQUE ID NO. (XXX)	UNIQUE ID NO. (XXX)	DROP PIPE SIZE	WELL NAME	UNIQUE ID NO. (XXX)	UNIQUE ID NO. (XXX)	DROP PIPE SIZE
	NORTH AREA				NORTH AREA				SOUTH AREA		
IW	IW1	IW17	1"	BIW	BIW1	BIW14	1"	IW	IW31	IW44	1"
	IW2	IW18	1"		BIW2	BIW15	1"		IW32	IW45	1"
	IW3	IW19	1"		BIW3	BIW16	1"		IW33	IW46	1"
	IW4	IW20	1"		BIW4	BIW17	1"		IW34	IW47	1"
	IW5	IW21	1"		BIW5	BIW18	1"		IW35	IW48	1"
	IW6	IW22	1"		BIW6	BIW19	1"		IW36	IW49	1"
	IW7	IW23	1"		BIW7	BIW20	1"		IW37	IW50	1"
	IW8	IW24	1"		BIW8	BIW21	1"		IW38	IW51	1"
	IW9	IW25	1"		BIW9	BIW22	1"		IW39	IW52	1"
	IW10	IW26	1"		BIW10	BIW23	1"		IW40	IW53	1"
	IW11	IW27	1"		BIW11	BIW24	1"		IW41	--	1"
	IW12	IW28	1"		BIW12	BIW25	1"		IW42	--	1"
	IW13	IW29	1"		BIW13	--	1"		IW43	--	1"
	IW14	IW30	1"								
	IW15	--	1"								
	IW16	--	1"								

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UNITED STATES ARMY CORPS OF ENGINEERS • CHARLOTTE, NORTH CAROLINA
CONCEPT DESIGN PHASE

PIPING AND INSTRUMENTATION DIAGRAM

PROCESS

Project No. GSCOE001.0001.00001
Date DECEMBER 2011

30 PATEWOOD DR.
SUITE 155
GREENVILLE, SC 29615
TEL: 864-987-3900

A-6 PFD

LIMITS OF INJECTION SYSTEMS (TYPICAL OF 2)

T-101N
NORTH MOLASSES STORAGE TANK (11,500 GAL)

VENT 2" 2" 2" 18" 2"

LT 101 — LALL 101 | LAL 101 | LAH 101 — LI 101

MOLASSES TANKER — 2" V-101

V-102 2"

2"-PVCP-101

HOA — HS 101 — VFD 101 — FIC 101 — YI 101

V-103 2" — 2"X1" — P-101N MOLASSES FEED PUMP

PI 103 — 1/4" — 1"X1/4"

FT 102 | FQ 102 | FIR 102 — FIT 102 — FE 102 — M

V-104 1" — V-105 1" — V-106 1"

V-120

1"-PVCP-102

1"-PVCP-103

PI 101 — 1/4" — 2"X1/4"

HOA — HS 103 — FV 103 — E

DILUTION WATER — V-107 2" — V-108 2" — DRAIN PORT 3/4" V-109 — V-110 2" — 2"-PVCP-104 2"

LAHH 103 | LSH 103 | LSL 103 | LALL 103

LT 103 — LIC 103

T-103N
DILUTION WATER STORAGE TANK (1,025 GAL)

2" 2" 2" 18" — V-111 2"

YI 102 — HOA — HS 102 — VFD 102 — FIC 102

P-102N
DILUTION WATER FEED PUMP

PI 102 — 1/4" — 2"X1/4"

FT 101 | FQ 101 | FIR 101 — FIT 101 — FE 101 — M

V-112 2" — V-113 2" — 2"X1" — 1"X2"

1"X 2"

STATIC MIXER

PAH 201A — 2 — PI 201A — PIT 201A — 1/4" — 2"X1/4" — V-114 2"

DPAH 201 — AND

PAH 201B — 2 — PI 201B — PIT 201B — 1/4" — 2"X1/4" — V-115 2" — V-116 2"

1/4" VENT — PI 201C — DRAIN 1"

BF-201N A/B
BAG FILTER

1/4" VENT — PI 202D — DRAIN 1"

BF-201B

2"-PVCP-105 — 2"-PETP-X01 — TO PID2

PVC TO HDPE TRANSITION

LIMITS OF INJECTION SYSTEMS (TYPICAL OF 2)

SCALE(S) AS INDICATED

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No.	Date	Revisions	By	Ckd

Professional Engineer's Name: **ANDREW DAVIS**
Professional Engineer's No.: NC#027773
State: NC — Date Signed: 12/9/11 — Project Mgr.: AD
Designed by: SS — Drawn by: SS — Checked by: MS

NORTH CAROLINA PROFESSIONAL SEAL 027773 ENGINEER ANDREW A. DAVIS

PIKA INTERNATIONAL, INC. — A JOINT VENTURE — MALCOLM PIRNIE
MALCOLM PIRNIE, INC.
ENGINEERING LICENSE # F-0299

UNITED STATES ARMY CORPS OF ENGINEERS • CHARLOTTE, NORTH CAROLINA
CONCEPT DESIGN PHASE

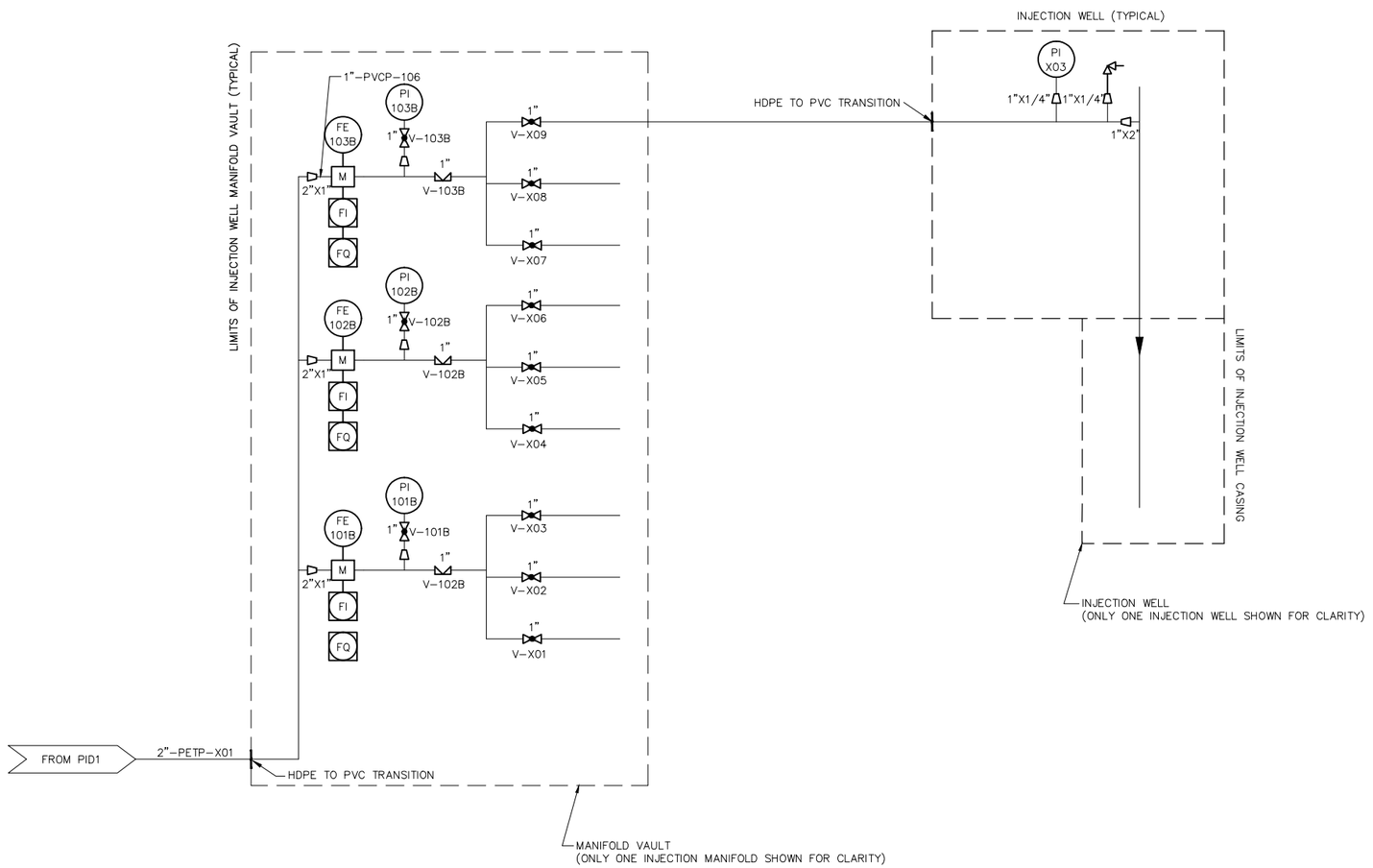
PIPING AND INSTRUMENTATION DIAGRAM

PROCESS

Project No. GSCOE001.0001.00001
Date DECEMBER 2011

30 PATEWOOD DR.
SUITE 155
GREENVILLE, SC 29615
TEL: 864-987-3900

A-7
PID1



1. CONTRACTOR SHALL USE A THREADED REDUCING TEE OR SOCKET TEE WITH REDUCER BUSHING TO REDUCE TO 1/4" THREADED CONNECTION.

INJECTION WELL MANIFOLD			
WELL NAME	UNIQUE ID NO. (XXX)	UNIQUE ID NO. (XXX)	INJECTION PIPE SIZE
IW	IW1	IW28	1"
	IW2	IW29	1"
	IW3	IW30	1"
	IW4	IW31	1"
	IW5	IW32	1"
	IW6	IW33	1"
	IW7	IW34	1"
	IW8	IW35	1"
	IW9	IW36	1"
	IW10	IW37	1"
	IW11	IW38	1"
	IW12	IW39	1"
	IW13	IW40	1"
	IW14	IW41	1"
	IW15	IW42	1"
	IW16	IW43	1"
	IW17	IW44	1"
	IW18	IW45	1"
	IW19	IW46	1"
	IW20	IW47	1"
	IW21	IW48	1"
	IW22	IW49	1"
	IW23	IW50	1"
	IW24	IW51	1"
	IW25	IW52	1"
	IW26	IW53	1"
	IW27		1"
	IW28		1"
BIW	BIW1	BIW14	1"
	BIW2	BIW15	1"
	BIW3	BIW16	1"
	BIW4	BIW17	1"
	BIW5	BIW18	1"
	BIW6	BIW19	1"
	BIW7	BIW20	1"
	BIW8	BIW21	1"
	BIW9	BIW22	1"
	BIW10	BIW23	1"
	BIW11	BIW24	1"
	BIW12	BIW25	1"
	BIW13		1"

SCALE(S) AS INDICATED

THIS BAR REPRESENTS ONE INCH ON THE ORIGINAL DRAWING: USE TO VERIFY FIGURE REPRODUCTION SCALE

No.	Date	Revisions	By	Ckd

Professional Engineer's Name: **ANDREW DAVIS**
Professional Engineer's No.: NC#027773

State	Date Signed	Project Mgr.
NC		AD
Designed by	Drawn by	Checked by
SS	SS	MS

NORTH CAROLINA PROFESSIONAL SEAL 027773 ENGINEER ANDREW A. DAVIS 12/9/11

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A-8
PID2

LEGEND:

- PROCESS PIPING
- PERIMETER
- BALL VALVE
- BUTTERFLY VALVE
- DIAPHRAGM VALVE
- SWING CHECK VALVE
- GLOBE VALVE
- PRESSURE CONTROL VALVE
- GATE VALVE
- 3 WAY VALVE
- ELECTRIC ACTIVATED VALVE
- FLOW METER (MECH.)
- CAM LOCK
- FLANGED CONNECTION
- REDUCER
- INSTRUMENT SIGNAL
- AIR/VACUUM RELEASE
- PRESSURE RELIEF VALVE
- STATIC MIXER
- CENTRIFUGAL PUMP
- PROGRESSIVE CAVITY PUMP

ABBREVIATIONS:

DPI	DIFFERENTIAL PRESSURE INDICATOR
E	ELECTRICAL ACTUATOR
F	FILTER
FAL	FLOW ALARM LOW
FE	FLOW ELEMENT
FI	FLOW INDICATOR
FIT	FLOW INDICATING TRANSMITTER
FQ	FLOW TOTALIZER
FIR	FLOW INDICATING RECORDER
FS	FLOW SWITCH
FT	FLOW TRANSMITTER
FV	FLOW VALVE
HOA	HAND/OFF/AUTO
HS	HAND SWITCH
LAL	LEVEL ALARM LOW
LALL	LEVEL ALARM LOW LOW
LAH	LEVEL ALARM HIGH
LAHH	LEVEL ALARM HIGH HIGH
LE	LEVEL ELEMENT
LSH	LEVEL SWITCH HIGH
LSL	LEVEL SWITCH LOW
LT	LEVEL TRANSMITTER
PAH	PRESSURE ALARM HIGH
PE	PRESSURE ELEMENT
PI	PRESSURE INDICATOR
PIR	PRESSURE INDICATING RECORDER
PIT	PRESSURE INDICATING TRANSMITTER
TK	TANK
V	VALVE
VFD	VARIABLE FREQUENCY DRIVE
YI	STATUS INDICATOR
P	PUMP

PROGRAMMABLE LOGIC:

1. FULL SYSTEM SHUTDOWN. NOTIFY SYSTEM OPERATOR.
2. SIGNAL ALARM TO SWITCH FROM PRIMARY BAG FILTER TO SECONDARY BAG FILTER. INITIATE CHANGE–OUT OF PRIMARY BAG FILTER.

INSTRUMENT SYMBOLS

	PRIMARY CONTROL PANEL NORMALLY ACCESSIBLE TO OPERATOR	FIELD MOUNTED	AUXILIARY PANEL OR RACK NORMALLY ACCESSIBLE TO OPERATOR
DISCRETE INSTRUMENTS			
SHARED DISPLAY, SHARED CONTROL			
COMPUTER FUNCTION INCLUDING DISTRIB. CNTL. SYS.			
PROGRAMMABLE LOGIC CONTROLLER FUNCTION			

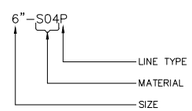
MATERIAL

BRZ – BRASS/BRONZE	PET – HIGH DENSITY POLYETHYLENE
CIR – CAST IRON	POP – POLYPROPYLENE
CST – CARBON STEEL	PVC – POLYVINYL CHLORIDE
CPR – COPPER	RUB – RUBBER
CVC – CHLORINATED POLYVINYL CHLORIDE	S04 – 304 STAINLESS STEEL
DIR – DUCTILE IRON	S4L – 304L STAINLESS STEEL
FRP – FIBERGLASS	S16 – 316 STAINLESS STEEL
GCS – GALVANIZED CARBON STEEL	S6L – 316L STAINLESS STEEL
LCS – LINED CARBON STEEL	TEF – TEFLON
	VIT – VITON

TYPE

D = DUCT P = PIPE C = DOUBLE WALL CONTAINMENT PIPE
H = HOSE T = TUBE

PIPE LINE DESIGNATION



INSTRUMENT IDENTIFICATION LETTERS

FIRST LETTER		SUCCEEDING LETTERS		
MEASURE OR INITIATING VARIABLE	MODIFIER	READOUT OR PASSIVE FUNCTION	OUTPUT FUNCTION	MODIFIER
A = ANALYSIS		ALARM		
B = BURNER, COMBUSTION		USER'S CHOICE	USER'S CHOICE	USER'S CHOICE
C = USER'S CHOICE			CONTROL, CLOSED	
D = USER'S CHOICE	DIFFERENTIAL			
E = VOLTAGE		SENSOR (PRIMARY ELEMENT)		
F = FLOW RATE	RATIO (FRACTION)			
G = USER'S CHOICE		GLASS, VIEWING DEVICE		
H = HAND				HIGH
I = CURRENT (ELECTRICAL)		INDICATE		
J = POWER	SCAN			
K = TIME, TIME SCHEDULE	TIME RATE OF CHANGE		CONTROL STATION	
L = LEVEL		LIGHT		LOW
M = USER'S CHOICE	MOMENTARY			MIDDLE, INTERMEDIATE
N = USER'S CHOICE		USER'S CHOICE	USER'S CHOICE	USER'S CHOICE
O = USER'S CHOICE		ORIFICE, RESTRICTION	OPEN	
P = PRESSURE, VACUUM		POINT (TEST) CONNECTION		
Q = QUANTITY	INTEGRATE, TOTALIZE			
R = RADIATION		RECORD	RUN	
S = SPEED, FREQUENCY	SAFETY	SWITCH	STOP	
T = TEMPERATURE			TRANSMIT	
U = MULTIVARIABLE		MULTIFUNCTION	MULTIFUNCTION	MULTIFUNCTION
V = VIBRATION, MECH. ANALYSIS			VALVE, DAMPER, LOUVER	
W = WEIGHT, FORCE		WELL		
X = UNCLASSIFIED	X AXIS	UNCLASSIFIED	UNCLASSIFIED	UNCLASSIFIED
Y = EVENT, STATUS OR PRESENCE	Y AXIS		RELAY, COMPUTE, CONVERT	
Z = POSITION, DIMENSION	Z AXIS		DRIVE, ACTUATOR, UNCLASSIFIED FINAL CONTROL ELEMENT	

NOTES:

1. ANY FIRST LETTER COMBINED WITH MODIFIER REPRESENTS A NEW AND SEPARATE MEASURED VARIABLE. EXAMPLES: PD = DIFFERENTIAL PRESSURE FQ = TOTALIZED OR INTEGRATED FLOW. EXCEPTION IS THE MODIFIER "J" FOR MULTIPOINT SCANNING.
2. FOR ANALYSIS NOT IDENTIFIED BY A SPECIFIC LETTER IN THE TABLE, USE FIRST LETTER "A" NEAR THE INSTRUMENT SYMBOL, SPECIFY THE NATURE OF THE ANALYSIS. EXAMPLE: PH
3. MEANING OF A "USER CHOICE" LETTER SHALL BE CONSISTENT THROUGHOUT A PROJECT, AND SHALL BE SPECIFIED IN THE DRAWING LEGEND.
4. UNCLASSIFED LETTER MAY HAVE A FEW DIFFERENT MEANINGS ON A PROJECT, THE MEANING SHALL BE SPECIFIED NEAR EACH INSTRUMENT SYMBOL USING THE UNCLASSIFIED LETTER.
5. THE MODIFIER "SCAN" APPLIES TO MULTIPOINT PRINTING INSTRUMENTS, SUCH AS CJRS (MULTIPOINT CONDUCTIVITY RECORDER WITH ALARM SWITCHES).

GENERAL NOTES:

1. ALL ANALOG SETPOINTS SHALL BE FIELD ADJUSTED BY OPERATOR AT HMI INTERFACE SCREEN.
2. ALARMS THAT SHUT DOWN EXTRACTION WELLS AND TREATMENT EQUIPMENT MUST BE CLEARED BY OPERATOR BEFORE BEING RESTARTED.
3. THIS DRAWING IS PROVIDED FOR INFORMATIONAL PURPOSES ONLY.

SCALE(S) AS INDICATED

THIS BAR REPRESENTS ONE INCH ON THE ORIGINAL DRAWING: USE TO VERIFY FIGURE REPRODUCTION SCALE

No.	Date	Revisions	By	Ckd

Professional Engineer's Name: ANDREW DAVIS		
Professional Engineer's No.: NC#027773		
State: NC	Date Signed:	Project Mgr.: AD
Designed by: SS	Drawn by: SS	Checked by: MS

NORTH CAROLINA PROFESSIONAL SEAL 027773 ENGINEER ANDREW A. DAVIS 12/9/11

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A-9
PID3

Appendix B

B-1 = Accident Prevention Plan (APP)

B-2 = Site Safety and Health Plan (SSHP)

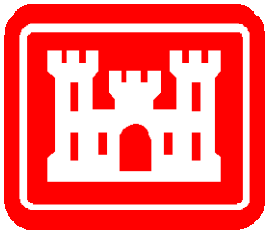
APPENDIX B-1
Accident Prevention Plan

Former Charlotte Naval Ammunition Depot
Charlotte, North Carolina

December 2011

DERP-FUDS Project No. I04NC080301
Contract No.:W912DY-10-D0025
Delivery Order No.: 0007

PREPARED FOR:



U.S. Army Corps of Engineers, Huntsville Center
U.S. Army Engineering and Support Center
4820 University Square
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Kevin Held
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PIKA-PIRNIE JV Certified Project Manager
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Accident Prevention Plan

Former Charlotte Naval
Ammunition Depot
Charlotte, North Carolina

Prepared for:
U.S. Army Corps of Engineers,
Huntsville Center

Prepared by:
PIKA-PIRNIE JV, LLC
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Our Reference:

DERP-FUDS Project No. I04NC080301
Contract No.: W912DY-10-D0025
Delivery Order No.: 0007

Date:
December 2011

The views, opinions, and/or findings contained in this report are those of the author(s) and should not be construed as an official Department of the Army position, policy, or decision, unless so designated by other documentation.

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Acronyms and Abbreviations

APP	Accident Prevention Plan
CNAD	Charlotte Naval Ammunition Depot
CO	Contracting Officer
COR	Contracting Officer's Representative
CPR	Cardio-Pulmonary Resuscitation
CVOC	Chlorinated Volatile Organic Compound
ERD	Enhanced Reductive Dechlorination
FRA	Federal Railroad Administration
H&S	Health and Safety
HAZWOPER	OSHA Hazardous Waste Operations and Emergency Response Standard
JSA	Job Safety Analysis
JV	Joint Venture
LPS	Loss Prevention System
MSDS	Material Safety Data Sheet
OSHA	Occupational Safety and Health Administration
PIKA	PIKA International, Inc. (PIKA)
PIKA-PIRNIE JV Team	PIKA International, Inc. (PIKA)/Malcolm Pirnie, Inc (Pirnie) JV (Joint Venture) LLC
Pirnie	Malcolm Pirnie, Inc
PM	Project Manager
PPE	Personal Protective Equipment
PWS	Performance Work Statement
QC	Quality Control
SHM	Safety and Health Manager
SSHO	Site Safety and Health Officer
SSHP	Site Safety and Health Plan
TCE	Trichloroethene
U.S.	United States
USACE	United States Army Corps of Engineers, Huntsville Center

1. Project Description

At the time of operation, the entire Charlotte Naval Ammunition Depot (CNAD) complex occupied approximately 2,266 acres. In June 1942 the United States (U.S.) Rubber Company began operating an ammunition shell loading and assembly plant on site that was taken over by the U.S. Navy in 1945. In 1956, the Naval Depot status was changed to inactive. In 1959, the former CNAD complex was sold to a development partnership and is currently occupied by light industrial and commercial businesses.

Two areas (1 and 2) were used for the production of munitions. Each area had multiple buildings with the largest buildings used for assembly, packaging and shipping of munitions. After 1945, operations in area 2 included reconditioning of munitions. This included degreasing operations in the southeast corner of Building 2-30 which utilized trichloroethene (TCE). TCE was found in groundwater plumes originating from this location, the mitigation of which is the objective of this work effort.

The PIKA International, Inc. (PIKA)/Malcolm Pirnie, Inc (Pirnie) JV (Joint Venture) LLC (the PIKA-PIRNIE JV Team) has been tasked under the Performance Work Statement (PWS) to implement the selected remedy, as presented in the Decision Document (USACE, 2011a) approved on April 18, 2011, for the cleanup of contaminated groundwater at the former CNAD complex. Specifically, in-situ enhanced reductive dechlorination (ERD) will be used to stimulate biological degradation of chlorinated volatile organic compounds (CVOCs) in groundwater within the impacted water bearing units that underlie the former CNAD complex.

The Site Safety and Health Plan (SSHP) (Appendix B-2) includes Activity Hazard Analyses for tasks associated with completion of this work. The work tasks associated with this project include:

- Mobilization and demobilization of equipment;
- Soil drilling and well installation;
- Installation of storage tanks and process equipment; and
- Operation of the injection equipment.

2. Global Health and Safety (H&S) Policy

The PIKA-PIRNIE JV Team commits to:

- Protecting our employees and clients from injury and illness;
- Placing H&S values on par with our other core business values;
- Supporting a H&S culture where employees share in this commitment to integrate H&S into their behaviors; and
- Integrating H&S into the solutions we offer to clients.

3. Responsibilities and Lines of Authorities

The PIKA-PIRNIE JV Team will develop, implement and maintain a current and effective H&S program (aligned with corporate objectives and policies and compliant with government regulations). Key roles in this effort include: Project Manager (PM), Safety and Health Manager (SHM), Construction Manager and Site Safety and Health Officer (SSHO). Resumes for the personnel assigned these roles are found in **Appendix A**.

3.1 All Personnel

Each person is responsible for completing tasks safely and reporting any unsafe acts or conditions to their supervisor. No person may work in a manner that conflict with these procedures. Prior to initiating site activities, all the PIKA-PIRNIE JV Team and subcontractor personnel will receive training in accordance with applicable regulations, and be familiar with the requirements and standards referenced in this Accident Prevention Plan (APP)/SSHP. In addition, all personnel will attend daily safety meetings (tailgate meetings) to discuss site-specific hazards prior to beginning each day's work. Every PIKA-PIRNIE JV Team employee, subcontractor, and client representative at the Site has the responsibility to stop the work of a coworker or subcontractor if the working conditions or behaviors are considered unsafe.

Additional qualifications and responsibilities with regard to implementation of this APP/SSHP exist for the PM, SSHO, SHM, and Construction Manager. Personnel assigned these roles are identified in Section 5 of the SSHP. Section 5 of the SSHP also includes further detail on the responsibilities of these positions with regard to APP/SSHP implementation.

3.2 Construction Task Manager

The Construction Task Manager reports to the PM. Additional responsibilities of the Construction Task Manager are as follows:

- Review all applicable H&S procedures, and ensure that project activities conform to all requirements.
- Obtain client-specific H&S information and communicate with the client on H&S issues.

- Communicate with the SSHO on H&S issues.
- Allocate resources for correction of identified unsafe work conditions.
- Ensure the PIKA-PIRNIE JV Team site workers and all subcontractors have all training necessary for the project.

The Construction Manager must report all injuries, illnesses and near-misses to the SHM, SSHO, lead incident investigations, and ensure that safety programs and recommendations are implemented.

3.3 SHM

The SHM oversees all aspects of the site safety program, and prepares and or reviews any site-specific H&S guidance documents or addenda to this plan. The SHM does not report to the construction manager, and is separately accountable to the PIKA-PIRNIE JV Team senior management for construction H&S. The SHM acts as the sole contact to regulatory agencies on matters of H&S. Other responsibilities include:

- Overall authority for H&S compliance and SSHP conformance for the project;
- General H&S program administration;
- Conducts on-site project H&S audits and participates in teleconferences as warranted;
- Remains available for project emergencies;
- Develops and approves modifications to the SSHP as needed;
- Evaluates exposure monitoring/air sampling data and adjusts the SSHP requirements as necessary;
- Serves as a Quality Control (QC) staff member;
- SSHP approval by signature;
- Determines the level of personal protective equipment (PPE) required;

- Updates equipment or procedures based on information obtained during site operations;
- Assists in injury, illness and near-miss investigation and follow-up;
- Assisting the SSHO in issues as they arise;
- Perform site audits and assessments;
- Assisting/conducting near-miss/incident investigation; and
- Serves as the liaison with corporate during H&S regulatory issues as they may arise.

3.4 SSHO

The SSHO reports to the SHM and has the following responsibilities and qualifications:

- The SSHO, or his designated representative, must be present anytime remedial activities are being performed to implement the SSHP.
- Ensures that this SSHP is available to and reviewed by all site personnel including subcontractors.
- Ensures that necessary site-specific training is performed (both initial and “tailgate” safety briefings).
- Has a minimum of one year experience implementing safety and occupational health procedures associated with remedial activities.
- Conducts exposure monitoring/air sampling and selects/adjusts protective equipment use.
- Inspects site activities to identify safety and occupational health deficiencies and correct them.
- Coordinates changes/modifications to the SSHP with the SHM, site superintendent, and Contracting Officer’s Representative (COR).

- Conducts project-specific training.
- Ensures site visitors have been informed of the hazards related to the PIKA-PIRNIE JV Team work and have signed the Site Visitors Log.
- Ensures that work is performed in a safe manner and has authority to stop work when necessary to protect workers and/or the public.
- Coordinates activities during emergency situations.
- Ensures that all necessary permits and safety information provided by the client is disseminated to other site personnel and is maintained in an organized manner.
- Reports all injuries, illnesses and near-misses to the PM, COR, and SHM.
- Ensures that necessary safety equipment is maintained and used at the site.
- Monitor the implementation of the SSHP.

4. Subcontractors and Suppliers

Subcontractors and suppliers anticipated to complete operations on site are listed in the Project Organization Chart (Figure 2-1) in the Project Management Plan (PMP) – Appendix F.

A copy of this APP/SSHP is to be provided to all PIKA-PIRNIE JV Team subcontractors or suppliers prior to the start of work so that the subcontractor is informed of the hazards at the site. While the PIKA-PIRNIE JV Team APP/SSHP will be the minimum H&S requirements for the work completed by the PIKA-PIRNIE JV Team and its subcontractors, each subcontractor, in coordination with the PIKA-PIRNIE JV Team H&S personnel, is expected to perform its operations in accordance with its own SSHP, policies and procedures unique to the subcontractor's work to ensure that hazards associated with the performance of the work activities are properly controlled. Copies of any required safety or training documentation for a subcontractor's work activities will be provided to the PIKA-PIRNIE JV Team for review prior to the start of on-site activities.

5. Training

5.1 Site-Specific H&S Training Orientation

Site-specific training will be accomplished by on-site personnel reading this APP/SSHP and associated job safety analyses (JSAs). The review must include a discussion of the planned work activities, chemical, physical and biological hazards; recommended controls, PPE; safe work procedures; and emergency procedures.

No person will be allowed in the work area during site operations without first receiving a site orientation. Following this initial site H&S orientation, safety meetings will be held at least daily as a component of the tailgate meeting. People entering the site work areas, including visitors, must document their attendance at this briefing, as well as any required safety meetings on the forms included with this APP/SSHP. No person will be allowed in the work area unless they are wearing the minimum PPE. A safety meeting must also be held prior to beginning new tasks, and repeated if new hazards are encountered.

5.2 Site-Required Railroad Safety Training and Security Clearance

Based on discussion with Norfolk Southern personnel, Federal Railroad Administration (FRA) training and e-RailSafe verification and training are not required to perform work on site. Should these certifications become necessary at a later date, training will be completed accordingly.

5.3 Site-Specific Training Requirements

All personnel working at the site must have the necessary training based on the hazards present. The following training is required for all site workers:

Training Required:	☒ 40-hour HAZWOPER	☐ Confined space
	☐ 24-hour HAZWOPER	☐ Lockout/tagout
	☐ HAZWOPER site supervisor	☐ Electricity
	☐ OSHA 30-hour Construction	☒ Fire extinguishers
	☐ OSHA 10-hour Construction	☐ Fall protection
	☒ PPE	☒ Noise exposure
	☐ Respiratory protection	☐ Forklifts
	☐ Chemical hygiene	☐ Asbestos
	☐ Hazard communication	☐ Lead
	☐ Hazardous waste	☐ Cadmium
	☒ First-aid/CPR/Bloodborne pathogens	☐ Radiation safety
	☐ DOT/IATA hazmat transportation	☐ Client specific
	☐ Diving	☐ Other: FRA Training
☐ None	☐ Boating safety	☐ Other: ERailSafe

All 40-hour OSHA Hazardous Waste Operations and Emergency Response Standard (HAZWOPER) trained personnel who are working at HAZWOPER project sites are required to participate in the PIKA-PIRNIE JV Team medical surveillance program as outlined in Section 7 of the SSHP. Any personnel who work on site, in a supervisory capacity, are required to have HAZWOPER Supervisor Training; in addition to the 40-hour HAZWOPER training and current annual refresher training.

The designated SSHO is required to have Occupational Safety and Health Administration (OSHA) 30-hour training on the Construction Standards and current certification in Cardio-Pulmonary Resuscitation (CPR) and First Aid issued by the Red Cross or American Heart Association.

Construction equipment operators including are required to have training on the particular type of equipment they are operating. Certain equipment items, such as drill rigs and cranes, have specific training and licensing requirements.

Personnel working on installation or repair of the electric service distribution system are required to have required OSHA training and competency.

The PIKA-PIRNIE JV Team does not believe there will be any Permit-Required Confined Space Entry associated with this project. The SSHO will have, at a minimum, Confined Space Awareness Training sufficient to determine if there are any permit required confined space locations on site. In the event that work tasks require permit required confined space entry, the training requirements would be applicable to the entry team.

5.4 Procedures for Periodic H&S Training for Supervisors and Employees

Daily safety meetings will be held to cover the work to be accomplished, hazards anticipated, PPE and procedures required to minimize site hazards, and emergency procedures. The SSHO must participate in these meetings prior to beginning the day's fieldwork. The safety meeting must also be held prior to conducting new tasks, and repeated if new hazards are encountered.

No one will start work before the daily safety meeting has been completed. If new tasks or new hazards arise, the SSHO will hold another tailgate safety meeting that day. The process for conducting these meetings follows the PIKA-PIRNIE JV Team TRACK process described in the SSHP.

5.5 Requirements for Emergency Response Training

Various site features may be encountered in an emergency response situation, such as active rail yards, industrial areas, commercial areas, vacant buildings, rural areas, streams, etc. Workers need to assess the various site features encountered and determine what hazards are associated with those features. Workers also need to determine what measures need to be taken to mitigate the hazards. Site features and hazards will be evaluated and response efforts will be planned accordingly.

Emergency telephone numbers will be posted at the site office. A site-specific hazard communication plan will be prepared that includes a hazardous substance inventory and material safety data sheet (MSDS) file. The SSHO will be responsible to ensure the MSDS file and hazard communication program is kept current and that the site has any specialized equipment (*e.g.,* spill response materials, fire extinguishers, PPE) to adhere to the manufacturer's recommendations.

6. H&S Inspections

6.1 Routine H&S Inspections

The SSHO will conduct a daily inspection of site activities, equipment and procedures to verify that the required elements are in place including compliance with OSHA Standards. The Daily Tailgate Form in Appendix C of the SSHP may be used as a guide for daily inspections. Deficiencies and recommended corrective measures will be noted by the SSHO in the Daily Report. Deficiencies will be categorized in the report and open or closed. Any open deficiencies will be discussed in subsequent daily reports until the deficiency is resolved and classified as closed. Deficiencies that remain open for more than one day will be brought to the attention of the PM and SHM.

Prior to the start and throughout the course of site operations the SSHO and Site Manager will evaluate the work area and project site for potential fire, contaminant release or other potential emergency events, including whether and/or other natural disasters.

6.2 H&S Audits

Project H&S Audits will be conducted and follow-up responses will be documented and evaluated in accordance with the PIKA-PIRNIE H&S Audit Program Manual (Revised July 2005). The Project SHM or designee (who must also be a PIKA-PIRNIE Corporate Approved Auditor) will complete at least one project audit during the first three months of operation and at least once H&S audit per year. The auditor will utilize the Project Audit Checklist during the audit and complete the Project Audit Findings Forms. As per the PIKA-PIRNIE H&S Audit Program Manual, the Project Audit Findings Form must be submitted to the PM, Regional SHM and Corporate SHM.

The PM has 10 days following receipt of the Project Audit Findings Form to prepare and submit a Project Corrective Actions Form. The Corrective Actions Form must be submitted to the SHM and Regional SHM, and it is intended to identify corrective and preventative actions and the schedule for completion. The SHM is required to follow-up the completion of the preventative and corrective actions and document the completions on the Project Corrective Actions Form in order to close out the corrective actions. Adherence to the Corporate H&S Audit Program is evaluated and documented by the Regional and Corporate SHMs annually.

7. H&S Reporting

7.1 Incident Reporting

The PIKA-PIRNIE JV Team will report and conduct an investigation following any work-related incident. Reporting notification times vary depending on the incident, but all incidents should be reported as soon as possible and no later than the time periods described here. All incidents will be reported to the Contracting Officer/Representative (CO/COR) as soon as possible, but not more than 24 hours afterwards. There are certain severe accidents, described later in this section, that require the Site Manager to immediately notify the COR, after notifying emergency responders.

The PIKA-PIRNIE JV Team will thoroughly investigate the accident and submit the findings of the investigation along with the appropriate corrective actions to the CO/COR on United States Army Corps of Engineers, Huntsville Center (USACE) Form ENG 3394, Accident Investigation Report as soon as possible but no later than five (5) working days following the accident. Corrective actions will be implemented as soon as reasonably possible.

The PM and/or SHM will follow the PIKA-PIRNIE Incident Reporting Standard ARC HSMS010 and will complete the PIKA-PIRNIE Incident Investigation Form utilizing the electronic PIKA-PIRNIE Loss Prevention System (LPS). This action will enter the incident into the LPS database. The entry initiates the senior management review process intended to ensure the investigation team agrees on the conclusions and contributing factors. The senior management review team must agree on appropriate response actions. The LPS system provides automated messages to personnel with specific incident follow-up response action items and automated messages to responsible parties when corrective measures are documented and accepted.

A work-related incident includes any of the following types of events that could occur during the conduct of the PIKA-PIRNIE JV Team work activities:

- Injuries and illnesses, including those needing only first aid
- Near misses/losses
- Spills or leaks
- Equipment and property damage

- Motor vehicle accidents
- Regulatory inspections and/or violations
- Fires
- Business interruptions

An accident or illness that has, or appears to have, any of the consequences listed below shall be immediately reported to the COR by the Site Manager or Task Manager. The PIKA-PIRNIE JV Team Site Manager will notify emergency responders, the PM and the COR. The PM will confirm the COR has been notified and will notify the PIKA-PIRNIE JV Team SHM and Corporate SHM via email and/or via telephone in accordance with the PIKA/PIRNIE Incident Reporting Standard ARC HSM-S010. The following accidents shall result in immediate notification to the COR:

- Fatal injury/illness
- Permanent totally disabling injury/illness
- Permanent partial disabling injury/illness
- Three (3) or more persons hospitalized as in-patients as a result of a single occurrence
- $200,000 or greater accident property damage due to an accident
- Injury or illness due to an arc flash incident

Except for rescue and emergency measures, the accident scene shall not be disturbed until it has been released by the COR. The PIKA-PIRNIE JV Team is responsible for obtaining appropriate medical and emergency assistance and for notifying fire, law enforcement, and regulatory agencies. The PIKA-PIRNIE JV Team shall assist the COR or other government agents conducting the government investigation(s) of the accident.

7.2 Hours Worked Reporting

The PM will provide the COR and SSHO with number of man hours worked on site on a monthly basis to facilitate maintenance of records on the incident and severity rates and the number of hours worked since a reportable incident occurred. The number of project hours, where the employee was in an on-duty pay status, will be reported by the PM to the COR by the 5th day of each month for the prior month.

7.3 Environmental Exposure Reporting

The air monitoring records provide data on potential exposure and non-exposure conditions to site contaminants. Air monitoring data will be summarized each month in a memorandum to the project file. The PIKA-PIRNIE JV Team and subcontractor personnel who worked within a site exclusion zone during the previous month will be issued a copy of the memorandum which will be discussed during a morning tailgate meeting.

The air monitoring summary memorandum will be prepared by the SSHO and distributed to affected personnel by the 5th day of each month. In additional to a summary of air monitoring data results, the memorandum will note any special events that may have affected environmental exposure conditions such as a spill or other unplanned release.

8. Plans (Programs, Procedures) Required by the Safety Manual

8.1 SSHP

A SSHP is required for remedial activities at the Former CNAD Complex Site developed in general accordance with the USACE Safety and Health Requirements Manual (EM385-1-1) The SSHP includes the following plans: Prevention of Alcohol and Drug Abuse, Night Operations Lighting, Hazardous Energy Control (Lockout-Tagout), Excavation and Trenching, and Confined Spaces.

8.2 Sanitation Plan

Drinking water, toilets and washing facilities are available in the Norfolk Southern office building, located on the project site. In addition, a portable toilet will be provided at the property for the duration of the remedial activities. The portable toilet would be equipped with an inside lock, a toilet seat and toilet seat cover, a urinal trough, a ready supply of toilet paper, and a hand sanitizer dispenser and will be compliant with EM-385-1-1 Section 02.E.02.

Areas will be kept as clean as possible, taking into consideration the nature of the work. Regular cleaning shall be conducted in order to maintain safe and sanitary conditions in the workplace including removal of packaging containers and loose items in work areas on a daily basis and cleaning the floors of offices and equipment storage areas at least weekly.

To facilitate cleaning, every floor, working place and passageway shall be kept free from protruding nails, splinters, loose boards, clutter and unnecessary holes and openings.

Office waste including used food cartons will be disposed of in tied plastic disposal bags with waste from the Norfolk Southern office building. Receptacles used for putrescible waste shall be constructed in order to prevent leakage and allow cleaning and maintenance. Such receptacles shall be equipped with solid tight fitting covers unless they can be maintained in sanitary condition without covers.

The workplace shall be maintained as clean as practical, in order to prevent the entrance of harborage of rodents, insects, or other vermin. If necessary, a continuing and effective extermination program shall be instituted. When the presence of vermin is detected, the use of licensed exterminators is required.

Drinking water will be supplied in plastic bottles sealed at the bottling plant or from dispensers intended to provide drinking water. Drinking water shall be provided according to the requirements of the Safe Drinking Water Act, as amended, and all applicable Federal State and local regulations.

9. Risk Management Processes

Detailed specific hazards and controls are provided in the project Activity Hazard Analyses found in Appendix B of the SSHP. The Activity Hazard Analyses are a fundamental component of the site-specific H&S orientation training and subsequent H&S meetings including occasions where a new task or new hazard has been identified.

Appendix A

Resumes

WILLIAM C. MENER, CSP

Manager, Industrial Hygiene

industrial hygiene
indoor air quality investigations
mold/asbestos/lead assessments
water intrusion
environmental site assessment

Mr. Mener has 21 years of experience with LFR Inc. in the Environmental Health and Safety (EHS) fields. He has managed numerous EHS projects involving health and safety, site investigation, air/water/noise/soil sampling, and remediation/abatement activities at residential, commercial, and industrial sites. He is experienced in EHS and consulting, industrial hygiene, mold/asbestos/lead surveys and remediation/abatement; water intrusion investigation; air and water permitting, and industrial ventilation design. His primary responsibilities include managing the Industrial Hygiene group; EHS; industrial hygiene; mold/asbestos/lead surveys and remediation/abatement; water intrusion investigation; design and installation of hoods, ductwork, and ventilation systems; ventilation system troubleshooting; stack design; evaluation of hood capture efficiency; independent compliance engineering; indoor air quality studies; air pollution control technology assessments; preparation of air and water permits; wastewater treatment specifications; dye-tracing operations; and particulate filter studies. He has also assisted in air dispersion modeling and risk assessment; stack emissions testing; and water/soil sampling.

EDUCATION

B.S. Mechanical Engineering, New Jersey Institute of Technology, 1987

A.S. Mechanical Engineering Technology, Somerset County College/Somerset County Technical Institute, 1985

REPRESENTATIVE EXPERIENCE

- **Assisted in accident investigations.** Provided engineering assistance in the investigation of a fatal industrial accident involving carbon monoxide poisoning as a result of not following confined space entry procedures. Provided assistance in determining the root cause, recommendations, and follow-up of work related injuries.
- **Preparation of Job Safety Analyses.** Prepared numerous Job Safety Analyses for activities within various industries.

- **Preparation of Risk Assessment and Hazard Control Plan.** Assisted in the preparation of Risk Assessment and Hazard Control Plans for printing operations to evaluate and eliminate risks associated with the exposure to volatile organic compounds.
- **Health and Safety Training.** Conducted numerous training classes pertaining to hazardous materials safety, hazardous waste operations, electrical safety, and liquid propane gas safety.
- **Health and Safety Plan Development and Review.** Developed and reviewed numerous Health and Safety Plans (HASPs) for various activities including excavations, well drilling, and soil and water sampling.
- **Site Auditing.** Conducted numerous health and safety site audits at job sites to ensure the safety of workers, general public, and property.
- **Evaluated and Specified Electrical Components For Use in Hazardous Locations.** Conducted an evaluation of existing electrical components within a hazardous atmosphere and specified new components as necessary.
- **Managed and Conducted Moisture Mapping Activities in Gulfport, MS and New Orleans, LA after Hurricanes Katrina and Rita.** Collected and reviewed moisture data obtained from building materials within flood impacted and hurricane damaged buildings. Provided remediation and building material drying recommendations to general contractor.
- **Managed and Conducted Mold/Asbestos/Lead Surveys, Abatement/Remediation Project Monitoring, and Contractor Oversight at Numerous Commercial, Industrial, and Residential Locations.**
- **Managed Comprehensive Lead-Based Paint Surveys.** Utilizing X-ray Fluorescence (XRF) real time instruments, under United States Environmental Protection Agency (U.S. EPA) and Housing and Urban Development (HUD) sampling protocol for various facilities in New York and New Jersey.
- **Managed and Conducted Noise Surveys.** Performed personal and ambient noise monitoring for various commercial, industrial, and manufacturing facilities. In some cases, the data obtained was used to develop a hearing conservation plan.
- **Developed Particulate Air Emission Factors.** Conducted field work and developed particulate air emission factors for welding and sand blasting operations for the ship building industry. The data and developed air emission factors are under consideration by the U.S. EPA to be included as U.S. EPA emission factors.
- **Environmental Site Assessments.** Conducted numerous Phase I Environmental Site Assessments at various facilities.
- **Engineering, Permitting and Construction/startup Oversight Services for an Air Emission/Odor Control System at a Vinyl Reprocessing Facility in NY.** Evaluated historical data and existing process operations, heating, ventilating, and air conditioning (HVAC) systems and administrative procedures to determine source of odors. Design effort involved improvement of hood capture efficiency, installation of supplementary emission controls, and installation of a taller stack.
- **Designed Complete Ventilation System for the Maintenance Shop.** Which included calculations to determine whether existing dust collector and fan were adequate at a medical

equipment manufacturing firm in New Jersey. Conducted airflow measurements and air balancing on various systems and prepared New Jersey Department of Environmental Protection (NJDEP) air permit applications for numerous sources. Designed various stack configurations for the boilers.

- **Designed and Installed a Complete Ventilation System for Sand Core Molding Department of a Cast Iron Pipe Manufacturer in NJ.** Assisted in the inspection and purchase of two dust collectors. Assisted in the preparation and review of contractor bid specification packages. Prepared the air permit applications for this system. Provided contractor supervision for the installation portion of the project.
- **Conducted Numerous Indoor Air Quality Investigations.** Investigated for carbon monoxide, carbon dioxide, temperature, relative humidity, bioaerosols, volatile organic compounds, formaldehyde, sulfur compounds, particulates and metals. Provided remedial recommendations as necessary.
- **Assisted in the Design of a Secondary Dust Collection System Including Fan Specification and Ductwork, for a Lead Chromate Manufacturer in NJ.** Prepared an air permit application for this system. Prepared screening risk assessments for air emissions of lead and chromium.
- **Conducted Air Flow Measurements and Balancing of Exhaust Streams From Four Coating Lines to a Catalytic Incinerator at a Foil Coating Facility in NJ.** Assisted the facility in complying with NJDEP capture efficiency requirements. Assisted in the design of the incinerator stack and associated ductwork.
- **Prepared Air Permit Applications and Supporting Calculations for a Site Remediation Contractor.** This was for several mobile, soil remediation system installations in New Jersey. Assisted in the preparation of screening risk assessments for air emissions of suspected carcinogens.
- **Prepared Superfund Amendments & Reauthorization Act (SARA) 312 Community Right-To-Know reports in NJ.** Reports were for a medical equipment manufacturer.
- **Designed a Carbon Adsorption System for Inhalation Toxicology Chambers for an Animal-testing Laboratory in NJ.** Assisted in the design of a thermal incinerator. Prepared air permit applications for the carbon adsorption system and thermal incinerator. Also prepared the air permit application for a pilot plant system.
- **Assisted in Review and Assessment of Baghouse Operations to Minimize Lead Emissions from a Battery Manufacturing Plant in New Jersey.** Conducted diagnostic measurements and reviewed maintenance, recordkeeping, and training procedures.
- **Conducted Sanitary Sewer and Storm Line Dye Tracing for a New Jersey-based Copper Powder Reclaimer.** Designed new sanitary sewer and storm line connections. Prepared NJPDES permit application for entire facility. Prepared contractor bid specifications. Designed a complete ventilation system for select sources in the copper powder department. Assisted in the preparation of dust collector refurbishment specifications, air recirculation studies, and inspection of on and off site dust collectors. Prepared the air permit applications for the above system. Prepared and reviewed contractor bid specification packages.

- **Assisted in the Design of Permanent Total Enclosures Around Two Coil Coating Lines to Satisfy NJDEP Capture Efficiency Requirements at a Vinyl Siding Manufacturing Plant in NJ.** Reviewed contractor bid specification package. Prepared the air permit application for this source.
- **Designed Permanent Total Enclosures Around Four Coating Lines to Satisfy NJDEP Capture Efficiency Requirements at a Coating Facility in NJ.** Prepared and reviewed contractor bid specification packages.
- **Conducted Airflow Measurements for Each Source at a Fabric Finishing Plant in NJ.** Assisted in the evaluation of an odor control device for a tentor frame. Prepared the air permit application for this source. Assisted in NJDEP negotiations.
- **Conducted Sanitary and Storm Line Dye Tracing at a Bus Garage in NJ.** Prepared new site drawing indicating sanitary and storm line configuration.
- **Assisted in Incinerator Performance Investigation and Air Permit Application Preparation for a NJ Coating Facility.** Assisted the facility in complying with NJDEP capture efficiency requirements. Provided technical engineering services during stack tests.
- **Served as an independent compliance engineer for a health waste processing facility in NY.**
- **Assisted an Auto Marine Terminal Facility in New Jersey in Complying with NJDEP Capture Efficiency Requirements.** Conducted air emission studies using an Organic Vapor Analyzer and an Organic Vapor Monitor to determine the fugitive emissions from the inlet and outlet of the de-waxing tunnel.
- **Served as the Borough of High Bridge Engineer Under the Supervision of a Licensed PE.** Conducted study of future sewer capacity requirements for residential, commercial, and industrial zones and assisted with the review of pumping station upgrade specifications and contract.
- **Participated in Review of a Proposed Shared Energy Savings Agreement Between a Local County Government Agency and an Independent Monitoring Company.** The evaluation consisted of reviewing all of the energy upgrades at the facility, contract review prior to county signature to assure that the monitoring company does not receive credit savings for energy projects initiated by the county, and proper apportioning of energy savings.
- **Provided Energy Evaluation Services for a Local County Government Agency.** Scope of work included review of three years of energy bills, review of the energy savings calculations for an independent monitoring company, and determination of whether the energy savings were calculated properly.
- **Provided Energy Audit Evaluation Services at a Postal Facility in Budd Lake, NJ.** Scope of work includes the review of electricity and propane usage, review of lighting equipment and HVAC systems, and recommendations for upgrades to the facility to reduce overall energy usage.

PUBLICATIONS

Author/co-author of over 400 technical reports for commercial clients.

REGISTRATIONS/CERTIFICATIONS

Certified Safety Professional, (Certificate #18458)
New York State Asbestos Inspector (Certificate #05-08209)
Virginia State Asbestos Inspector (License #3303 003094)
Pennsylvania State Asbestos Inspector (Certificate #038209)

SEMINARS/COURSES

Department of Transportation Compliance Training

3-Day NY State/EPA/AHERA Asbestos Building Inspector Program

Occupational Safety and Health Administration (OSHA) Hazardous Waste Site Safety Training (40-hour and annual refreshers)

OSHA Hazardous Waste Site Supervisor

National Institute for Occupational Safety and Health (NIOSH) Industrial Ventilation Training

NIOSH/EOHSI Indoor Air Quality in Non-Industrial Buildings

Environmental Protection Agency Control of Particulate Emissions

The Center for Professional Advancement, Indoor Air Quality

American Industrial Hygiene Association (AIHA) New Jersey, Assessing and Remediation of Microbial Contamination in the Indoor Environment

Underground Tank Management and Design, The University of Wisconsin

PROFESSIONAL HISTORY

ARCADIS Inc., Manager-Industrial Hygiene, November 1987 to present

Kevin Held

Senior Environmental Scientist

Education

M.S. Environmental Science and Toxicology, New Jersey Institute of Technology, 1990

B.A. Psychobiology, University of California Santa Cruz, 1984

Years of Experience

Total - 26

With ARCADIS –2

Professional Registrations

New Jersey UST Closure and Subsurface Evaluator License

U.S. Army Corps of Engineers Construction QC Manager

American Society of Heating, Refrigeration and Air-conditioning Engineers (ASHRAE), Member

National Environmental Health Association Industrial Toxicologist

Professional Qualifications

Occupational Safety and Health Administration (OSHA) Train-the-trainer, Construction Standards

Hazardous Waste Operations and Emergency Response (HAZWOPER), Incident Command, USEPA

Shipper of Dangerous Goods, USDOT, IATA

Permit Required Confined Space Entry

Advanced Microbiology Investigations, EMSL Analytical, Inc.

Underground Storage Tank Management, Professional Engineering Society

Mr. Kevin Held's broad EHS work experience includes various roles at superfund sites, managing environmental compliance at industrial facilities, industrial hygiene, ambient air and indoor air quality investigations. Clients have cited Mr. Held for providing exceptional environmental services and contributing to receipt of major awards through creative air monitoring strategies including the 2007 award of Environmental Excellence from the US EPA and 1999 US Army Corps Regional Safety Award.

Mr. Held provided health and safety management services for high-risk activities including: stabilization of hydrogen sulfide gas emitting refuse, installation of gas extraction wells, abandonment of subsurface leachate collection systems, extraction of experimental rocket fuels, clean-up of abandoned vaccine supplies, evaluation of USPS mail suspected to contain critical biological agents, and the excavation and sampling of buried drums. Many high hazard activities were completed at national priority superfund sites. He participated in various municipal-scale projects compliant with National Ambient Air Quality Standards (NAAQS) State and Local Air Monitoring (SLAM) networks and State standards relevant to fence line air quality at construction and hazardous material sites. Experience includes the development and implementation of a state-of-the-art ambient air and noise monitoring program for the lower Manhattan redevelopment program that began operation in 2005.

Representative Experience

Ingersoll-Rand Company – Outsourced EHS Manager during the last year of manufacturing and closing of a 115-year old powered tool manufacturing 32-acre facility. Responsibilities included discharge compliance monitoring and reporting, waste management, oversight of wastewater treatment system, cleaning and transport of manufacturing equipment including vessels with hazardous wastes, phase I assessment,

phase II investigations, closure of storage tanks and related equipment and remediation actions.

Lower Manhattan Construction Command Center (LMCCC) - Designed and implemented a program to monitor dust and noise in a 1-square mile area of Lower Manhattan during the $30B Lower Manhattan Redevelopment Program in and around the World Trade Center Site. Program included the installation and operation of $300,000 of ambient monitors that feed real-time data to a secured web site that provides alarms and status conditions to project scientists. This rapid evaluation of neighborhood-scale conditions allows prompt investigations of suspect sources of fugitive emissions, such as particular construction sites and operations. Program also includes mobile monitoring of construction sites for use of low-sulfur fuels, diesel particulate filters, tracking pads, tire wash, dust suppression, noise, and fugitive emissions. Data from the air monitoring stations was incorporated into the SLAMS and the project approach helped the client (LMCCC) receive the 2007 Environmental Excellence Award from US EPA.

US Army Corps of Engineers and US Environmental Protection Agency – Worked as site safety officer and manager during investigative and remedial actions at over 25 superfund sites. Activities included management of health and safety programs, air monitoring, sample collection and analysis, construction quality control, heavy equipment operation and laborer.

North Princeton Development Center - Planned and implemented community-scale environmental monitoring program for township with an elementary school inside an abandoned 256-acre property. The abandoned property had been a former epileptic community and NJ State Psychiatric Hospital in Montgomery Township, New Jersey. Project entailed real-time monitoring plus collection of air samples for laboratory analysis during demolition of 98 buildings and excavation of an ash fill. Demolition activities included remediation of five-miles of outdoor steam pipes with asbestos containing insulation.

U.S. Postal Service - Preparation and implementation of comprehensive OSHA compliance programs at 27 US Postal Service Facilities following US Presidential executive order mandating US Postal Service to comply with all OSHA provisions following 20-years of exemption. Facility-specific programs included all written programs required by OSHA standards and USPS Standard Operating Procedures as well as training. Training included awareness, operational, and supervisory level training for USPS personnel, including emergency response drills. With addition of other projects completed at over 100 facilities, Mr. Held was cited for outstanding service with a very diverse array of projects over a 7-year period, including emergency response, anthrax and critical biological agent testing, indoor air quality investigations, radon mitigation, permitting, chemical storage, chemical waste, asbestos delineation and remediation, as well as the various OSHA compliance programs.

IEQ Investigations **-**Multiple Locations - Comprehensive IEQ investigations at numerous bank facilities, hospitals, medical clinics, USACE Headquarters, GAO Building, Four Seasons Hotels, and more than 1,000 buildings in total, including class A office buildings, landmark and institutional buildings. Mr. Held specializes in addressing IEQ-related health complaints.

Selected Publications

"Implementing Environmental Performance Commitments (EPCs) During the Lower Manhattan Redevelopment Project," EM – The Magazine for Environmental Managers, Air & Waste Management Association, February 2008, pgs. 24-27.

"Particulate Monitoring and Control in Lower Manhattan During Large Urban Redevelopment" Air &Waste Management Association's 2007 Conference Proceedings.

Ground Zero Focus Shifting to Indoor Environments, Indoor Environment Connections, vol. 3, Issue 11, September 2002, page 1. http://www.ieconnections.com/archive/sept_02/sept_02.htm#article_1

Increasing Outside Air to Comply with ASHRAE 62, Indoor Environment Connections, vol. 3, Issue 10, August 2002, page 14.

Allergist Makes Calls to Sick Buildings, Indoor Environment Connections, vol. 3, Issue 10, August 2002, page 13.

The First Line of Defense: Maintenance Departments Can Make or Break IAQ, Indoor Environment Connections, vol 3, Issue 8, June 2002, page 1. http://www.ieconnections.com/archive/jun_02/june_2002.htm#article2

Presentations

"Particulate Monitoring and Control in Lower Manhattan During Large Urban Redevelopment" at the Air & Waste Management Association 2007 annual conference in Pittsburgh, PA, June 28, 2007.

"Moisture-Mold Problems: Prevention, Investigation and Remediation" approved for 0.1 CEU by the International Facilities Management Association at the Mid-Atlantic Plant Engineering & Facilities Management Conference, November 1, 2006.

Appendix B-2
Site Safety and Health Plan

Former Charlotte Naval Ammunition Depot
Charlotte, North Carolina

December 2011

Revision 2 – May 2012

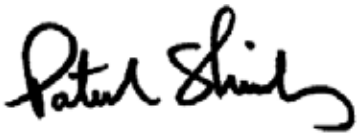
DERP-FUDS Project No. I04NC080301
Contract No.:W912DY-10-D0025
Delivery Order No.: 0007

PREPARED FOR:

U.S. Army Corps of Engineers, Huntsville Center
U.S. Army Engineering and Support Center
4820 University Square
Huntsville, Alabama 33816-1822

Kevin Held
PIKA-PIRNIE JV Site Safety and Health Plan Writer

William Mener, CSP
PIKA-PIRNIE JV Site Safety and Health Plan Reviewer



Patrick Shirley, PG
PIKA-PIRNIE JV Certified Project Manager
Second Plan Reviewer

Site Health & Safety Plan

Former Charlotte Naval
Ammunition Depot
Charlotte, North Carolina

Prepared for:
U.S. Army Corps of Engineers,
Huntsville Center

Prepared by:
PIKA-PIRNIE JV, LLC
12723 Capricorn Drive
Suite 500
Stafford, Texas 77477

Our Reference:
DERP-FUDS Project No. I04NC080301
Contract No.: W912DY-10-D0025
Delivery Order No.: 0007

Date:
December 2011
Revision 2 – May 2012

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Acronyms and Abbreviations

ACGIH	American Conference of Industrial Hygienists
AHA	Activity Hazard Analysis
ANSI	American National Standards Institute
ASIP	Arrowood Southern Industrial Park
ASTM	American Society for Testing and Materials
APP	Accident Prevention Plan
BEI	Biological Exposure Indices
C°	Celcius
cm^3	Cubic Centimeter
CFR	Code of Federal Regulations
CNAD	Charlotte Naval Ammunition Depot
COC	Constituent of Concern
CO	Contracting Officer
COR	Contracting Officer's Representative
CPR	Cardiopulmonary Resuscitation
CRZ	Contaminant Reduction Zone
dBA	Decibel
DEET	N,N-diethyl-meta-toluamide
EMS	Emergency Medical Services
EpiPen	Epinephrine Auto Injector
ERD	Enhanced Reductive Dechlorination
ERP	Emergency Response Plan
eV	Electron Volt
eZ	Exclusion Zone
°F	Fahrenheit
FHSHB	Field Health and Safety Handbook
FRA	Federal Railroad Administration
H&S	Health and Safety
HARC	Hazard Assessment and Risk Control
HazCom	Hazardous Communication

HazMat	Hazardous Materials
HAZWOPER	Hazardous Waste Operations and Emergency Response
HEPA	High-Efficiency Particulate Air
HSE	H&S Environment
HSU	Hydrostratigraphic Unit
IDLH	Imminently Dangerous to Life and Health
IP	Ionization Potential
JV	Joint Venture
LEL	Lower Explosive Limit
LI	Loss Investigation
mg/m^3	Milligram per Cubic Meter
MNA	Monitored Natural Attenuation
mph	Miles per Hour
MSDS	Material Safety Data Sheet
NA	Not Applicable
NIOSH	National Institute for Occupational Safety and Health
NOAA	National Oceanic and Atmospheric Administration
NRR	Noise Reduction Rating
OSHA	Occupational Safety and Health Administration
PEL	Permissible Exposure Limit
PID	Photoionization Detector
PIKA	PIKA International, Inc.
PIKA-PIRNIE JV Team	PIKA International, Inc./Malcolm Pirnie, Inc. Joint Venture LLC Team
Pirnie	Malcolm Pirnie, Inc.
PM	Project Manager
PPE	Personal Protective Equipment
ppm	Parts per Million
PVC	Polyvinyl Chloride
QC	Quality Control
OV	Organic Vapor

RAWP	Remedial Action Work Plan
RMSF	Rocky Mountain Spotted Fever
SHM	Safety and Health Manager
SSHO	Site Safety and Health Officer
SSHP	Site Safety and Health Plan
STEL	Short Term Exposure Limit
TCE	Trichloroethene
TLV	Threshold Limit Value
TWA	Time Weighted Average
µg/L	Micrograms per Liter
U.S.	United States
USACE	U.S. Army Corps of Engineers, Huntsville Center
USEPA	U.S. Environmental Protection Agency
VOC	Volatile Organic Compound

1. Introduction

All work on this project will be carried out in compliance with the PIKA International, Inc. (PIKA)/Malcolm Pirnie, Inc. (Pirnie) Joint Venture (JV), LLC (the PIKA-PIRNIE JV Team) Health and Safety (H&S) Standards, Occupational Safety and Health Administration (OSHA) Standards for General Industry and Construction and United States (U.S.) Army Corps of Engineers (USACE) Safety and Health Requirements found in Consolidated EM385-1-1 (2008 with Errata and Changes) and USACE Technical Guidelines for Hazardous & Toxic Waste Treatment and Cleanup Activities EM 111-1-502.

The design of this Site Safety & Health Plan (SSHP) conforms to the requirements of the ARC HSFS010-H&S Plan Standard, the OSHA Hazardous Waste Operations and Emergency Response (HAZWOPER) Standard 29 Code of Federal Regulations (CFR) 1926.65(b)(4), and USACE EM385-1-1 Section 28.B (SSHP) and the Accident Prevention Plan (APP).

Specific H&S information for the project is contained in this SSHP. All personnel working on hazardous operations or in the area of hazardous operations shall read and be familiar with this SSHP before doing any work. All project personnel shall sign the certification page acknowledging that they have read and understand this SSHP.

Changes in the scope of the project or introduction of new hazards to the project shall require revision of the SSHP approved by the Project Manager (PM) and Safety and Health Manager (SHM). The SSHP Addendum Form and log table are included as **Appendix A** of this plan.

The objectives of this SSHP are to increase individuals' awareness, promote sound H&S behavior and eliminate incidents and injuries. The PIKA-PIRNIE JV Team is committed to providing a healthy and safe working environment by ensuring employees:

- Are an integral part of our existing H&S program and help to strengthen it;
- Emphasize proactive activities;
- Capitalize on the job expertise of personnel;
- Maximize the use of positive reinforcement;

- Integrate behavior-based tools with daily business activities; and
- Practice "providing stewardship in H&S Environment (HSE) from the top down while developing HSE solutions from the bottom up".

All PIKA-PIRNIE JV Team employees and sub consultants are expected to understand and use this SSHP and apply the H&S best practices and principles contained herein when performing their daily work activities.

The SSHP is intended to supplement the PIKA-PIRNIE JV Team Employee Field H&S Handbook (FHSHB) (required sections included in Section 6.2). The Handbook covers a wide variety of project types and potential hazards. This Plan has been tailored to address the specific policies and hazards present for the work at this project.

We are committed to:

- Protecting our employees and clients from injury and illness;
- Placing H&S values on par with our other core business values;
- Supporting a H&S culture where employees share in this commitment to integrate H&S into their behaviors; and
- Integrating H&S safety into the solutions we offer to clients.

2. Project Scope of Work and Site Description

At the time of operation, the entire Charlotte Naval Ammunition Depot (CNAD) complex occupied approximately 2,266 acres. In June 1942 the United States Rubber Company began operating an ammunition shell loading and assembly plant on site that was taken over by the U.S. Navy in 1945. In 1956, the Naval Depot status was changed to inactive. In 1959, the former CNAD complex was sold to a development partnership and is currently occupied by light industrial and commercial businesses.

Two areas (1 and 2) were used for the production of munitions. Each area had multiple buildings with the largest buildings used for assembly, packaging and shipping of munitions. After 1945, operations in area 2 included reconditioning of munitions. This included degreasing operations in the southeast corner of Building 2-30 which utilized trichloroethene (TCE). TCE was found in groundwater plumes originating from this location, the mitigation of which is the objective of this work effort.

2.1 Scope of Work/ Project Type

The remedial technical approach involves implementing active enhanced reductive dechlorination (ERD) treatment in areas where TCE concentrations are present at concentrations greater than 500 micrograms per liter (µg/L), followed by monitored natural attenuation (MNA) to achieve the long-term remedial goals. The active ERD treatment period will include periodic injections of a carbohydrate solution to provide an organic carbon source to increase the rate at which naturally occurring bacteria breakdown TCE under anaerobic conditions. The carbohydrate solution injected in the treatment area will consist of dilute molasses. The remedial approach includes subsurface injections to address TCE impacts in the transition zone and bedrock hydrostratigraphic units (HSUs). The hydrogeology and nature and extent of TCE impacts in each of these HSUs are further detailed the Remedial Action Work Plan (RAWP).

2.2 Site Description

Site Type:

X	Active	X	Secure	X	Industrial	X	Building		Ports
	Inactive		Unsecured	X	Commercial		Highway/ Bridges		Water work
			Uncontrolled		Residential	X	Rail		Undeveloped
Other specify:									

The Former CNAD complex covers over 2,000 acres of land that currently includes the Arrowood Southern Industrial Park (ASIP) which has a combination of light industrial and commercial tenants. Other areas of the Former CNAD site with planned work activities include a series of parallel rail road spurs associated with a bulkmatic loading facility operated by the Norfolk Southern Railway.

3. PIKA-PIRNIE JV Team Organization and Responsibilities

3.1 All Personnel

Each person is responsible for completing tasks safely, and reporting any unsafe acts or conditions to their supervisor. No person may work in a manner that conflict with these procedures. Prior to initiating site activities, all PIKA-PIRNIE JV Team and subcontractor personnel will receive training in accordance with applicable regulations, and be familiar with the requirements and standards referenced in this SSHP. In addition, all personnel will attend daily safety meetings (tailgate meetings) to discuss site-specific hazards prior to beginning each day's work. Every PIKA-PIRNIE JV Team employee, subcontractor, and client representative at the Site has the responsibility to stop the work of a coworker or subcontractor if the working conditions or behaviors are considered unsafe. Workers will be required to comply with requirements of the Drug and Alcohol program, set forth in **Appendix E**.

3.2 Construction Manager

Additional responsibilities of the construction manager are as follows:

- Review all applicable H&S Procedures, and ensure that project activities conform to all requirements.
- Obtain client-specific H&S information and communicate with the client on H&S issues.
- Communicate with the Site Safety and Health Officer (SSHO) on H&S issues.
- Allocate resources for correction of identified unsafe work conditions.
- Ensure PIKA-PIRNIE JV Team site workers have all training necessary for the project.

The Construction Manager must report all injuries, illnesses and near-misses to the SHM, SSHO, lead incident investigations, and ensure that safety programs and recommendations are implemented.

3.3 SHM

The SHM oversees all aspects of the site safety program, and prepares and or reviews any site-specific H&S guidance documents or addenda to this plan. The SHM does not report to the Construction Manager, and is separately accountable to the PIKA-PIRNIE JV Team project team senior management for construction H&S. The SHM acts as the sole contact to regulatory agencies on matters of H&S. Other responsibilities include:

- Overall authority for H&S compliance and SSHP conformance for the project.
- Conducts General H&S program administration.
- Conducts on-site project H&S audits and participates in teleconferences as warranted.
- Remains available for project emergencies.
- Develops and approves modifications to the SSHP as needed.
- Evaluates exposure monitoring/air sampling data and adjusts the SSHP requirements as necessary.
- Serves as a quality control (QC) staff member.
- SSHP approval by signature.
- Determines the level of personal protection required.
- Updates equipment or procedures based on information obtained during site operations.
- Assists in injury, illness and near-miss investigation and follow-up.
- Assists the SSHO in issues as they arise.
- Perform site audits and assessments.

- Assists/conducts near-miss/incident investigation.

- Serves as the liaison with corporate during H&S regulatory issues as they may arise.

3.4 SSHO

The SSHO has the following responsibilities and qualifications:

- The SSHO or his designated representative must be present anytime clean-up operations are being performed to implement the SSHP. After the groundwater injection system becomes operational and is automated, the PIKA/PIRNIE JV Team anticipates having one field technician on site periodically. This individual will be a collateral SSHO.

- Ensures that this SSHP is available to and reviewed by all site personnel including subcontractors.

- Ensures that necessary site-specific training is performed (both initial and "tailgate" safety briefings).

- Has a minimum of 1 year experience implementing safety and occupational health procedures at clean-up operations.

- Conducts exposure monitoring/air sampling and selects/adjusts protective equipment use.

- Inspects site activities to identify safety and occupational health deficiencies and correct them.

- Coordinates changes/modifications to the SSHP with the SHM, site superintendent, and Contracting Officer's Representative (COR).

- Conducts project-specific training.

- Ensures site visitors have been informed of the hazards related to the PIKA-PIRNIE JV Team work, and have signed the Site Visitors Log.

- Ensures that work is performed in a safe manner and has authority to stop work when necessary to protect workers and/or the public.
- Coordinates activities during emergency situations.
- Ensures that all necessary permits and safety information provided by the client is disseminated to other site personnel and is maintained in an organized manner.
- Reports all injuries, illnesses and near-misses to the PM, COR, and SHM.
- Ensures that necessary safety equipment is maintained and used at the site.
- Monitor the implementation of the SSHP.

4. Hazard Analysis

Table 4-1. Hazard Ranking Chart

Severity	Consequence		Probability				
	Property Damage	Injury	Frequent	Likely	Occasional	Seldom	Unlikely
	> $100,000	Fatality	H	H	H	H	M
	> $10,000	Injury Requiring Hospitalization	H	H	H	M	L
	> $1000	Injury Requiring Medical Treatment Beyond First Aid	H	M	M	L	L
	< $1000	Injury Requiring First Aid	M	L	L	L	L

☒ Hazards are ranked using the the PIKA-PIRNIE JV Team hazard assessment and risk control (HARC) Process: *ARC_HSMS002*

Biological		***Mechanical***		***Gravity***	
L	Biting/stinging insects	L	Cuts on equipment/tools	M	Slip, Trip, Fall
L	Biting animals	L	Pinch points on equipment	L	Fall from height
L	Poisonous plants	L	Burns from equipment	L	Ladders or scaffolds
L	Phys. damaging plants	L	Struck by equipment	L	Struck by falling object
Driving		***Motion***		***Environmental***	
L	Night driving	L	Lifting/awkward body positions	L	Heat
L	Off-road driving	L	Struck by vehicle/traffic	L	Cold
L	Urban driving			L	Inclement Weather
L	All terrain vehicle	***Personal Safety***		L	High Wind
NA	Boat	L	Working late/night	L	Water/Sea
		L	Working alone		
Electrical		L	High Crime Area		
L	Wet environments				
L	Electrical panels	***Pressure***		***Sound***	
L	Electric utilities	L	Utilities (gas, water, etc)	L	Equipment Noise
L	Electric power tools	L	Hydraulic	L	Tool Noise
		L	Compressed air	L	Traffic Noise

4.1 Constituent of Concern (COC)

The Constituent of Concern (COC) at the site is TCE.

Chemical Hazard Information

Chemical Name	IP (eV) Ionization Energy	Odor Threshold (ppm)	Routes of Entry/ Exposure Symptoms	8-hour TWA[1] (ppm)	IDLH (NIOSH) (ppm)	STEL (ppm)	Source TLV/PEL
TCE	9.45 eV	110 ppm	Inhalation, skin absorption, ingestion, skin/eye contact. Irritation to the eyes, skin; visual disturbance, lassitude (weakness, exhaustion), dizziness, tremor, drowsiness, nausea, vomiting; dermatitis; cardiac arrhythmias, paresthesia; liver injury (potential occupational carcinogen).	10 ppm	1,000 ppm	25 ppm	TLV

Notes:
[1]The TLV from the American Conference of Industrial Hygienists (ACGIH) is listed unless the PEL, designated by OSHA, is lower.
NIOSH = National Institute for Occupational Safety and Health
IP = ionization potential
cm^3 = cubic centimeter
eV = electron volt
IDLH = Imminently dangerous to life and health
mg/m^3 = milligrams per cubic meter
NA = not applicable
PEL = Permissible Exposure Limit
ppm = parts per million
STEL = Short- term exposure limit
TLV = Threshold Limit Value
TWA = time weighted average

See Section 10 for information on air monitoring requirements.

4.2 Activity Hazard Analyses (AHAs)

An AHA has been completed for each safety critical task on this project, and are included in **Appendix B**. Each AHA contains specific H&S information for each task, and; therefore, takes precedence over other H&S documents. Hazards identified in the table above are addressed specifically in the AHAs as well as control methods to protect employees and property from hazards. The AHA also lists the type of personal protective equipment (PPE) required for the completion of the project. A detailed list of PPE for the project is located in **Appendix D**.

AHAs are available in **Appendix B** of this SSHP for the following tasks.

- Mobilizing and demobilizing equipment
- Soil drilling and well installation

- Installation of storage tanks and process equipment

- Operation of injection equipment

If additional H&S information is needed for the project, refer to the applicable the PIKA-PIRNIE JV Team H&S Standards and FHSHB sections that are listed below.

4.3 PIKA-PIRNIE JV Team H&S Standards

These PIKA-PIRNIE JV Team H&S Standards provide standard operating procedures for various H&S tasks such as utility clearance and hot work. The H&S Standards will be used as an additional source to the AHAs and this SSHP. All PIKA-PIRNIE JV Team field staff have access to the H&S Standards, as well as an electronic copy of the FHSHB, via the PIKA-PIRNIE JV Team intranet site, APEX.

- ARC HSFS019 – Utility Clearance – This operational procedure requires personnel to obtain a minimum of three lines of evidence prior to excavation or intrusive subsurface operations with potential to contact underground utilities and that the evidence be documented on the Utility and Structures Checklist found in **Appendix C**, SSHP forms. Additional detail on excavation and trenching is provided in the Excavation and Trenching Plan, provided as **Appendix H**.

- ARC HSCS013 – Hot Work (including welding and cutting) – This standard provides procedures for preparing a work area for hot work including the completion of a clearance inspection and a hot works permit. A copy of the hot works permit is included in **Appendix C**, SSHP forms.

- ARC HSFS003 – Confined Space Entry – This procedure requires identification of confined space locations on-site and identification of permit required confined spaces. Includes requirements for labeling confined spaces and a permit-required confined space program in the event that permit-required entries may occur on-site. Permit-required confined space entries are not anticipated so a copy of the permit is not included with the SSHP forms in **Appendix C**. The Confined Space Entry Program is detailed in **Appendix I**.

- ARC HSFS004 – Control of Hazardous Energy (Lockout/Tagout) – Program requires identification of activities that require isolation of hazardous

energies such as temporary isolation of electrical service to an electric-powered motor before conducting tasks that expose personnel to hazards associated with inadvertent operation of the equipment. The Hazardous Energy Control Plan has been included as **Appendix G**.

- ARC HSGE024 – Defensive Driving – Program requires site personnel to participate in the Smith Driving Safety Program and that PIKA-PIRNIE JV Team personnel refrain from phone conversations or operation of electric communication devices while driving.

- ARC HSFS006 – Electrical safety – Program includes qualifications and competent person requirements for restricted work activities including installation of electrical service to sight equipment. ARC HSIH008 – Hearing Conservation – Standard includes provisions for ensuring hearing protection is available, personnel receive training, and noise monitoring is conducted when appropriate and affected personnel are enrolled in a hearing conservation program.

- ARC HSMS010 – Incident Investigation – Program includes PIKA-PIRNIE JV Team forms to be completed for corporate records, investigative techniques, and required corporate notifications.

- ARC HSMS011 – Root Cause Analysis – Procedure with guidelines and procedures for performing root cause analysis on various system failures including safety incidents.

- ARC HSGE015 – PPE Includes requirements for identification of PPE requirements and formal review of determinations prior to task implementation. Includes requirements for designated person to oversee storage, training and inspection of PPE.

- ARC HSGE007 – Hazard Communication (HazCom) – Program includes requirements for training, preparation of a site-specific program that includes a hazardous substance inventory, material safety data sheet (MSDS) file, and provisions to ensure proper labeling.

- ARC HSFS021 – Ladder Safety Standard – This standard includes provisions for the selection, use, storage and inspection of ladders on project sites.

- ARC HSFS007 – Elevated Work and Fall Protection – This procedure includes requirements for fall arrest systems including: railings, leading edge restriction, safety nets and personal fall arrest systems during construction activities where personnel may access a walking or working surface with an unprotected side or edge more than six feet above a lower level or at any height where personnel could fall into or onto dangerous equipment. The procedure includes an Elevated Work Permit to be completed prior to conducting work activities on elevated work surfaces. A copy of the Elevated Work Permit is included in **Appendix C**, SSHP Forms.

4.4 FHSHB

The FHSHB will be used as an additional source to the AHAs and this SSHP. The FHSHB provides technical information that enhances the knowledge and thereby facilitates the effectiveness of personnel tasked with implementing specific H&S procedures. The FHSHB addresses topics that are also addressed in EM385-1-1. In any instances where the provisions of EM385-1-1 differ from requirements or guidance found in the FHSHB, the requirements of EM385-1-1 will take precedence. Following are lists of section topics from the FHSHB that are anticipated to be applicable to the Former CNAD Complex Site H&S program.

<u>General Field H&S Requirements (Section III)</u>

III.A. Daily Safety Meetings/Tailgates
III.F. General Housekeeping, Personal Hygiene and Field Sanitation
III.I. Severe Weather
III.J. Fire Prevention
III.K. HazCom
III-L. Noise
III-M. Heat and Cold Stress
III-N. Biological Hazards
III-O. Illumination
III-Q. Field Office General H&S Requirements
III-R. PPE
III-U. Driving
III-Y. Confined Spaces
III-Z. Control of Hazardous Energy
III-AA. Electrical Safety
III-BB. Fall Protection
III-CC. Hand and Power Tools
III-EE. Ergonomics

III-JJ. Ladders (Portable/Fixed)
III-KK. Signs, Signals and Barricades
III-LL. Traffic Control
III-MM. Utility Location

General Field Construction H&S (Section IV)

IV-A. Elevated Work Surfaces
IV-B. Scaffolds
IV-C. Lifts
IV-E. Heavy Equipment
IV-F. Hoisting and Rigging, Cranes and Derricks
IV-G. Forklifts
IV-H. Concrete and Masonry
IV-I. Demolition
IV-J. Blasting and Explosives
IV-K. Welding and Cutting
IV-L. Temporary Working Surfaces and Railings
IV-M. Underground Construction (Tunnels)
IV-N. Steel Erection
IV-O. Rollover and Overhead Protection
IV-P. Power Transmission and Distribution
IV-Q. Permit to Work

5. Training

5.1 Authorization to Enter

Only personnel with the appropriate training and medical certifications will be allowed to work at the site. The SSHO will maintain a list of authorized workers; only personnel on the work authorized list, regulatory agency personnel, authorized employees, and others authorized by the PM will be allowed to enter the site work areas.

5.2 AHAs

Hazards and controls have been identified for each work task. An AHA is a tool used to identify potential hazards and develop corrective or protective systems to eliminate the hazard. AHAs list operational steps, potential hazards and controls associated with an activity. Hazards may be physical (*e.g.,* lifting hazards, eye hazards) or environmental (*e.g.,* weather or biological [*i.e.,* stinging insects, snakes]). AHAs are reviewed periodically to ensure that the procedures and protective equipment specified for each activity are current and technically correct. Changes in site conditions and/or the scope of work may require a review and modification to the AHA in question. During this review process, comments on the AHA and its procedures will be obtained from personnel associated with the activity being analyzed.

The AHA must be reviewed and signed by persons involved in performing the described task. The task must not be started until personnel understand that task, the hazards and how hazards will be mitigated.

5.3 HAZWOPER

On-site project personnel who work in areas where they may be exposed to site contaminants must be trained as required by OSHA Regulation 29 CFR 1910.120 HAZWOPER. Field employees also must receive a minimum of 3 days of actual field experience under the direct supervision of a trained, experienced supervisor. Personnel who completed their initial training more than 12 months prior starting the project must have completed an 8-hour refresher course within the past 12 months. Specialty services contractors (*e.g.,* surveyor) are not required to have HAZWOPER training; however, they must have a full-time, trained escort. Personnel who act in a supervisory capacity within exclusion zone areas must be trained as HAZWOPER Supervisor in accordance with 29 CFR 1910.120(e).

5.4 Site-Specific Railroad Safety Training and Security Clearance

Based on discussion with Norfolk Southern personnel, Federal Railroad Administration (FRA) training and e-RailSafe verification and training are not required to perform work on site. Should these certifications become necessary at a later date, training will be completed accordingly.

5.5 Site Orientation Training

Site-specific training will be accomplished by onsite personnel reading this SSHP and associated AHAs. The review must include a discussion of the chemical, physical and biological hazards; PPE; safe work procedures; and emergency procedures for the project.

No person will be allowed in the work area during site operations without first receiving a site orientation, safety briefing and/or refresher training. Following this initial site H&S orientation, safety meetings will be held daily as a component of the tailgate meeting. People entering the site work areas, including visitors, must document their attendance at this briefing, as well as any required safety meetings on the forms included with this SSHP. No person will be allowed in the work area unless they are wearing the minimum PPE. A safety meeting must also be held prior to beginning new tasks, and repeated if new hazards are encountered.

5.6 First Aid and Cardiopulmonary Resuscitation (CPR)

At least two site personnel that are current in first aid/CPR will be assigned to the work crew and will be on site during operations, except during the periodic inspections of the automated site operations by the Field Technician/SSHO. The Field Technician/SSHO is anticipated to conduct routine inspections of the plant during automated operations by his/herself. This individual will be CPR/First Aid trained, have a mobile phone with them, and will have scheduled call-in times to the PM or Task Manager.

Refresher training in first aid (triennially) and CPR (annually) are required to keep the certificate current. These individuals must also receive training regarding the precautions and PPE necessary to protect against exposure to blood-borne pathogens.

5.7 Certification Documents

A training and medical file has been established for this project and will be kept with field personnel and in the office during site operations. Specialty training, such as first aid/CPR certificates, as well as current medical clearances for all project field personnel required to wear respirators, will be maintained within that file. The PIKA-PIRNIE JV Team and subcontractor personnel must provide their training and medical documentation to the SHM prior to starting work.

5.8 HazCom

All project required chemicals must be handled in accordance with OSHA 29 CFR 1910.1200, the PIKA-PIRNIE JV Team-HazCom Procedure (ARC HSGE007), and the requirements outlined in the FHSHB. The tasks coved by this SSHP are expected to be completed without the use of hazardous chemicals as defined by the regulation and the PIKA-PIRNIE JV Team procedure. However, there may be many chemicals brought onto the site by other contractors that meet the definition of hazardous. For this reason, the PIKA-PIRNIE JV Team SSHO will request that information be exchanged from the other contractors regarding hazardous chemical present on the site and communicated to the affected PIKA-PIRNIE JV Team employees and subcontractors.

Any MSDSs obtained for informational exchange with affected the PIKA-PIRNIE JV Team employee/subcontractor are to be kept by the SSHO. Affected employees will be notified of any hazardous chemicals being used in their immediate work area or in any area where it is likely they will travel through during the completion of observation tasks before exposure potential and in general at staff meeting/ progress meetings.

5.9 Tailgate Meetings

Tailgate safety meetings must be conducted at least once daily. Each tailgate safety meeting must be documented on the form included in **Appendix C** and maintained with the project files. The tailgate safety meeting will serve as a final review for hazard identification and controls to be utilized. AHAs and the PIKA-PIRNIE JV Team FHSHB controls should be reviewed as part of the meeting to ensure hazard controls are adequate for planned work.

6. PPE Selection

PPE will be selected based on the potential for contact, site conditions, ambient air quality and the judgment of supervising site personnel and H&S professionals. The PPE used will be chosen to be effective against the COCs present on site.

Specifically, the level of PPE selected will be based on the work environment and an assessment by the SSHO of the potential for skin or eye contact with COCs. The PPE selection matrix is presented below.

PPE Selection Matrix

Task	Anticipated Level of Protection
Mobilization and demobilization of equipment	Level D
Site assessment (walking and accessing site locations without intrusive actions)	Level D
Remedial activities (*i.e.,* well installation, water sampling, operation of injection equipment)	Level D (with air monitoring)
Installation of storage tanks, process equipment and electrical service	Non-HAZWOPER contingent upon task not having potential for contact with COCs.

6.1 Using PPE

Depending on the level of protection selected, specific donning and doffing procedures may be required. The procedures presented in this section are mandatory if Modified Level D PPE is used. All personnel entering the exclusion zone (EZ) must use the required PPE in accordance with the requirements of this SSHP. When leaving the EZ, PPE will be removed in accordance with the procedures listed to minimize the spread of COCs.

6.1.1 Donning Procedures

These procedures are mandatory only when Modified Level D or Level C PPE is used on site:

- Remove bulky outerwear.

- Don work clothes or coveralls.
- Don the required chemical-protective coveralls.
- Don the required chemical-protective boots or boot covers.
- Tape the legs of the coveralls to the boots with duct tape.
- Don the required chemical-protective gloves.
- Tape the wrists of the protective coveralls to the gloves.
- Don the required respirator and perform the appropriate fit check (Level C).
- Put hood or head covering over head and respirator straps and tape hood to facepiece (Level C).
- Don remaining PPE, such as safety glasses or goggles and hard hat.
- When these procedures are instituted, one person must remain outside the work area to provide that each person entering has the proper protective equipment.

6.1.2 Doffing Procedures

The following procedures are only mandatory when Modified Level D or Level C PPE is required for the site. Whenever a person leaves the work area, the following decontamination sequence will be followed:

- Upon entering the contaminant reduction zone (CRZ), rinse contaminated materials from the boots or remove contaminated boot covers.
- Clean reusable protective equipment.
- Remove protective garments, equipment and respirator (Level C) and place all disposable clothing in plastic bags, labeled with contaminated waste labels.

- Wash hands, face and neck (or shower if necessary).
- Proceed to clean area and dress in clean clothing.
- Clean and disinfect respirator for next use.
- All disposable equipment, garments and PPE must be bagged in plastic bags and labeled for disposal.

7. Medical Surveillance

7.1 Baseline Medical Examination

All potentially exposed personnel must have completed a comprehensive medical examination prior to assignment and periodically thereafter as defined by applicable regulations including 29 CFR 1910.120(f) and 29 CFR 1910.134.

The examining physician will provide the employee with a letter summarizing findings and recommendations, and confirming the worker's fitness for work and ability to wear a respirator. Documentation of current medical clearance will be available for each employee during project site work.

Subcontractors will certify that all their employees have successfully completed a physical examination by a qualified physician. The physical examinations must meet the requirements of 29 CFR 1910.120 and 29 CFR 1910.134. Subcontractors will supply copies of the medical examination certificate for each on-site employee.

7.2 Other Medical Examinations

In addition to pre-employment, annual and exit physicals, personnel may be examined: at the employee's request after known or suspected exposure to toxic or hazardous materials (HazMat) and at the discretion of the Construction Task Manager or SSHO or occupational physician after known or suspected exposure to, toxic or HazMat.

7.3 Medical Restriction

When the examining physician identifies a need to restrict work activity, the employee's supervisor must communicate the restriction to the employee. The terms of the restriction will be discussed with the employee and the supervisor.

8. Levels of Protection

PPE will be selected based on the potential for contact, site conditions, ambient air quality and the judgment of supervising site personnel and H&S professionals. The PPE used will be chosen to be effective against the COCs present on site.

The level of PPE selected will be based on air monitoring of the work environment and an assessment of the potential for skin contact with the COCs.

8.1 Level D Protection

The minimum level of protection that is required of personnel and subcontractors at the site is Level D, which is worn when activities do not involve potential dermal contact with contaminants and there is no indication that an inhalation hazard exists. Level D protection includes the following equipment:

- work clothing as prescribed by weather
- leather work gloves
- steel-toe work boots (over the ankle), meeting American National Standards Institute (ANSI) Z41
- safety glasses with side shields and goggles, meeting ANSI Z87
- hard hat, meeting ANSI Z89
- safety/reflector vests
- hearing protection (if noise levels exceed 85 decibels [dBA], then hearing protection with a U.S. Environmental Protection Agency (USEPA) noise reduction rating [NRR] of at least 20 dBA must be used)

8.2 Modified Level D Protection

Modified Level D protection will be used when airborne contaminants are not present at levels of concern for inhalation, but site activities present the potential for skin contact with contaminated materials. Modified Level D protection consists of the following equipment:

- nitrile outer gloves when handling potentially contaminated media
- leather work gloves
- latex or polyvinyl chloride (PVC) overboots when contact with COC-impacted media is anticipated
- steel-toe work boots (over the ankle), meeting ANSI Z41
- safety glasses with side shields and goggles, meeting ANSI Z87
- face shield in addition to safety glasses or goggles when projectiles or splash hazards exist
- hard hat, meeting ANSI Z89
- safety/reflector vests
- hearing protection (if noise levels exceed 85 dBA, then hearing protection with a USEPA NRR of at least 20 dBA must be used)

8.3 Level C Protection

Level C protection will be required when the airborne concentration of COCs reaches the Level C action level or if strong odors, action level readings on air monitoring equipment, or related site conditions warrant use of respiratory protection. The following equipment will be used for Level C protection:

- half-face or full-face, air-purifying respirator with high-efficiency particulate air/organic vapor (HEPA/OV) cartridge
- disposable coveralls (*e.g.,* Tyvek suit), with ankles and cuffs taped to boots and gloves
- nitrile outer gloves when handling potentially contaminated media
- leather work gloves

- steel toe work boots, meeting American Society for Testing and Materials (ASTM) International F2412 and F2413; puncture-proof inserts are recommended
- chemical-resistant boots with steel toes or latex/PVC overboots over steel toe boots
- hard hat, meeting ANSI Z89 when falling object hazards are present
- high-visibility vests
- hearing protection (if noise levels exceed 85 dBA, then hearing protection with a USEPA NRR of at least 20 dBA must be used)

9. Respiratory Protection

Respiratory protection is an integral part of employee H&S at the site due to potentially hazardous concentrations of airborne COCs. The site respiratory protection program will consist of the following (as a minimum):

- All on-site personnel who may use respiratory protection will have an assigned respirator.
- All on-site personnel who may use respiratory protection will have been fit tested and trained in the use of a full-face air-purifying respirator within the past 12 months.
- All on-site personnel who may use respiratory protection must, within the past year, have been medically certified as being capable of wearing a respirator. Documentation of the medical certification must be provided to the SHM prior to commencing site work.
- Only cleaned, maintained, NIOSH-approved respirators will be used.
- If respirators are used, the respirator cartridge must be properly disposed of at the end of each work shift, or when load-up or breakthrough occurs.
- All on-site personnel who may use respiratory protection must be clean-shaven. Mustaches and sideburns are permitted, but they must not touch the sealing surface of the respirator.
- Respirators will be inspected and a negative pressure test will be performed prior to each use.
- After each use, the respirator will be wiped with a disinfectant cleansing wipe. When used, the respirator will be thoroughly cleaned at the end of the work shift. The respirator will be stored in a clean plastic bag, away from direct sunlight in a clean, dry location, in a manner that will not distort the face piece.

10. Air Monitoring

Air monitoring will be conducted to evaluate airborne constituent levels. The monitoring results will be used to determine exclusion zone boundaries, work procedures and the selection of PPE. Air monitoring equipment will include a photo ionization detector (PID) with a 10.6 eV lamp or greater, and a Combustible Gas Indicator that measures lower explosive limit (LEL)/oxygen. At a minimum, visual observations will be used to monitor airborne dust. The PIKA-PIRNIE JV Team SSHO will be responsible for using the air monitoring results to determine appropriate H&S precautions for PIKA-PIRNIE JV Team personnel and subcontractors. Monitoring for OVs for the purpose of estimating worker exposure level will be conducted in the breathing zone during field activities and at exclusion boundaries to determine the acceptable zone boundaries (see Appendix J). At a minimum, readings will be recorded at least hourly or more frequently as determined by the SHM. Air monitoring data must be recorded on an air monitoring log or in a field logbook by the SSHO.

The SSHO must record air monitoring data, including the recording of instrument settings, on an air monitoring log or in the field logbook, even when the permit required confined space is monitored by subcontractor personnel.

All direct-reading instrumentation calibrations must be conducted under the approximate environmental conditions the instrument will be used. Instruments must be calibrated before and after use, noting the reading(s) and any adjustments that are necessary. All air monitoring equipment calibrations, including the standard used for calibration, must be documented on a calibration log or in the field logbook. The SHM will review and maintain all completed documentation/forms.

Air monitoring equipment will be maintained, calibrated and repaired in accordance with the specific manufacturers' procedures. When applicable, only manufacturer-trained and/or authorized personnel will be allowed to perform instrument repairs or preventive maintenance.

If an instrument is found to be inoperative or suspected of giving erroneous readings, the SSHO is responsible for removing the instrument from service and obtaining a replacement unit. If the instrument is essential for safe operation during a specific activity, that activity must cease until an appropriate replacement unit is obtained. The PM is responsible for ensuring that a replacement unit is obtained and/or repairs are initiated on the defective equipment.

10.1 Action Levels

The table below presents airborne contaminant action levels that will be used to identify the procedures and protective equipment necessary based on conditions as measured at the site.

Airborne Constituent Action Levels

Exposure Hazard	Monitoring Equipment	Monitoring Frequency	Action Level	Required Action
Total volatile organic compound (VOC)	PID (10.6 eV lamp or greater)	Continuous in breathing zone/ work zone perimeter	0 - 5 ppm	Normal operations. Ensure PID levels at exclusion zone boundary remain at background concentration for PID.
			> 5 ppm, ≤ 10 ppm	Upgrade to Level C PPE.
			≥ 10 ppm ≤ 100 ppm	Stop work and investigate cause of reading. Modify work practices as practical to reduce levels; contact SHM/PM.
			≥ 100 ppm	Cease operations resulting in contaminant release. Modify work area as necessary to prevent or manage VOC emissions. Contact SHM/PM.
Oxygen	Oxygen Sensor, Typically Integrated into Combustible Gas Indicator	Continuous in breathing zone prior to and during excavation entry	< 19.5%	Stop work, evacuate confined spaces/work area, investigate cause of reading and ventilate area.
			> 19.5% to < 23.5%	Normal operations.
			> 23.5%	Stop work, evacuate confined spaces/work area, investigate cause of reading and ventilate area.
Flammable vapors (LEL)	Combustible Gas Indicator	Continuous in breathing zone prior to and during excavation entry	< 10% LEL	Normal operations.
			> 10% LEL	Stop work, ventilate area, investigate source of vapors.

11. General Safety Practices

General safety rules for site activities include, but are not limited to, the following:

- At least one copy of this SSHP must be kept in a location on site that is readily available to personnel, and all project personnel will review the SSHP prior to starting work.
- Wear PPE as required, and stop work and replace damaged PPE immediately.
- Upon skin contact with materials that may be impacted by COCs, remove contaminated clothing and wash the affected area immediately. Contaminated clothing must be changed. Skin contact with materials potentially impacted by COCs must be reported to the PM or the SSHO immediately. If needed, seek medical attention.
- Food, drink and smoking are permitted in designated areas. Food, drink and smoking are not permitted in the EZ or CRZ.
- Practice contamination avoidance. Avoid contact with surfaces either suspected or known to be impacted by COCs, such as mud or discolored soil.
- Remove PPE as required before leaving the work area to limit the spread of COC-containing materials.
- Dispose of all soiled gloves in designated receptacles designated for this purpose.
- Report all injuries, illnesses, near misses and unsafe conditions or work practices to the SHM.

11.1 Buddy System

On-site personnel involved in construction phase activities or any operations with active handling of hazardous waste must use the buddy system. Use of the buddy system is required during operations. Additionally, the buddy system must be used during work along roadways or any other area where vehicles may pose a risk to field

personnel. Visual contact must be maintained between crew members at all times, and crew members must observe each other for signs of chemical exposure, and heat or cold stress. Indications of adverse effects include, but are not limited to:

- changes in complexion and skin coloration
- changes in coordination
- changes in demeanor
- excessive salivation and pupillary response
- changes in speech pattern

Team members must also be aware of potential exposure to possible safety hazards, unsafe acts or noncompliance with safety procedures. Employees must inform their fellow team members of potential effects of exposure to toxic materials. The symptoms of such exposure may include:

- headaches
- dizziness
- nausea
- blurred vision
- cramps
- irritation of eyes, skin or respiratory tract

If protective equipment or noise levels impair communications, pre-arranged hand signals must be used for communication. Personnel must stay within line of sight of another team member.

During the automated operational phase of the treatment system the Field Technician/SSHO may complete inspection related activities while on site alone. This

individual with have a mobile phone with them and designated call-in times to the Task Manager to confirm arrival and departure from the site.

11.2 Site Security

If work is to be conducted during hours of darkness, adequate lighting will be utilized in accordance with **Appendix F** – Night Operations Plan. Upon arrival, vehicles will be parked inside the site and the parking area will serve as the muster point in the event of site emergencies.

11.3 Heat Stress

Heat stress monitoring will be performed in accordance with PIKA\PIRNIE H&S Procedure ARC HSIH013. Work/rest cycles will be used as necessary to minimize exposure to the elements and overheating, resulting in any of the illnesses described above. Heat stress monitoring will be performed by both individuals and the SHM. Monitoring may include, but is not limited to, checking individual heart rates, body temperature, sweating, rashes, change in disposition *(i.e.,* giddiness), nausea and vomiting. Additionally, one or more of the following control measures may be used to help control heat stress and are mandatory if any site worker is suspected of exhibiting any of the early symptoms of heat stress:

- Site workers will be encouraged to drink plenty of water and electrolyte replacement fluids throughout the day.
- On-site drinking water will be kept cool (50 to 60 degrees Fahrenheit [°F]).
- A work regimen that will provide adequate rest periods for cooling down will be established, as required.
- All personnel will be advised of the dangers and symptoms of heat stroke, heat exhaustion and heat cramps.
- Employees will be instructed to monitor themselves and coworkers for signs of heat stress and to take additional breaks as necessary.
- Frequent breaks can be taken in either a shaded rest area or in an air-conditioned vehicle.

- Employees must not be assigned to other tasks during breaks.

- Employees must remove impermeable garments during rest periods. This includes white Tyvek®-type garments.

All employees must be informed of the importance of adequate rest, acclimation and proper diet in the prevention of heat stress disorders.

11.3.1 Heat Rashes

Heat rashes are one of the most common problems in hot work environments. Commonly known as prickly heat, a heat rash is manifested as red papules and usually appears in areas where the clothing is restrictive. As sweating increases, these papules give rise to a prickling sensation. Prickly heat occurs in skin that is persistently wetted by un-evaporated sweat, and heat rash papules may become infected if they are not treated. In most cases, heat rashes will disappear when the affected individual returns to a cool environment.

11.3.2 Heat Cramps

Heat cramps are usually caused by performing hard physical labor in a hot environment. These cramps have been attributed to an electrolyte imbalance caused by sweating. It is important to understand that cramps can be caused both by too much or too little salt.

Cramps appear to be related to a lack of water replenishment. Because sweat is a hypotonic solution (plus or minus 0.3 percent sodium chloride), excess salt can build up in the body if the water lost through sweating is not replaced. Thirst cannot be relied on as a guide to the need for water; instead, water must be taken every 15 to 20 minutes in hot environments.

Under extreme conditions, such as working for 6 to 8 hours in heavy protective gear, a loss of sodium may occur. Drinking commercially available carbohydrate electrolyte replacement liquids is effective in minimizing physiological disturbances during recovery.

11.3.3 Heat Exhaustion

Heat exhaustion occurs from increased stress on various body organs due to inadequate blood circulation, cardiovascular insufficiency or dehydration. Signs and symptoms include:

- pale, cool, moist skin
- heavy sweating
- dizziness
- nausea
- headache
- vertigo
- weakness
- thirst
- giddiness

Fortunately, this condition responds readily to prompt treatment. However, heat exhaustion must not be dismissed lightly, for several reasons. The fainting associated with heat exhaustion can be dangerous because the victim may be operating machinery or controlling an operation that must not be left unattended; moreover, the victim may be injured when he or she faints. Also, the signs and symptoms seen in heat exhaustion are similar to those of heat stroke, which is a medical emergency.

Workers suffering from heat exhaustion must be removed from the hot environment, given fluid replacement and be encouraged to get adequate rest.

11.3.4 Heat Stroke

Heat stroke is the most serious form of heat stress. Heat stroke occurs when the body's system of temperature regulation fails and the body's temperature rises to

critical levels. This condition is caused by a combination of highly variable factors and its occurrence is difficult to predict.

Heat stroke is a medical emergency. Primary signs and symptoms of heat stroke are:

- confusion
- irrational behavior
- loss of consciousness
- convulsions
- a lack of sweating (usually)
- hot, dry skin
- an abnormally high body temperature (*e.g.,* a rectal temperature of 41degrees Celsius (°C) [105.8°F])

If body temperature is too high, it causes death. The elevated metabolic temperatures caused by a combination of workload and environmental heat load, both of which contribute to heat stroke, are also highly variable and difficult to predict.

If a worker shows signs of possible heat stroke, professional medical treatment must be obtained immediately. The worker will be placed in a shady area and the outer clothing must be removed. The worker's skin must be wetted and air movement around the worker must be increased to improve evaporative cooling until professional methods of cooling are initiated and the seriousness of the condition can be assessed. Fluids must be replaced as soon as possible. The medical outcome of an episode of heat stroke depends on the victim's physical fitness and the timing and effectiveness of first-aid treatment.

Regardless of the worker's protestations, no employee suspected of being ill from heat stroke will be sent home or left unattended unless a physician has specifically approved such an order.

Proper training and preventive measures will help avert serious illness and loss of work productivity. Preventing heat stress is particularly important because once someone

suffers from heat stroke or exhaustion, that person may be predisposed to additional heat injuries.

11.3.5 Heat Stress Monitoring

Heat stress monitoring and work/rest cycle implementation will commence when the ambient adjusted temperature exceeds 72°F. Screening criteria for heat stress exposure and examples of activities within metabolic rate categories are provided in the tables below. Cooling vests are available on site and will be used by PIKA\PIRNIE personnel during operations requiring Level C PPE to maintain body core temperature at safe levels.

Screening Criteria for Heat Stress Exposure for 8-Hour Work Day, 5 Days Per Week with Conventional Breaks

	Acclimatized				Unacclimatized			
Work Demands	**Light**	**Moderate**	**Heavy**	**Very Heavy**	**Light**	**Moderate**	**Heavy**	**Very Heavy**
100% work	85.1ºF (29.5ºC)	81.5ºF (27.5ºC)	78.8ºF (26ºC)		81.5ºF (27.5ºC)	77ºF (25ºC)	72.5F (22.5ºC)	
75% work; 25% rest	86.9ºF (30.5ºC)	83.3ºF (28.5ºC)	81.5ºF (27.5ºC)		84.2ºF (29ºC)	79.7ºF (26.5ºC)	76.1ºF (24.5ºC)	
50% work; 50% rest	88.7ºF (31.5ºC)	85.1ºF (29.5ºC)	83.3ºF (28.5ºC)	81.5ºF (27.5ºC)	86ºF (30ºC)	82.4ºF (28ºC)	79.7ºF (26.5ºC)	77ºF (25ºC)
25% work, 75% rest	90.5ºF (32.5ºC)	87.8ºF (31ºC)	86ºF (30ºC)	85.1ºF (29.5ºC)	87.8ºF (31ºC)	84.2ºF (29ºC)	82.4ºF (28ºC)	79.7ºF (26.5ºC)

Source: 2004 TLVs and Biological Exposure Indices (BEIs) – TLVs for Chemical Substances and Physical Agents and BEIs (ACGIH 2004).

Examples of Activities within Metabolic Rate Categories

Categories	Example Activities
Resting	Sitting quietly
	Sitting with moderate arm movements
Light	Sitting with moderate arm and leg movements
	Standing with light work at machine or bench while using mostly arms
	Using a table saw
	Standing with light or moderate work at machine or bench and some walking about
Moderate	Scrubbing in a standing position
	Walking about with moderate lifting or pushing
	Walking on a level at 6 kilometers per hour while carrying 3 kilogram weight load
Heavy	Carpenter sawing by hand
	Shoveling dry sand
	Heavy assembly work on a non-continuous basis
	Intermittent heavy lifting with pushing or pulling (*e.g.,* pick-and-shovel work)
Very heavy	Shoveling wet sand

Source: 2004 TLVs and BEIs – Threshold Limit Values for Chemical Substances and Physical Agents and Biological Exposure Indices (ACGIH 2004).

11.4 Cold Stress

Cold stress normally occurs in temperatures at or below freezing, or under certain circumstances, in temperatures of 40°F. Extreme cold for a short time may cause severe injury to exposed body surfaces or result in profound generalized cooling, causing death. Areas of the body that have high surface area-to-volume ratio, such as fingers, toes and ears, are the most susceptible. Two factors influence the development of a cold weather injury: ambient temperature and wind velocity. For example, 10°F with a 15 mile per hour (mph) wind is equivalent in chilling effect to still air at 18°F. An equivalent chill temperature chart relating the actual dry bulb temperature and wind velocity is presented in the table below.

Chill Temperature Chart

Estimated Wind Speed (mph)	Actual Temperature Reading (°F)											
	50	**40**	**30**	**20**	**10**	**0**	**-10**	**-20**	**-30**	**-40**	**-50**	**-60**
	Equivalent Chill Temperature (°F)											
Calm	50	40	30	20	10	0	-10	-20	-30	-40	-50	-60
5	48	37	27	16	6	-5	-15	-26	-36	-47	-57	-68
10	40	28	16	4	-9	-24	-33	-46	-58	-70	-83	-95
15	36	22	9	-5	-18	-32	-45	-58	-72	-85	-99	-112
20	32	18	4	-10	-25	-39	-53	-67	-82	-96	-110	-121
25	30	16	0	-15	-29	-44	-59	-74	-88	-104	-118	-133
30	28	13	-2	-18	-33	-48	-63	-79	-94	-109	-125	-140
35	27	11	-4	-20	-35	-51	-67	-82	-98	-113	-129	-145
40	26	10	-6	-21	-37	-53	-69	-85	-100	-116	-132	-148
(Wind speeds greater than 40 mph have little additional effect.)	**LITTLE DANGER** **Maximum danger of false sense of security.**				**INCREASING DANGER** **Danger from freezing of exposed flesh within 1 minute.**			**GREAT DANGER** **Flesh may freeze within 30 seconds.**				
	Trench foot and immersion foot may occur at any point on this chart.											

This chart was developed by the U.S. Army Research Institute of Environmental Medicine, Natick, MA (Source: ACGIH 2004).

Local injury resulting from cold is included in the generic term “frostbite.” Several degrees of tissue damage are associated with frostbite. Frostbite of the extremities can be categorized into:

- *Frost Nip or Incipient Frostbite*. Characterized by sudden blanching or whitening of skin.

- *Superficial Frostbite*. Skin has a waxy or white appearance and is firm to the touch, but tissue beneath is resilient.

- *Deep Frostbite*. Skin tissue is cold, pale and solid; extremely serious injury.

Systemic hypothermia is caused by exposure to freezing or rapidly dropping temperature. It can be fatal. Its symptoms are usually exhibited in five stages:

1. shivering

2. apathy, listlessness, sleepiness and (sometimes) rapid cooling of the body to less than 95°F

3. unconsciousness, glassy stare, slow pulse, and slow respiratory rate

4. freezing of the extremities

5. death

Trauma sustained in freezing or sub-zero conditions requires special attention because an injured worker is predisposed to secondary cold injury. Special provisions must be made to prevent hypothermia and secondary freezing of damaged tissues in addition to providing for first-aid treatment. To avoid cold stress, site personnel must wear protective clothing appropriate for the level of cold and physical activity. In addition to protective clothing, preventive safe work practices, additional training and warming regimens may be used to prevent cold stress.

11.4.1 Safety Precautions for Cold Stress Prevention

For air temperature of 0°F or less, mittens will be used to protect the hands. For exposed skin, continuous exposure will not be permitted when air speed and temperature results in a wind chill temperature of -25°F.

At air temperatures of 36°F or less, field personnel who become immersed in water or whose clothing becomes wet must be immediately provided with a change of clothing and be treated for hypothermia.

If work is done at normal temperature or in a hot environment before entering the cold, the field personnel must ensure that their clothing is not wet as a consequence of sweating. If wet, field personnel must change into dry clothes prior to entering the cold area.

If the available clothing does not give adequate protection to prevent hypothermia or frostbite, work must be modified or suspended until adequate clothing is made available or until weather conditions improve.

Field personnel handling evaporative liquid (*e.g.,* gasoline, alcohol or cleaning fluids) at air temperatures below 40°F must take special precaution to avoid soaking of clothing or gloves with the liquids because of the added danger of cold injury due to evaporative cooling.

11.4.2 Safe Work Practices for Cold Work Areas

Direct contact between bare skin and cold surfaces (<20°F) must be avoided. Metal tool handles and/or equipment controls will be covered by thermal insulating material.

For work performed in a wind chill temperature at or below 10°F, workers must be under constant protective observation (buddy system). The work rate will be established to prevent heavy sweating that will result in wet clothing. For heavy work, rest periods must be taken in heated shelters and workers must be provided with an opportunity to change into dry clothing if needed.

Field personnel must be provided the opportunity to become accustomed to cold-weather working conditions and required protective clothing. Work will be arranged to minimize sitting or standing still for long periods.

During the warming regimen (rest period), field personnel will be encouraged to remove outer clothing to permit sweat evaporation or to change into dry work clothing. Dehydration, or loss of body fluids, occurs insidiously in the cold environment and may increase susceptibility to cold injury due to a significant change in blood flow to the extremities. Fluid replacement with warm, sweet drinks and soups is recommended. The intake of coffee will be limited because of diuretic and circulatory effects.

11.5 Biological Hazards

Biological hazards may include black bears, snakes, thorny bushes and trees, poison ivy, ticks, mosquitoes, chiggers, fire ants, and other pests.

11.5.1 Snakes

The possibility of encountering snakes exists, specifically for personnel working in wooded/vegetated areas. Snake venoms are complex and include proteins, some of which have enzymatic activity. The effects produced by venoms include neurotoxic effects with sensory, motor, cardiac and respiratory difficulties; cytotoxic effects on red blood cells, blood vessels, heart muscle, kidneys and lungs; defects in coagulation; and effects from local release of substances by enzymatic actions. Other noticeable effects of venomous snake bites include swelling, edema and pain around the bite, and the development of ecchymosis (the escape of blood into tissues from ruptured blood vessels).

Control. To minimize the threat of snake bites, all personnel walking through vegetated areas must be aware of the potential for encountering snakes and the need to avoid actions potentiating encounters (*e.g.,* turning over logs). If a snake bite occurs, an attempt will be made to safely identify the snake via size and markings. The victim must be transported to the designated hospital within 30 minutes; first aid consists of applying a constriction band and washing the area around the wound to remove any unabsorbed venom.

11.5.2 Mosquitoes

Personnel may be exposed to mosquitoes during work activities. Typical exposure to mosquitoes does not present a significant hazard. However, if West Nile virus is prevalent in the area, exposure to this virus is increased. West Nile virus results in flu-like symptoms and can be serious if not treated, or in immune-compromised individuals.

Control. To minimize the threat of mosquito bites, all personnel working outside must be aware of the potential for encountering mosquitoes and implement the basic precautions listed below:

- Avoid working at dawn or dusk, when mosquitoes are most active.

- Prevent accumulation of standing water at the work site.
- Apply an insect repellent that contains N,N-diethyl-meta-toluamide (DEET) to exposed skin.
- Wear light-colored clothes, preferably with long sleeves and full-length pants.
- Do not touch any dead birds or animals that you encounter.

If dead birds are detected near the site, report this to the local County Health Department. If flu-like symptoms are present in onsite personnel, contact your doctor or the HSM for more information.

11.5.3 Spiders

The possibility of personnel encountering spiders exists during work activities. Two spiders are of concern: the black widow and the brown recluse. Both prefer dark sheltered areas such as basements, equipment sheds and enclosures, and around woodpiles or other scattered debris.

The black widow is shiny black, approximately 1 inch long and found throughout the United States. There is a distinctive red hourglass marking on the underside of the black widow's body. The bite of a black widow is seldom fatal to healthy adults, but effects include respiratory distress, nausea, vomiting and muscle spasms.

The brown recluse is smaller than the black widow and gets its name from its brown coloring and behavior. The brown recluse is more prevalent in the Southern United States. The brown recluse has a distinctive violin shape on the top of its body. The bite of the brown recluse is painful and the bite site ulcerates and takes many weeks to heal completely.

The following photographs identify brown recluse and black widow spiders:

Brown recluse spider

Female black widow spider

Control. To minimize the threat of spider bites, all personnel walking through vegetated areas must be aware of the potential for encountering these arachnids. Personnel need to avoid actions that may result in encounters such as turning over logs and placing hands in dark places (*e.g.,* behind equipment or in corners of equipment sheds or enclosures). To prevent the possibility of a spider bite, all enclosures/equipment will be visually inspected prior to opening. Leather or equivalent work gloves will be worn at all times when working in and around enclosures/ equipment where spiders could be encountered. Any items removed from an enclosure will be carefully inspected and/or shaken out before use (*e.g.,* donning of a vest, use of a manual). If a preponderance of spiders is noted in a particular location (*e.g.,* control panels, sheds), an exterminator will be contacted to provide removal services.

If a spider bite occurs, the victim must be transported to the designated hospital as soon as possible; field first aid consists of washing the area around the wound to remove any unabsorbed venom and applying ice packs. Acetaminophen may be given for pain, and the affected limb may be elevated to reduce swelling.

11.5.4 Tick-Borne Diseases

During work activities, personnel may be exposed to ticks and the tick-borne diseases described below.

Lyme Disease. The disease commonly occurs in summer and is transmitted by the bite of infected ticks. "Hot spots" in the United States include New York, New Jersey, Pennsylvania, Massachusetts, Connecticut, Rhode Island, Minnesota and Wisconsin.

Erlichiosis. The disease commonly occurs in summer and is transmitted by the bite of infected ticks. "Hot spots" in the United States include New York, Massachusetts, Connecticut, Rhode Island, Minnesota and Wisconsin.

These diseases are transmitted primarily by the deer tick, which is smaller and redder than the common wood tick. The disease may be transmitted by immature ticks, which are small and hard to see. The tick may be as small as a period on this page.

Symptoms of Lyme disease include a rash or a peculiar red spot, like a bull's eye, which expands outward in a circular manner. The victim may have headache, weakness, fever, a stiff neck, and swelling and pain in the joints, and eventually,

arthritis. Symptoms of erlichiosis include muscle and joint aches, flu-like symptoms, but there is typically no skin rash.

Rocky Mountain Spotted Fever (RMSF). This disease is transmitted via the bite of an infected tick. The tick must be attached for 4 to 6 hours before the disease-causing organism (Rickettsia rickettsii) becomes reactivated and can infect humans. The primary symptom of RMSF is the sudden appearance of a moderate-to-high fever. The fever may persist for 2 to 3 weeks. The victim may also have a headache, deep muscle pain and chills. A rash appears on the hands and feet on about the third day and eventually spreads to all parts of the body. For this reason, RMSF may be confused with measles or meningitis. The disease may cause death, if untreated, but if identified and treated promptly, death is uncommon.

Control. Tick repellant containing DEET must be used when working in tick-infested areas and pant legs must be tucked into boots. Avoid walking in heavily vegetated areas; stick to trails if they are present. If the work area is known to be heavily infested with ticks, employees will wear a white Tyvek® liner with taped ankles and wrists to keep ticks out and ease in identification. In addition, workers, using the buddy system, must search the entire body every 3 to 4 hours for attached ticks. Workers will check themselves with a mirror upon arriving home for the evening. Ticks removed within 24 hours are less likely to transmit disease if they are carriers. Ticks must be removed promptly and carefully without crushing, because crushing can squeeze the disease-causing organism into the skin. A gentle and steady pulling action must be used to avoid leaving the head or mouth parts in the skin. Hands must be protected with surgical gloves when removing ticks. Deer ticks removed from the skin will be placed in a plastic bag and brought to the HSM, who may send the recovered tick for disease testing.

11.5.5 Bees

Personnel may be exposed to bees and other stinging insects during work activities. Bee stings are painful and can be very serious when individuals with an allergy are stung. Employees that have an allergy and have a prescribed Epinephrine Auto Injector (EpiPen) must let coworkers know where it is in the event they are stung. You will only assist in administering the EpiPen if you are trained to do so or given permission by the injured person. If an individual has received an EpiPen injection, he/she must still be transported to the hospital regardless if allergic reaction symptoms began to fade.

To prevent bee stings there are several steps to take:

- Wear dull-colored clothing when outdoors. Brightly colored clothing attracts certain bees, wasps, ants and other insects. *(Remember flowers are brightly colored and many bees and wasps feed on the nectar and pollen of flowers.)* Wear orange vests and white hardhats (not yellow).

- Look around the area to see if there are any large groups of bees.

- Avoid wearing perfumes or colognes because they attract bees; use unscented sunblock and insect repellant for field work.

- If you see a bee, do not run away or make sudden movements, which tend to scare the bees.

- If a bee is flying around you, do not swat at it. Let it fly around until it decides to leave.

- DO NOT KILL A BEE UNLESS IT IS ABSOLUTELY NECESSARY. Bees emit an odor when they are killed that tells other bees there is danger. When other bees smell it, they may attack and sting in large groups.

- If you see a large group of bees, leave them alone and try to complete your task in another area. If this is not feasible, you may have to postpone work until the bees can be properly removed.

- Wear long sleeves and long-legged pants outdoors where practical. Bees are attracted to sweaty skin.

- Avoid disturbing likely beehive sites, such as large trees, tree stumps, logs and large rocks.

- If a colony is disturbed, run and find cover as soon as possible. Running in a zigzag pattern may be helpful. Cover as much of the head and face as possible, without obscuring vision, while running. Once clear of the bees, remove stingers and seek medical care if necessary, especially if there is a history of allergy to bee venom.

- Do not try and hide in water such as ponds, swimming pools or creeks. Bees know that you are in there and will wait for you to exit the water.

If an employee has been stung by a bee, wasp, hornet or yellow jacket, follow these instructions closely:

- Bees leave behind a stinger attached to a venom sac. Do not try to pull out the stinger because this may release more venom; instead gently scrape it out with a blunt-edged object, such as a credit card or dull knife.
- Wash the area carefully with soap and water. This must be continued several times a day until the skin is healed.
- Apply a cold or ice pack, wrapped in cloth, for a few minutes.
- Apply a paste of baking soda and water and leave it on for 15 to 20 minutes.
- Telephone 911 to summon paramedics if the victim is having an allergic reaction, and use a bee sting emergency survival kit if previously prescribed.
- Treat swelling by elevating the swollen body part above the heart.
- Do not squeeze the sting or rub mud into it. This increases the risk of infection.
- Do not administer drugs that are not prescribed for the patient.

Seek immediate medical attention if you are stung in the mouth or nose, because swelling may block airways. Also seek emergency care if any of the following symptoms are present, which could indicate an allergic reaction:

- large areas of swelling
- abnormal breathing
- tightness in throat or chest
- dizziness

- hives
- fainting
- nausea or vomiting
- persistent pain or swelling

Every tailgate safety meeting must include a discussion of which field personnel are allergic to bees, bug bites or other insects.

11.5.6 Poisonous Plants

Poisonous plants may be present in the work area. Personnel will be alerted to their presence and instructed on methods to prevent exposure.

Poison sumac grows as a shrub or small tree with large alternate, compound leaves having 7 to 13 leaflets without teeth. All plant parts are poisonous. The lack of leaflet glands, "wings" between the leaflets and teeth on the leaves, in addition to this species' red stems supporting the leaflets and leaves, help to distinguish this plant from similar-looking nonpoisonous species such as other sumacs and tree-of-heaven. Flowers are shades of green, white and yellow and appear in late spring. Fruits are small white berries that mature in late summer and may last through winter. Poison sumac is occasionally found in moist or wet soils. The following photographs are for identification of poison sumac.

Unripe Berries of Poison Sumac


Compound Leaf with Red Petiole

Poison ivy is a woody shrub or vine with hairy looking aerial roots. It grows to 10 feet or more in height, climbing high on trees, walls and fences, or trails along the ground. All parts of poison ivy, including the roots, are poisonous at all times of the year. The following photographs are for identification of poison ivy:

Compound Leaf with Three Leaflets

White Berries of Poison Ivy

The main control for both poison ivy and poison sumac is to avoid contact with the plant, cover arms and hands, and frequently wash potentially exposed skin. Particular attention must be given to avoiding skin contact with objects or protective clothing that have touched the plants. Treat every surface that may have touched the plant as contaminated, and practice contamination avoidance.

Poison ivy and sumac are easy to treat if you identified your contact with the irritating plant within a few hours of the incident. Rash symptoms can appear within a few hours, but can take 2 to 5 days to appear. The rash starts as a red, annoyingly itchy area that starts to swell. The area then gets inflamed and will get covered in clusters of tiny pimples; the pimples eventually merge and turn into blisters. The fluid in the blisters turns yellow, dries up and becomes crusty. Left completely untreated, this cycle can last as short as 5 days and in severe cases as long as 5 to 6 weeks.

If an employee comes in contact with poison ivy, oak or sumac, or an animal exposed to any of these, or tools, gear or clothing exposed to any of these, they must wash with hot water (not so hot that it burns) and strong soap as soon as possible. Washing within the first 6 hours before the first symptoms appear, will help to avoid or may minimize an outbreak.

11.6 Noise

Exposure to noise over the OSHA action level can cause temporary impairment of hearing; prolonged and repeated exposure can cause permanent damage to hearing. The risk and severity of hearing loss increases with the intensity and duration of exposure to noise. In addition to damaging hearing, noise can impair voice communication, thereby increasing the risk of accidents on site.

Control. All personnel must wear hearing protection, with an NRR of at least 20, when noise levels exceed 85 dBA. When it is difficult to hear a coworker at normal conversation distance, the noise level is approaching or exceeding 85 dBA, and hearing protection is necessary. All site personnel who may be exposed to noise must also receive baseline and annual audiograms and training as to the causes and prevention of hearing loss. Noise monitoring is discussed in Section 6.3.

Whenever possible, equipment that does not generate excessive noise levels will be selected for this project. If the use of noisy equipment is unavoidable, barriers or increased distance will be used to minimize worker exposure to noise, if feasible.

11.7 Spill Control

All personnel must take every precaution to minimize the potential for spills during site operations. All on-site personnel will immediately report any discharge, no matter how small, to the Construction Task Manager or SSHO.

Spill control equipment and materials will be located on site at locations that present the potential for discharge. All sorbent materials used to clean up spills will be containerized and labeled appropriately.

11.8 Sanitation

Site sanitation will be maintained according to OSHA requirements.

11.8.1 Break Area

Breaks must be taken in the Support Zone, away from the active work area after site personnel go through decontamination procedures. There will be no smoking, eating, drinking or chewing gum or tobacco in any area other than the Support Zone.

11.8.2 Potable Water

The following rules apply to the use of potable water during field operations:

- An adequate supply of potable water will be provided at each project site. Potable water must be kept away from HazMat or media, and contaminated clothing or equipment.

- Portable containers used to dispense drinking water must be capable of being tightly closed and must be equipped with a tap dispenser. Water must not be consumed directly from the container (drinking from the tap is prohibited) and may not be removed from the container by dipping.

- Containers used for drinking water must be clearly marked and will not be used for any other purpose.

- Disposable drinking cups must be provided. A sanitary container for dispensing cups and a receptacle for disposing of used cups is required.

11.8.3 Sanitary Facilities

Access to facilities for washing before eating, drinking or smoking, or alternate methods such as waterless hand-cleaner and paper towels, will be provided.

11.9 Traffic Safety

To minimize the likelihood of project personnel and activities being affected by traffic, the following traffic safety procedures will be implemented. When working adjacent to roadways, cones will be placed along the perimeter of the work area to alert drivers to the presence of personnel and equipment. All crewmembers will remain behind the equipment and the traffic barrier. All site personnel will wear an outer layer of orange or safety yellow warning garments, such as vests, jackets or shirts meeting ANSI Class II or Class III requirements. Traffic Control requirements from Norfolk Southern Railroad will be complied with in conjunction with these traffic safety procedures.

11.10 Lifting Safety

Using proper lifting techniques may prevent back strain or injury. The fundamentals of proper lifting include:

- Consider the size, shape and weight of the object to be lifted.
- The hands and the object will be free of dirt or grease that could prevent a firm grip.
- Fingers must be kept away from points that could crush or pinch them, especially when putting an object down.
- The load must be kept as low as possible, close to the body with the knees bent.
- To lift the load, grip firmly and lift with the legs, keeping the back as straight as possible.
- When putting an object down, the stance and position are identical to that for lifting: legs are bent at the knees and the back is straight as the object is lowered.
- Two-person lifts are required to lift objects greater than 50 pounds.

11.11 Emergency Equipment

Adequate emergency equipment will be available for the activities being conducted on site and as required by applicable sections of 29 CFR 1910. Personnel will be provided with access to emergency equipment, including, but not limited to, the following:

- fire extinguishers of at least 5 pounds, of the class, number and location as required by applicable sections of 29 CFR 1910
- industrial first aid kits of adequate size for the number of personnel on site
- emergency eyeflush bottle, at a minimum, and eyewash and/or shower if required by operations being conducted on site

11.12 Lockout/Tagout Procedures

Only fully qualified and trained personnel will perform maintenance procedures. Before maintenance begins, lockout/ tagout procedures per ARC HSFS004 and OSHA 29 CFR 1910.147 will be followed.

Lockout is the placement of a device that uses a positive means, such as lock, to hold an energy- or material-isolating device such that the equipment cannot be operated until the lockout device is removed. If a device cannot be locked out, a tagout system will be used. Tagout is the placement of a warning tag on an energy- or material-isolating device indicating that the equipment controls may not be operated until the tag is removed by the personnel who attached the tag.

11.13 Electrical Safety

Electricity may pose a particular hazard to site workers due to the installation and use of permanent and portable electrical equipment. If wiring or other electrical work is needed, a qualified electrician must perform the work. Electrical safety will comply with PIKA\PIRNIE H&S Standard HSFS006. Requirements include:

- Portable and semiportable tools and equipment must be grounded by a multiconductor cord having an identified grounding conductor and a multicontact polarized plug-in receptacle.
- Electric wire or flexible cord passing through work areas must be protected from foot traffic, vehicles, sharp corners, projections or pinching.
- All circuits must be protected from overload.
- Plugs and receptacles must be kept out of water unless of an approved submersible construction.
- All extension cord outlets must be equipped with ground fault circuit interrupters.
- Attachment plugs or other connectors must be equipped with a cord grip and be constructed to endure rough treatment.

- Extension cords or cables must be inspected prior to each use and replaced if worn or damaged. Cords and cables must not be fastened with staples, hung from nails or suspended by bare wire.

- Flexible cords must be used only in continuous lengths without splice, with the exception of molded or vulcanized splices made by a qualified electrician.

11.14 Confined Space Entry

The site confined space program will include surveys for confined spaces, designation as permit-required or non-permit required confined space, and labeling or signage at confined space entrances in accordance with PIKA/PIRNIE H&S Standard HSFS003, Confined Space Entry. Permit-required confined space entries are not anticipated for this project. However, in the event personnel plan to enter a permit-required confined space than a site-specific program will be implement that includes requirements for current confined space entry training with documentation of training maintained on site. Pre-entry air monitoring will be performed prior to entering the space and continuous air monitoring will be performed for the duration of the entry.

11.15 Ladder Safety

Use of ladders on-site will comply with PIKA\PIRNIE H&S Standard HSFS021, Ladder Safety. When portable ladders are used to access an upper landing surface, the ladder side rails must extend at least 3 feet above the upper landing surface to which the ladder is used to gain access; or, when such an extension is not possible because of the ladder's length, the ladder must be secured at its top to a rigid support that will not deflect, and a grasping device (such as a grab rail) will be provided to assist employees in mounting and dismounting the ladder. In no case will the extension be such that ladder deflection under a load would, by itself, cause the ladder to slip off its support. Additional ladder safety procedures are described below:

- Ladders will be maintained free of oil, grease and other slipping hazards.

- Ladders will not be loaded beyond the maximum intended load for which they were built, or beyond their manufacturer's rated capacity.

- Ladders will be used only for the purpose for which they were designed.

- Non-self-supporting ladders will be used at an angle such that the horizontal distance from the top support to the foot of the ladder is approximately ¼ of the working length of the ladder (the distance along the ladder between the foot and the top support).

- Wood job-made ladders with spliced side rails will be used at an angle such that the horizontal distance is ⅛ the working length of the ladder.

- Fixed ladders will be used at a pitch no greater than 90 degrees from the horizontal, as measured to the back side of the ladder.

- Ladders will be used only on stable and level surfaces unless secured to prevent accidental displacement.

- Ladders will not be used on slippery surfaces unless secured or provided with slip-resistant feet to prevent accidental displacement. Slip-resistant feet will not be used as a substitute for care in placing, lashing or holding a ladder that is used upon slippery surfaces, including, but not limited to, flat metal or concrete surfaces that are constructed so they cannot be prevented from becoming slippery.

- Ladders placed in any location where they can be displaced by workplace activities or traffic, such as in passageways, doorways or driveways, will be secured to prevent accidental displacement, or a barricade will be used to keep the activities or traffic away from the ladder.

- The area around the top and bottom of ladders will be kept clear.

- The top of a non-self-supporting ladder will be placed with the two rails supported equally unless it is equipped with a single support attachment.

- Ladders will not be moved, shifted or extended while occupied.

- Ladders will have nonconductive side rails if they are used where the employee or the ladder could contact exposed energized electrical equipment.

- The top, top step will not be used as a step, and the step labeled that it or any step above it must not be used as a step.

- Cross-bracing on the rear section of stepladders will not be used for climbing, unless the ladders are designed and provided with steps for climbing on both front and rear sections.

- Ladders will be inspected by the HSM for visible defects daily and after any occurrence that could affect their safe use.

- Portable ladders with structural defects, such as, but not limited to, broken or missing rungs, cleats or steps; broken or split rails; corroded components; or other faulty or defective components will either be immediately marked in a manner that readily identifies them as defective, or tagged with "Do Not Use" or similar language, and will be withdrawn from service.

- When ascending or descending a ladder, the user will face the ladder.

- Each employee will use at least one hand to grasp the ladder when progressing up and/or down the ladder to maintain three points of contact.

- Employees will not climb any ladder while carrying items with their hands.

11.16 Lightning

Outdoors is the most dangerous place to be during a lightning storm. When lightning is seen or thunder is heard, or when dark clouds are observed, quickly move indoors or into a hard-topped vehicle and remain there until 30 minutes after the last visible lightning or audible thunder. Listen to forecasts and warnings through National Oceanic and Atmospheric Administration (NOAA) Weather Radio or your TV and radio stations. If lightning is forecast, plan an alternate work activity or know where you can take cover quickly. Check on client/site-specific procedures regarding lightning prior to starting work.

11.16.1 Hazards

Burns, nervous system damage, broken bones, loss of hearing or eyesight, electrocution and/or death may occur from contact with lightning.

11.16.2 Control

1. **Postpone activities promptly. Don't wait for rain.** Many people take shelter from the rain, but most people struck by lightning are not in the rain! Go quickly inside a completely enclosed building, not a carport, open garage or covered patio. If no enclosed building is convenient, get inside a hard-topped all-metal vehicle. A cave is a good option outside, but move as far as possible from the cave entrance.

2. **Be the lowest point. Lightning tends to hit the tallest object.** In the mountains if you are above tree line, you ARE the highest object around. Quickly get below tree line and get into a grove of small trees. Do not be the second tallest object during a lightning storm! Crouch down if you are in an exposed area.

3. **Keep an eye on the sky.** Look for darkening skies, flashes of lightning or increasing wind, which may be signs of an approaching thunderstorm.

4. **Listen for the sound of thunder.** If you can hear thunder, go to a safe shelter immediately.

5. **If you see or hear a thunderstorm coming or your hair stands on end, immediately suspend work and instruct everyone to go inside a sturdy building or car.** Sturdy buildings are the safest place to be. Avoid sheds, picnic shelters, baseball dugouts and bleachers. If no sturdy building is nearby, a hard-top vehicle with windows closed will offer some protection. The steel frame of the vehicle provides some protection if you are not touching metal. Work will not be resumed until at least 30 minutes have passed since thunder has been heard or lightning has been seen.

6. **Listen to the weather radio**. Listen for alerts.

7. **Set up weather alerting**. PIKA\PIRNIE has a lightning and severe weather alerting system that, when enabled for a project site, sends automated text messages to cell phones when lightning is occurring within 25 miles of project sites.

8. **If you cannot get to a shelter, stay away from trees.** If there is no shelter, crouch in the open, keeping twice as far away from a tree as it is tall.

9. **Avoid leaning against vehicles**. Get off bicycles and motorcycles.

10. **Get out of the water; it is a great conductor of electricity.** Stay off the beach and out of small boats. If caught in a boat, crouch down in the center of the boat away from metal hardware. Wading and scuba diving are NOT safe. Lightning can strike the water and travel some distance beneath and away from its point of contact. Do not stand in puddles of water, even if wearing rubber boots.

11. **Avoid metal!** Drop metal backpacks, stay away from clothes lines, fences, exposed sheds and electrically conductive elevated objects. Do not hold on to metal items such golf clubs, fishing rods, tennis rackets or tools. Large metal objects such as drill rigs or excavators can conduct lightning. Small metal objects can cause burns.

12. **Move away from a group of people.** Stay several yards away from other people. Do not share a bleacher bench or huddle in a group.

Make sure you are not the highest object. Lighting tends to strike the highest object. Crouch down. Do not lie down! What you most want to avoid is lightning going through your heart. Lightning follows the path of least resistance. When lightning strikes the ground near you, a ground current will set up in the area nearby. If you are lying flat your chances of being “hit” by this ground lightning increases. Not only that, the lightning will run through your whole body including your heart. If you are in a crouching position with your heels together, the ground current will enter one foot, but it will then return to the ground through your other foot on the ground. The current does not go through your heart.

11.17 HazCom

All project-required chemicals will be handled in accordance with OSHA 29 CFR 1910.1200 and the PIKA-PIRNIE H&S Standard ARC HSGE007. The SSHO will act as the HazCom Program Coordinator for the site and will maintain the Master Inventory List of hazardous chemicals kept on site. The SSHO will maintain MSDSs on site for all chemicals. MSDSs will be located in each PIKA-PIRNIE JV Team field vehicle. The SSHO will communicate the location of the MSDSs and the hazards associated with these chemicals to all project PIKA-PIRNIE JV Team employees and subcontractors during the safety orientation. This information will be reviewed during tailgate briefings, especially if new chemicals or materials are introduced on site.

The SSHO will ensure that all containers of chemicals (*e.g.,* drums, bags, pails, tanks, vessels) are labeled appropriately with the contents of the container, proper chemical name, associated hazards and hazard warnings, and name and address of the manufacturer/importer. Chemicals that are not properly labeled will not be accepted or allowed on site. If transferred to a secondary container, the new container will be labeled as described.

The SSHO will ensure that the PPE necessary for work around the particular chemical is available and that project employees have been trained in its use.

The PM will ensure that all project personnel have received HazCom training as required in OSHA 29 CFR 1910.1200 (h).

11.18 Work within Railroad Easements

Personnel who access within 25 feet of railroad tracks will comply with the provision of PIKA-PIRNIE H&S Standard HSSP0002l Railroad Workplace H&S Standard including provisions for site access, PPE, training, and documentation. Anytime personnel plan to be within 25 feet of railroad tracks, they will first confer with the on-site H&S representative from Norfolk Southern Railroad.

12. General Site Access and Control

Only authorized personnel will be allowed beyond the perimeter of the work site. The SSHO will coordinate access and control security at the work site. Other site workers and visitors to the work site should be kept out of the work site. If visitors under the PIKA-PIRNIE JV Team control need access to the work site, they must sign in on the the PIKA-PIRNIE JV Team or General Contractor sign in sheet and must be escorted by the designated the PIKA-PIRNIE JV Team employee. The visitor log sheet is included in **Appendix C**.

No person will be allowed access to railroad easements without Federal Railroad Administration (FRA) prior training. All on-site personnel working on or near active tracks will have the Norfolk Southern specific eRailSafe verification and training prior to coming on site.

A Norfolk Southern Safety Representative is required to be on site at all times and to identify instances where a railroad flagman is required. Whenever personnel or equipment will be within 25 feet of the centerline of the tracks, the SSHO is required to ensure a railroad flagman is on site and aware of the operations within 25 feet of tracks.

13. Decontamination

Part of required reading for this SSHP includes reviewing the FHSHB, Section III-G Site Security, Work Zones and Decontamination for HAZWOPER site zones. The decontamination procedures outlined in the FHSHB are provided for typical Level D and Level C ensembles.

13.1.1 Personnel Decontamination

All personnel wearing Modified Level D or Level C PPE in the EZ must undergo personal decontamination prior to entering the Support Zone. The personnel decontamination area will consist of the following stations, at a minimum:

- *Station 1*. Personnel leaving the contaminated zone will remove the gross contamination from their outer clothing and boots.

- *Station 2*. Personnel will remove their outer garment and gloves and dispose of them in properly labeled containers. Personnel will then decontaminate their hard hats and boots with an aqueous solution of detergent or other appropriate cleaning solution. These items will be hand carried to the next station.

- *Station 3*. Personnel will thoroughly wash their hands and face before leaving the CRZ. Respirators, when used, will be sanitized and then placed in a clean plastic bag.

13.1.2 Equipment Decontamination

All vehicles and equipment that have entered the EZ will be decontaminated at the decontamination pad prior to leaving the EZ. If the level of contamination is low, decontamination may be limited to rinsing the vehicle or equipment with water. If the vehicle or equipment is significantly contaminated, steam cleaning or pressure washing may be required.

Decontamination water will be captured, contained and staged for characterization and disposal.

13.1.3 Personal Protective Equipment Decontamination

Where and whenever possible, single-use, external protective clothing must be used for work within the EZ or CRZ. This protective clothing must be disposed of in properly labeled containers. Reusable protective clothing will be rinsed at the site with detergent and water. The rinsate will be collected for disposal.

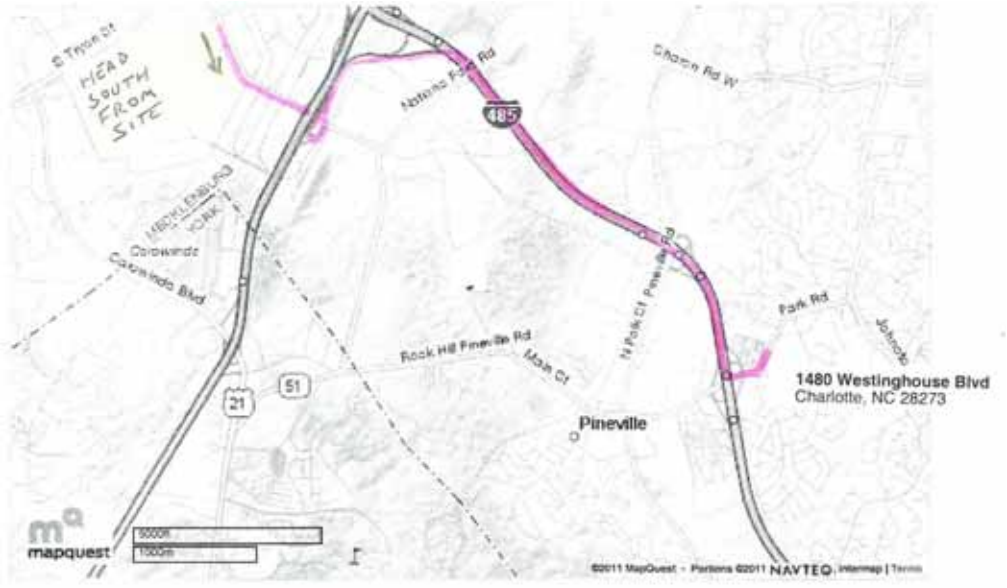
When removed from the CRZ, all respirators will be thoroughly cleaned with soap and water. The face piece, straps, valves and covers must be thoroughly cleaned at the end of each work shift and be ready for use prior to the next shift. Respirator parts may be disinfected with a solution of bleach and water, or by using a spray disinfectant.

14. Emergency Response Plan (ERP)

14.1 Route to Mercy Hospital

Mercy Hospital
10620 Park Rd. Charlotte, NC 28210

1. Go southeast on Westinghouse Blvd. towards Wilmar Blvd.
2. Continue going southeast on Westinghouse Blvd. and pass under I-77, then make the first right turn after the underpass onto a jug handle turn. This will put you on the highway access road (unnamed) heading north towards I-485.
3. Merge onto I-485 East towards Pineville. Go 4.5 miles to Exit 64A.
4. Take Exit 64A toward Matthews. Merge onto Pineville-Matthews Rd/Route 51N. Go 0.6 miles to Park Rd.
5. Make a left on Park Rd and go 0.1 miles and the entrance to Mercy Hospital. 10620 Park Rd Charlotte, NC 28210
(704) 543-1467



14.2 Emergency Contact Information and Procedures

Work Care	1-800-455-6155
Local Police –	911 and Charlotte Police Department (Non-Emergency) (704) 336-8301
Local Ambulance –	911 and (704) 543-1467
Local Fire Department –	911 and (704) 336-2851
Local Hospital –	Mercy Hospital 10620 Park Rd Charlotte, NC 28210 **(704) 543-1467**
Local Weather Data	www.Weather.com
Poison Control	800.332.3073
PIKA-PIRNIE JV Team Construction Manager –	TBD
PIKA-PIRNIE JV Team Safety and Health Manager – William C. Mener, CSP	908.526.1000 ext 209, 908.685.7859 (cell)
PIKA-PIRNIE JV Team H&S Contact – Mija Coppola, Director of Health Safety	720.344.3500
Site Safety and Health Officer – Kevin Held	908.526.1000 ext 208, 908.304.5236 (cell)
General Contractor Contact –	TBD

14.3 Emergency Supplies and Equipment List

Emergency Supplies and Equipment	Location on Project Site
☒ First Aid Kit (type):OSHA 29CFR1926.50 & ANSI Z308.1 Compliant	Operations Trailer
☒ Fire Extinguisher	Trailer, Injection System,
☒ Mobile Phone ☐ Satellite Phone	Site Manager, HSO
☒ Traffic Cones	Storage Box/Trailer
☒ Walkie Talkies	Operations Trailer
☒ Water or Other Fluid Replenishment	Operations Trailer
☐ Eye Wash/Quick Drench Station	
☒ Eye Wash Bottle	Operations Trailer
☒ Wash and Dry Towelettes	Operations Trailer
☒ Sunscreen (SPF 15 or higher)	Operations Trailer
☒ Insect Repellant	Operations Trailer
☒ Chemical Spill Kit (Bulk Chemicals including injection compounds)	Storage Box/Trailer
☒ Other (specify): ANSI/OSHA Compliant Blood-borne Pathogen Kit	Operations Trailer

14.4 Site Evacuation

If evacuation of the site is required, follow the procedure described below:

- Provide verbal notification to personnel that evacuation is necessary. Personnel are expected to be working in small groups and air horn alarms are not considered to be necessary.
- Upon notification to evacuate, all personnel will meet in the parking lot and determine the means of evacuation.
- The Construction Task Manager or SSHO will take a head count and will report the count to the SSHO via radio.
- The SSHO (or designee) will use the site sign-in sheet to coordinate the evacuation and ensure that all personnel are accounted for.
- After investigation, the Construction Task Manager, SSHO, or PM will determine the next steps to take based on the nature of the incident.

- When the incident is completed, the Construction Task Manager or SSHO will make the determination of "All Clear." No personnel will enter any of the buildings prior to the "All Clear."

- If outside resources (*e.g.,* fire, rescue, police) are required, the Construction Task Manager or SSHO will coordinate with the emergency services lead person to make a determination of "All Clear" prior to allowing site personnel to re-enter the site buildings.

14.5 Fire

In the case of a fire at the site, the Construction Task Manager or SSHO will assess the situation and direct fire-fighting activities. The Construction Task Manager or SSHO will provide that the PM is immediately notified of fires. Site personnel will attempt to extinguish the fire with available extinguishers, if safe to do so. In the event of a fire that site personnel are unable to safely extinguish with one fire extinguisher, the local fire department will be summoned by calling 911 and site personnel will meet in the parking lot and determine the Construction Task Manager or SSHO will determine safe distances for personnel to gather.

14.6 Contaminant Release

In the event of a contaminant release, take the following steps:

- Evacuate the immediate area of the release.

- Secure the impacted area.

- Notify the Construction Task Manager or SSHO immediately.

- Assemble spill response team and equipment. Conduct air monitoring and reference MSDS if appropriate, to identify the PPE needed.

- Initiate spill response efforts that may not require specialized PPE such as positioning waste containers and applying absorbent to spill boundaries.

- Don the required level of PPE and prepare to implement control procedures that entail waste handling such as shoveling absorbent into waste storage containers.

The SM/SSHO has the authority to commit resources as needed to contain and control released material and to prevent its spread to off-site areas.

14.7 Medical Emergency

All employee injuries must be promptly reported to the SSHO, who will:

- Provide the injured employee prompt first aid and medical attention.
- In emergency situations, transport the employee by appropriate (self-transport to the hospital is not allowed) means to the designated urgent care facility (normally a hospital emergency room), accompanied by a coworker.
- If the injured employee is a PIKA/Pirnie JV Team employee, notify the Workers Compensation coordinator at 720.344.3844 as soon as possible after an injured worker has left the site.

14.7.1 Emergency Care Steps

In the event of an employee injury, perform the following emergency care steps:

- Survey the scene. Determine if it is safe to proceed. Try to determine if the conditions that caused the incident are still a threat. Protect yourself from exposure before attempting to rescue the victim.
- Do a primary survey of the victim. Check for airway obstruction, breathing and pulse. Assess likely routes of chemical exposure by examining the eyes, mouth, nose and skin of the victim for symptoms.
- Phone Emergency Medical Services (EMS). Give the location, telephone number used, caller's name, what happened, number of victims, victim's condition and help being given.
- Maintain airway and perform rescue breathing as necessary.
- Perform CPR as necessary.

- Do a secondary survey of the victim. Check vital signs and do a head-to-toe exam.

Treat other conditions as necessary. If the victim can be moved, take him/her to a location away from the work area where EMS can gain access.

14.8 First Aid – General

All persons must report injury or illness to their immediate supervisor and the Construction Task Manager or SSHO. Trained personnel will provide first aid. Injuries and illnesses requiring medical treatment must be documented. The SM must conduct a Loss Investigation (LI) as soon as emergency conditions no longer exist and first aid and/or medical treatment have been obtained. LIs must be completed and submitted to the PM within 24 hours after the incident.

If first aid treatment is required, obtain a first aid kit from the work vehicle. If treatment beyond first aid is required, transport the injured person(s) to Mercy Hospital. If the injured person is not ambulatory, or shows any sign of not being in a comfortable and stable condition for transport, summon an ambulance/ paramedics. If there is doubt as to the injured worker's condition, it is best to let the local paramedic or ambulance service examine and transport the worker.

14.8.1 First Aid – Inhalation

Any employee complaining of symptoms of chemical overexposure will be removed from the work area and transported to the designated medical facility for examination and treatment.

14.8.2 First Aid – Ingestion

If an employee has or may have ingested a poisonous substance, call EMS and consult a poison control center for advice. If available, refer to the chemical's MSDS for treatment information. If the victim is unconscious, keep them on their side and clear the airway if vomiting occurs.

14.8.3 First Aid – Skin Contact

Employees who have had skin contact with contaminants will, unless the contact is severe, proceed to the wash area. Employees will remove any contaminated clothing

and flush the affected area with water. Transport the employee to the designated medical facility if he/she shows signs of skin reddening, irritation or if he/she requests a medical examination.

14.8.4 First Aid – Eye Contact

Employees who have had contaminants splashed in their eyes or who have experienced eye irritation must immediately notify nearby personnel to assist and utilize the portable eye flush bottle. Do not decontaminate prior to using the eye flush. Remove whatever protective clothing is necessary to use the eye flush. Arrange prompt transport to Mercy Hospital.

14.9 Reporting Injuries, Illnesses and Near-Miss Incidents

Report injuries and illnesses, however minor, to the Construction Task Manager or SSHO immediately. The SSHO will complete an injury report and submit it to the SHM, PM and Contracting Officer (CO)/COR within 24 hours. A completed Form 3394 (**Appendix C**) will be attached to the PIKA-PIRNIE JV Team incident report (see ARC HSMS010 - Incident Investigation).

Near-loss incidents are situations in which no injury or property damage occurred, but under slightly different circumstances an injury or property damage could have occurred. Near losses are caused by the same factors as injuries; therefore, they must be reported and investigated in the same manner. An assessment must be conducted immediately after an injury, illness, near loss or other incident to determine if it is safe to proceed with the work.

14.10 WorkCare

WorkCare is a 24-hour service that provides professional medical assistance on the telephone for non-emergency injuries or illness. The WorkCare nurse or doctor may assess the injury or symptoms, advise the employee as to appropriate care and assist in identifying a medical facility if necessary. Contact WorkCare at 800.455.6155.

Note: First aid will be administered and, when needed, emergency medical treatment will be sought prior to contacting WorkCare (800.455.6155).

15. TRACK

TRACK is a tool that enables personnel to probe day-to-day operational and procedural systems to identify hazards that may have been overlooked in project or task planning; that may have developed after the job/task started; or that may exist due to a lack of proper procedures, training, equipment or process modifications or because of site-specific concerns such as weather, location or traffic. The PIKA-PIRNIE JV Team TRACK process is undocumented. It is done in the mind of the employee or communicated among co-workers. This tool is based on the principle that personnel must take responsibility for their own H&S in all daily activities and take the time to conduct TRACK.

"*Think through the task!"*

- First, think about the task to determine whether an incident could occur:
- What are the steps in the task?
- How is the job going to be done?
- What tools will be used; what environment are we in; what techniques will be used?
- Who is involved and who needs to be involved?

"Recognize the hazards!"

- Next, recognize the hazards associated with the task and its individual steps:
- What is the worst that could happen?
- Is the work area safe?
- What hazards might I encounter while performing these tasks?
- Are tools and equipment in good repair and working properly?
- Which physical hazards are present (*e.g.,* heat, noise, vibration, awkward positions, lifting)?

"*Assess the risks!"*

- Be sure you understand the risks associated with the identified hazards. If a hazard is likely to occur, how badly could I or anybody else be hurt?
- How often might I or anybody else be exposed to that hazard as I am performing this task?
- What is the likelihood of an injury or damage?

"Control the hazards!"

- Now, take the necessary steps to eliminate or control the hazards.
- Is there a safer way to do the job?
- Can the hazard be eliminated?
- Can the hazard be engineered out of the task or work area (*e.g.,* guardrails, ventilation, material substitution or piece of equipment)?
- Can administrative controls be implemented to eliminate or minimize the hazard (*e.g.,* rest periods, signage, job rotation, training)?
- If engineering or administrative controls are not practical, will the use of PPE minimize the hazard and risk?

"Keep H&S first in all things!"

- Finally, always put H&S first in all things.
- Correct or report H&S concerns.
- Suggest ways to improve H&S and/or eliminate unsafe conditions.
- Monitor H&S controls for effectiveness.
- Look out for yourself and others.
- Continually be aware of your surroundings, and, when things change or you have a concern, stop and redo TRACK.
- STOP WORK IF IT IS NOT SAFE.

16. Subcontractors

A copy of this SSHP is to be provided to all subcontractors prior to the start of work so that the subcontractor is informed of the hazards at the site. While the PIKA-PIRNIE JV Team SSHP will be the minimum H&S requirements for the work completed by the PIKA-PIRNIE JV Team and its subcontractors, each subcontractor, in coordination with the PIKA-PIRNIE JV Team H&S personnel, is expected to perform its operations in accordance with its own SSHP, policies and procedures unique to the subcontractor's work to ensure that hazards associated with the performance of the work activities are properly controlled. Copies of any required safety documentation for a subcontractor's work activities will be provided to the PIKA-PIRNIE JV Team for review prior to the start of on-site activities.

In the event that the subcontractor's procedures/requirements conflict with requirements specified in this SSHP, the more stringent guidance will be adopted after discussion and agreement between the subcontractor and the PIKA-PIRNIE JV Team project H&S personnel. Hazards not listed in this SSHP, but known to the subcontractor or known to be associated with the subcontractor's services, must be identified and addressed to the PIKA-PIRNIE JV Team project or Task Manager and SSHO prior to beginning work operations.

If the subcontractor prefers to adopt this SSHP, the "Subcontractor Acknowledgement Memo" must be signed and dated by the subcontractor's management and placed in the project file. Once the signed memo is received by the project manager, an electronic version of our SSHP can be submitted to the subcontractor to use as their own. Subcontractors working at the site will need to have this plan with them, and will also need to sign the Subcontractor SSHP receipt signature page of the PIKA-PIRNIE JV Team SSHP (**Appendix C**). Subcontractors are responsible for the H&S of their employees at all times, and have the authority to halt work if unsafe conditions arise.

The PM/Task Manager and SSHO (or authorized representative) has the authority to halt the subcontractor's operations and to remove the subcontractor or subcontractor's employee(s) from the site for failure to comply with established H&S procedures or for operating in an unsafe manner.

17. Project Personnel SSHP Certification

All site project personnel will sign the certification signature page provided below.

17.1 PIKA-PIRNIE JV Team Personnel Signature Page

I certify that I have read, understand, and will abide by the safety requirements outlined in this SSHP.

Printed Name	Signature	Date

17.2 Subcontractor Acknowledgement: Receipt of SSHP

The PIKA-PIRNIE JV Team claims no responsibility for the use of this SSHP by others although subcontractors working at the Site may use this SSHP as a guidance document. In any event, the PIKA-PIRNIE JV Team does not guarantee the health and/or safety of any person entering this Site. Strict adherence to the H&S guidelines provided herein will reduce, but not eliminate, the potential for injury at this Site. To this end, H&S becomes the inherent responsibility of personnel working at the Site.

Printed Name	Company	Signature	Date

17.3 Visitor Acknowledgement and Acceptance of SSHP

By signing below, I waive, release and discharge the Owner of the Site and the PIKA-PIRNIE JV Team and their employees from any future claims for bodily and personal injuries which may result from my presence at, entering, or leaving the Site and in any way arising from or related to any and all known and unknown conditions on the Site.

Name	Company	Reason for Visit	Date/Time On Site	Date/Time Off Site

Appendix A

SSHP Addendum Pages and Log Table

Addendum Page

This form should be completed for new tasks associated with the project. The project manager and/or task manager should revise the Project Hazard Analysis Worksheet with the new task information and attach to this addendum sheet. JLAs should be developed for any new tasks and attached as well.

Review the addendum with all site staff, including subcontractors, during the daily tailgate briefing, and complete the tailgate briefing form as required. Attach a copy of the addendum to all copies of the SSHP including the site copy, and log in the Addendum Log Table A-1 on the next page.

Addendum Number: _____ Project Number: _____

Date of Changed Conditions: _____ Date of Addendum: _____

Description of Change that Results in Modifications to SSHP:

Signed: _____ Signed: _____
Project Manager Site Safety Officer

Signed: _____ Signed: _____
H&S Plan Writer H&S Plan Reviewer

Addendum Log Table

Addendums are to be added to every copy of the SSHP, and logged on Table A-1 to verify that all copies of the SSHP are current:

Table A-1 Addendum Log Table

Addendum Number	Date of Addendum	Reason for Addendum	Person Completing Addendum
1			
2			
3			
4			
5			
6			
7			
8			
9			
10			

Make additional copies of this form as necessary.

Appendix B

AHAs

ACTIVITY HAZARD ANALYSIS #1

Activity:	Date Prepared	Date Revised	Revision No.
Mobilization and Demobilization of Equipment	October 2011		

Project	Analysis prepared by:	Reviewed by:	Accepted by:
Former Charlotte Naval Ammunition Depot (CNAD) Complex	Kevin Held		

Activity	Hazards	Recommended Controls
1. Utilize Hand Tools to Disassemble Equipment	Contact with site contaminants. Strike or impact hazards with other workers, equipment or structures.	A. Ensure all process equipment that has contacted contaminated materials has been flushed and cleaned prior to disassembly. B. Inspect hand tools and only use tools in good operating condition C. Exercise caution when applying manual pressure with hand tools. D. Wear gloves appropriate for hazard while maintaining dexterity E. Watch for and keep hands clear of obvious hazards like door or cover closures.
2. Disassemble and Load Equipment Utilizing Manual Lifting and Powered Industrial Vehicles	Strike or impact hazards with other workers, equipment or structures. Rollovers on slopes. Dropped Loads. Muscle Strains	A. Maintain a minimum 15-operation zone around active powered industrial equipment. B. Follow equipment manufacturer instructions for use on slopes or load capacities. C. Wear seatbelt at all times. D. Utilize safe lifting practices. Observe 50-lb personal weight limit. E. Ensure rigging personnel are qualified and maintain one-to-one communication with equipment operators.
3. Ingress and egress from equipment	Slips and falls from accessing or egress from equipment	A. Maintain 3 points of contact when access or egress equipment

Activity	Hazards	Recommended Controls
		B. Keep any ladder or steps on equipment clean and dry to extent practical
4. Equipment inspections and disassembly	A. Pinch/crush hazards to hands from doors or covers	A. Wear gloves appropriate for hazard while maintaining dexterity B. Watch for and keep hands clear of obvious hazards like door or cover closures. C. Do not hurry during the removal or placement of covers or equipment components.
Equipment to be used.	**Inspection Requirements**	**Training Requirements**
1. Powered Industrial Vehicles 2. Construction Equipment 3. Hand Tools	1. PIVs and other construction vehicles. 2. Fire extinguisher 3. Hand Tools	1. Equipment operation 2. Operator and pedestrian hand signals 2. Hazard communication 3. Fire extinguisher

ACTIVITY HAZARD ANALYSIS #2

Activity: Soil Drilling and Well Installation	Date Prepared	Date Revised	Revision No.
	October 2011		

Project	Analysis prepared by:	Reviewed by:	Accepted by:
Former Charlotte Naval Ammunition Depot (CNAD)	Kevin Held		

Activity	Hazard	Recommended Controls
1. Railroad clearance	Contact with trains	A. Identify instances where personnel or equipment will be within 25-feet of the centerline of the tracks. Ensure railroad flagman is on-site and aware of operations within 25-feet of tracks as discussed in AHA for work near railroad tracks.
2. Utility Clearance	Inadvertent contact with utilities	A. Complete utility clearance in accordance with the PIKA-PIRNIE JV Team H&S procedure ARCHSF019.
3. Delineate work area with traffic cones. Mobilize drill rig to drill location.	Vehicular accidents including rollover and contact with overhead utilities	A. Walk through work area with drill crew prior to mobilization. B. Do not move drill rig with raised derrick.
4. Utilize drill rig to advance borehole	Rotary movement of auger, moving parts Contact with site contaminants or subsurface gases Exposure to loud noise Presence of slippery surfaces; slip, trip and fall potential Contact with raised derrick	A. Utilize Modified Level D protection. B. Stay at least 5 feet away from moving parts of the drill rig. C. Know where the kill switch is, and have drillers test it. D. Do not wear loose clothing, and tie long hair back. Avoid wearing jewelry inside drilling operations work area. E. Utilize hearing protection in addition to Modified Level D protection. F. Air monitoring for organic vapors (PID), lower explosive limit and hydrogen sulfide

Activity	Hazard	Recommended Controls
		(CGI). G. Ensure derrick maintains safe clearance from obstructions and utilities. H. Ensure work area is kept clear of slip, trip and fall hazards.
5. Install well screen, piping and packing in borehole	Lifting strains Presence of slippery surfaces; slip, trip and fall potential Exposure to well packing material, such as bentonite dust.	A. Practice proper lifting techniques. B. Ensure work area is kept clear of slip, trip and fall hazards. C. Comply with provisions of MSDS for well packing materials.

Equipment to be used.	Inspection Requirements	Training Requirements
1. Drill rig	1. Drill rig including safety features such as kill switch, outriggers and level indicator.	1. Licensed driller 2. Drillers helper on-the-job training 3. HAZWOPER 4. Hazard Communication

ACTIVITY HAZARD ANALYSIS #3

Activity:	Date Prepared	Date Revised	Revision No.
Installation of Storage Tanks and Process Equipment	October 2011		

Project	Analysis prepared by:	Reviewed by:	Accepted by:
Former Charlotte Naval Ammunition Depot (CNAD)	Kevin Held		

Activity	Hazard	Recommended Controls
1. Mobilize equipment	Lifting strains, impacts, lacerations Presence of slippery surfaces; slip, trip and fall potential	A. Safe work practices
2. Install electrical service and distribution system.	Exposure to electrical hazards Lifting strains Presence of slippery surfaces; slip, trip and fall potential	A. Installation by a licensed contractor with competent electrician on-site when potential exposure to live electrical connections exist. B. Isolation of hazardous energies with lockout-tagout procedure. The PIKA-PIRNIE JV Team H&S procedure ARCHSF019.
3. Grade surfaces, install concrete and gravel pads	Handling concrete mix Lifting strains Presence of slippery surfaces; slip, trip and fall potential	A. Practice proper lifting techniques & safe work practices. B. Ensure work area is kept clear of slip, trip and fall hazards including water and cement slurries or powders. C. Comply with provisions of MSDS for cement and other materials being utilized including requirements for eye wash and skin washing facilities.
4. Modify and install process equipment components using hand and power tools and acetylene torch cutting or welding.	Lifting strains Slip, trip and fall potential Contact with moving parts on power tools Electric shock	A. Practice proper lifting techniques & safe work practices. B. Ensure work area is kept clear of slip, trip and fall hazards including water and cement

Activity	Hazard	Recommended Controls
	Falls from heights Fire Hazard Burns Damage to compressed gas cylinders	slurries or powders. C. Comply with provisions of MSDS for hazardous materials used for construction. D. Personal fall arrest system or other approved fall protection system for work on surfaces more than 5-ft above walking surface. E. Remove or isolate flammable materials in work area before hot works. F. Ensure cylinders are chained and utilized properly. G. Specialized PPE for hot works including welding helmet with shaded lens rated for type of hot work being performed.
5. Utilization of powered industrial vehicles including forklifts, man lifts, and related construction equipment	Lifting strains Hitting personnel, equipment or structures Equipment roll over Contact with utilities Dropped loads	A. Plan and manage movement of equipment and personnel to minimize exposure of pedestrians to vehicular traffic. B. Ensure work area is kept clear of slip, trip and fall hazards. C. Comply with provisions of MSDS for well packing materials.

Equipment to be used.	Inspection Requirements	Training Requirements
1. Ladders 2. Hand tools 3. Powered tools 4. Powered industrial vehicles 5. Compressed gases with torch cutting and/or welding equipment	1. Ladder components 2. Hand and power tools 3. Powered industrial vehicles 4. Compressed gases 5. Fire extinguisher	1. Ladder Safety 2. Hazard communication 3. Fire extinguisher 4. PPE 5. Hot works procedures/Fire watch

ACTIVITY HAZARD ANALYSIS #4

Activity:	Date Prepared	Date Revised	Revision No.
Low Pressure Injection of Colloidal Molasses Suspension Into Groundwater Aquifer	October 2011		

Project	Analysis prepared by:	Reviewed by:	Accepted by:
Former Charlotte Naval Ammunition Depot (CNAD)	Kevin Held		

Activity	Hazard	Recommended Controls
1. Mobilize materials and equipment for low pressure injection of colloidal molasses suspension.	A. Slips, trips and falls B. Chemical release during transport across site C. General contact and splashes D. Strain, lifting injuries E. Chemical release during transfers and mixing F. Unexpected reaction between chemicals/solutions	A. All hoses crossing vehicle access / escape route to be mechanically protected by industial “cable protectors”. Attempt to minimize length of hose on surface. B. Assess all manual handling actions before undertaking. C. Two-person lift required to move, coil/uncoil and position hoses that weigh more than 30-lbs. D. Ensure GFCI protection on all outdoor electrical service. E. All fittings and connections to be secured and inspected for proper locking of connections. E. Complete an integrity test with water from the on-site supply before pumping the molasses solution. F. Adhere to safety provisions on product MSDSs including provisions for spill response. G. Rubber boots with approved toe-protection or boot covers and nitrile rubber gloves required.
2. Pressure injection of molasses suspension	A. Slips, trips and falls	A. Minimize length of hose and mark trip

Activity	Hazard	Recommended Controls
	B. Spillage from hoses and connections. C. Chemical reaction leading to a temperature increase in groundwater D. Electrical shock	hazards with traffic cones. B. Personnel must remain in vicinity of flow controls during injection and monitor flow, pressure, temperature and other parameters for comparison to pre-established limits. D. Adhere to safety provisions on product MSDSs including provisions for spill response. E. Level D protection with rubber gloves and boots/boot covers. Splash goggles and splash shield for tasks with splash hazards.
3. System decommissioning 3A. Flush the system with copious amounts of water from the on-site supply. 3B. Drain excess fluid from hoses into injection site. 3C. Rinse containers with clean water and dispense in accordance with waste disposal requirements.	A. Slips, trips and falls B. Spillage from hoses and connections. C. Residue from molasses suspension on or in hoses or equipment. D. Release of residue from used containers	A. Pump should be used to pump residual solution from secondary containment into aquifer. B. Prior to decommissioning, contents of part open containers will be dispensed through aquifer injection system. No part used containers to be transported from site. C. Level D protection with rubber gloves and boots/boot covers. Splash goggles and splash shield for tasks with splash hazards.

Equipment to be used.	Inspection Requirements	Training Requirements
1. Chemical storage and transfer containers 2. Hoses with cam-lock connections 3. Mixing tanks 4. Low pressure injection system	1. Chemical storage, handling, and spill response equipment 2. Eye wash and field wash facilities 3. Fire extinguisher 4. GFCI test 5. Hose connections and integrity of injection system.	1. HAZWOPER 2. Operation of groundwater injection system. 3. Hazard communication 4. Fire extinguisher

Appendix C

SSHP Forms

- Tailgate Meeting Form
- Utilities and Structures Checklist
- Elevated Work Permit
- Hot Work Permit
- USACE Form 3394, Accident Investigation Report

PIKA INTERNATIONAL, INC. — A JOINT VENTURE — MALCOLM PIRNIE

Document Control Number:TGM - _____

TGM + *project number plus date as follows: xxxxxxxx.xxxx.xxxxx - dd/mm/year*

TAILGATE HEALTH & SAFETY MEETING FORM

This form documents the tailgate meeting conducted in accordance with the Project HASP. Personnel who perform work operations on-site during the day are required to attend this meeting and to acknowledge their attendance, at least daily.

Project Name:			**Project Location:**
Date:	**Time:**	**Conducted by:**	**Signature/Title:**
Client:		**Client Contact:**	**Subcontractor companies:**

TRACKing the Tailgate Meeting

Think through the Tasks (list the tasks for the day):

1 _____ 3 _____ 5 _____

2 _____ 4 _____ 6 _____

Other Hazardous Activities - Check the box if there are any other PIKA-PIRNIE JV Team, Client or other party activities that may pose hazards to PIKA-PIRNIE JV Team operations ☐ If there are none, write "None" here: _____

If yes, describe them here: _____

How will they be controlled? _____

Prework Authorization - check activities to be conducted that require permit issuance or completion of a checklist or similar before work begins:

	Doc #		Doc #		Doc #
☐ Not applicable		☐ Working at Height	_____	☐ Confined Space	_____
☐ Energy Isolation (LOTO)	_____	☐ Excavation/Trenching	_____	☐ Hot Work	_____
☐ Mechanical Lifting Ops	_____	☐ Overhead & Buried Utilities	_____	☐ Other permit	_____

Discuss following questions (for some review previous day's post activities). **Check if yes :**

☐ Incidents from day before to review?	☐ Lessons learned from the day before?	☐ Any Stop Work Interventions yesterday?
☐ Any corrective actions from yesterday?	☐ Will any work deviate from plan?	☐ If deviations, notify PM & client
☐ JLAs or procedures are available?	☐ Field teams to "dirty" JLAs, as needed?	☐ All equipment checked & OK?
☐ Staff has appropriate PPE?	☐ Staff knows Emergency Plan (EAP)?	☐ Staff knows gathering points?

Comments: _____

Recognize the hazards (check all those that are discussed) (Examples are provided) and **A**ssess the Risks (Low, Medium, High - circle risk level) - Provide an overall assessment of hazards to be encountered today and briefly list them under the hazard category.

☐ Gravity (i.e., ladder, scaffold, trips) (L M H)	☐ Motion (i.e., traffic, moving water) (L M H)	☐ Mechanical (i.e., augers, motors) (L M H)
☐ Electrical (i.e., utilities, lightning) (L M H)	☐ Pressure (i.e., gas cylinders, wells) (L M H)	☐ Environment (i.e., heat, cold, ice) (L M H)
☐ Chemical (i.e., fuel, acid, paint) (L M H)	☐ Biological (i.e., ticks, poison ivy) (L M H)	☐ Radiation (i.e., alpha, sun, laser) (L M H)
☐ Sound (i.e., machinery, generators) (L M H)	☐ Personal (i.e. alone, night, not fit) (L M H)	☐ Driving (i.e. car, ATV, boat, dozer) (L M H)

Continue TRACK Process on Page 2

TAILGATE HEALTH & SAFETY MEETING FORM - Pg. 2

Control the hazards (Check all and discuss those methods to control the hazards that will be implemented for the day): Review the HASP, applicable JLAs, and other control processes. Discuss and document any additional control processes.

- [x] **STOP WORK AUTHORITY** (Must be addressed in every Tailgate meeting - (*See statements below*)

☐ Elimination	☐ Substitution	☐ Isolation
☐ Engineering controls	☐ Administrative controls	☐ Monitoring
☐ General PPE Usage	☐ Hearing Conservation	☐ Respiratory Protection
☐ Personal Hygiene	☐ Exposure Guidelines	☐ Decon Procedures
☐ Emergency Action Plan (EAP)	☐ Fall Protection	☐ Work Zones/Site Control
☐ JLA to be developed/used ***(specify)***	☐ LPO conducted ***(specify job/JLA)***	☐ Traffic Control
☐	☐	☐ Other ***(specify)***
☐	☐	☐

Signature and Certification Section - Site Staff and Visitors

Name/Company/Signature	Initial & Sign in Time	Initial & Sign out Time	I have read and understand the HASP

Important Information and Numbers

All site staff should arrive fit for work. If not, they should report to the supervisor any restrictions or concerns.

In the event of an injury, employees will call WorkCare at 1.800.455.6155 and then notify the field supervisor who will, in turn, notify Corp H&S at 1.720.344.3844.

In the event of a motor vehicle accident, employees will notify the field supervisor who will then notify Corp H&S at 1.720.344.3844 and then Corp Legal at 1.720.344.3756.

In the event of a utility strike or other damage to property of a client or 3rd party, employees will immediately notify the field supervisor, who will then immediately notify Corp Legal at 1.678.373.9556 and Corp H&S at 1.720.344.3500

Visitor Name/Co - not involved in work

	In	Out
	In	Out
	In	Out
	In	Out

I will STOP the job any time anyone is concerned or uncertain about health & safety or if anyone identifies a hazard or additional mitigation not recorded in the site, project, job or task hazard assessment.

I will be alert to any changes in personnel, conditions at the work site or hazards not covered by the original hazard assessments.

If it is necessary to **STOP THE JOB,** I will perform **TRACK**; and then amend the hazard assessments or the HASP as needed.

I will not assist a subcontractor or other party with their work unless it is absolutely necessary and then only after I have done TRACK and I have thoroughly controlled the hazard.

Post Daily Activities Review - Review at end of day or before next day's work (Check those applicable and explain:)

- ☐ Lessons learned and best practices learned today: _____
- ☐ Incidents that occurred today: _____
- ☐ Any Stop Work interventions today? _____
- ☐ Corrective/Preventive Actions needed for future work: _____
- ☐ Any other H&S issues: _____

Keep H&S 1st in all things

WorkCare - 1.800.455.6155
Near Loss Hotline - 1.866.242.4304

Utilities and Structures Checklist

Project: _____
Project Number: _____
Date: _____
Work locations applicable to this clearance checklist:

Pre-Field Work

One Call or "811" notified 48-72 hours in advance of work? ☐ Yes ☐ No
Utility companies notified during the One Call process ☐ See attached ticket

_____ _____ _____ _____
_____ _____ _____ _____
_____ _____ _____ _____

List any other utilities requiring notification: ☐ None

_____ _____ _____ _____

Client provided utility maps or "as built" drawings showing utilities? ☐ Yes ☐ No

Field Work

Markings present: ☐ Paint ☐ Pin flags/stakes ☐ Other ☐ None

Subsurface Utility Lines of Evidence Used (3 Minimum):

☐ One Call/"811"
☐ Client Provided Maps/Drawings
☐ Client Clearance
☐ Interviews: Name(s)/Affiliation(s) _____
_____
_____

Did persons interviewed indicate depths of any utilities in the subsurface?
☐ Yes, depths provided:

☐ Did not know or refused to answer

Comments:

☑ Site Inspection
☐ GPR
☐ Air-Knife
☐ Hydro-Knife
☐ Public Records/Maps
☐ Radiofrequency
☐ Metal Detector
☐ Handauger
☐ Potholing
☐ Probing
☐ Private Locator: Name and Company: _____
☐ Marine Locator: Name and Company: _____
☐ Other:

_____
_____

Tips for Successful Utility Location:
1. No excessive turning or downward force of handaugers/shovels, etc.
2. No hammering- no pickaxes-no digging bars-no hurrying or shortcutting
3. Select alternate/backup locations for clearance
4. Utilities may run directly under asphalt/concrete or be > 5 ft depth
5. Be on site when utilizing private utility locators

T R A C K

Site Inspection

During inspections look for the following ("YES" requires follow up investigation):

		Utility color codes		
a)	Natural gas line present (evidence of a gas meter)?	Yellow	☐ Yes	☐ No
b)	Evidence of subsurface electric lines :	Red		
	i) Conduits to ground from electric meter?		☐ Yes	☐ No
	ii) Overhead electric lines absent		☐ Yes	☐ No
	iii) Light poles, electric devices with no overhead lines?		☐ Yes	☐ No
c)	Evidence of water lines:	Blue		
	i) Water meter on site?		☐ Yes	☐ No
	ii) Fire hydrants in vicinity of work?		☐ Yes	☐ No
	iii) Irrigation systems?		☐ Yes	☐ No
d)	Evidence of sewers or storm drains:	Green		
	i) Restrooms or kitchen on site?		☐ Yes	☐ No
	ii) Gutter down spouts going into ground		☐ Yes	☐ No
	iii) Grates in ground in work area		☐ Yes	☐ No
e)	Evidence of telecommunication lines:	Orange		
	i) Fiber optic warning signs in areas?		☐ Yes	☐ No
	ii) Lines from cable boxes running into ground?		☐ Yes	☐ No
	iii) Conduits from power poles running into ground?		☐ Yes	☐ No
	iv) Aboveground boxes or housings in work area?		☐ Yes	☐ No
f)	Underground storage tanks:			
	i) Tank pit present?		☐ Yes	☐ No
	ii) Product lines running to dispensers/buildings?		☐ Yes	☐ No
	iii) Vent present away from tank pit?		☐ Yes	☐ No
g)	Proposed excavation markings in work area?	White	☐ Yes	☐ No
h)	Other:			
	i) Evidence of linear asphalt or concrete repair		☐ Yes	☐ No
	ii) Evidence of linear ground subsidence or change in vegetation?		☐ Yes	☐ No
	iii) Manholes or valve covers in work area?		☐ Yes	☐ No
	iv) Warning signs ("Call Before you Dig", etc) on or adjacent to site?		☐ Yes	☐ No
	v) Utility color markings not illustrated in this checklist?		☐ Yes	☐ No
i)	Aboveground lines in or near the work area:			
	i) < 50 kV within 10 ft of work area?		☐ Yes	☐ No
	ii) >50 - 200 kV within 15 ft of work area?		☐ Yes	☐ No
	iii) >200-350 kV within 20 ft of work area?		☐ Yes	☐ No
	iv) >350-500 kV within 25 ft of work area?		☐ Yes	☐ No
	v) >500-750 kV within 35 ft or work area?		☐ Yes	☐ No
	vi) >750-1000 kV within 45 ft of work area?		☐ Yes	☐ No

Comments:

Do not initiate intrusive work if utilities are suspected to be present in area and are not located, markings are over 14 days old, or if clearance methods provide incomplete or conflicting information. Do not perform intrusive work within 30 inches of a utility marking without hand clearing.

Name and signature of person completing the checklist:

Name: _____

Signature: _____

Date: _____

PIKA INTERNATIONAL, INC. A JOINT VENTURE MALCOLM PIRNIE

ELEVATED WORK PERMIT

Project Name:		**Project Location:**	
Permit Developed By:		**Date:**	
Project Start Date:		**Project Completion Date:**	
Client:		**Client Contact:**	
Subcontractor Companies			

TRACKing the Elevated Work Permit

THINK THROUGH THE TASK

Job Task: (Summary of what elevated work is proposed)

WORKFORCE INVOLVED IN ELEVATED WORK		Check all the apply						
Name	**Company**	Elevated Work Qualified	Competent Person	Experienced	Can Work Alone	Short Service Employee	Training Required	Supervision Required
		☐	☐	☐	☐	☐	☐	☐
		☐	☐	☐	☐	☐	☐	☐
		☐	☐	☐	☐	☐	☐	☐
		☐	☐	☐	☐	☐	☐	☐
		☐	☐	☐	☐	☐	☐	☐
		☐	☐	☐	☐	☐	☐	☐
		☐	☐	☐	☐	☐	☐	☐

Comments/Additional Details:

RECOGNIZE THE HAZARDS (check those that apply) and ASSESS THE RISK (Low - Moderate - High)

YES	NO		SELECT ↓	YES	NO		SELECT
☐	☐	FRAGILE ROOF OR SURFACES		☐	☐	POSSIBLE FALL FROM A HEIGHT BELOW 6' (1.83M)	
☐	☐	MATERIALS OR TOOLS AT HEIGHTS		☐	☐	POSSIBLE FALL FROM A HEIGHT OF 6' (1.83M) OR MORE	
☐	☐	LIFTING, PUSHING OR PULLING		☐	☐	POSSIBLE FALL FROM A LADDER	
☐	☐	WORK NEAR ELECTRICAL LINES		☐	☐	POSSIBLE FALL FROM A WORK PLATFORM	
☐	☐	MANUAL HANDLING MATERIALS		☐	☐	POSSIBLE FALL INTO A HAZARDOUS SUBSTAN	
☐	☐	GROUND LEVEL OBSTRUCTION		☐	☐	POSSIBLE FALL INTO EXCAVATION	
☐	☐	MOVING MATERIALS		☐	☐	POSSIBLE FALL INTO WATER	
☐	☐	OVERHEAD OBSTRUCTIONS		☐	☐	RESTRICTED SPACE	
☐	☐	FALLING OBJECTS		☐	☐	VEHICLES OR TRAFFIC	
☐	☐	SLIPS, TRIPS, FALLS		☐	☐	WEATHER OR TEMPERATURE	
☐	☐	WORKING ALONE		☐	☐	FALLING OBJECTS	
☐	☐	UNGUARDED EDGES		☐	☐	LACK OF SPACE	
☐	☐	UNEVEN FLOOR SURFACES		☐	☐	WORKING ABOVE A HAZARD	

CONTROL THE WORKING ENVIRONMENT

	YES	NO		YES	NO
GENERAL INDUSTRY PROJECT	☐	☐	CONSTRUCTION INDUSTRY PROJECT	☐	☐
CONES/BARRIERS	☐	☐	ISOLATE EQUIPMENT	☐	☐
EMERGENCY RESCUE PROCEDURES IN PLACE	☐	☐	THREE FEET OF LADDER ARE ABOVE STEPPING-OFF POINT	☐	☐
EQUIPMENT MAINTAINED	☐	☐	LADDER PLACED AT 4:1 ANGLE	☐	☐
FIRST AID PROVISION	☐	☐	WEATHER	☐	☐
RESCUE AT HEIGHTS AVAILABLE WITHIN FIVE MINUTES	☐	☐	LADDER SECCURED AT TOP AND ON LEVEL FIRM BASE TO SUPPORT LOAD	☐	☐
SAFE WORKING AREA	☐	☐	WORK EQUIPMENT INSPECTED	☐	☐

Note: General Industry requires fall protection at heights of 4 feet and greater, whearas Construction Industry requires protection at 6 feet and higher.

CONTROL THE HAZARDS: TYPE OF FALL PROTECTION SYSTEM TO BE USED

	YES	NO		YES	NO
GUARDRAILS	☐	☐	FENCES	☐	☐
PFAS	☐	☐	BARRICADES	☐	☐
SAFETY NET	☐	☐	CAGE LADDER SYSTEM	☐	☐
POSITIONING DEVICE SYSTEM	☐	☐	RIGID RAIL	☐	☐
COVERS	☐	☐	WIRE RAIL SYSTEM	☐	☐
CONTROLLED ACCESS ZONE	☐	☐	WARNING LINE SYSTEM	☐	☐

EQUIPMENT REQUIRED

Personal Fall Arrest System	YES	NO	**Guardrail System**	YES	NO
6' (1.83M) FALL-LIMITING LANYARD WITH SHOCK ABSORBER	☐	☐	MID-RAIL PLACED WITH NO GAP OF 19" (48cm)	☐	☐
ANCHORAGE POINTS DESIGNED	☐	☐	GUARD RAILS (DOUBLE ABOVE 6'	☐	☐
FALL ARREST (INSPECTED)	☐	☐	GUARD RAILS (TOP RAIL A MINIMUM OF 39" [1m] ABOVE PLATFORM)	☐	☐
HARNESS (INSPECTED)	☐	☐	TOE BOARDS	☐	☐
Positioning/ Restraint System	YES	NO	**Scaffolding**	YES	NO
DOUBLE LANYARD FOR 100% TIE-OFF	☐	☐	TAGGED	☐	☐
FALL RESTRAINT (INSPECTED)	☐	☐	FIXED SCAFFOLDING ERECTED BY CP	☐	☐
Other Equipment	YES	NO			
LADDERS	☐	☐			
AERIAL LIFT/ MAN LIFT/ SCISSOR LIFT	☐	☐			
RAMPS/STAIRWAYS/STEPS	☐	☐			

KEY ITEMS CHECKLIST

Fall Protection Program	YES	NO	N/A
Has a Competent Person been designated?	☐	☐	☐
Have employees received training (site specific as needed) by a Competent Person?	☐	☐	☐
Is a Qualified Person available for assistance if needed?	☐	☐	☐
Fall Protection Systems	YES	NO	N/A
Are midrails being used with guardrail systems?	☐	☐	☐
Are toeboards being used with guardrail systems?	☐	☐	☐
Will guardrails withstand a 200-pound force from an outward or downward direction?	☐	☐	☐
Are openings on safety nets no greater than 6-inch squares?	☐	☐	☐
Does warning line have a minimum tensile strength of 500 pounds (2,220 N)?	☐	☐	☐
Is warning line capable of supporting, without breaking, loads applied to the stanchions?	☐	☐	☐

Are positioning devices used for fall protection?	☐	☐	☐
Are covers appropriately marked or adequately fastened?	☐	☐	☐
If a fall protection plan is in use, has it been developed by a Qualified Person?	☐	☐	☐
Personal Fall Arrest System	**YES**	**NO**	**N/A**
Are only full body harnesses being used?	☐	☐	☐
Are lanyards with a deceleration device being used?	☐	☐	☐
Are only double-locking safety-type snap hooks being used?	☐	☐	☐
Are anchorage points capable of supporting 5,000 pounds (22.2 kilonewtons)?	☐	☐	☐
Are horizontal lifelines engineered by a Qualified Person?	☐	☐	☐
Are horizontal lifelines designed to support 5,000 pounds for each employee attached?	☐	☐	☐
Is no more than one employee being attached to a single vertical lifeline?	☐	☐	☐
Are personal fall arrest systems being adequately inspected before each use?	☐	☐	☐
Self-Retracting Lifelines	**YES**	**NO**	**N/A**
Do they automatically limit free fall distances to 2 feet?	☐	☐	☐
Are they capable of sustaining a tensile load of 3,000 pounds (13.3 kilonewtons)?	☐	☐	☐
Are self-retracting systems being inspected before and after each use?	☐	☐	☐
Rescue Plan	**YES**	**NO**	**N/A**
Has an effective rescue plan been developed?	☐	☐	☐
Have personnel been trained in the rescue plan?	☐	☐	☐

EMERGENCY CONTACT LIST

Emergency Contact:	**Phone 1:**	**Phone 2:**	**Location:**
Local Police:			
Local Ambulance:			
Local Fire Dept.			
Project Manager:			
Site Manager:			
Client Contact:			
Site Safety Officer:			
H&S Manager:			
Work Care	800-455-6155		

***Include any Task Specific JLA's with this permit.**

KEEP H&S FIRST IN ALL THINGS

I understand the nature of the work for this permit, and certify that this permit meets the requirements specified in the ARCADIS Elevated Work and Fall Protection Standard.

APPROVAL OF ELEVATED WORK PERMIT- By ARCADIS Competent Person:

Name: _____

Title: _____

Office Location: _____

Date: _____

(For Safety Staff only)	REPORT NO.	EROC CODE	UNITED STATES ARMY CORPS OF ENGINEERS ACCIDENT INVESTIGATION REPORT *(For Use of this Form See Help Menu and USACE Suppl to AR 385-40)*	REQUIREMENT CONTROL SYMBOL: CEEC-S-8(R2)

1. ACCIDENT CLASSIFICATION

PERSONNEL CLASSIFICATION	INJURY/ILLNESS/FATAL	PROPERTY DAMAGE	MOTOR VEHICLE INVOLVED	DIVING
GOVERNMENT ☐ CIVILIAN ☐ MILITARY	☐	☐ FIRE INVOLVED ☐ OTHER	☐	☐
☐ CONTRACTOR	☐	☐ FIRE INVOLVED ☐ OTHER	☐	☐
☐ PUBLIC	☐ FATAL ☐ OTHER		☐	

2. PERSONAL DATA

a. Name *(Last, First, MI)*	b. AGE	c. SEX ☐ MALE ☐ FEMALE	d. SOCIAL SECURITY NUMBER	e. GRADE

f. JOB SERIES/TITLE	g. DUTY STATUS AT TIME OF ACCIDENT	h. EMPLOYMENT STATUS AT TIME OF ACCIDENT
	☐ ON DUTY ☐ TDY ☐ OFF DUTY	☐ ARMY ACTIVE ☐ ARMY RESERVE ☐ VOLUNTEER ☐ PERMANENT ☐ FOREIGN NATIONAL ☐ SEASONAL ☐ TEMPORARY ☐ STUDENT ☐ OTHER *(Specify)*

3. GENERAL INFORMATION

a. DATE OF ACCIDENT *(month/day/year)*	b. TIME OF ACCIDENT *(Military time)* hrs	c. EXACT LOCATION OF ACCIDENT	d. CONTRACTOR'S NAME (1) PRIME: (2) SUBCONTRACTOR:

e. CONTRACT NUMBER	f. TYPE OF CONTRACT	g. HAZARDOUS/TOXIC WASTE ACTIVITY
☐ CIVIL WORKS ☐ MILITARY ☐ OTHER *(Specify)*	☐ CONSTRUCTION ☐ SERVICE ☐ A/E ☐ DREDGE ☐ OTHER *(Specify)*	☐ SUPERFUND ☐ DERP ☐ IRP ☐ OTHER *(Specify)*

4. CONSTRUCTION ACTIVITIES ONLY *(Fill in line and corresponding code number in box from list - see help menu)*

a. CONSTRUCTION ACTIVITY	(CODE) #	b. TYPE OF CONSTRUCTION EQUIPMENT	(CODE) #

5. INJURY/ILLNESS INFORMATION *(Include name on line and corresponding code number in box for items e, f & g - see help menu)*

a. SEVERITY OF ILLNESS/INJURY	(CODE) #	b. ESTIMATED DAYS LOST	c. ESTIMATED DAYS HOSPITALIZED	d. ESTIMATED DAYS RESTRICTED DUTY

e. BODY PART AFFECTED	(CODE) #	g. TYPE AND SOURCE OF INJURY/ILLNESS	(CODE) #
PRIMARY		TYPE	
SECONDARY		SOURCE	
f. NATURE OF ILLNESS/INJURY			

6. PUBLIC FATALITY *(Fill in line and correspondence code number in box - see help menu)*

a. ACTIVITY AT TIME OF ACCIDENT	(CODE) #	b. PERSONAL FLOATATION DEVICE USED?
		☐ YES ☐ NO ☐ N/A

7. MOTOR VEHICLE ACCIDENT

a. TYPE OF VEHICLE	b. TYPE OF COLLISION	c. SEAT BELTS	USED	NOT USED	NOT AVAILABLE
☐☐ PICKUP/VAN ☐☐ AUTOMOBILE ☐☐ TRUCK ☐☐ OTHER *(Specify)*	☐ SIDE SWIPE ☐ HEAD ON ☐ REAR END ☐ BROADSIDE ☐ ROLL OVER ☐ BACKING ☐ OTHER *(Specify)*	(1) FRONT SEAT			
		(2) REAR SEAT			

8. PROPERTY/MATERIAL INVOLVED

a. NAME OF ITEM	b. OWNERSHIP	c. $ AMOUNT OF DAMAGE
(1)		
(2)		
(3)		

9. VESSEL/FLOATING PLANT ACCIDENT *(Fill in line and correspondence code number in box from list - see help menu)*

a. TYPE OF VESSEL/FLOATING PLANT	(CODE) #	b. TYPE OF COLLISION/MISHAP	(CODE) #

10. ACCIDENT DESCRIPTION *(Use additional paper, if necessary)*

11. CAUSAL FACTOR(S) *(Read Instruction Before Completing)*

a. (Explain YES answers in item 13)	YES	NO
DESIGN: Was design of facility, workplace or equipment a factor?	☐	☐
INSPECTION/MAINTENANCE: Were inspection & maintenance procedures a factor?	☐	☐
PERSON'S PHYSICAL CONDITION: In your opinion, was the physical condition of the person a factor?	☐	☐
OPERATING PROCEDURES: Were operating procedures a factor?	☐	☐
JOB PRACTICES: Were any job safety/health practices not followed when the accident occurred?	☐	☐
HUMAN FACTORS: Did any human factors such as, size or strength of person, etc., contribute to accident?	☐	☐
ENVIRONMENTAL FACTORS: Did heat, cold, dust, sun, glare, etc., contribute to the accident?	☐	☐

a. *(CONTINUED)*	YES	NO
CHEMICAL AND PHYSICAL AGENT FACTORS: Did exposure to chemical agents, such as dust, fumes, mists, vapors or physical agents, such as, noise, radiation, etc., contribute to accident?	☐	☐
OFFICE FACTORS: Did office setting such as, lifting office furniture, carrying, stooping, etc., contribute to the accident?	☐	☐
SUPPORT FACTORS: Were inappropriate tools/resources provided to properly perform the activity/task?	☐	☐
PERSONAL PROTECTIVE EQUIPMENT: Did the improper selection, use or maintenance of personal protective equipment contribute to the accident?	☐	☐
DRUGS/ALCOHOL: In your opinion, was drugs or alcohol a factor to the accident	☐	☐

b. WAS A WRITTEN JOB/ACTIVITY HAZARD ANALYSIS COMPLETED FOR TASK BEING PERFORMED AT TIME OF ACCIDENT?

☐ YES *(If yes, attach a copy.)* ☐ NO

12. TRAINING

a. WAS PERSON TRAINED TO PERFORM ACTIVITY/TASK?	b. TYPE OF TRAINING.	c. DATE OF MOST RECENT FORMAL TRAINING.
☐ YES ☐ NO	☐ CLASSROOM ☐ ON JOB	(Month) (Day) (Year)

13. FULLY EXPLAIN WHAT ALLOWED OR CAUSED THE ACCIDENT; INCLUDE DIRECT AND INDIRECT CAUSES *(See instruction for definition of direct and indirect causes.) (Use additional paper, if necessary)*

a. DIRECT CAUSE

b. INDIRECT CAUSE(S)

14. ACTION(S) TAKEN, ANTICIPATED OR RECOMMENDED TO ELIMINATE CAUSE(S).

DESCRIBE FULLY:

15. DATES FOR ACTIONS IDENTIFIED IN BLOCK 14.

a. BEGINNING (Month/Day/Year)	b. ANTICIPATED COMPLETION (Month/Day/Year)		
c. SIGNATURE AND TITLE OF SUPERVISOR COMPLETING REPORT CORPS _____ CONTRACTOR _____	d. DATE *(Mo/Da/Yr)*	e. ORGANIZATION IDENTIFIER *(Div, Br, Sect)*	f. OFFICE SYMBOL

16. MANAGEMENT REVIEW *(1st)*

a. ☐ CONCUR b. ☐ NON CONCUR c. COMMENTS

SIGNATURE	TITLE	DATE

17. MANAGEMENT REVIEW *(2nd - Chief Operations, Construction, Engineering, etc.)*

a. ☐ CONCUR b. ☐ NON CONCUR c. COMMENTS

SIGNATURE	TITLE	DATE

18. SAFETY AND OCCUPATIONAL HEALTH OFFICE REVIEW

a. ☐ CONCUR b. ☐ NON CONCUR c. ADDITIONAL ACTIONS/COMMENTS

SIGNATURE	TITLE	DATE

19. COMMAND APPROVAL

COMMENTS

COMMANDER SIGNATURE	DATE

*U.S. GOVERNMENT PRINTING OFFICE: 1993-0-791-757

10.	ACCIDENT DESCRIPTION *(Continuation)*

13a.	DIRECT CAUSE *(Continuation)*

13b.	INDIRECT CAUSES *(Continuation)*

14.	ACTION(S) TAKEN, ANTICIPATED, OR RECOMMENDED TO ELIMINATE CAUSE(S) *(Continuation)*

Appendix D

PPE Equipment List

PPE CHECKLIST

This is a comprehensive list of all PPE that is required for the project. Refer to the AHA for task specific PPE requirements. Subcontractors must have the same equipment listed as a minimum.

Description **(Specify Material or Type in Box)**	**Level Of Protection** **R = Required O = Optional**	
	D	**C**
Body		
Coveralls	O	R
Chemical Protective Suit		R
Splash Apron		
Rain Suit	O	O
Traffic Safety Vest (reflective)	R	R
Head		
Hard Hat (if does not create other hazard)	R	R
Head Warmer (depends on temperature and weather	O	O
Eyes & Face		
Safety Glasses (incorporate sun protection as necessary)	R	R
Goggles (based on hazard)		
Splash Guard (based on hazard)		
Ears		
Ear Plugs	O (as required)	
Ear Muffs	O	
Hands and Arms		
Outer Chemical Resistant Gloves	O	R
Inner Chemical Resistant Gloves (i.e. Nitrile)	R	R
Insulated Gloves	O	O
Work Gloves	O	O
Foot		
Safety Boots (steel toe and shank)	R	R
Rubber, Chemical Resistant Boots	R (near waterways)	R
Rubber Boots		
Disposable Boot Covers		O
Respiratory Protection (indicate cartridge type where applicable)		
Dust Protection	O	R
1/2 Mask APR	O (take with you)	R
Full Face APR	O (take with you)	O
Full Face Canister APR		
Powered APR		
Other Supplies		

Description (Specify Material or Type in Box)	Level Of Protection R = Required O = Optional	
	D	C
First Aid Kit	R	R
Fire Extinguisher	R	R
Mobile Phone	R	R
Traffic Cones	R	R
Walkie Talkies	O	O
Water or Other Fluid Replenishment	O	R
Eye Wash Station		R
Eye Wash Bottle	R	R
Wash and Dry Towelettes	R	R
Sunscreen (SPF 15 or higher)	O	O
Insect Repellant	O	O

Appendix E

Alcohol and Drug Abuse Program

Prevention of Alcohol and Drug Abuse

The purpose of this policy is to promote a safe, healthy and productive work environment for all employees free from the affects of substance abuse. To ensure a safe and productive work environment, the company prohibits the use, sale, dispersion, manufacture, distribution or possession of alcohol, drugs, controlled substances or drug paraphernalia on the Project Site including company owned or leased equipment and vehicles.

Abuse of alcohol, drugs and controlled substances impairs employee's judgment resulting in increased safety risks, injuries and faulty decision making.

No employee will report to work or be at work with alcohol or any detectable amount of prohibited drugs in the employees system, (a detectable amount refers to the standards generally used in workplace drug and alcohol testing).

Personnel will, when drugs are prescribed by a medical professional, inquire of the prescribing professional whether the drug prescribed has any side effects that may impair the employee's ability to safely perform the employee's job duties. If the answer from the medical professional is yes, the employee will obtain a statement and provide to the project manager prior to going to the site.

Illegal use of drugs off duty and off company premises or worksites is not acceptable. It can affect on-the-job performance and the confidence of the public and our customers in the company's ability to meet its responsibilities.

Any violation of this policy will result in disciplinary action, up to and including termination.

Appendix F

Night Operations Plan

Night Operations Lighting Plan

The key issue facing personnel working on-site at night will be ensuring sufficient light for the work areas. Illumination for all site operations, including any work activities conducted at night, are required to meet OSHA Regulation 1926.56(a). When night time work is being performed, mobile floodlights and/or balloon lights will be used to illuminate the work area, flagger stations, equipment crossings, and other areas.

The SSHO shall have a light meter, calibrated and maintained in accordance with manufacturer's instructions, on-site when construction activities are conducted at night in order to ensure compliance with OSHA requirements for illumination on construction sites.

Construction areas, ramps, runways, corridors, offices, shops, and storage areas shall be lighted to not less than the minimum illumination intensities listed below in Table 1 while any work is in progress:

TABLE 1 – MINIMUM ILLUMINATION INTENSITIES

FOOT-CANDLES	AREA OF OPERATION
5	General construction area lighting
3	General construction areas, concrete placement, excavation and waste areas, access ways, active storage areas, loading platforms, refueling, and field maintenance areas.
5	Indoors: warehouses, corridors, hallways, and exitways.
5	Tunnels, shafts, and general underground work areas: (Exception: Minimum of 10 foot-candles is required at tunnel and shaft heading during drilling, mucking, and scaling. Bureau of Mines approved cap lights shall be acceptable for use in the tunnel heading.)
10	General construction plant and shops (*e.g.,* batch plants, screening plants, mechanical and electrical equipment rooms, carpenter shops, rigging lofts and active store rooms, mess halls, and indoor toilets and workrooms.)
30	First aid stations, infirmaries, and offices.

Desired illumination levels vary depending upon the nature of the task involved. An average horizontal luminance of 5 foot candles can be adequate for general activities. Tasks requiring high levels of precision and extreme care can require an average horizontal luminance of 20 foot candles.

Except in emergency situations, flagger stations shall be illuminated at night.

Floodlighting shall not produce a disabling glare condition for approaching road user, flaggers, or workers.

The adequacy of the floodlight placement and elimination of potential glare should be determined by the SSHO by driving through and observing the floodlighted area from each direction on all approaching roadways after the initial floodlight setup, at night, and periodically.

Any personnel working outside the lights will be directed to return to a lighted area or the operation shall be stopped.

All vehicles utilized at night, including heavy equipment, backhoes, trenching machines, etc, are required to have two working headlights and taillights.

Due to limited visibility at night, a signaler is required for all backing operations.

Any stockpiles or construction equipment outside the active lighted work zone shall be delineated with cones or barrier fencing.

All personnel working at night are required to wear, at a minimum, ANSI Class 2 traffic vests, hard hats, and clear safety glasses.

Appendix G

Hazardous Energy Control Plan

Equipment Specific LOTO Procedure

LOCKOUT / TAGOUT PROCEDURE

PIKA-PIRNIE Office:	Greenville, South Carolina		
Written by (Name/Job Title):	Kevin Held/Scientist	Date Written:	3 Dec. 2011
Revised By (Name/Job Title):		Date Revised:	

INTRODUCTION

This procedure is specific to machines/equipment with an energy source and covers the safety rules and procedures to follow while installing, servicing or performing maintenance on any equipment or machines in which unexpected energization or start up, or release of stored energy could cause injury to employees. This procedure includes the following machine(s)/equipment(s):

- Main Electric Supply Cut-Off
- Electric Powered Pumps and Equipment

The circuits that energize the equipment or machines will be locked with a personally assigned lock and a disposable tag per the PIKA/PRINIE Control of Hazardous Energy (Lockout/Tagout) Standard-ARC HSFS004.

PURPOSE

This procedure establishes the minimum requirements for the lockout of energy isolating devices whenever maintenance or servicing is performed on machines or equipment. It shall ensure that the machine or equipment is stopped, isolated from all potentially hazardous energy sources and locked out before any servicing or maintenance where the unexpected energization or start-up of the machine or equipment or releases of stored energy could cause injury.

COMPLIANCE

All employees are required to comply with the limitations and restrictions imposed on them during the use of this lockout/tagout procedure. The authorized employees are required to perform the lockout/tagout in accordance with this standard. All employees, upon observing a machine or piece of equipment which is locked/tagged out to perform servicing or maintenance shall not attempt to use that machine or equipment. This standard is written in accordance with the Occupational Safety and Health Administration (OSHA) Standard 1910.147.

Any person who willfully violates this standard is subject to disciplinary action including termination.

Prior to removing a **machine/equipment** from service for servicing or maintenance the following steps shall be taken:

1. Notify all affected employees that servicing or maintenance is required on a machine or piece of equipment, and that the machine or equipment must be shut down and locked/tagged out to perform the servicing or maintenance.

2. The authorized employees (*e.g.,* Site Manager) shall refer to the manufacturer's manual to identify the type and magnitude of the energy source that the machine or equipment utilizes, shall understand the hazards of the energy source, and shall know how to control the energy.

3. If the machine or equipment is operating, shut it down through the normal procedures as specified in the manufacturer's manual.

4. Deactivate the electrical energy isolating device so that the machine or equipment is isolated from the energy source. Most equipment and machines that require this procedure have a separate circuit box that can be locked/tagged out for that specific piece of equipment or machine.

5. Lock/tag out the energy isolating device with an assigned individual lock and disposable red tag. The Equipment Operator/Technician will place the first lock followed by the SSHO or designee.

6. Any stored or residual electrical energy must be dissipated or restrained.

7. Ensure that the equipment is disconnected from its energy source. Make sure no personnel are or will be exposed; then verify the isolation of the equipment by attempting to operate through the normal controls. CAUTION: The operating controls must be returned to the neutral or "off' position after verification of isolation.

8. The machine/equipment is now locked/tagged out and servicing or maintenance can proceed.

RESTORING EQUIPMENT TO SERVICE

1. After servicing or maintenance is completed and the machine/equipment is ready to be returned to normal operation, the following steps shall be taken.

    a. Check the machine/equipment and the area immediately around the machine/equipment to ensure that non-essential items have been removed and the machine/equipment and/or components are operationally intact.

    b. Check the area to ensure that all non-essential personnel are in a safe place or are well clear of the area.

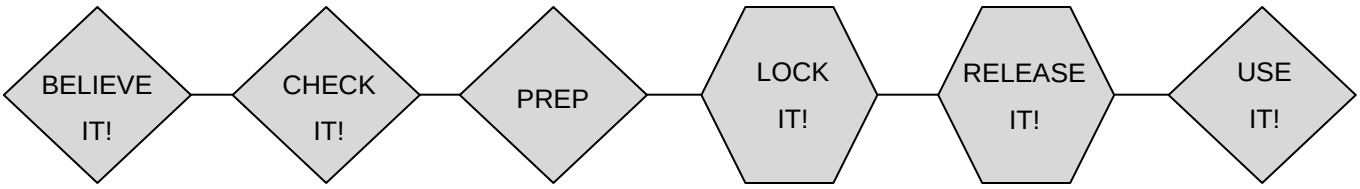
    c. Verify that all operating controls are in the "off" position or are in neutral.

    d. Remove the lock/tag out devices. The Equipment Operator/Technician will remove his lock first followed by the SSHO.

    e. Notify affected employees that the servicing or maintenance has been completed and that the machine/equipment is ready for use.

    f. Re-energize the machine/equipment.

Lockout / Tagout Equipment-Specific Energy Control Procedure



Equipment Identification:					
Hazardous Energy Source		**Isolation Device**			**Verifying Lockout**
Type and Magnitude	**Function**	**Type**	**Location**	**I.D. No.**	**Means of Verification of Lockout**
Electrical (*i.e.,* 120, 220, 480)					
Pneumatic					
Hydraulic					
Mechanical					
Potential Energy (springs, tension, etc.)					
Gravity					
Chemical					
Other					
Area:		**Date of Last Review:**			**Authorized by:**

Appendix H

Excavation and Trenching Plan

1. POLICY

It is PIKA-PIRNIE policy to be proactive in the identification, assessment and control of health and safety hazards and associated risks. To those means, any work involving trenching and excavation that is under the control or direction of PIKA-PIRNIE or an PIKA-PIRNIE subcontractor will be accomplished following, at a minimum, this procedure.

It is PIKA-PIRNIE's policy that PIKA-PIRNIE staff will not enter excavations and trenches unless it is absolutely necessary. If there are no suitable alternatives and it becomes necessary to enter excavations or trenches, this procedure, at a minimum will be strictly followed.

It is also the policy of PIKA-PIRNIE to ensure an OSHA-defined Excavation Competent Person is on-site for all excavation work under PIKA-PIRNIE contractual control. The competent person will be provided by the entity on site responsible for performing the excavation work unless otherwise required by the client. Thus, if an PIKA-PIRNIE subcontractor is conducting the excavation work, that subcontractor will provide the competent person. If PIKA-PIRNIE is self-performing the excavation services, then PIKA-PIRNIE will provide a competent person whether a specialized subcontractor or authorized employee.

2. PURPOSE AND SCOPE

2.1 Purpose

To effectively control or eliminate the hazards presented by working near or entry into excavations or trenches, this procedure sets forth the accepted practice for and establishes the requirements for workplace safety near excavations and trenches and employee and subcontractor entry into such.

2.2 Scope

This procedure along with associated checklists and the Utility Location procedure (ARC HSFS019) apply to all employees of PIKA-PIRNIE. Only trained and authorized personnel are permitted to work near or enter excavations and trenches, perform rescue services, or act as the excavation competent person.

3. DEFINITIONS

Exhibit 1 includes relevant definitions to this procedure including that for competent person qualifications.

4. RESPONSIBILITIES

4.1 Corporate H&S with Division and Practice Experts

On an annual basis, review and update, as necessary, this procedure. In addition, review cancelled checklists periodically to ensure conformance to this procedure. Provide the excavation competent person and qualified person training and retraining, or recommend qualified training provider. Provide technical assistance regarding excavation and trench protocol, atmospheric testing equipment, PPE, hazard assessment and research

information on unusual hazards. Audit project-specific excavation sites for compliance with this procedure.

4.2 Principal in Charge (PIC), Project Manager (PM), and Task Manager (TM)

PIC, PM and TMs are responsible to:

- Verify that all excavation and trench protocols are properly identified and addressed within the project work plan, project health & safety plan, and/or other project-related documents.
- Verify that their divisional or project team employees have received the proper training provided by Corporate Health & Safety or qualified training source prior to conducting excavation/trenching entry activities.
- Verify that any PIKA-PIRNIE employee acting as the Excavation Competent person has been authorized and trained to do so as noted in Exhibit 1
- Verify that the proper entry equipment, including personal protective equipment (PPE), atmospheric testing equipment and safety equipment, is available for use by their divisional employees.
- Verify that copies of the completed checklists are available for Corporate Health and Safety review and retained with the project files

4.3 Health and Safety Plan Writers and Reviewers

Utilize this procedure as guidance to ensure the appropriate identification, assessment and control of excavation and trenching hazards for documentation in project HASPs

4.4 Entry/Work Supervisors (also see Training and Duties of Entry Supervisor)

- Work in direct coordination with and under the direction of the project excavation competent person
- Interface with the client representative to identify hazards associated with the client's excavation and trenching and/or work permit programs.
- Review existing soil sampling (if any) data or other pertinent hazard characterization information recorded by the client.
- Investigate the client's excavation/trenching protocol, to verify that any identified hazards and previous experience with earthwork at the site is properly communicated.
- Coordinate entry operations with the client's employees when both client and PIKA-PIRNIE employees will be working in or near an excavation/trench.
- Coordinate necessary rescue assistance with either the client's in-house rescue team and/or the offsite rescue assistance specified by the client. The offsite rescue

assistance specified by the client must have applicable rescue experience and be within a reasonable response distance.

- Verify that the client takes the necessary precautions in notifying their employees that our employees will be installing an excavation or trench.
- Review the lockout/tagout and isolation measures implemented by the client as necessary based on proximity of utilities or other energy sources in the area of the excavation/trench
- Immediately report any unusual or unforeseen excavation or trenching hazards to Corporate Health and Safety prior to authorizing entry
- Verify that all tests and precautionary measures identified on the Daily/Periodic Inspection Checklist located in Exhibit 1 and the PIKA-PIRNIE Utility Location Policy and Procedure ARC HSFS019 has been performed prior to authorizing subsurface work or entry into an excavation or trench
- Offer all entrants an opportunity to review the applicable control measures and testing results and an opportunity to request a reevaluation as necessary
- Issue, authorize, and have the Utility Clearance and Daily/Periodic Inspection forms readily available for review
- Verify that copies of the completed clearance forms and checklists are properly disseminated to Corporate Health and Safety and retained with the project files, as specified in Section 8.0 – Records.

4.5 Entrants

- Qualified Employee Entrants must have training and instruction in their duties and responsibilities regarding the following:
- Recognize the hazards which may be faced during entry, as well as the signs and symptoms of exposure to the hazard(s).
- Maintain visual contact and/or verbal communications with the attendant at all times.
- Use the PPE, air monitoring and testing equipment that has been provided or have access to the information.
- Maintain an awareness of all required hazard controls and consult with the Competent Person as necessary
- Obey evacuation orders given by the Attendant, automatic alarm activation, or when self-perceived.

4.6 Competent Person

- The Competent Person will be responsible for the anticipation, identification and control of excavation and trenching hazards, as well as the signs and symptoms of

exposure to the hazard(s), and the Authority to implement all corrective actions including Stopping Work.

- Meet all of the requirements specified for the Qualified Employee Entrants plus adequate training and experience for their duties and responsibilities.
- Implement the PIKA-PIRNIE Utility Clearance Policy and Procedure and complete the Daily/Periodic Excavation Inspection Checklist
- Verify adequate training and experience of all Entrants prior to entry
- Verify that the safety procedures identified in this Standard, the site specific HASP, and applicable regulatory requirements are utilized when required to protect employees during excavation activities.

PIKA-PIRNIE employees must meet the following requirements to be considered a Competent Person:

- Attend an Excavation Competent Person training course approved by Corporate Health and Safety or have equivalent training to that provided in the course; and
- Approval by Corporate Health and Safety through demonstration of practical field experience and/or knowledge of the subject matter.
    - If on an Environmental project where HAZWOPER training is required by PIKA-PIRNIE, completed a 40 Hour HAZWOPER and HAZWOPER Supervisor training course and be current on their annual 8 Hour refresher.
    - If a hazardous atmosphere is present, or there is limited entry or exit and the excavation or trench must be entered as a confined space, the person must also be Confined Space trained and authorized as per the PIKA-PIRNIE Confined Space procedure ARC HSFS003.

4.7 Attendants

- An attendant must be stationed outside the excavation and be available to monitor operations above and below ground. The attendant may have no other duties besides those listed in this section.
- All attendants must have training and instruction in their duties and responsibilities regarding excavation/trenching entry. The following are assigned duties.
- Maintain an accurate count of all entrants in the excavation
- Monitor activities both inside and outside the excavation/trench to verify the continued safety of entrants
- Maintain visual contact or verbal communication with all entrants

- Order evacuation of the excavation/trench if an uncontrolled hazard develops, either within or outside the space, or upon observing a behavioral effect of hazard exposure among entrants
- Keep unauthorized persons away from the excavation area
- Participate in non-entry rescue as appropriate
- Summon rescue and other emergency services
- Attendants must maintain current certification in basic first aid and cardiopulmonary resuscitation (CPR).

4.8 All PIKA-PIRNIE Employees

Use the TRACK process described below regularly and frequently. In addition, employees read and understand all documented hazard identification and risk assessments conducted using the HARC process and documented in HASPs, JSAs, and other written plans that are associated with their work. PIKA-PIRNIE employees will:

- Recognize the hazards of trenches and excavations
- Understand and follow the methods for working near trenches and excavations
- Notify the PIC, PM, TM or entry/work supervisor if they have not received appropriate training
- Participate in entry operations only if trained and authorized to do so
- Never enter an excavation/trench without completion of the required Utility Location Procedure, Daily/Periodic Inspection Checklist and have an authorized attendant
- Never attempt entry rescue within a excavation unless trained in entry rescue with appropriate equipment available
- If unexpected conditions arise during entry, immediately notify other entrants, evacuate the space and inform the designated Competent Person

5. PROCEDURE

5.1 General Safety Requirements for all Excavations

- All surface obstructions must be moved or supported so as to protect employees and equipment.
- Prior to excavation, all underground installations (water, electric, telephone, gas, etc.) must be located and documented in accordance with PIKA-PIRNIE Utility Clearance Policy and Procedure ARC HSFS019.

- When excavating in areas near underground installations, proper precautions must be taken to determine the exact location of the installations and to adequately protect and support them. While an excavation is open, underground installations shall be protected, supported or removed as necessary to protect employees.

- Structural ramps that are used solely by employees as a means of access or egress from excavations shall be designed by a competent person.

- Structural ramps used for access or egress of equipment shall be designed by a competent person qualified in structural design, and shall be constructed in accordance with the design.

- Ladders used for access and egress from the excavation must extend at least 36” (3 feet) above the landing surface.

- If personnel are working in a location exposed to vehicular traffic they must be provided with and be required to wear reflective safety vests. Adequate, signs, barriers or other equivalent traffic controls must be used to protect employees.

- Personnel are not permitted to be beneath elevated loads handled by equipment or be in excavations when heavy equipment is digging in or near the excavation.

- Mobile equipment located near open excavations must be adequately protected from falling or rolling into excavations by the use of barricades or warning devices.

- All excavations over 4 feet in depth must be tested for hazardous atmospheres whenever personnel are required to enter and a potential exists for the existence of hazardous contaminants or oxygen deficiency. Excavations less than 4 feet in depth must be evaluated by the competent person and at the competent person's discretion be tested for hazardous atmospheres whenever personnel are required to enter and a potential exists for the existence of hazardous contaminants or oxygen deficiency.

- Means of rescue including a lifeline and body harness must be used by personnel entering excavations with a potential for air hazards. A standby person must be stationed outside the excavation to tend the lifeline(s).

- Water must not be allowed to accumulate in open excavations where employees are working. When necessary, means such as diverting natural drainage around the excavation or actively pumping water must be used to prevent or control water accumulation.

- All structures adjacent to an open excavation must be supported, or a registered professional engineer (PE) must determine that the structure will not be affected by the excavation activities.

- Excavated materials (spoil) must be placed no closer than 2 feet from the edge of an open excavation, and otherwise retained to prevent loose material from falling into the excavation.

- Protection such as guardrails, barricades or covers must be in place to protect personnel from possible falls into open excavations, pits, wells and shafts.

- Work tasks will be designed to limit the number of personnel required to enter any excavation. All tasks that can be completed remotely from outside the excavation (such as soil sampling) will be conducted in such a manner.

- Personnel will not be allowed to enter any excavation unless adequate protective systems and procedures are utilized to prevent accidents and injury.

- All excavations over four feet in depth shall be provided with a stairway, ladder, ramp, or other safe means of egress so as to require no more than 25 feet of lateral travel. As deemed necessary by the competent person, excavations less than 4 feet in depth will be provided with a stairway, ladder, ramp, or other safe means of egress so as to require no more than 25 feet of lateral travel.

5.2 Excavations Requiring Protective Systems

This section defines excavations that require protective systems.

- All excavations into which employees will enter, regardless of depth, where the potential for cave-in exists.

- Any excavation over 4 feet in depth into which employees will enter that is not entirely in stable rock as defined in this procedure.

- Any excavation near a structure, (e.g. foundations, piers, footers, walls, sidewalks, tanks, roadways, etc.), as required by the registered professional engineer reviewing the stability of the excavation and the structure.

- All excavations over 20 feet in depth must be designed by a registered professional engineer regardless of whether personnel will enter it or not.

- All excavations with adjacent structures which are located a distance less than 6 times the depth of the excavation away shall be reviewed by a registered professional engineer to determine if the stability of the structure will be affected by the excavation.

- Support systems for an adjacent structure must be designed by a registered professional engineer.

5.3 Selection and Use of Protective Systems

5.3.1 Shoring or Shielding

- If shoring or shielding is selected as the protective system for an excavation, soil classification in accordance with 1926 Subpart P Appendix A (see Section 9 of this procedure) is required.

- One of the following options must be utilized for all excavations which will be shored or shielded.

    - Timber shoring as specified in 1926 Subpart P Appendix C must be utilized

    - Hydraulic shoring, trench jacks, air shores, or shields as required in 1926.652 (c)(2) must be utilized following the system manufacturer's data

    - A system which follows other tabulated data (approved by a registered professional engineer) must be utilized

    - The excavation must be designed by a registered professional engineer

5.3.2 Sloping

- If sloping is selected as the protective system for an excavation, the excavation sides must be sloped at a maximum of 34 degrees (1.5 Horizontal: 1 Vertical), unless the procedure listed above is followed.

- Soil classification in accordance with Section 10 of this procedure) is required for all excavations with sides which will be sloped greater than 34^{o} (1.5 Horizontal: 1 Vertical). If it will be sloped greater than 34^{o}, the one of the following options must be utilized:

    - Option 1 - assume Type C and slope 1.5/1 - probably the most common and preferred method for us

    - Option 2 - classify soil according to the standard and use Type A/B sloping requirements

    - Option 3 – use other tabulated data with PE approval

    - Option 4 – PE approval of sloping/benching design

5.4 Atmospheric Testing for Entry

Any excavation over 4 feet in depth with a potential for hazardous contaminants or oxygen deficiency must be tested for hazardous atmospheres prior to and during activities involving entry. After atmospheric testing, if the area is found to be oxygen deficient or a hazardous atmosphere exists or could exist a confined space permit must be obtained if the area will be entered.

The site designated "competent person" will document initial and periodic air monitoring results for all activities requiring entry into the excavation. All atmospheric testing of excavations must be conducted in the following sequence and meet the following air quality criteria.

- Oxygen content must be 19.5 to 23.5%
- Combustible gas or vapor must not exceed 10% of its lower explosive limit (LEL)
- Toxic air contaminant levels must not exceed 50% of the PEL or TLV for the specific contaminant whichever is lower
- Carbon monoxide must not exceed 10 ppm for a 5 minute average or ceiling value of 25 ppm
- Hydrogen sulfide must not exceed 0.5 ppm

5.5 Location of Underground/Overhead Utilities

- The competent person and the project manager shall both verify that local underground facilities location/protection agencies are notified within the required time frame prior to the initiation of excavation activities and meet all requirements in the PIKA-PIRNIE Utility Location Policy and Procedure ARC HSFS019.
- Prior to initiation of excavation or trenching operations the competent person shall verify that all utilities have been located.

5.6 Daily/Periodic Inspections

- Prior to initiation of daily excavation or trenching operations the competent person shall complete a daily inspection of the excavation.
- During excavation or trenching operations the competent person shall complete a periodic inspection after any event (e.g., thunderstorm, vibration, excessive drying) that may affect excavation stability.
- The competent person shall complete the daily/periodic inspection checklist (A copy of the checklist is attached to this Policy as Exhibit A– Subcontractors must complete an equivalent inspection form) is completed for each inspection of excavation and trenching activities.

5.7 Soil Classification for Selection of Protective Systems

5.7.1 Soil Classification

This section describes a method of classifying soil and rock deposits based on site and environmental conditions, and on the structure and composition of the earth deposits. This

section contains definitions, sets forth requirements, and describes acceptable visual and manual tests for use in classifying soils.

This section applies when a sloping, benching or shoring system is utilized as a method of protection for employees from cave-ins.

5.7.2 Soil Classification Definitions

5.7.2.1 Types/Classes of Soil

Type/Class A Soils are cohesive soils with an unconfined, compressive strength of 1.5 ton per square foot (tsf) (144kPa) or greater. Examples of cohesive soils are: Clay, silty clay, sandy clay, clay loam and in some cases, silty clay loam and sandy clay loam. Cemented soils such as caliche and hardpan are also considered Type A. However, no soil is Type A if the following apply.

- The soil is fissured
- The soil is subject to vibration from heavy traffic, pile driving, or similar effects
- The soil has been previously disturbed
- The soil is part of a sloped, layered system where the layers dip into the excavation on a slope of four horizontal to one vertical (4 Horizontal:1 Vertical) or greater
- The material is subject to other factors that would require it to be classified as a less stable material

5.7.2.1.1 Type Class B Soils

- Cohesive soils with an unconfined compressive strength greater than 0.5 tsf (48 kPa) but less than 1.5 tsf (144 kPa)
- Granular cohesionless soils including angular gravel (similar to crushed rock), silt, silt loam, sandy loam and, in some cases, silty clay loam and sandy clay loam
- Previously disturbed soils except those which would otherwise be classed as Type C soil
- Soil that meets the unconfined compressive strength or cementation requirements for Type A, but is fissured or subject to vibration
- Dry rock that is not stable
- Material that is part of a sloped, layered system where the layers dip into the excavation on a slope less steep than four horizontal to one vertical (4 Horizontal:1 Vertical), but only if the material would otherwise be classified as Type B

5.7.2.1.2 Type/Class C Soils

- Cohesive soil with an unconfined compressive strength of 0.5 tsf (48 kPa) or less
- Granular soils including gravel, sand, and loamy sand
- Submerged soil or soil from which water is freely seeping
- Submerged rock that is not stable
- Material in a sloped, layered system where the layers dip into the excavation or a slope of four horizontal to one vertical (4 Horizontal:1 Vertical) or steeper

5.7.2.2 Methods for Classifying Soils

Each soil and rock deposit shall be classified by a competent person as Stable Rock, Type A, Type B, or Type C in accordance with the definitions set forth in this section. The classification of the deposits shall be made based on the results of at least one visual and at least one manual analysis conducted by a competent person using tests described below, or in other recognized methods of soil classification and testing such as those adopted by the American Society for Testing Materials, or the U.S. Department of Agriculture textural classification system.

The visual and manual analyses, such as those noted as being acceptable in this section, shall be designed and conducted to provide sufficient quantitative and qualitative information as may be necessary to identify properly the properties, factors, and conditions affecting the classification of the deposits. Visual analysis is conducted to determine qualitative information regarding the excavation site in general, the soil adjacent to the excavation, the soil forming the sides of the open excavation, and the soil taken as samples from excavated material.

Observe the following:

- Samples of soil that are excavated and soil in the sides of the excavation. Estimate the range of particle sizes and the relative amounts of the particle sizes. Soil that is primarily composed of fine grained material is cohesive material. Soil composed primarily of coarse grained sand or gravel is granular material.
- Soil as it is excavated. Soil that remains in clumps when excavated is cohesive. Soil that breaks up easily and does not stay in clumps is granular.
- The side of the open excavation and the surface area adjacent to the excavation. Crack like openings such as tension cracks could indicate fissured material. If chunks of soil spall off a vertical side, the soil could be fissured. Small spalls are evidence of moving ground and are indications of potentially hazardous situations.

- The area adjacent to the excavation and the excavation itself for evidence of existing utility and other underground structures, and to identify previously disturbed soil.

- The open side of the excavation to identify layered systems. Examine layered systems to identify if the layers slope toward the excavation. Estimate the degree of slope of the layers.

- The area adjacent to the excavation and the sides of the opened excavation for evidence of surface water, water seeping from the sides of the excavation, or the location of the level of the water table.

- The area adjacent to the excavation and the area within the excavation for sources of vibration that may affect the stability of the excavation face.

Manual analysis of soil samples is conducted to determine quantitative as well as qualitative properties of soil and to provide more information in order to classify soil properly.

5.7.2.3 Classifications

A. Plasticity. Mold a moist or wet sample of soil into a ball and attempt to roll it into threads as thin as 1/8 inch in diameter. Cohesive material can be successfully rolled into threads without crumbling. For example, if at least a two inch (50 mm) length of 1/8 inch thread can be held on one end without tearing, the soil is cohesive.

B. Dry strength. If the soil is dry and crumbles on its own or with moderate pressure into individual grains or fine powder, it is granular (any combination of gravel, sand, or silt). If the soil is dry and falls into clumps which break up into smaller clumps, but the smaller clumps can only be broken up with difficulty, it may be clay in any combination with gravel, sand or silt. If the dry soil breaks into clumps which do not break up into small clumps and which can only be broken with difficulty, and there is no visual indication the soil is fissured, the soil may be considered unfissured.

C. Thumb penetration. The thumb penetration test can be used to estimate the unconfined compressive strength of cohesive soils. Type A soils with an unconfined compressive strength of 1.5 tsf can be readily indented by the thumb; however, they can be penetrated by the thumb only with very great effort. Type C soils with an unconfined compressive strength of 0.5 tsf can be easily penetrated several inches by the thumb, and can be molded by light finger pressure. This test should be conducted on an undisturbed soil sample, such as a large clump of spoil, as soon as practicable after excavation to keep to a minimum the effects of exposure to drying influences. If the excavation is later exposed to wetting influences (rain, flooding), the classification of the soil must be changed accordingly.

D. Other strength tests. Estimates of unconfined compressive strength of soils can also be obtained by use of a pocket penetrometer or by using a hand operated shearvane.

E. Drying test. The basic purpose of the drying test is to differentiate between cohesive material with fissures, unfissured cohesive material, and granular material. The procedure for the drying test involves drying a sample of soil that is approximately one inch thick (2.54 cm) and six inches (15.24 cm) in diameter until it is thoroughly dry:

1. If the sample develops cracks as it dries, significant fissures are indicated.

2. Samples that dry without cracking are to be broken by hand. If considerable force is necessary to break a sample, the soil has significant cohesive material content. The soil can be classified as an unfissured cohesive material and the unconfined compressive strength should be determined by using the thumb penetration or other test.

5.7.2.4 If a sample breaks easily by hand, it is either a fissured cohesive material or a granular material. To distinguish between the two, pulverize the dried clumps of the sample by hand or by stepping on them. If the clumps do not pulverize easily, the material is cohesive with fissures. If they pulverize easily into very small fragments, the material is granular.

5.7.2.5 Layered system

A layered system shall be classified in accordance with its weakest layer. Each layer may be classified individually where a more stable layer lies under a less stable layer.

5.7.2.6 Reclassifying Soils

A layered system shall be classified in accordance with its weakest layer. Each layer may be classified individually where a more stable layer lies under a less stable layer.

In most instances the PIKA-PIRNIE designated Excavation/Trenching Competent person will assume Type C soil, unless they have conclusive data to validate Type A or B.

5.7.2.7 Excavation Construction Based on Soil Type

The Maximum allowable slope means the steepest incline of an excavation face that is acceptable for the most favorable site conditions as protection against cave-ins, and is expressed as the ratio of horizontal distance to vertical rise (H:V). Short-term exposure means a period of time less than or equal to 24 hours that an excavation is open. Soil and rock deposits must be classified in accordance with Appendix A to Subpart P of Part 1926. The maximum allowable slope for a soil or rock deposit must be determined from the table provided below. The actual slope

must not be steeper than the maximum allowable slope. The actual slope must be less steep than the maximum allowable slope, when there are signs of distress. If that situation occurs, the slope must be cut back to an actual slope which is at least horizontal to one vertical (1/2H:1V) less steep than the maximum allowable slope. When surcharge loads from stored material or equipment, operating equipment, or traffic are present, a competent person must determine the degree to which the actual slope must be reduced below the maximum allowable slope, and must assure that such reduction is achieved. Surcharge loads from adjacent structures must be evaluated in accordance with 1926.651(I). Configurations of sloping and benching systems must be in accordance with 29 CFR 1926 Subpart P, Appendix B.

EXCAVATION SLOPE TABLE 2 29 CFR 1926 SUBPART P APPENDIX B MAXIMUM ALLOWABLE SLOPES	
Soil or Rock Type	**Maximum Allowable Slopes (H:V)[1] for Excavations Less Than 20 Feet Deep[2]**
Stable Rock	Vertical (90 degrees)
Type A[3]	¾:1 (53 degrees)
Type B	1:1 (45 degrees)
Type C	1:½ (34 degrees)

1. Numbers shown in parentheses next to maximum allowable slopes are angles expressed in degrees from the horizontal. Angles have been rounded off.
2. Sloping or benching for excavations greater than 20 feet deep must be designed by a registered professional engineer.
3. A short-term maximum allowable slope of 1/2H:1V (63 degrees) is allowed in excavations in Type A soil that are 12 feet (3.67 m) or less in depth. Short-term maximum allowable slopes for excavations greater than 12 feet (3.67 m) in depth must be 3/4H:1V (53 degrees).

6. TRAINING

6.1 Project - Specific Training

All staff working on a site where trenching and excavation activities are being conducted by PIKA-PIRNIE or its subcontractors will be provided with site orientation on excavation projects, and shall include a discussion of the following:

- Site excavation hazards and procedures
- Requirements for conducting activities remotely whenever possible
- Client requirements and procedures for excavation activities
- This Procedure

Daily Safety Meetings on projects involving excavation activities shall include a discussion of:

- Site excavation hazards and procedures
- Requirements for conducting activities remotely whenever possible
- Client requirements and procedures for excavation activities
- This Excavation and Trenching Procedure, as appropriate

6.2 Additional Training

Besides site orientation training, additional training will be provided as follows based on the employee's activities:

- All employees who work in the area of potential excavation/trenching sites will receive awareness level training as provided and/or approved by PIKA-PIRNIE Corporate H&S in order to recognize and to understand the hazards.
- Entrants, Attendants, and Entrant Supervisors will receive additional training as approved by Corporate H&S. This training will be classroom in nature and cover the details of trenching and excavation hazards and controls
- Qualified Competent Persons will be provided training as follows:

  In order to be assigned duties as a competent person with respect to excavation and trenching, in addition to the criteria noted in section 4.6, personnel must attend an Excavation Competent Person training course approved by Corporate Health and Safety or have equivalent training to that provided in the course. The course shall include, but is not limited to the following:

  - Introduction to trenches and excavations
    - Definition of trenches and excavations
    - General requirements of OSHA 29 CFR 1926 Subpart P
  - Responsibilities and requirements of a competent person
    - Necessary authority
    - When other/outside resources may be necessary
  - Hazard Identification and Assessment
    - Cave-In Hazards including nearby structures
    - Underground utilities
    - Confined Space
    - Hazardous atmospheres

- Water accumulation
- Vehicular traffic and falling loads

- Hazard controls
  - Soil analysis and testing (visual and manual
  - Protective systems
    - Shoring
    - Sloping
    - Shielding
    - Benching
  - Personal protective equipment
  - Utility location
  - Atmospheric testing
  - Water drainage and pumping
  - Site housekeeping and management
    - Spoils
    - Traffic control
    - Overhead hazard protection
  - Communications
    - Verbal
    - Signaling
  - Access and egress
- Emergency Procedures
  - Warning signs of cave-in
  - Evacuation procedures
  - Rescue
- Inspections
  - Checklists

- Potential deficiencies

All training provided must be reviewed and approved by Corporate Health & Safety and will be managed through the Training Team.

Documentation of training certification received by attendance at any training course including externally provided training courses will be kept by the employee with copies provided to the Training Team.

7. REFERENCES

PIKA-PIRNIE Health and Safety Procedure ARC HSFS010 – Health and Safety Planning

PIKA-PIRNIE Health and Safety Procedure ARC HSFS004 – Control of Hazardous Energy (Lockout/Tagout)

PIKA-PIRNIE Utility Clearance Policy and Procedure ARC HSF019

PIKA-PIRNIE Confined Space Policy and Procedure ARC HSF003

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8. RECORDS

8.1 Training records will be kept by the individual employee with copies of such certificates kept by the Training Team. Training dates and times will be kept by the Training Team.

8.2 Completed clearance forms and checklists will be kept in the project files with copies available for Corporate H&S review.

8.3 Copies of all HASPs that document excavation trenching procedures will be kept in the project files.

9. APPROVALS AND HISTORY OF CHANGE

Approved By: Michael Thomas, CIH, CPEA

History of Change

Revision Date	Revision Number	Reason for change
12 May 2008	01	Original document

Revision Date	Revision Number	Reason for change
13 June 2008	02	Modified Section 5.1 – 4th bullet related to structural ramps. Modified Section 5.2 to designate a 6x factor for structural integrity of structures near the excavation. Revised Exhibit 1 to modify the definition of a Competent person
9 January 2009	03	Cleaned up definitions, deleted training requirements from Section 5.0 and moved them to Section 6.0, modified purpose statement
31 March 2011	04	Updated Competent Person training and qualification requirements in section 4.6, section 6.2 and definition in Exhibit 1.

Exhibit 1 – Definitions

Attendant is a trained qualified individual stationed outside the excavation whose duty is to monitor authorized entrants inside the excavation or trench and have a means of communication with the designated rescue services.

Benching/Benching system means a method of protecting employees from cave-ins by excavating the sides of an excavation to form one or a series of horizontal levels or steps, usually with vertical or near-vertical surfaces between levels.

Cave-in means the separation of a mass of soil or rock material from the side of an excavation, or the loss of soil from under a trench shield or support system, and its sudden movement into the excavation, either by falling or sliding, in sufficient quantity so that it could entrap, bury or otherwise injure and immobilize a person.

Competent person means one who, through education, training, and/or experience, is capable of identifying existing and predictable hazards or working conditions which are unsanitary, hazardous, or dangerous to employees and who has authorization to take prompt corrective measures to eliminate them.

Excavation means any man-made cut, cavity, trench, or depression in an earth surface formed by earth removal into which a person can bodily enter. **Entry** constitutes the act by which an employee proceeds into an excavation or trench. Consideration of hazards, especially cave-ins and fall protection must still be considered and accounted for when equipment or personnel are near an excavation or trench, even if personnel will not be entering.

Entrants are employee's who are trained and authorized to enter a trench or excavation. Entrants must have attended a Qualified Excavation Training course offered or approved by Corporate Health and Safety.

Failure means the breakage, displacement, or permanent deformation of a structural member or connection so as to reduce its structural integrity and its supportive capabilities.

Hazardous Atmosphere is an atmosphere which exposes employees to a risk of death, incapacitation, injury, or acute illness from one or more of the following:

- An atmospheric concentration of any substance in excess of 50% of its established permissible exposure limit (PEL); or its assigned threshold limit value (TLV) or other value listed on the Material Safety Data Sheet (MSDS) for the chemical constituent, whichever is lower.
- A flammable gas, vapor, or mist in excess of 10% of its lower explosive limit (LEL).
- An airborne combustible dust at a concentration that obscures vision at a distance of 5 feet or less.
- An atmospheric oxygen concentration below 19.5% (oxygen-deficient atmosphere) or above 23.5% (oxygen-enriched atmosphere).
- An atmosphere which is immediately dangerous to life and health.

Immediately Danger to Life and Health (IDLH) means any condition which poses an immediate threat to loss of life; may result in irreversible or immediate-severe health effects; may result in eye damage, irritation, or other conditions which could impair escape from the space.

Protective system means a method of protecting employees from cave-ins, from material that could fall or roll from an excavation face or into an excavation, or from the collapse of adjacent structures. Protective systems include support systems, sloping and benching systems, shield systems and other systems that provide protection.

Ramp means an inclined walking or working surface that is used to gain access to one point from another, and is constructed from earth or from structural materials such as steel or wood.

Registered Professional Engineer means a person who is registered as a professional engineer in the state where the work is to be performed. However, a professional engineer, registered in any state is deemed to be a "registered professional engineer" within the meaning of this standard when approving designs for "manufactured protective systems" or "tabulated data" to be used in interstate commerce. To oversee an excavation/trench activity the PE must have experience with and expertise in excavation, soil and stability considerations.

Sheeting means the members of a shoring system that retain the earth in position and in turn are supported by other members of the shoring system.

Shield (Shield system) means a structure that is able to withstand the forces imposed on it by a cave-in and thereby protect employees within the structure. Shields can be permanent structures or can be designed to be portable and moved along as work progresses. Additionally, shield can be either pre-manufactured or job-built in accordance with 1926.652 (c)(3) or (c)(4). Shields used in trenches are usually referred to as "trench boxes" or "trench shields".

Shoring (Shoring system) means a structure such as a metal hydraulic, mechanical or timber shoring system that supports the sides of an excavation and which is designed to prevent cave-ins.

Sloping (Sloping system) means a method of protecting employees from cave-ins by excavating to form sides of an excavation that are inclined away from the excavation so as to prevent cave-ins. The angle of incline required to prevent a cave-in varies with differences in such factors as the soil type, environmental conditions of exposure, and application of surcharge loads.

Stable rock means natural solid mineral material that can be excavated with vertical sides and will remain intact while exposed. Unstable rock is considered to be stable when the rock material on the side or sides of the excavation is secured against caving-in or movement by rock bolts or by another protective system that has been designed by a registered professional engineer.

Support system means a structure such as underpinning, bracing, or shoring, which provides support to an adjacent structure, underground installation, or the sides of an excavation.

Trench means a narrow excavation (in relation to its length) made below the surface of the ground to which a person can bodily enter. In general, the depth is greater than the width, but the width of a trench (measured at the bottom) is not greater than 15 feet (4.6 meters). If forms or other structures are installed or constructed in an excavation so as to reduce the dimension measured from the forms or structure to the side of the excavation to 15 feet (4.6 meters) or less (measured at the bottom of the excavation), the excavation is considered to be a trench.

Cemented soil means a soil in which the particles are held together by a chemical agent, such as calcium carbonate, such that a hand size sample cannot be crushed into powder or individual soil particles by finger pressure.

Cohesive soil means clay (fine grained soil), or soil with a high clay content, which has cohesive strength. Cohesive soil does not crumble, can be excavated with vertical sides, and is plastic when

moist. Cohesive soil is hard to break up when dry, and exhibits significant cohesion when submerged. Cohesive soils include clayey silt, sandy clay, silty clay, clay and organic clay.

Dry soil means soil that does not exhibit visible signs of moisture content.

Fissured means a soil material that has a tendency to break along definite planes of fracture with little resistance, or a material that exhibits open cracks, such as tension cracks, in an exposed surface.

Granular soil means gravel, sand, or silt (coarse grained soil) with little or no clay content. Granular soil has no cohesive strength. Some moist granular soils exhibit apparent cohesion. Granular soil cannot be molded when moist and crumbles easily when dry.

Layered system means two or more distinctly different soil or rock types arranged in layers. Micaceous seams or weakened planes in rock or shale are considered layered.

Moist soil means a condition in which a soil looks and feels damp. Moist cohesive soil can easily be shaped into a ball and rolled into small diameter threads before crumbling. Moist granular soil that contains some cohesive material will exhibit signs of cohesion between particles.

Plastic means a property of a soil which allows the soil to be deformed or molded without cracking, or appreciable volume change.

Saturated soil means a soil in which the voids are filled with water. Saturation does not require flow. Saturation, or near saturation, is necessary for the proper use of instruments such as a pocket penetrometer or sheer vane.

Soil classification system means, for the purpose of this procedure, a method of categorizing soil and rock deposits in a hierarchy of Stable Rock, Type A, Type B and Type C, in decreasing order of stability. The categories are determined based on an analysis of the properties and performance characteristics of the deposits and the characteristics of the deposits and the environmental conditions of exposure.

Submerged soil means soil which is underwater or is free seeping.

Unconfined compressive strength means the load per unit area at which a soil will fail in compression. It can be determined by laboratory testing, or estimated in the field using a pocket penetrometer, by thumb penetration tests, and other methods.

Wet soil means soil that contains significantly more moisture than moist soil, but in such a range of values that cohesive material will slump or begin to flow when vibrated. Granular material that would exhibit cohesive properties when moist will lose those cohesive properties when wet.

Exhibit 2 – Daily / Periodic Excavation Inspection Checklist

		Daily / Periodic Excavation Inspection Checklist
Project Name:		**Date / Time:**
Project Number:		**Location:**
Prepared By:		**Project Manager:**

This checklist must be completed for all excavations. It documents that daily and post-event / periodic inspections are conducted.

Soil Classified As: Stable Rock Type A Type B Type C

Soil Classified On: By:

Type of Protective System in Use: Sloping Shoring Other _____

Description:

Inspection Item	YES	NO	Comments
Has PIKA-PIRNIE Utility Clearance Procedure been completed?			
Are underground installations protected from damage?			
Are adequate means of entry / exit available in the excavation – at least every 25 feet?			
If exposed to traffic, are personnel wearing reflective vests and adequate barriers/traffic controls installed?			
Do barriers exist to prevent equipment from rolling into the excavation?			
Was air monitoring conducted prior to and during excavation entry?			
Was the stability of adjacent structures reviewed by a registered P.E.?			
Are spoil piles at least 2 feet from the excavation edge?			
Is fall protection in use near excavations deeper than 6 feet?			
Are work tasks completed remotely if feasible?			
Is a protective system in place and in good repair?			
Is emergency rescue (lifeline / body harness) equipment used due to potential atmospheric hazard?			
Is excavation exposed to vibration?			
Are employees protected from falling / elevated material?			
Is soil classification adequate for current environmental / weather conditions?			
Do portable ladders extend at least 4 feet above the excavation?			
Are portable ladders or ramps secured in place?			
Have all personnel attended safety meeting on excavation hazards?			
Are support systems for adjacent structures in place?			
Is the excavation free from standing water?			
Is water control and diversion of surface runoff adequate?			
Are employees wearing required protective equipment?			
PIKA-PIRNIE Excavation Competent Person:			**Date/Time:**

Appendix I

Confined Space Entry Program

EXECUTIVE SUMMARY

It is PIKA-PIRNIE's policy that our staff will not enter confined spaces of any type unless it is absolutely necessary. If it becomes necessary to enter confined spaces, this standard, at a minimum will be strictly followed. A summary of the important requirements for managing the risks of confined spaces are summarized below.

- Potential confined spaces in the work area that could be purposely or mistakenly entered by PIKA-PIRNIE or subcontractor staff must be identified as confined spaces. Entry is forbidden into these spaces until they are classified. If no entry is necessary, classification is not required.

- If entry is or may be required, the spaces must be evaluated and classified as to being permit-required or non-permit required confined spaces. The client or property owner may have done this already, but this must be verified.

- Non-Permit Required Confined Spaces

    - Entry into non-permit required confined spaces is allowed but only it has been appropriately evaluated and verified that all hazards are adequately controlled - **TRACK**

    - Personnel entering non-permit required spaces must complete confined space awareness training per PIKA-PIRNIE training requirements

- Permit-Required Confined Spaces

    - Permit-required confined spaces must be marked to identify them as such if not already.

    - A thorough hazard analysis of the space and the activities that could create hazards in the space must be completed – **TRACK**

    - A permit package must be completed prior to any entry, including the evaluation form, the permit form and the confined space entry checklist

    - All hazards must be appropriately controlled and verified before entry

    - Permit must be reissued for each entry and cancelled at the completion of an entry. Cancelled permits should be maintained in the project files.

    - An air monitoring plan and implementation of the plan is required for the entry

    - Entrants must be outfitted with appropriate equipment including PPE and rescue

    - A pre-planned rescue plan is required including identification of rescue services and trained personnel

- Awareness level confined space training is required for all staff who work where confined spaces may be present. Those who enter or serve as Attendants, Entry Supervisors or Rescuers must complete classroom training approved by Corporate H&S

1. POLICY

It is PIKA-PIRNIE's policy that staff will not enter confined spaces of any type unless it is absolutely necessary. If it becomes necessary to enter confined spaces, this standard, at a minimum will be strictly followed.

2. PURPOSE AND SCOPE

2.1 Purpose

This standard sets forth the accepted practice for confined space entry and establishes the requirement for a Confined Space Entry Permit protocol to effectively mitigate or eliminate the hazards presented by entry into confined spaces.

2.2 Scope

This standard applies to all employees of PIKA-PIRNIE who may work around or in confined spaces. Only trained and authorized personnel are permitted to enter confined spaces, supervise confined space activities, serve as an attendant during confined space activities and perform entry or non-entry rescues from confined spaces.

3. DEFINITIONS

See Definitions in Exhibit 1.

4. RESPONSIBILITIES

4.1 Attendants

- An Attendant is prohibited from monitoring the activities of more than one confined space entry.
- An Attendant must be stationed and remain stationed outside the permit space at all times during entry operations. The Attendant may have no other duties besides those listed in this section.
- All Attendants must have training and instruction in their duties and responsibilities regarding confined space entry. The following are assigned duties:
    - Maintain an accurate count of all entrants in the confined space
    - Monitor activities both inside and outside the confined space to verify the continued safety of entrants
    - Maintain visual contact or verbal communication with all entrants in the confined space at all times

- Order evacuation of the confined space if an uncontrolled hazard develops, either within or outside the confined space, or upon observing a behavioral effect of hazard exposure among entrants
- Warn unauthorized persons away from the confined space
- Participate in non-entry rescue
- Summon rescue and other emergency services

- Attendants must maintain current certification in basic first aid and cardiopulmonary resuscitation (CPR).
- Under no circumstances should the Attendant attempt rescue of entrants by entering the confined space.
- In addition to a dedicated Attendant a second standby employee must be present in the area within sight or call to assist as necessary. This standby person could be the Entry Supervisor if different than the Attendant.

4.2 Authorized Entrants

Entrants must have training and instruction in their duties and responsibilities regarding confined space entry. All authorized entrants must:

- Recognize the hazards which may be faced during entry, as well as the signs and symptoms of exposure to the hazard(s).
- Shall confirm that all isolation, Lock/Out and Tag/outs have been completed prior to entry into confined space.
- Maintain visual contact and/or verbal communications with the Attendant at all times.
- Use the PPE, air monitoring and testing equipment that has been provided.
- Maintain an awareness of all external barriers required to protect from external hazards (e.g., blanking, blocking and lockout) and the proper use of those barriers.
- Obey evacuation orders given by the Attendant, Entry Supervisor, automatic alarm activation, or when self-perceived.

4.3 Entry Supervisors (also see Training and Duties of Entry Supervisor)

- Issue, authorize, and post the Entry Permit prior to any confined space entry.
- Interface with the client representative to identify hazards associated with the client's confined space.
- Review existing confined space data (if any) recorded by the client.

- Review the client's confined space standard.
- Investigate the client's permit entry protocol, ensuring that any identified hazards and previous experience with the confined space is properly communicated.
- Coordinate entry operations with the client's employees when both client and PIKA-PIRNIE employees will be working in or near a permit space.
- Coordinate rescue assistance with either the client's in-house rescue team and/or the offsite rescue assistance specified by the client. The offsite rescue assistance specified by the client must have direct rescue experience in the client's identified confined space or be provided an opportunity to examine the space and practice a rescue.
- Verify that the client takes the necessary precautions in notifying their employees that our employees will be entering the confined space.
- Review the lockout/tagout and isolation measures implemented by the client.
- Immediately report any unusual or unforeseen confined space entry hazard to Corporate Health and Safety prior to authorizing entry.
- Should test all atmosphere conditions prior to entry and shall complete and maintain the confined space permit form, and have it accessible for review on the job site at all times.
- Offer all entrants an opportunity to review the confined space entry testing results and an opportunity to request a reevaluation of the permit space in the presence of the entrant if the entrant has reason to believe that the evaluation of the space may not have been adequate.
- Upon completion of the entry covered by the permit, and after all entrants have exited the permit space, cancel the Entry Permit.
- Verify that copies of the completed and canceled Entry Permits are properly disseminated to Corporate Health and Safety and retained with the project files, as specified in Section 8.0I – Records.
- Identify and label confined spaces under PIKA-PIRNIE long-term control.

The Entry Supervisor may also function as the Attendant; therefore, the Entry Supervisor must have the training specified for an Attendant and will assume the duties listed for either the Entry Supervisor or Attendant.

4.4 Corporate H&S with Division and Practice Experts

On an annual basis, review and update, as necessary, this standard. In addition, review cancelled entry permits periodically to ensure conformance to this standard. Provide the initial confined space entry training and retraining, or recommend qualified training provider, to all Entry Supervisors, entrants and Attendants. Provide technical assistance regarding confined space entry protocol, atmospheric testing equipment, PPE, hazard assessment

and research information on unusual hazards. Audit project-specific confined space entry for compliance with this SOP. Retain a file of cancelled Confined Space Entry Permits for annual review.

4.5 Principal in Charge (PIC), Project Manager (PM), and Task Manager (TM)

Are responsible to:

- Verify that all confined spaces and entry protocols are properly identified and addressed within the project work plan, project health & safety plan, and/or other project-related documents.
- Verify that their divisional or project team employees have received the proper confined space training provided by Corporate Health & Safety or qualified training source prior to conducting confined space entry activities.
- Verify that the proper confined space entry equipment, including PPE, atmospheric testing equipment and safety equipment, is available for use by their divisional employees.
- Verify that copies of the completed and canceled Entry Permits are properly disseminated to Corporate Health and Safety and retained with the project files.
- Identify client requirements and assure they are communicated to the project team.

4.6 Health and Safety Plan Writers and Reviewers

Utilize this standard as guidance to ensure the appropriate identification, assessment and control of confined space spaces and associated entries for documentation in project HASPs

4.7 Rescue Services

Two types of rescue may be initiated during confined space work, entry rescue and non-entry rescue. Rescue services in this standard refer to both entry rescue and non-entry rescue. Entry rescue is typically provided by an outside service such as a local fire brigade. PIKA-PIRNIE employees are prohibited from conducting entry rescues, and will only participate in non-entry rescue if trained to do so.

Whenever PIKA-PIRNIE or subcontractor personnel enter a permit-required confined space, a written plan must be in place for the rescue of those employees from the space, as needed. The rescue service must:

- Be available and always be on alert for all confined space entries as required.
- Be familiar with all equipment used for the task.
- Have proper training and preparation for confined space rescue.
- Use the PPE and rescue equipment necessary for making rescues from confined spaces.

- Know basic first aid and cardiopulmonary resuscitation (CPR); at least one member of the rescue service shall hold current certification in first aid and CPR.
- Perform assigned rescue duties competently.
- Receive the required authorized entrants' training.
- Practice making confined space rescues using the following:
    - Dummies, mannequins or actual people
    - Representatives spaces to simulate the types of confined spaces from which the rescue is be performed
- Complete training at least once every 12 months, with training certification reviewed by the Entry Supervisor.
- Have ready access to the appropriate PPE and equipment necessary to safely retrieve injured or collapsed personnel from the confined space
- Consider ready access to first aid provisions during job planning
- Gather information on every confined space entry task, including exact location, immediately prior to work commencing and before and after breaks

4.8 All PIKA-PIRNIE Employees

Use the TRACK process described below regularly and frequently. In addition, employees read and understand all documented hazard identification and risk assessments conducted using the HARC process and documented in HASPs, JSAs, and other written plans that are associated with their work. PIKA-PIRNIE employees will:

- Participate in entry operations only if trained and authorized to do so
- Never enter a confined space without an authorized Attendant, Entry Supervisor, and a completed Entry Permit
- Never attempt entry rescue within a confined space unless trained in entry rescue
- If unexpected conditions arise during entry, immediately notify other entrants, evacuate the space and inform the Entry Supervisor

5. PROCEDURES AND PRACTICES

Structures or facilities that could be deemed confined spaces may include but not be limited to:

- Enclosed drains or sewers
- Excavations and trenches
- Process vessels and exchanges

- Road and rail tankers
- Silos
- Storage tanks
- Sumps
- Well vaults
- Crawl spaces

A confined space can be permitted or non-permitted, which is determined by through the completion of a comprehensive evaluation of hazards to appropriate controls are implemented to ensure entry is completed safely.

5.1 What is a Confined Space?

A Confined Space is any enclosed space which:

- Is large enough and so configured that an employee can bodily enter and perform work.
- Has limited or restricted means for entry or exit.
- Is not intended for continuous employee occupancy.

5.2 What is a Permit-Required Confined Space?

A Permit-Required Confined Space is a confined space that has one or more of the following characteristics:

- Contains or has a known potential to contain a hazardous atmosphere.
- Contains a material with the potential for engulfment of an Entrant.
- Has an internal configuration such that an Entrant could be trapped or asphyxiated by inwardly converging walls or a floor which slopes downward and tapers to a smaller cross-section.
- Contains any recognized safety or health hazard capable of causing injury or death.
- Contains job-introduced hazards such as welding, cutting, grinding, hot riveting, burning, heating, or the introduction or sources of ignition within the confined space, asbestos or lead containing material removal, or the use of flammable or toxic cleaning solvents.

5.3 What is Confined Space Entry?

Entry constitutes the act by which an employee intentionally passes through an opening into a permit-required confined space. Entry is considered to have occurred as soon as any part of the employee's body breaks the plane of the opening into the space.

5.4 What are the Potential Hazards of a Confined Space?

All parties involved in confined space entry will be competent to recognize hazards that may be associated with a confined space. The parties involved, as part of the evaluation process, will perform a hazard analysis and assessment of the space to identify associated hazards for each space identified if entry is required. Appropriate controls will then be implemented before entry. The potential hazards of a confined space include but are not limited to:

- Presence of flammable substances and oxygen enrichment which can lead to fire or explosion (Chemical).
- Toxic gases, fumes or vapors which can result in acute local or systemic health effects (Chemical).
- Inert gases which can result in asphyxiation (Chemical).
- Oxygen-deficient atmospheres which can result in asphyxiation (Chemical).
- Liquids or solids that can engulf an Entrant (Mechanical, Motion).
- Extreme temperatures which can result in heat-stress, cold stress, or mental acuity decline (Environment).
- Mechanical or electrical equipment which can result in bodily injury if contacted (Mechanical, Electrical).
- Working at heights when entering and exiting which can result in falls (Gravity).
- Slippery or uneven walking surfaces (Gravity, Mechanical).
- Elevated noise levels from activities and echoing (Sound).
- Low light levels (Radiation, Environment).
- Stressful, confined work areas (Personal safety).
- Poisonous or biting insects or animals, bacteria, biological materials, sanitary wastes (Biological).

5.5 Confined Space Identification and Evaluation

On an PIKA-PIRNIE project site or work location, information about confined spaces, including their location and characteristics, near the work site must be obtained from the owner or operator of the confined space. In turn, PIKA-PIRNIE will then inform its subcontractors, as appropriate.

In addition, prior to the initiation of activities, PIKA-PIRNIE will:

- Verify the location of the identified confined spaces and confirm no others exist in its work area
- Evaluate each space in accordance with the criteria defined in this Program utilizing the Confined Space Evaluation Form and Instruction Guide. This form then becomes part of the Entry permit package if entry is necessary.
- Classify the confined spaces as to whether they are permit- or non-permit required. This may include additional consulting with the client, outside agencies and other necessary entities, as appropriate. PIKA-PIRNIE can upgrade the classification beyond the client's classification; however PIKA-PIRNIE will not downgrade the classification.
- Ensure the spaces are marked accordingly by either the client, site owner, or PIKA-PIRNIE, as appropriate.

Permit-required confined spaces will be marked as indicated below or similarly:

Danger - - Permit-Required Confined Space, Do Not Enter

Non-permit required spaces may also be marked as appropriate to notify others that it is a confined space.

5.6 General Requirements for Confined Space Entry

5.6.1 Communications

Prior to any confined space entry, a communication system will be established.

For communication during a permit-required space, there should be an established system between:

- the Attendant and the Entrant
- the Attendant and the Entry Supervisor
- The Attendant and the assigned Rescuers. The Attendant will be in constant and direct communication with the Entrants.

Acceptable forms of communication between the Entrant and the Attendant are:

- Hand signals, as long as the Entrants are in constant view of the Attendant

- Intrinsically safe portable radios

The appointed rescue team(s) shall be informed of every permit-required confined space entry task, including the exact location, both immediately prior to work commencing and before and after work breaks. Intrinsically safe portable radios or cellular telephones of summoning help from a rescue service shall be available.

5.6.2 Confined Space Entry Equipment

Confined space entry, whether it be permit or non-permit required entry may require a variety of equipment. This will be evaluated as part of the hazard analysis and evaluation process. This equipment may include, but not limited to the following:

- Signs, barricades or other devices to control access to the confined space and to control the area around the confined space where people will be working.

- Energy control devices as specified by the PIKA-PIRNIE Lockout/Tagout (LOTO) Control of Hazardous Energy standard.

- Entry devices – ladder, tripod and winch, harness or other mechanism to allow safe entry and non-entry rescue – rescue equipment is required for all permit-required spaces.

- Safety equipment – fire extinguishers, lights, cooling vests.

- Ventilation equipment – fans, blowers or other methods to maintain safe atmospheric conditions within the space.

- Air monitoring – meters to measure the air quality, at a minimum confined space entry meters will be able to measure oxygen, lower explosive limit (LEL), hydrogen sulfide (H_2S) and Carbon monoxide (CO) – these may not be necessary for non-permit required spaces.

- Personal Protective Equipment – selected based on the hazards.

Other equipment may be required based on the nature of the entry and the activities. For example, self-contained breathing apparatus may be necessary for external rescuers for entry rescuers. All equipment must be appropriate for the activities of the entry and be approved or certified as specified by regulatory requirements.

5.7 Non-Permit-Required Confined Space Requirements

If the site, as a result of the evaluation process, is revealed to contain any non-permit required confined spaces, PIKA-PIRNIE will inform affected employees. Entry into these spaces can be made as follows:

- Entry into a non-permit confined space will be allowed only after review of the inventory/ evaluation form to determine what potential hazards may exist.

- Entry into the non-permit confined space will occur only after all hazards, if any, have been eliminated.

If there are changes in the use or configuration of a non-permit confined space, the space in question must be re-evaluated to determine if any hazards exist. If the hazards cannot be eliminated, the space must be reclassified as a permit required confined space.

Non-Permit Entry is permitted when all atmospheric, egress and other recognized hazards can be eliminated. If this is a sustainable situation, the confined space can be entered as a non-permit required entry. **The confined space entry checklist must still be completed.**

5.8 Reclassifying a Permit-Required to a Non-Permit-Required Confined Space

To reclassify a space from permit required to non-permit required, the following procedures must be used:

- Without entering the space, if all the hazards in the space can be eliminated, (including atmospheric or potential atmospheric hazards), the space may be reclassified as a non-permit required.
- If it is necessary to enter the space to eliminate existing hazards, all requirements for permit required confined space entry must be followed. Once testing or inspection during this entry demonstrates that the hazards within the space have been eliminated, the space may be reclassified as a non-permit required.
- Prior to reclassifying a permit required confined space to a non-permit required confined space, all relevant data that demonstrates the basis for the reclassification must be documented and referenced on the evaluation form.

5.9 Permit-Required Confined Space Entry

After a confined space is determined to be permit-required and entry is necessary to complete the activities, PIKA-PIRNIE staff will:

- Complete the entry permit package which includes the completed evaluation form, the entry permit, and the entry checklist.
- Evaluate the hazards external to the confined space that may impact the confined space operations including but not limited to pedestrians and vehicles
- Determine the appropriate hazard controls appropriate for the space using the hazard control hierarchy of eliminate, substitute, isolate, engineer out, administratively manage, and provide personal protective equipment
- Develop an appropriate air monitoring program based on the characteristics of the space
- Identify competent Entry Supervisor, Attendant, Entrants and Rescue personnel and establish procedures for the coordination of these staff

5.9.1 Entry Permit Package and Process

The Confined Space Entry Permit Package is made up of the completed Evaluation form, the Entry Permit, and the Entry Checklist. The Entry Permit will be:

- Completed, signed and issued by the Entry Supervisor who will verify that the confined space has been properly evaluated, isolated, drained, washed, purged, flushed ventilated, and tested as appropriate, and that the Entry Checklist and the Evaluation form are completed and part of the permit package.
- Posted at the entrance to the space and remain for the duration of the entry
- Weather-protected to maintain integrity.

In addition, prior to authorizing the Entry Permit, the Entry Supervisor, along with the attendant and Entrants must ensure the following, as applicable.

- All mechanical apparatus (such as agitators) within or connected to the confined space are de-energized, locked-out, and tagged as per the PIKA-PIRNIE Lockout/Tagout standard.
- All lines connected to the confined space where the nature of the service could present a hazard, such as nitrogen, steam, solvent, acid, or hot water, are isolated from the confined space. Acceptable isolation methods include removing a valve, spool piece, or expansion joint, and blanking or capping the opened end; inserting a suitable full-pressure blank in the piping between connecting flanges; and/or closing and locking at least two valves in the pipeline and locking open to atmosphere a chain valve between the two closed and locked valves.
- All electrical equipment in and around the confined space is de-energized and locked out.
- For confined spaces which have contained a known hazardous chemical (e.g., vessels, storage tanks), have been thoroughly cleaned by appropriate means, e.g., overflowing with water, steaming, etc.
- For confined spaces containing known atmospheric hazards, mechanical ventilation is operating to maintain atmospheric hazards within permit parameters.
- The atmosphere of the confined space is initially checked to verify that it contains acceptable levels of oxygen (19.5 to 23.5%) and is free of hazardous levels of explosive/combustible or toxic gases or vapors. The Atmospheric Testing Section, of this standard lists the air quality specifications which must be met. These specifications are also listed on the Entry Permit. Continuous air monitoring may be required depending on the nature of the confined space, as well as the activity(ies) to be conducted within the confined space.
- All necessary entry equipment (e.g., retrieval lines, PPE, respiratory protective equipment) is available, in good condition, and functional.

- All Entrants and Attendants have received the appropriate confined space entry training.
- All rescue arrangements are in-place as per the section on Outside Rescue Assistance, and that an adequate means of communicating with outside assistance is immediately available to the attendant.

The Entry Permit must be canceled and all Entrants ordered to evacuate the confined space when any one of the following conditions arises:

- A change in initial atmospheric conditions which may jeopardize the continued health and safety of Entrants is detected.
- The attendant must leave the work station.
- The attendant is called on to perform duties which do not allow him/her to fulfill his/her duties as an attendant.
- Whenever ordered by the attendant due to factors external to the confined space which may jeopardize the continued safety and health of Entrants.
- At the end of the work shift and/or whenever a different group of Entrants and Attendants will take charge of the confined space.
- Whenever Entrants self-perceive danger and self-initiate evacuation.
- At the termination of confined space entry.
- At the end of the work shift in which the entry occurs.

5.9.2 Other Confined Space Entry Permit Requirements

Additional Entry Permit package requirements are:

- A separate Entry Permit must be generated for each confined space. However, a single Entry Permit may be generated for entry into multiple sewer system manholes in a continuous sewer system.
- The completed permit package is valid for one shift only. A new permit and checklist must be completed with each new entry. However, if the activities are the same as those evaluated on the evaluation form, a new evaluation form is not required.
- A new completed and signed Entry Permit Package must be issued for each new crew of Entrants and Attendants.

5.9.3 Evaluation of External Hazards

Prior to any confined space entry, hazards external to the space will be evaluated to ensure protection of Entrants and others. This evaluation is done as part of the PIKA-PIRNIE TRACK process, the development of JLAs for the job, and the daily tailgate meeting on site. These external hazards may also be evaluated through the development of the site specific health and safety plan.

5.9.4 Atmospheric Testing

All confined spaces will be tested for atmospheric hazards as outlined below. Results of this testing will be documented on the Entry permit.

- Each confined space will be initially tested prior to the Entry Supervisor authorizing entry.
- Each confined space will also be tested continuously or at intervals as specified by the Entry Supervisor based on the characteristics and hazards identified associated with the space, even when mechanical ventilation is utilized.
- The following are the testing sequence and acceptable air quality criteria:
    - Oxygen content for all confined space entry must be 19.5 to 23.5% (Oxygen must be measured first)
    - Combustible gas or vapor must not exceed 10% of its Lower Explosive Limit (LEL)
    - Toxic gas or vapor must not exceed 50% of the OSHA Permissible Exposure Limit (PEL) or other published exposure guidelines whichever is lower
    - Carbon monoxide must not exceed 10 parts per million (ppm)
    - Hydrogen sulfide must not exceed 0.5 ppm
- If it is necessary to enter a confined space where any of the following atmospheric conditions exist, all Entrants must wear either a NIOSH approved self-contained breathing apparatus (SCBA) of at least 60-minute duration or an air line respirator with emergency SCBA, as delineated below.
    - Initial atmospheric testing indicates conditions outside the parameters listed on the Entry Permit. However, no entry will be made, even with respiratory protection, if combustible gases/vapors are greater than 10% of the LEL or oxygen is greater than 23%
    - Initial atmospheric testing indicates conditions within permit parameters, but where the quality of the atmosphere remains questionable
    - Despite initial atmospheric testing results, activities to be performed while in the confined space would endanger Entrants by creating a sudden change in atmospheric conditions within the space. This activities may include but are

not limited to welding, painting, coating, cutting, using solvents, or agitating bottom sediments

- o Mechanical ventilation will not maintain atmospheric hazards within permit limits

- Under no circumstances is entry into a confined space having an IDLH condition (less than 19.5% oxygen or >5% of the LEL) permitted by any employee of PIKA-PIRNIE. (10% of the LEL is safe from a flammability standpoint, but may pose certain other hazards from a health risk, thus, entry is not permitted if conditions measure more than 5% of the LEL)

- Results of all atmospheric testing must be recorded on the Confined Space Entry Permit and/or an attached air monitoring log found in the HASP.

- Entrants and their representatives have the opportunity to participate in all air monitoring and air monitoring instrument calibration, and to review all air monitoring data prior to entry to the space.

5.9.5 Mechanical Ventilation

Mechanical ventilation may be:

- Utilized to maintain the atmospheric conditions hazards within entry limits.

- Used to force clean air into a space or remove contaminated air from the space.

Ventilation systems must be set up to adequately ventilate all areas of the space and be locked in the “on” position. The space must be evacuated if the system fails.

Air intake must be positioned to prevent the introduction of air contamination into the confined space (e.g., away from vehicle exhaust, tank vents).

5.9.6 Work Practices

The following Work Practices must be followed for Permit-Required Confined Space Entry.

- All Entrants must wear a retrieval line secured on one end to the Entrant by a full-body harness, or parachute harness, and the end secured outside the space unless this creates more of a hazard.

- If there is not a fixed retrieval line, a suitable means for rescue appropriate for the configuration of space, must be provided.

- For vertical-entry spaces, the lifeline must be secured to a lifting or other mechanical retrieval device affixed to a suitable anchor point. Reliance on manually lifting an Entrant from a vertical confined space is prohibited. If more than one Entrant is entering the space, each line shall be clearly marked to identify the Entrant and the mechanical retrieval system must be rated for multiple Entrant use.

- Whenever a ladder is required for entry, it must be secured and not withdrawn while anyone remains within the confined space, except as necessary to permit extraction during rescue.
- Adequate, explosion proof illumination must be provided for entry as necessary.
- Electrical equipment used within a confined space must be explosion-proof and be inspected prior to use to verify good working condition. The equipment must utilize a ground fault interrupt and/or be properly grounded.
- Whenever the confined space is structured such that visual contact cannot be maintained between Entrants and the attendant, intrinsically-safe, two-way radios must be utilized to maintain continuous contact between Entrants and Attendants.
- All confined spaces must be isolated prior to entry.
- Prior to opening or removing lids, covers, access doors, or hatches of a confined space, precautions must be taken to determine if it is safe to do so.
- Whenever entering spaces with permanent ladders, all rungs must be inspected to verify they are in safe and useable condition.
- When working in a vertical confined space, precautions must be taken to prevent equipment and personnel from falling into the confined space opening. Tools should be lowered and removed from the space using a basket or sling to prevent falls and falling objects.
- A re-evaluation of the hazards associated with the space will be conducted if it is believed or known that conditions in the space have changed, or if an Entrant requests re-evaluation or air monitoring.
- If PIKA-PIRNIE staff must enter a space with staff from one or more other employers, no entry will take place until the entry supervisors from each employer coordinate activities and determine and communicate the entry, operations, and exit and rescue procedures for the multi-employer space. Responsibilities and designation of each authorized position will be reviewed and authorized positions will be appropriate staffed with qualified personnel.

5.10 Confined Space Rescue

Rescue services will be provided and be prepared for permit-required confined space entries. Non-entry rescues can be performed by qualified and trained PIKA-PIRNIE staff. Entry rescues will be performed by external sources that are qualified and competent to perform entry rescues.

5.10.1 Non-Entry Rescue

PIKA-PIRNIE staff or an external rescue team can provide non-entry rescue if they are property trained and qualified. This training must be completed at least annually and include the actual use of the rescue equipment that will be used during the confined

space entry over which they will provide rescue services. In addition, at least one person on the rescue team must be currently certified in first aid and CPR.

Non-entry rescue will involve the use of retrieval systems or methods to assist in the rescue unless the retrieval equipment increases the overall risk of the entry or does not contribute to the rescue of the Entrants.

For non-entry retrieval, non-entry rescuers shall:

- Ensure the use of a full body harness with a retrieval line attached at the center of the Entrant's back near shoulder level or above the Entrant's head by each authorized Entrant.
- Attach the other end of the retrieval line to a mechanical device or fixed point outside the confined space in such a manner that rescue can begin as soon as the rescuer becomes aware that rescue is necessary.
- Set up a mechanical device and make it ready to retrieve personnel from vertical type confined spaces more than 4 feet deep.
- Use the material safety data sheets or written information if an injured Entrant is exposed to a hazardous substance.
- Provide medical personnel treating the exposed Entrant with the material safety data sheet or written information.

5.10.2 Outside Entry Rescue Assistance

If the Entry Supervisor determines entry rescue may be necessary, an assessment will be completed to verify that the designated rescue service:

- Has adequate resources, training and equipment to provide services for the scope of the planned entry.
- Is within a reasonable response distance/time.

Such rescue assistance must be coordinated with either the client's designated confined space rescue team and/or with a local emergency response team. The selected rescue services must be offered an opportunity to inspect the confined space prior to initiating the entry. Documentation of this offer and the status of the site visit, if conducted, must be maintained by the entry supervisor.

As appropriate, entry shall progress only after proper notification and verification of adequacy of outside rescue assistance prior to the actual entry activity.

An adequate means of communication (e.g., cellular telephone for contacting offsite emergency assistance, air horn, or two-way radio for summoning the rescue team) must be immediately available to the attendant.

5.11 Annual Review of the Confined Space Entry Program

At least annually, PIKA-PIRNIE will review the confined space program to determine if revisions are required to ensure it is adequate. This review will be accomplished using cancelled entry permits from within the last year, reading and updating this written standard, and via on-site assessments of entry activities. Based on this review, the program and procedures will be revised as appropriate, and then communicated to appropriate staff.

6. TRAINING

All employees who work in the area of potential confined spaces or who enter non-permit required spaces will be trained in awareness level training in order to recognize confined spaces and to understand their hazards. This training is provided during initial and refresher HazWoper training or through specific confined space awareness training.

For entry to permit-required confined spaces, all parties involved including the entry supervisor, Attendants, Entrants and rescuers will take classroom, hands-on training pursuant to their activities. Additional training is provided to these employees if their duties change, or if new hazards are encountered, or if special procedures or activities occur. Site specific training is also provided to address those site-specific hazards and confined spaces encountered on each project.

Rescuers will receive hands-on training pertinent to the type of rescue services they will provide as described in the Rescue section of this document.

All training provided to PIKA-PIRNIE employees must be reviewed and approved by Corporate Health & Safety and will be managed through the PIKA-PIRNIE training center.

Documentation of training certification received by attendance at any training course including externally provided training courses will be kept by the employee with copies provided to the PIKA-PIRNIE training center.

7. REFERENCES

- Confined Space Entry Evaluation Form and Instruction Guide – found on the Community Page of the H&S Team Site
- Confined Space Entry Checklist – found on the Community Page of the H&S Team Site
- Confined Space Entry Permit – found on the Community Page of the H&S Team Site
- PIKA-PIRNIE Health and Safety Standard ARC HSFS010– Health and Safety Planning
- PIKA-PIRNIE Health and Safety Standard ARC HSFS004 – Control of Hazardous Energy (Lockout/Tagout)

8. RECORDS

- Training records will be kept by the individual employee with copies of such certificates kept by the PIKA-PIRNIE training system. Training dates and times will be kept by the PIKA-PIRNIE training system.
- Confined Space entry permits and cancelled permits will be kept in the project files with copies of cancelled permits kept by Corporate H&S.
- Copies of all HASPs that document confined space procedures will be kept in the project files.

9. APPROVALS AND HISTORY OF CHANGE

Approved By: Michael Thomas, CIH, CPEA, Director H&S Environmental Division

History of Change

Revision Date	Revision Number	Reason for Change
5 May 2008	01	Original document
21 Jan 2009	02	Removed reference to Sewer System Manhole Checklist
27 Jan 2009	03	Corrected discrepancy in acceptable CO levels between Section 5.3.3 and checklist in Exhibit1
29 April 2009	04	Modified and added to several sections to provide more detail per the request of several client via ISN
1 November 2009	05	Modified to include enhancements to the procedures and to meet BP Control of Work Defined Practice for Confined Spaces
1 November 2010	06	Modified format to include an Executive Summary. Also, reviewed and edited text to simplify the text.

Exhibit 1 – Definitions

Attendant is a trained **authorized** individual stationed outside the confined space whose sole duty is to monitor authorized entrants inside the confined space.

Blanking or Blinding is the absolute closure of a pipe, line or duct by the fastening of a solid plate that completely covers the bore and that is designed to withstand the pressure of the pipe, line or duct with no leakage beyond the plate.

Confined Space is any enclosed space which is large enough and so configured that an employee can bodily enter and perform work, has limited or restricted means for entry or exit, and is not intended for continuous employee occupancy. Confined spaces include, but are not limited to, storage tanks, vessels, pits, boilers, flues, manholes, ventilation system ductwork, sewers, vaults, pipelines, silos, storage hoppers, diked areas, and wells greater than 4 feet deep.

Confined Space Entry Permit is the document which defines the conditions of confined space entry, the reasons for entering the confined space, the anticipated hazards of the entry, a listing of atmospheric monitoring equipment, and acceptable atmospheric conditions. The Entry Permit identifies the rescue and other contacts which must be summoned in the case of an emergency, provides a listing of authorized attendants and entrants, the date of entry to the confined space, and the expiration of the Entry Permit. For the purposes of this HSP, the Confined Space Entry Permit package consists of the Confined Space Entry Checklist (Exhibit 1), the Confined Space Entry Permit (Exhibit 2), and the Confined Space Evaluation Form (Exhibit 3). The Entry permit package must be re-issued at the beginning of each shift.

Double block and bleed – the closure of a line, duct, or pipe by closing and locking or tagging two inline valves and by opening and locking or tagging a drain or vent valve in the line between the two closed valves.

Engulfment is the surrounding and effective capturing of a person by a liquid or finely divided (flow-able) solid substance that can cause death by filling or plugging the respiratory system or exert force on the body to cause death by strangulation, constriction or crushing

Entrants are employee's who are trained and **authorized** to enter a confined space.

Entry constitutes the act by which an employee intentionally passes through an opening into a permit-required confined space. Entry is considered to have occurred as soon as any part of the employee's body breaks the plane of the opening into the space.

Entry Supervisor is the trained, competent and **authorized** employee responsible for determining if acceptable entry conditions are present at a permit space where entry is planned, for authorizing entry and overseeing entry operations, and for terminating entry. The Entry Supervisor may also serve as an authorized attendant.

Hazardous Atmosphere is an atmosphere which exposes employees to a risk of death, incapacitation, injury, or acute illness from one or more of the following:

- An atmospheric concentration of any substance in excess of 50% of its established permissible exposure limit (PEL); or in the absence of a PEL, its assigned threshold limits value (TLV) or other value listed on the Material Safety Data Sheet (MSDS) for the chemical constituent whichever is lower.

- A flammable gas, vapor, or mist in excess of 5% of its lower explosive limit (LEL).
- An airborne combustible dust at a concentration that obscures vision at a distance of 5 feet or less or is above the LEL.
- An atmospheric oxygen concentration below 19.5% (oxygen-deficient atmosphere) or above 23.5% (oxygen-enriched atmosphere).
- An atmosphere which is immediately dangerous to life and health.

Hot Work Permit – the written authorization to perform operations (for example, riveting, welding, cutting, burning, and heating) capable of providing a source of ignition.

Immediately Danger to Life and Health (IDLH) means any condition which poses an immediate threat to loss of life; may result in irreversible or immediate-severe health effects; may result in eye damage, irritation, or other conditions which could impair escape from the confined space.

Inerting – the displacement of the atmosphere in a permit space by a noncombustible gas (such as nitrogen) t such an extent that the resulting atmosphere is non-combustible. This procedure produces an IDLH oxygen deficient atmosphere.

Isolation involves removing equipment/systems in and around the space from service. This includes but is not limited to lockout/tagout, double blanking and bleeding, disconnecting and securing or restraining equipment.

Lower Explosive Limit (LEL) is the minimum concentration (percentage) of a flammable gas that will propagate a flame in the presence of an ignition source. The more explosive the gas, the lower the LEL. LEL is usually expressed as a percentage (from zero to 100 percent explosive) and is often used interchangeably with lower flammability limit

Non-Permit Confined Space is a confined space that does not contain or have the potential to contain any hazards capable of causing death or serious physical harm.

Oxygen-deficient Atmosphere is an atmosphere containing less than 19.5 percent oxygen by volume.

Oxygen-enriched Atmosphere is an atmosphere containing more than 23.5 percent oxygen by volume.

Permit-Required Confined Space (Permit Space) is a confined space that has one or more of the following characteristics:

- Contains or has a known potential to contain a hazardous atmosphere
- Contains a material with the potential for engulfment of an entrant
- Has an internal configuration such that an entrant could be trapped or asphyxiated by inwardly converging walls or a floor which slopes downward and tapers to a smaller cross-section
- Contains any recognized safety or health hazard capable of causing injury or death.

- Contains job-introduced hazards such as welding, cutting, grinding, hot riveting, burning, heating, or the introduction or sources of ignition within the confined space, asbestos or lead containing material removal, or the use of flammable or toxic cleaning solvents

- Is an excavation or trench that is deeper than three feet.

Prohibited Condition is any condition in a permit space that is not allowed by the permit during the period when entry is authorized

Rescue Service is the team or entity designated to rescue personnel from confined spaces

Self-rescue is an entrant's ability to escape unaided from a confined space

Retrieval system – the equipment (including a retrieval line, chest or full-body harness, wristlets, if appropriate, and a lifting device or anchor) used for non-entry rescue of persons from confined spaces.

Appendix J

Perimeter Air Monitoring Plan

PERIMETER AIR MONITORING PLAN

Former Charlotte Naval Ammunition Depot (CNAD)
Charlotte, North Carolina
PIKA-PIRNIE JV, LLC

The *Perimeter Air Monitoring Plan* was developed to supplement the air monitoring requirements set forth in Section 10 of the Site Safety and Health Plan (SSHP), which was included as Appendix B-2 of the *Remedial Action Work Plan*. This plan establishes Exclusion Zone requirements and thresholds for the protection of Norfolk-Southern/RSI operations and staff at the former CNAD in Charlotte, North Carolina. This plan is to be implemented during remedial activities to address potential vapors encountered during course of drilling, sampling or other remedial activities as a result of trichloroethene (TCE) and vinyl chloride (VC) present in groundwater.

Site Control:

An active Exclusion Zone boundary will be established around each area of daily activity. Access within this zone will be restricted to PIKA-PIRNIE project staff, approved visitors and contractors. Personnel entering this zone will be subject to the requirements of the SSHP (PIKA-PIRNIE, 2011) established for protection of project staff. No Norfolk-Southern/RSI staff will be permitted within the Exclusion Zone without prior approval, appropriate health and safety training, and appropriate personal protective equipment (PPE). In the event that it is necessary for Norfolk-Southern/RSI staff to enter the Exclusion Zone to complete an assigned task, PIKA-PIRNIE will Stop Work until the area is cleared and activities can resume.

Air Monitoring:

Air monitoring stations will be set up in the breathing zone at a location(s) between the remedial activity and any Norfolk Southern/RSI workers. If necessary, multiple stations will be established, depending on the remedial action location and on site activities. Photo Ionization Detector (PID) and Combustible Gas Indicator (CGI) readings will be collected continuously through data logging, with manual readings recorded every 30 minutes at a minimum.

Air monitoring will be conducted at the extents of the Exclusion Zone using a PID with a 10.6 eV bulb and a CGI, as designated in the SSHP. The PID detects a broad range of volatile organic compounds (VOCs) including TCE and VC below 1 part per million (ppm) in air. The Occupational Safety and Health Administration (OSHA) permissible exposure limit (PEL) for TCE is 100 ppm and for VC is 1 ppm. The CGI will be used to measure the lower explosive limit (LEL)/oxygen levels. In addition to the PID and CGI, colorimetric tubes will used as needed to confirm the specific presence of TCE and/or VC.

Prior to initiating any activities at the site, background concentrations will be established for all areas of intended work. Background values will be established daily by collecting CGI, and PID readings at multiple locations in and around the Exclusion Zone established for that particular task. These values will be used during the corresponding field activities as background values for comparison.

During the course of remedial activities, the following chart will be used for Exclusion Zone Action Levels:

Exclusion Zone PID/CGI Readings	Action
<1 ppm/background	Continue work with continuous air monitoring
Detected concentrations on the PID are >1 ppm CGI > 10% LEL	Stop work. Collect colorimetric tubes to confirm the presence or absence of TCE and VC in the breathing zone at the perimeter of the Exclusion Zone. • If colorimetric tubes are non-detect, continue work. • If colorimetric tubes confirm the presence of TCE less than 5 ppm and VC less than 0.5 ppm, expand the perimeter of the Exclusion Zone to an area where colorimetric tubes are non-detect or consistent with the established background. Work may continue after the Exclusion Zone has been expanded. • If Exclusion Zone cannot be expanded without interfering with Norfolk-Southern/RSI operations, work will be discontinued, the SHM/PM will be notified, and alternative engineering controls to manage emissions will be evaluated. • *Air monitoring of the breathing zone at the drill rig will be completed in accordance with Section 10.1 of the SSHP.* If CGI is >10% LEL, immediately stop work, ventilate area, and investigate source of vapors.

Appendix C

Sampling and Analysis Plan (SAP) consisting of the following:

C-1 = Field Sampling Plan (FSP)

C-2 = Quality Assurance Project Plan (QAPP)

C-3 = Data Management Plan (DMP)

APPENDIX C-1
Field Sampling Plan

Former Charlotte Naval Ammunition Depot
Charlotte, North Carolina

December 2011

Revision 1 – February 2012

DERP-FUDS Project No. I04NC080301
Contract No.:W912DY-10-D0025
Delivery Order No.: 0007

PREPARED FOR:

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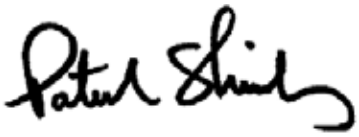

Patrick Shirley, PG
PIKA-PIRNIE JV Team Certified Project Manager

Field Sampling Plan

Former Charlotte Naval Ammunition Depot
Charlotte, North Carolina

Prepared for:
U.S. Army Corps of Engineers,
Huntsville Center

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DERP-FUDS Project No. I04NC080301
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Date:
December 2011
Revision 1 – February 2012

The views, opinions, and/or findings contained in this report are those of the author(s) and should not be construed as an official Department of the Army position, policy, or decision, unless so designated by other documentation.

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Acronyms and Abbreviations

ASTM	American Society for Testing and Materials
bgs	Below Ground Surface
C°	Celsius
CMUD	Charlotte Municipal Utility Department
CNAD	Charlotte Naval Ammunition Depot
COC	Constituent of Concern
CQC	Construction Quality Control
CQCP	Construction Quality Control Plan
DMP	Data Management Plan
DO	Delivery Order
DOT	Department of Transportation
EM	Engineering Manual
ERD	Enhanced Reductive Dechlorination
FFS	Focused Feasibility Study
FRA	Federal Railroad Administration
FSP	Field Sampling Plan
H&S	Health and Safety
HazMat	Hazardous Materials
HSU	Hydrostratigraphic Unit
HTRW	Hazardous, Toxic and Radioactive Waste
IATA	International Air Transport Association
ID	Inside Diameter
IDW	Investigation-Derived Waste
JV	Joint Venture
M&E	Metcalf and Eddy, Inc.
mL/min	Milliliters per Minute
MNA	Monitored Natural Attenuation
MS/MSD	Matrix Spike/Matrix Spike Duplicate
MV	Millivolt
NCAC	North Carolina Administrative Code

NCDENR	North Carolina Department of Environment and Natural Resources
NTU	Nephelometric Turbidity Unit
OD	Outside Diameter
ORP	Oxidation Reduction Potential
OSHA	Occupational Safety & Health Administration
PIKA	PIKA International, Inc.
PIKA-PIRNIE JV Team	PIKA International, Inc./Malcolm Pirnie, Inc. Joint Venture LLC Team
Pirnie	Malcolm Pirnie, Inc.
PM	Project Manager
PMP	Project Management Plan
PPE	Personal Protective Equipment
PVC	Polyvinyl Chloride
PWR	Partially Weathered Rock
PWS	Performance Work Statement
QA	Quality Assurance
QA/QC	Quality Assurance/Quality Control
QAPP	Quality Assurance Project Plan
QC	Quality Control
QCR	Quality Control Report
RA	Remedial Action
RAWP	Remedial Action Work Plan
RI	Remedial Investigation
SAP	Sampling and Analysis Plan
SESDPROC	Science and Ecosystem Support Division, Soil Gas Sampling Operating Procedure
SOP	Standard Operating Procedure
SOW	Scope of Work
SSHP	Site Safety and Health Plan
TCE	Trichloroethene
TOC	Total Organic Carbon

µg/L	Micrograms per Liter
U.S.	United States
USACE	U.S. Army Corps of Engineers, Huntsville Center
USEPA	U.S. Environmental Protection Agency
VOC	Volatile Organic Compound
WERS	Worldwide Environmental Remediation Services

1. Project Background

This Field Sampling Plan (FSP) was prepared by the PIKA International, Inc. (PIKA)/Malcolm Pirnie, Inc. (Pirnie) Joint Venture (JV), LLC (the PIKA-PIRNIE JV Team) to provide field personnel with detailed instructions and procedures regarding field activities to be performed in support of the Performance Work Statement (PWS) and Delivery Order (DO) DM0007 under the United States (U.S.) Army Corps of Engineers, Huntsville Center (USACE) Worldwide Environmental Remediation Services (WERS) Contract W912DY-10-D-0025 for the remedial activities at the Former Charlotte Naval Ammunition Depot (CNAD) in Charlotte, North Carolina.

The FSP was prepared, and is organized, in accordance with the Army guidance document Engineering Manual (EM) 200-1-3 (Requirements for the Preparation of Sampling and Analysis Plans [SAPs]). This project-specific FSP provides a detailed description of the field methodologies that will be used to complete remedial activities at the site, as specified in the DO and WERS Contract. The Remedial Action Work Plan (RAWP) further defines the scope of activities to be performed at the Site.

The PIKA-PIRNIE JV Team field personnel will use the procedures described in this FSP to produce accurate, comparable, and reproducible data for evaluation. This FSP is presented in the following 10 sections:

- Section 1 – Project Background;
- Section 2 – Project Organization and Responsibilities;
- Section 3 – Project Scope and Objectives;
- Section 4 – Non-Measurement Data Acquisition;
- Section 5 – Field Activities;
- Section 6 – Field Operations Documentation;
- Section 7 – Sample Packaging and Shipping Requirements;
- Section 8 – Investigation-Derived Waste (IDW);

- Section 9 – Field Assessment, Three-Phase Inspection Procedures; and
- Section 10 – Nonconformance/Corrective Actions.

1.1 Site History and Contaminants

A comprehensive summary of the Site's operational history was included in the Phase I Remedial Investigation (RI) Report. The reference for the document is included below.

- Metcalf and Eddy, Inc. (M&E). 1995. Phase I Remedial Investigation Final Report for the Former Naval Ammunition Depot Areas 1 and 2, Mecklenburg County, Charlotte, North Carolina. April.

A brief summary of the site history has also been included in Section 2.1 of the RAWP.

1.2 Summary of Existing Site Data

A summary of previous investigations is provided as Section 2.2 of the RAWP. The primary environmental investigations and studies at CNAD include:

- Phase I RI (M&E, 1995);
- Phase II RI (M&E, 2000);
- Site-Wide Groundwater Sampling for the Future Remedial Design (SAIC, 2008); and
- Focused Feasibility Study (FFS) Report (SAIC, 2009).

The Phase II RI (M&E, 2000) concluded that soil associated with the former CNAD was not impacted; however, groundwater was contaminated with volatile organic compounds (VOCs), specifically trichloroethene (TCE) and daughter products. Concentrations of TCE were observed to be present in both the transition zone and in bedrock hydrostratigraphic units (HSUs). The contaminant plume is located under the former CNAD Areas 1 and 2, which is now occupied by light industrial/commercial businesses and the bulkmatic terminal. The selected remedial alternative, as described in the 2009 Final FFS Report (SAIC, 2009), includes implementation of an in-situ remedy using enhanced reductive dechlorination (ERD) to treat VOCs in groundwater followed by a transition to monitored natural attenuation (MNA) until

concentrations in groundwater are in compliance with North Carolina Administrative Code (NCAC) 2L groundwater quality standards.

The Final FFS Report (SAIC, 2009) presents a summary of the activities conducted to date. There are two distinct TCE plumes at the site; one in the transition zone and one in the bedrock zone. The transition zone, extending to 42 feet below ground surface (bgs), contains TCE concentrations from non-detect to 6,200 micrograms per liter (µg/L) while the bedrock zone, extending to 305 feet bgs, contains TCE concentrations from 2.0 to 40,000 µg/L. Although no source was identified, the highest concentrations are located in the area of the former vapor degreaser building.

1.3 Site-Specific Definition of Problems

As a result of RIs and subsequent studies completed, TCE and daughter products have been identified as the primary constituents of concern (COCs) in the groundwater at the Site. Based on the FFS, a Decision Document (USACE, 2011) was prepared identifying the selected remedy as ERD to treat COCs in groundwater followed by MNA. Treatment of groundwater was to target both the transition zone and bedrock zones of the site.

2. Project Organization and Responsibilities

The project organization as well as the roles and responsibilities of key personnel are presented in Section 2 of the Project Management Plan (PMP) which is included as Appendix D of the RAWP

2.1 Applicable Training

The PIKA-PIRNIE JV Team and subcontractor personnel that will be conducting field work associated with the CNAD project will be required to complete the following training:

- 40-hour Occupational Safety & Health Association (OSHA) Hazardous Materials (HazMat) Training (and subsequent 8-hour updates;
- Department of Transportation (DOT)/International Air Transport Association (IATA) training (HazMat #1 course), if material shipping is required; and
- Federal Railroad Administration (FRA) training/E-RailSafe certification.

3. Project Scope and Objectives

The technical approach for remedial activities at the CNAD facility was developed to be in accordance with the approved remedy for the site. Implementation of this remedy has been refined to include eight injection events to reduce the site-related constituents (TCE) to a level where MNA can be implemented.

3.1 Task Description

The overall scope of work covered by this FSP is outlined in the RAWP. The following section presents a summary of the individual tasks:

- Monitoring Well Installation – A total of 11 additional monitoring wells (six bedrock and five transition zone wells) will be installed. The detailed scope of work is included in Section 4 of the RAWP.
- Injection Well Installation – The ERD injection system includes 53 injection wells to be completed in the transition zone and 25 injection wells to be completed in the bedrock zone. The detail scope of work (SOW) is presented in Section 3 of the RAWP.
- Baseline Groundwater Monitoring Event – Includes sampling and analyses of groundwater from 30 monitoring wells. The detailed SOW is presented in Section 4.2 of the RAWP.
- Methane Monitoring – Includes the routine monitoring of soil gas probes to assess potential risk associated with methane generated as part of the ERD injections. The detailed SOW is presented in Section 4.3.2 of the RAWP.
- Performance Monitoring Events – Includes sampling and analyses of groundwater from 20 monitoring wells to evaluate the performance of the ERD system. The detailed SOW is presented in Section 4.3.1 of the RAWP.
- MNA Monitoring Events – Includes sampling and analyses of groundwater from 30 monitoring wells to evaluate natural attenuation of the groundwater

once active remediation is complete. The detailed SOW is presented in Section 4.4 of the RAWP.

3.2 Applicable Regulations/Standards

Project quality assurance (QA) includes project chemical quality data assessment via laboratory validation, field and laboratory audits and data validation procedures. These procedures are discussed in the Quality Assurance Project Plan (QAPP), included with this FSP as part of the SAP.

All field sampling activities will be conducted in accordance with Section 3.3.4.5 of the EM 300-1-3 (Requirements for the Preparation of SAPs) and the laboratory will meet the requirements of the ER 1110-1-263 (Chemical QA for Hazardous, Toxic and Radioactive Waste [HTRW] Projects).

Level D personal protective equipment (PPE) will be required throughout the work (*i.e.,* hard hat, steel toed boots, and safety glasses). Work completed in high traffic areas will require Type II traffic vests during site work. Detailed safety procedures are presented in the Site Safety and Health Plan (SSHP), which is included as Appendix B-2 to the RAWP.

All site drilling activities will be completed by a North Carolina certified driller. Work will be completed in accordance with the NCAC Well Construction Standards (NCDENR, 2009).

3.3 Project Schedule

The Project Schedule has been included as Section 6 of the RAWP.

4. Non-measurement Data Acquisition

As part of the SOW, it was not necessary to compile historical data from sources outside of the previously prepared Site reports. Non-measurement data acquisition is not anticipated at this time.

5. Field Activities

5.1 Geophysics

No geophysical investigations are included in this SOW.

5.2 Methane Monitoring

5.2.1 Rationale/Design

Methane monitoring has been incorporated into this remedial action (RA) in order to assess potential risk associated with by-products generated as part of the ERD in the groundwater. Six soil gas probes will be installed in the vadose zone, adjacent to the buildings where ERD is occurring, to monitor the potential for methane accumulation.

5.2.1.1 Methane Monitoring Locations

A total of six soil gas probes will be installed adjacent to occupied buildings to assess the potential for methane generation as a result of ERD. Proposed locations of soil gas probes are included on Figure 4-1 of the RAWP. Locations were selected in order to provide a means of evaluating methane generation and to assess potential risk associated with possible migration into occupied buildings in the vicinity of the site.

5.2.1.2 Sample Collection and Field and Laboratory Analysis

The potential for methane generation will be monitored on a monthly basis using a landfill gas meter during the timeframe when ERD injection events are being performed. Methane monitoring will be reduced to a quarterly frequency after ERD injections are complete, throughout the MNA monitoring period.

The soil gas will be measured in-situ using a Landtec GEM 2000 Gas Analyzer or equivalent device. Methane results will be recorded in percent methane. No samples will be collected for laboratory analysis.

5.2.1.3 Background, QA/QC, and Blank Samples and Frequency

Background methane levels will be established through the performance of a methane monitoring event, prior to the initiation of ERD injections. These values will be

compared to measurements collected during ERD and MNA phases of the project to determine the potential methane generation as a result of ERD.

5.2.2 Field Procedures

All field procedures for methane measurement will be in accordance with the U.S. Environmental Protection Agency (USEPA) Field Branches Quality System and Technical Procedures (USEPA, 2009) and the PIKA-PIRNIE JV Team standard operating procedures (SOPs).

5.2.2.1 Drilling Methods and Equipment

Utility protection procedures as described in Section 5.3.2 will be completed prior to any intrusive work. A decontaminated 2 to 3-inch diameter stainless steel hand auger will be used to advance soil gas probes to a depth of 6 feet bgs.

5.2.2.2 Materials (Casing, Screen, etc.)

Stainless steel soil gas probes will be constructed using 6-inch long, 1.25-inch inside diameter (ID) stainless steel screens. Teflon® tubing, 3/8-inch outside diameter (OD) by ¼-inch ID, will be inserted into each stainless steel soil gas implant to surface to extract the soil gas to ground surface. The end of the Teflon® tubing will be capped with a water tight Swagelok® fitting at ground surface.

5.2.2.3 Installation

The annular space surrounding the stainless steel soil gas implant will consist of filter pack sand (No. 1 sand) from the bottom of the soil gas probe to 6 inches above the top of the screen. Granular bentonite will be set from the top of the filter pack to surface as a seal, and will be hydrated in lifts. An 8-inch diameter flush-mounted manhole will be installed flush with the ground surface, encased in a 2-foot x 2-foot by 6-inch thick concrete pad.

5.2.2.4 Sampling Methods

Soil gas sampling will be conducted in accordance with the Soil Gas Sampling Operating Procedure, (SESDPROC)-307-R2 (USEPA, 2009) and the EM 200-1 SAP Preparation document, and the PIKA-PIRNIE JV Team SOPs.

Before initiating field activities, evaluate the anticipated subsurface conditions and potential methane concentrations to select the proper equipment for monitoring soil vapor. In general, the following equipment is required for soil vapor/methane monitoring:

- Appropriate health and safety (H&S) equipment.
- Tools and/or keys required for opening soil vapor vaults and probes.
- Logbook, marking pens.
- Appropriate monitoring instrument/meter with adequate detection levels.
- Charcoal filters to ensure that readings are methane specific.
- Tubing/hoses for connecting to the soil vapor probe.
- Disposable nitrile gloves.
- Towels.

The portable gas meter selected for monitoring soil vapor/methane measurements has a detection range capable of measuring the anticipated methane concentrations in the subsurface. The Landtec GEM 2000 has an internal pump capable of drawing sufficient volume to purge the soil gas probe void space and acquire a sample of soil vapor that is representative of the subsurface. If the pump is incapable of purging adequate soil vapors from the probes due to unforeseen subsurface conditions, a separate pump will be utilized to evacuate the soil vapor probe until a casing volume has been removed or soil vapor/methane readings stabilize.

The monitoring instrument/meter will be checked for calibration accuracy prior to each use, and recalibrated according to manufacturer's instructions if the calibration measurements are outside of the acceptable error range. This calibration check will be documented in the field book. The serial number of the equipment being used should also be recorded in the field book. Prior to collecting any soil vapor measurements, a background reading of the ambient air in the field must be collected and the measurement recorded in the field book and on any logging sheets.

When collecting measurements from installed soil vapor probes using portable meters, the following procedures should be followed:

- Inspect the soil vapor probe for damage, paying special attention to conditions that may compromise the atmospheric integrity of the soil vapor probe such as cracked or heaved surface seals, and bent or cracked riser pipes.

- Remove the Swagelok® fitting from the end of the Teflon® tubing and connect it to the portable gas meter. Monitor the methane until the concentration remains stable for approximately 45 seconds. A stable reading is one that does not vary more than 0.2 percent on the instrument/meter's scale. Record the stabilized reading, as well as any peak/highest reading. Note when the peak reading occurred, relative to the stabilized reading.

- In addition to recording the methane reading, record the sample location, date, time, observed weather conditions, barometric pressure, and the purge/ monitoring duration. Atmospheric pressure data can be obtained from a local weather station for the monitoring period.

5.2.2.5 Field Measurement Procedures and Criteria

Prior to collecting methane reading, the SSHP (Appendix B-2 of the RAWP) will be reviewed for all H&S protocol.

The sampler shall wear clean, disposable nitrile gloves at each sampling station. The gloves will be changed when their cleanliness is compromised.

5.2.2.6 Documentation

All field notes will be recorded in bound field logbooks, designated for the maintenance of field records. Field records will document aspects of the site setup and sample collection, preparation, and handling, and field analytical data (*i.e.,* calibration data, sample and quality control [QC] results, etc.) as outlined in Instruction F-1 Appendix F in the EM 200-1-3 SAP Preparation guidance document. **Appendix A** has the field sampling logs that will be used.

5.3 Ground Water

5.3.1 Rationale/Design

The implementation of the RA at the Site includes the installation of additional monitoring and injection wells and the subsequent monitoring of groundwater in order to monitor the performance of the RA. The following sections detail the methods that will be used for the groundwater monitoring portion of the project, including well installation and monitoring activities. The rational and design of the groundwater RA are discussed in Sections 3 and 4 of the RAWP.

5.3.1.1 Monitoring Well Location and Installation

Figures 4-1 and 4-2 of the RAWP present the well layouts for the transition zone and bedrock monitoring wells. The layout of the proposed injection wells are included on Figures 3-1 and 3-2 of the RAWP for the transition zone and bedrock HSUs, respectively. Figures 4-3 and 4-4 of the RAWP present the well construction diagrams for the transition zone and bedrock monitoring wells, respectively. Well construction details for the injection wells are included with the remedial system design drawings in Appendix A of the RAWP.

5.3.1.2 Sample Collection and Field and Laboratory Analysis

The baseline groundwater sampling event will include collection of samples from 32 monitoring wells, detailed in Appendix H of the RAWP. These wells provide a baseline measurement of the TCE in both the transition and bedrock zones. Performance groundwater monitoring events, conducted on a quarterly basis after each injection event, will include 20 wells listed on Appendix H of the RAWP. Results of performance monitoring will be utilized to determine if additional modifications to injection volumes or locations are necessary. In addition, the data will be used to evaluate the TCE and daughter product degradation as a result of the injections. MNA monitoring will be completed following the conclusion of ERD injections and will include sampling of the 32 wells listed on Appendix H of the RAWP. These wells will be monitored to gauge the continued effects of ERD after injection and the TCE will degrade into the daughter products.

Field parameters (pH, electrical conductivity, temperature, dissolved oxygen, oxidation-reduction potential [ORP], and turbidity) will be collected from each well using a calibrated water quality meter *(i.e.,* YSI 556 meter), prior to sample collection.

Groundwater samples will be collected in accordance with Appendix H of the RAWP. Groundwater samples will be analyzed using the analytical methods included on Table 4-1 of the QAPP.

Laboratory analysis of groundwater samples will be by a certified North Carolina Laboratories (TestAmerica [VOCs, total organic carbon (TOC), and biogeochemical parameters] and Microseeps [dissolved gases: Methane, Ethane, and Ethene]). Specific method preservation requirements, size, and type of sample containers to be used, and holding times for each parameter are presented in Table 5-1 of the QAPP.

5.3.1.3 Upgradient, Quality Assurance/Quality Control (QA/QC), and Blank Samples and Frequency

To monitor sampling and laboratory performance it will be necessary to collect several types of field QA/QC samples. The field QA/QC samples include trip blanks, equipment rinsate blanks, matrix spike/matrix spike duplicates (MS/MSDs), and field duplicates. Table 4-1 of the QAPP presents the estimated frequency for collection of QA/QC samples. The specific number and type of QA/QC samples that will be collected during each monitoring event may be more or less than the criteria stated below, based upon data quality objectives and professional judgment.

A trip blank is a container filled with distilled and organic-free water prepared in, and provided by the analytical laboratory. A trip blank is sent from the analytical laboratory to the field sampling site, and is returned to the laboratory for analysis. The trip blank results are used to evaluate whether contamination by VOCs occurred during shipment of samples and/or during container transport. One trip blank is required in each sample cooler transporting samples for VOC analysis.

Equipment rinsate blanks are collected immediately after the equipment has been decontaminated. Equipment rinsate blanks are collected by gently pouring distilled or deionized water over selected clean non-dedicated equipment and collected for laboratory analysis. For example, the equipment rinsate blank for soil and sediment sampling programs will be collected by gently pouring distilled or deionized water over clean core barrels or soil core samplers. The equipment rinsate blank for surface water and groundwater sampling programs will be collected by gently pouring distilled or deionized water over clean non-dedicated bailers or sampling cups. Equipment rinsate blanks will be collected at a frequency of 10 percent of the field samples at critical points in the sampling program, such as the sampling of a background well or the end of the sampling program.

The frequency requirements for collecting equipment rinsate blanks are a minimum of 10 percent of the environmental samples. The blank shall be analyzed for all laboratory analyses requested for the environmental samples collected at the Site. When an analyte is detected in the equipment rinsate blank, the appropriate validation flag, as described in the data validation section, shall be applied to all sample results from samples collected. It should be noted that the laboratory will supply the organic free water. A sample aliquot of the organic free water will be submitted for the analysis of all parameters of interest.

A field duplicate sample is a second sample collected at the same location as the original sample. Duplicate samples are collected simultaneously or in immediate succession, using identical recovery techniques, and treated in an identical manner during storage, transportation, and analysis. The sample containers are assigned an identification number in the field such that they cannot be identified (blind duplicate) as duplicate samples by laboratory personnel performing the analysis. Specific locations are designated for collection of field duplicate samples prior to the beginning of sample collection. A field duplicate will be collected at a rate of 1 per 10.

Field duplicate sample results are used to assess precision, including variability associated with both the laboratory analysis and the sample collection process. Field duplicates will be collected at a frequency of 10 percent of samples collected. Analytical results for field duplicate will be assessed during the data validation process. Specific locations will be designated for collection of field duplicate samples prior to the beginning of sample collection. Control limits for evaluation of precision for field duplicates will be 40 percent for aqueous samples and 70 percent for soil/sediment samples.

Laboratory QA protocols including the performance of laboratory control samples and matrix spikes relating to method acceptance criteria are included in the QAPP. The QAPP also defines the data qualification guidelines for evaluating potential matrix interferences identified during matrix spike analyses. The parent and field duplicate sample will be included in all reporting.

5.3.2 Well Installation

The PIKA-PIRNIE JV Team will be responsible for notifying designated USACE personnel of planned activities at least two weeks prior to the initiation of field activities. Property owners will also be notified in advance of field activities in accordance with their specific access agreement terms. At the time of notification, field personnel will

communicate to the USACE environmental staff the SOW to be completed, tentative schedule, and drilling locations.

Prior to mobilization for drilling activities, a utility clearance will be completed with three lines of evidence per the PIKA-PIRNIE JV Team SOP Utility Clearance ARCHSFS019. The first line of evidence will require the PIKA-PIRNIE JV Team to submit a utility locate request through the North Carolina 811 utility protection service. North Carolina 811 will accept these locate requests either by phone or internet. The phone number is 811 or (800) 632-4949. The North Carolina 811 web address is http://nc811.org/homepage.htm. In order to submit a request using the website, pre-registration and training is required.

Permits will be issued within three business days following the receipt of the request by North Carolina 811. The permits are valid for 21 days with renewal requests required a minimum of three days prior to expiration. Requesting contractors are responsible for maintaining marks during the 21-day period. If, after acquiring a permit, a utility is damaged during field activities, the appropriate utility company must be notified. The contractor should be prepared to submit proof of a valid permit at that time.

The second line of evidence includes the use of a private utility locator to clear each boring location. The locator will use a combination of electromagnetic and ground penetrating radar to evaluate the subsurface for potential utilities.

The third line of evidence includes conducting a detailed visual site inspection sheet to identify and confirm utility locations. The site representative and occupants should be consulted with to identify any potential utilities that may not be readily identified by non-intrusive clearing methods.

If any of these lines of evidence are deemed inadequate, alternative lines of evidence must be used (*i.e.,* air knife vacuuming to five feet, hydro knife vacuuming). As standard protocol, all borings are cleared to a depth of 5 feet bgs using a decontaminated hand auger. A Utilities and Structures Checklist (**Appendix B**) will be completed by the Field Geologist or Engineer for each area to be sampled prior to commencement of field activities. A copy of the completed checklist will be retained in the PIKA-PIRNIE JV Team project file.

5.3.2.1 Drilling Methods and Equipment

All monitoring and injection wells will be installed by a North Carolina-licensed water well driller. The contractors selected for this project shall comply with any and all installation, local, state, and federal H&S regulations and requirements. The contractors are responsible, per the PIKA-PIRNIE JV Team's contractual agreements, for securing and/or complying with permits required by state or local authorities.

All borings will be hand cleared to a depth of five feet to the OD of hollow stem augers to be used to install in the transition and bedrock zone monitoring and injection wells. A hand auger can be used to advance three holes orientated in a triangular pattern that are sufficient to clear the OD of the hollow stem augers to be used.

5.3.2.1.1 Hollow Stem Auger

The upper reaches of each well will be drilled using hollow-stem auger techniques (American Society for Testing and Materials [ASTM] 1452), from ground surface until auger refusal. Additional flights of auger will continue to be added in 5-foot increments to achieve the desired borehole depth. At that time refusal in encountered, the drilling technique will be modified to air rotary.

All soil cuttings generated during the drilling of the boreholes will be containerized (drum or roll-off box) and temporarily stored onsite while awaiting characterization. Additional details regarding the soil cuttings are detailed in the IDW Management Plan (Appendix F of the RAWP).

5.3.2.1.2 Air Rotary

Air rotary drilling uses air as a means of cooling the bit and removing drill cuttings. The air hammer is a drill bit that pulverizes the material as it is advanced through the subsurface. Initially, the drilling equipment will be inserted through the hollow stem augers, which act as a casing to hold back the unconsolidated saprolite and partially weathered rock (PWR). The air rotary will be used to advance the boreholes, in both transition zone and bedrock wells, to the desired total depth. Once the design depth is reached, the air rotary tools are removed and the well can be completed as designed.

5.3.2.1.3 Method by Well Type

The drilling methods for each well type include:

- Transition zone monitoring and injection wells:
    - Overburden will be drilled with 6.25-inch ID hollow stem augers to refusal. Once refusal is encountered, the drilling will switch to air rotary drilling using a 6-inch diameter air rotary bit to drill through the PWR to an estimated depth of 25 feet bgs.
- Bedrock monitoring wells:
    - The overburden will be drilled with 10.25-inch ID hollow stem augers to refusal. Once refusal is encountered, the drilling will switch to air rotary using a 10-inch diameter air rotary bit to drill through the PWR to competent bedrock.
    - A 6-inch polyvinyl chloride (PVC) surface casing will be installed in to competent bedrock, which is anticipated at a depth of 25 feet bgs. Then, the casing will be grouted in place using neat Portland Type 1 cement. The grout around the casing will be allowed to cure for a minimum of 12 hours.
    - The well will then be drilled using 6-inch air rotary to a depth of 250 feet bgs.
- Bedrock injection wells:
    - The overburden will be drilled with 10.25-inch ID hollow stem augers to refusal. Once refusal is encountered, the drilling will switch to air rotary using a 10-inch diameter air rotary bit to drill through the PWR to competent bedrock.
    - A 6-inch PVC surface casing will be installed in to competent bedrock, which is anticipated at a depth of 25 feet bgs. Then, the casing will be grouted in place using neat Portland Type 1 cement. The grout around the casing will be allowed to cure for a minimum of 12 hours.
    - The well will then be drilled using 6-inch air rotary to a depth of 100 feet bgs.

5.3.2.2 Materials

5.3.2.2.1 Casing/Screen/Centralizers

The well construction materials for each well type include:

- Transition zone monitoring wells:
    - 10-foot long, 2-inch ID, 10-slot, 304 stainless steel, wire-wrapped screens.
    - Estimated screened interval from 15 feet to 25 feet bgs, but may vary based on subsurface conditions encountered.
    - 2-inch ID Schedule 80 PVC riser to grade.
- Transition zone injection wells:
    - 17-foot long, 2-inch ID, 20-slot, 304 stainless steel, wire-wrapped screens.
    - Estimated screened interval from 8 feet to 25 feet bgs, but may vary based on subsurface conditions encountered.
    - 2-inch ID Schedule 80 PVC riser to grade.
- Bedrock monitoring wells:
    - 20-foot long, 2-inch ID, 10-slot, 304 stainless steel, wire-wrapped screens.
    - Estimated screened interval from 230 to 250 feet bgs, but may vary based on subsurface conditions encountered.
    - 2-inch ID Schedule 80 PVC riser to grade.
- Bedrock injection wells:

- 70-foot long, 2-inch ID, 20-slot, 304 stainless steel, wire-wrapped screens.
- Estimated screened interval from 30 to 100 feet bgs, but may vary based on subsurface conditions encountered.
- 2-inch ID Schedule 80 PVC riser to grade.

5.3.2.2.2 Filter Pack, Bentonite Seal, Cement/Bentonite Grout

The filter pack and well seal materials for each well type include:

- Transition zone monitoring wells:
  - #1 filter sand (#1) will be added in the annular space around the screen from the base of the well and extend approximately 2 feet above the top of the screen.
  - Six inches of very fine sand will be added on top of the #1 filter sand.
  - Approximately 2 feet of bentonite pellets will be added above the very fine sand.
  - The remaining annular space will be grouted using neat Portland Type 1 cement.
- Transition zone injection wells:
  - #2 filter sand (#2) will be added in the annular space around the screen and extend approximately 2 feet above the top of the screen.
  - Two feet of very fine sand will be added on top of the #2 filter sand.
  - The remaining annular space will be grouted using neat Portland Type 1 cement.

- Bedrock monitoring wells:
    - #1 filter sand (#1) will be added in the annular space around the screen and extend approximately 2 feet above the top of the screen.
    - One foot of very fine sand will be added on top of the #1 filter sand.
    - Approximately 2 feet of bentonite pellets will be added above the very fine sand.
    - The remaining annular space will be grouted using neat Portland Type 1 cement. Given the length of the annular space, the grout will be added in three lifts.
- Bedrock injection wells:
    - #2 filter sand (#2) will be added in the annular space around the screen and extend approximately 2 feet above the top of the screen.
    - Two feet of very fine sand will be added on top of the #2 filter sand.
    - The remaining annular space will be grouted using neat Portland Type 1 cement.

5.3.2.2.3 Surface Completion

The surface completions for each well type include:

- Transition zone monitoring wells:
    - 8-inch diameter flush-mounted manhole
    - 2-foot by 2-foot by 6-inch concrete pad
- Transition zone injection wells:

- 12-inch diameter flush-mounted manhole
- 3-foot by 3-foot by 6-inch concrete pad

- Bedrock monitoring wells:
  - 12-inch diameter flush-mounted manhole
  - 3-foot by 3-foot by 6-inch concrete pad
- Bedrock injection wells:
  - 12-inch diameter flush-mounted manhole
  - 3-foot by 3-foot by 6-inch concrete pad

5.3.2.2.4 Water Source

Potable water will be obtained on-site from the Charlotte Municipal Utility Department (CMUD) via two separate water line connections. One connection will be from the water line located along Nevada Boulevard, the second from a water line located along Cordage Street.

5.3.2.2.5 Delivery, Storage, and Handling of Materials

All materials shipped or carried onto the site will be stored in a temporary lay down area, at a location designed by the respective property owners. Materials will be properly stored in accordance with the manufacturer's requirements and general industry standards.

5.3.2.3 Installation

5.3.2.3.1 Soil Sampling and Rock Coring During Drilling

Soil samples will not be collected for laboratory analysis during drilling activities as soils and bedrock characterization has been previously completed at the site However, lithologic descriptions will be prepared based on auger cuttings, air rotary cuttings and observations during drilling.

Composite samples will be collected for disposal purposes in accordance with the IDW Management Plan, included in the RAWP as Appendix F.

5.3.2.3.2 Borehole Diameter and Depth

Proposed well construction diagrams providing the borehole diameter and boring depth for the monitoring wells are presented in Figures 4-3 and 4-4 of the RAWP for the transition zone and bedrock wells, respectively. Well construction details for the injection wells are included with the remedial system design drawings in Appendix A of the RAWP.

5.3.2.3.3 Screen and Well Casing Placement

Proposed well construction diagrams providing the screen and well casing placement for the monitoring wells are presented in Figures 4-3 and 4-4 of the RAWP for the transition zone and bedrock wells, respectively. Well construction details for the injection wells are included with the remedial system design drawings in Appendix A of the RAWP.

5.3.2.3.4 Filter Pack Placement

Proposed well construction diagrams providing the filter pack placement for the monitoring wells are presented in Figures 4-3 and 4-4 of the RAWP for the transition zone and bedrock wells, respectively. Well construction details for the injection wells are included with the remedial system design drawings in Appendix A of the RAWP.

5.3.2.3.5 Bentonite Seal

Proposed well construction diagrams providing the bentonite seal placement for the monitoring wells are presented in Figures 4-3 and 4-4 of the RAWP for the transition zone and bedrock wells, respectively. No bentonite seal will be used for the injection wells.

5.3.2.3.6 Cement/Bentonite Grout Placement

Proposed well construction diagrams providing the Portland Type 1 (neat) cement placement for the monitoring wells are presented in Figures 4-3 and 4-4 of the RAWP for the transition zone and bedrock wells, respectively. Well construction details for the

injection wells are included with the remedial system design drawings in Appendix A of the RAWP.

5.3.2.3.7 Protective Cover Placement

Proposed well construction diagrams providing the surface completion details for the monitoring wells are presented in Figures 4-3 and 4-4 of the RAWP for the transition zone and bedrock wells, respectively. Well construction details for the injection wells are included with the remedial system design drawings in Appendix A of the RAWP.

5.3.2.3.8 Well Identification

A numbering system has been developed for the monitoring wells installed during this phase of field investigation. Well nomenclature for this phase of remedial action will be as follows:

- The abbreviation for the site – NAD;
- The monitoring well designation – MW followed by;
- The well number (*e.g.*, 01, 10).

For example, the PIKA-PIRNIE JV Team field personnel will identify the first proposed monitoring well as NAD MW-66.

The nomenclature for the injection wells will include:

- Transition zone injection wells will be labeled as IW-# (ex. IW-12)
- Bedrock injection wells will be labeled as BIW-# (ex. BIW-7)

Figures 3-1, 3-2, 4-1, and 4-2 of the RAWP provide the proposed well locations/names for the proposed wells.

5.3.2.3.9 Well Development

Each monitoring and injection well will be properly developed after installation in order to:

- Restore the natural permeability of the formation adjacent to the borehole;
- Remove fines from the filter pack and well casing so that subsequent water samples will not be abnormally turbid or contain undue suspended matter; and
- Remove remnant drilling fluids/contaminants from the well, filter pack, and aquifer that may have been introduced during the drilling process.

The following equipment will be utilized to complete well development activities:

- PPE;
- submersible or centrifugal pump;
- discharge or suction tubing;
- bailer with rope;
- generator;
- air compressor (dependant on method used);
- surge block arrangement;
- jetting equipment;
- water quality meter (pH, conductivity, temperature, and turbidity meter);
- equipment for monitoring or determining flow rate;
- disposable beaker;
- well development log; and
- arrangements for storage or disposal of development water.

Each newly installed transition zone monitoring and injection wells will be developed no earlier than 24 hours after well installation. In addition to following the PIKA-PIRNIE JV

Team SOP Monitoring Well Development, the procedures used for development are as follows:

- An initial specific capacity test will be performed prior to the development of the well. The test will be completed using a constant flow rate and will continue until the water level is stabilized. The anticipated duration of the test will be 1 to 2 hours depending on the stabilization of the water level. A constant flow rate of 1 to 2 gallons per minute is estimated based on existing well data.

- Starting at the bottom of the well, the well will be surged in 2-foot depth intervals. A total of 10 minutes will be spent on each 2-foot depth interval (80 to 100 minutes total for entire well screen). A pump will be used above the jetting assembly to maintain a constant water level *(i.e.,* volume of water injected through the jetting process should be removed at an equivalent rate during pumping).

- At the end of the jetting/scrubbing, the solids will be removed from the well until water is free of visible sediment. This will be completed using an air-lift methodology.

- A second specific capacity test will be completed to determine the amount of change post-development. This test will be completed using the same procedures and flow rate as the initial specific capacity test.

The development techniques that will be used for the bedrock injection and monitoring wells are included below:

- The well will be surged along the screened portion of the well for a period of 30 minutes prior to pumping.

- The well will then be pumped for a period of 30 minutes at a constant rate. This will allow the sediment to be removed from the base of the well and provide an estimate of the specific capacity of the well.

- Repeat the surging for another 30 minute period.

- Repeat 30 minutes of pumping (same pumping rate. If the second specific capacity value is consistent with the first value, development will be considered

complete. If there is a significant difference, an additional surging and pumping cycle will be completed.

5.3.2.3.10 Well Survey

A survey of newly installed monitoring wells will be conducted by a North Carolina registered land surveyor to measure elevations (X, Y, and Z coordinates) of any new wells. The north side of the top of the monitoring well casing and the land surface adjacent to each well will be surveyed relative to mean sea level to the nearest 0.01 foot. The horizontal location of each monitoring point and well will also be determined relative to the North Carolina State Plane coordinate system (northing, easting coordinates) to the nearest 0.1 foot. The survey coordinates and elevations will be tied into the existing on-site benchmark and/or existing wells.

5.3.2.4 Documentation

Field activities will be documented on the PIKA-PIRNIE JV Team pre-printed forms or a bound field logbook. Notes will be compiled into a field log, consisting of notes and drawings describing the location, field conditions, and activities completed. Examples of the field log forms are provided in **Appendix A**.

All aspects of sample collection and handling as well as visual observations will be documented on designated forms or field logbooks. All sample collection equipment (where appropriate), field analytical equipment, and equipment utilized to make physical measurements shall be identified in the field logbooks. All calculations, results, and calibration data for field sampling, field analytical, and field physical measurement equipment shall be recorded on the forms or in the field logbooks. In addition, the Field Operations Leader will fill out a daily site activity log that details the activities and/or issues that occurred that day.

Entries in field logbooks or the field forms will be dated, legible, and contain accurate and inclusive documentation of an individual's project activities. At the end of each day's activity the documents in the field will be secured by the Field Operations Leader for each task. Once completed, these field logbooks and/or preprinted forms will be maintained as a part of the project files.

Data forms will be completed in indelible ink. Each blank of the data form should be completely filled out. Where there is no data entry, enter "UNK" for Unknown, "NA" for Not Applicable, or "ND" for Not Done. To revise an entry, the person making the

revision will strike a single line through the mistake, add the correct information above or adjacent to it, and initial the change.

5.3.2.4.1 Logs and Well Installation Diagrams

Soil borings, boreholes, and monitoring well installations completed by the field team will be documented on Boring/Well Construction Logs (**Appendix A**). The logs will document the drilling location, drilling dates and times, drilling personnel, logging personnel, soil descriptions, sample depths, recovery, boring location and volatile organic vapor content. The log will also document the well identification, drilling method, development technique, well construction materials, material depths, and abandonment, if any.

5.3.2.4.2 Development Records

Development records of monitoring and injections wells completed by the field team will be documented on Development Well Log (**Appendix A**). The logs document the monitoring well location, depth to fluid levels before and after developing the wells, water quality data, field personnel, logging personnel, and development technique.

5.3.2.4.3 Decommission/Abandonment Records

In the event that a borehole is not able to be completed for use as a monitoring or injection well, the borehole will be properly abandoned. Abandonment will be completed by a licensed North Carolina well driller in accordance with the NCAC Well Construction Standards (Title 15A, Subchapter 2C, Section .0100 (NCDENR, 2009).

The procedures for abandoning boreholes are as follows:

- The entire borehole will be grouted with a cement and bentonite slurry containing high solids or neat Portland Type 1 cement mixed to the manufacturer's specifications. The bentonite slurry will be placed with a tremie pipe from the bottom of the annular area to be grouted to ensure proper placement of the slurry.

5.3.2.4.4 Photographs

Relevant events, findings, or activities during the remedial action will be documented with photographs. Photographs will be compiled into photo logs with each photo identified with the following information:

- Date;
- Time;
- Photographer (signature);
- Name of site; and
- General direction faced and description of the subject in the photo.

5.3.2.5 Well Decommission/Abandonment

Well decommissioning and abandonment procedures have been documented in Section 5.3.2.4.3.

5.3.2.6 Water Level Measurement

Water level measurements will be referenced to a surveyed elevation point located on the top of the well casing. An electronic water level probe will be used to gauge the water level in the new wells, in addition to the existing monitoring wells and piezometers at the facility.

Site wide water level measurements will be collected at the site wells within a 24-hour period, where no measureable rainfall has occurred. The total well depth may also be measured at this time to verify if sediment has accumulated in the well, resulting in a reduction in the effective well depth. Water level measurements will begin with downgradient wells (*i.e.,* inferred least contaminated wells) and proceed toward the estimated source areas (*i.e.,* inferred most contaminated wells). Water-level measurements will be collected within a single 24-hour period and will be measured twice to check the reproducibility of the data. This measurement validation helps ensure accuracy with regard to the water level data collection. The procedure for obtaining water level measurements is as follows:

1. Describe the area surrounding the well, whether or not the lock was secure (if applicable), if the well could have been impacted by surface water runoff,

ambient weather conditions and other factors that could affect the final data analysis. This documentation is recorded on a Water Level Measurement Form (**Appendix A**).

2. Decontaminate the gauging equipment prior to collecting the initial water level measurement and between all wells. Decontamination procedures are described in Section 5.3.9.

3. Unlock the protective casing and remove the inner cap on the riser. Caution should be used when opening well caps around the injection zone because the well may be under pressure.

4. Check the probe to verify that it is operational, then lower down the monitoring well.

5. If the well is not vented, allow the water level to equilibrate for a few minutes prior to collecting the first measurement. Take fluid level measurements from a fixed reference point (the north side of the top of the PVC riser) using an electric tape graduated in 0.01-foot intervals.

6. Repeat the measurements until two measurements are obtained that are within 0.01 ft.

7. Remove and decontaminate the probe, replace the inner cap, and lock the protective casing.

5.3.3 Aquifer Testing

No aquifer testing is planned for this scope of work.

5.3.4 Field Measurement Procedures and Criteria

Several instruments may be used to collect field analytical data. These instruments include a pH meter, specific conductance meter, a thermometer, dissolved oxygen meter, and turbidity meter (nephelometer). A YSI 556 water quality meter or equivalent will be used to measure pH meter; specific conductivity; temperature; ORP; and dissolved oxygen. An additional nephelometer will be required to measure turbidity.

Field instruments will be calibrated at the beginning of each day, and more often if conditions warrant. Calibration procedures will follow manufacturer's specifications and will be documented by field personnel on the Field Instrument Calibration Log (**Appendix A**).

5.3.5 Sampling Methods for Ground Water – General

The following protocol has been developed to obtain groundwater samples that are representative of formation conditions and is intended for use in sampling monitoring wells during the field activities. New monitoring wells will not be sampled for at least 48 hours following well development. Monitoring wells will be purged prior to collecting groundwater samples to ensure that representative formation water is being sampled. The monitoring wells will be purged and sampled in the same order as that for water-level measurements (downgradient to upgradient, or least contaminated to most contaminated where known based upon prior sampling results). Prior to introduction into the well, all non-dedicated equipment and materials will be decontaminated in accordance with the procedures outlined in Section 5.3.9.

The following low flow groundwater sampling procedures will be implemented when performing well purging prior to sample collection:

- Put on clean nitrile gloves.
- Unlock the metal protective casing, remove the well cap, and document the general condition of the well.
- Determine static fluid-level elevation using electronic probe (see Section 5.3.2.6). Record on Groundwater Sampling Form (**Appendix A).**
- Insert the pre-cleaned bladder (or peristaltic) pump and tubing into the well to the midpoint of the well screen. Record installation time in field notes.
- Start pump at the lowest possible flow rate and adjust the pumping rate to approximately 100 milliliters per minute (mL/min). Record pump start time in field notes. Verify the flow rate with the graduated cylinder or equivalent by collecting the water from the discharge line for one minute. Record results in field notes.

- Monitor water level to verify that little or no drawdown (0 to 0.1 foot) is occurring in the well. If desired, the flow rate may be increased to up to 300 mL/min in more permeable formations as long as little or no drawdown is observed in the well. Record measurements and flow rates in field notes.

- Obtain field parameter measurements (temperature, specific conductance, pH, dissolved oxygen, ORP, and turbidity) every 5 minutes and record on the Groundwater Sample Log. Purge until the criteria listed below have been met (unless low well recovery precludes this).

- The field parameters are considered stable when the following conditions are met for three consecutive meter readings taken at least 5 minutes apart:

    - pH within 0.1 standard units

    - specific conductance within 3 percent

    - Dissolved oxygen within ± 10 percent

    - Turbidity is less than 10 nephelometric turbidity units (NTUs) or within ± 10 percent if the turbidity is greater than 10 NTUs

- Collect VOC and dissolved gas samples for laboratory analysis at a low flow rate (100 mL/min) directly into the appropriate sample container. If a peristaltic pump is used, the downhole tubing will be filled using suction and removed from the well to prevent the sample from contacting the pump head. The pump speed is reduced and the direction reversed to push the sample out of the tubing and into the sample containers. Ensure that no air bubbles are present in the vial. Secure sample container lid and store sample containers in chilled cooler after filling out the sample label.

- Collect additional samples for non-VOC analysis. Dissolved metals will be field filtered using an in-line 0.45 micron filter.

- Secure sample container lids and store sample containers in chilled cooler.

- Complete sampling documentation on the Groundwater Sampling Form, record the collection date and time on the sample key, and fill out the Well Sampling Summary form (**Appendix A**).

- If inadequate water is present to fill the required sample containers, return periodically within 24 hours until adequate sample volume is obtained and field parameters measured. Collect groundwater for individual analyses in the appropriate sample order. If required, collect VOCs and store first, then metals and other indicator parameters. If the recharge is no adequate after 24 hours, the well will be considered dry and no sample will be collected.

- If drawdown in the well cannot be maintained within the 0.3-foot requirement, sample collection will be performed after three well volumes of groundwater have been purged. Begin sample collection with VOC analysis unless otherwise noted in the site-specific work plan. For wells that purge dry before all of the samples are collected, allow the well to recover and then make one more attempt to collect the remaining samples within a 24-hour period.

- Turn off pump. Remove portable pump from well and decontaminate. Tubing will be disposed of after use.

- Replace cap on well and protective casing lock well.

- Grab samples will be collected using bailers for TOC monitoring.

5.3.6 Sample Handling Methods for Ground Water – Filtration

Dissolved metals will be field filtered using an in-line 0.45 micron filter.

5.3.7 Sample Containers and Preservation Techniques

All samples will be stored at approximately 4°C from immediately after collection until analysis. In the field and during transportation to the laboratory, samples will be kept in coolers on ice, not "blue ice". Ice for coolers will be double-bagged in self-sealing plastic bags. Protective foam or Styrofoam packing will be used to minimize the risk of breakage during transport. When packaging samples for commercial transport, individual bottles will be wrapped separately in padded materials. The top two copies of the original chain-of-custody form will be placed in a plastic bag secured inside the shipping container closed with a chain-of-custody seal.

5.3.8 Field Quality Control Sampling Procedures

See Section 5.3.1.3.

5.3.9 Decontamination Procedures

The cleaning procedures outlined in this section will be used by all personnel to clean sampling and other field equipment to prevent cross-contamination during separate phases of the investigation. Documentation regarding decontamination will be recorded on the Daily Log (**Appendix A**). Specific cleaning procedures are presented in the following section.

A decontamination area will be established where steam cleaning of the drilling and well construction equipment and materials will be completed with proper containment and disposal of wash water. An impervious decontamination area will be utilized and the water used to clean the equipment will be containerized for off-site disposal. A decon pad will be constructed at a designated location on site. A steam cleaner will be used to decon the hollow stem augers, and air rotary drilling equipment and any other associated drilling equipment before and after they are used. All water needs to be contained during decontamination. At the conclusion of decontamination, the water will be properly containerized in the frac tanks.

The laboratory detergent used to wash the equipment will be a standard brand of phosphate-free laboratory-grade detergent such as Micro or Liquinox. The use of any other detergent must be justified and documented in the field logbooks and inspection or investigative reports.

Potable water is defined as tap water fit for human consumption from a known source. Deionized water is defined as tap water that has been treated by passing through a standard deionizing resin column. The deionized water should contain no metals or other inorganic compounds (*i.e.,* at or above analytical detection limits). The brushes used to clean equipment as outlined in the following sections, will be stiff plastic bristled and will not be wire-wrapped.

Field or sampling equipment that needs to be repaired shall be identified with a tag indicating date repair requested, problem if known, personnel requesting repair, and if the equipment has been decontaminated. Field equipment needing cleaning or repairs will not be stored with clean equipment or sample containers. Field equipment and/or disposable sample containers that are not used during the course of an investigation may not be placed in storage without being recleaned unless it is the opinion of the field investigator that the materials have not become contaminated during the course of the field investigation. However, equipment and sample containers must be labeled as such.

The materials used to implement the cleaning procedures outlined in this section can be dangerous if improperly handled. Caution must be exercised by all personnel, and all applicable safety procedures shall be followed. At a minimum, the following precautions will be taken in the field during these cleaning operations:

- Safety glasses with side shields or goggles, and latex or vinyl surgical gloves or nitrile rubber gloves will be worn during all cleaning operations;
- All rinsing operations will be conducted in the open (never in a closed room); and
- No eating, smoking, drinking, chewing, or any hand-to-mouth contact shall be permitted during cleaning operations.

6. Field Operations Documentation

6.1 Daily Quality Control Reports (QCR)

During the RA activities, QCRs will be prepared daily, dated, signed by the project contractor quality control representative, and sent to USACE at a frequency specified in the PMP (Appendix D of the RAWP). These reports should include weather information at the time of sampling, field instrument measurements, calibrations, identification of all field and control samples taken, departures from the approved SAP necessary, deviations from approved drilling procedures (such as well installation specifications), any problems encountered, and instructions from government personnel. Any deviations that may affect data quality objectives must be conveyed to USACE personnel (technical manager, project geologist, project chemist, etc.) immediately.

The following should be attached to the daily contractor QCRs (if applicable):

- Copies of field/project forms generated
- QA sample summary
- Copies of chain-of-custody forms
- Field-generated analytical results

6.2 Field Logbook and/or Sample Field Sheets

Bound field logbooks will be used for the maintenance of field records. Field records include all aspects of the site setup and sample collection, preparation, and handling, and all field analytical reportable (*i.e.,* calibration data, sample and QC results, etc.) as outlined in Instruction F-1 Appendix F in the EM 200-1-3 SAP Preparation guidance document. Project-specific field sheets are included in **C-1 Appendix A.**

6.3 Photographic Records

See Section 5.3.2.4.4.

6.4 Sample Documentation

See Data Management Plan (DMP) and QAPP which are included as part of the SAP (Appendix C) to the RAWP.

6.5 Field Analytical Records

See DMP and QAPP which are included as part of the SAP (Appendix C) to the RAWP.

6.6 Documentation Procedures/Data Management and Retention

See DMP which is included as part of the SAP (Appendix C) to the RAWP.

7. Sample Packaging and Shipping Requirements

All samples will be placed in a sample cooler full of ice immediately after collection until analysis to be stored at approximately 4°C. In the field and during transportation to the laboratory, samples will be kept in coolers on ice, not "blue ice". Ice for coolers will be double-bagged in re-sealable plastic bags. Protective foam, bubble wrap, or Styrofoam packing will be used to minimize the risk of breakage during transport. When packaging samples for commercial transport, individual bottles will be wrapped separately in padded materials.

The top two copies of the original chain-of-custody form will be placed in a plastic bag secured inside the shipping container closed with a chain-of-custody seal.

After the coolers are delivered to the laboratory, the samples are logged in, the chain-of-custody is signed, and the samples are checked for breakage or leakage. The temperature of the ice bath is checked. If the temperature exceeds 4º C or if any other problems are noted, this information is recorded on the chain-of-custody and the Field Operations Leader or Project Manager (PM) will be notified of the problem.

8. IDW

All IDW management procedures are described in the IDW Management Plan, included as Appendix F of the RAWP.

9. Field Assessment/Three-Phase Inspection Procedures

Quality will be maintained throughout all phases of field work during execution of the remedial actions at the Former CNAD facility. QC procedures for RA including drilling, remedial system construction, groundwater monitoring, and information management have been detailed in the Construction Quality Control Plan (CQCP), included as Appendix G to the RAWP.

9.1 Construction Quality Control (CQC)

The PIKA-PIRNIE JV Team will maintain contractor QC as identified in the CQCP. The CQC Manager and the remainder of the field team will provide a continuous oversight of any on site activities. Each phase of work will be evaluated in accordance with the three-phase approach (preparatory, initial and follow up) methodology in order to insure that execution of activities is in accordance with the PWS, DO, and approved RAWP.

9.2 Sampling Apparatus and Field Instrumentation Checklist

Details of sampling equipment, field procedures, and quality control are provided in Section 5.3.1.3 of this plan.

10. Nonconformance/Corrective Actions

In the event that a QC inspection identifies a nonconformance with the specification, contract document or RAWP, corrective measures will be taken in order restore compliance with the associated requirement. Typical nonconformances associated with projects of this type include but are not limited to deviations in sampling procedures, faulty equipment as denoted by instrument calibration procedures, incomplete or improper sample preservation, and laboratory analysis. The QC program in place will provide a means of identifying these nonconformances early in the process, allowing for resolution of them without significant environmental, financial, or scheduling impacts to the project.

Nonconformances identified will be documented and tracked until project completion. The nonconformance tracking will document that any issues, identified as part of the QC program, were resolved in a complete and timely manner.

Appendix A

Field Sampling Logs

Daily Contractor Quality Control Reports

Project Name _____ Project No. _____

Project Location _____ Contract No. _____

Prepared By _____ Date/Time _____

Signature _____ Weather _____

1. Field instrument measurements: _____

2. Calibration of field equipment: _____

3. Field and control samples collected: _____

4. Departures from approved SAP: _____

5. Deviations from procedures: _____

6. Problems encountered: _____

7. Instructions from USACE personnel: _____

Attachments:

QA sample tables
Chain-of-custody records
Field-generated analytical results
Project forms

PIKA INTERNATIONAL, INC. — A JOINT VENTURE — MALCOLM PIRNIE

Daily Log

Project Name _____ Project No. _____

Project Location _____ Contract No. _____

Prepared By _____ Date _____

Title _____ Arrival Time _____

Weather _____ Departure Time _____

Name of site of investigation:
(e.g., NAD MW-66) _____

Location of site of investigation
GPS/northing/easting) _____

Purpose of site visit/sampling activity: _____

Name/address of field contact: _____

Name/responsibilities of all site field staff: _____

Name/affiliations/purpose of visitors: _____

Appropriate PPE for site work: _____

Equipment used for site work:
(brand name, serial number)
(source/quality, lot numbers)
(retain certs of info supplied) _____

Sample location:
(provide sketch) _____

Sample collection method: _____

Field measurements
(field results, calibration methods) _____

Chemicals to be analyzed:
(constituents/lab methods) _____

QA/QC Samples _____

Decontamination procedures: _____

Photologs/Site maps _____

Scheduling modifications/change
orders/changes in drilling/sampling
procedures _____

Number of sample coolers, COCs,
mode of shipping transportation/track
tracking numbers _____

Receiving laboratories
(name/address) _____

Type of waste to be handled: _____

IDW Documentation _____

Signature _____

METHANE AND
BREATHING ZONE MONITORING FORM

Site: _____

Date Measured: _____

Meter Type: _____

Sampler: _____

METHANE MONITORING

Monitoring Point	Sampling Time	Parameter	Units	Initial/Peak Reading	Steady-State Reading	Comments
		Methane	% LEL			
		Methane [1]	ppmv			
		Methane	% LEL			
		Methane [1]	ppmv			
		Methane	% LEL			
		Methane [1]	ppmv			
		Methane	% LEL			
		Methane [1]	ppmv			
		Methane	% LEL			
		Methane [1]	ppmv			
		Methane	% LEL			
		Methane [1]	ppmv			
		Methane	% LEL			
		Methane [1]	ppmv			

BREATHING ZONE MONITORING

Monitoring Point	Sampling Time	Parameter	Units	Initial/Peak Reading	Steady-State Reading	Comments
		Methane	% LEL			
		Methane [1]	ppmv			
		Methane	% LEL			
		Methane [1]	ppmv			
		Methane	% LEL			
		Methane [1]	ppmv			
		Methane	% LEL			
		Methane [1]	ppmv			
		Methane	% LEL			
		Methane [1]	ppmv			
		Methane	% LEL			
		Methane [1]	ppmv			

Calibration Information (required): _____

Notes:

Collect initial/peak readings directly following the removal of the well cap.
Collect steady-state readings after five minutes with the well cap removed.
Collect breathing zone monitoring readings from an approximate height of 4 feet.

% LEL = % Lower Explosive Limit
ft bgs = feet below ground surface
ppmv = parts per million by volume

[1] Methane is measured in the field for % LEL and converted to methane concentration by: (% LEL/100) x 50,000 ppmv
10% LEL = 0.5% methane by volume = 5,000 parts per million by volume
25% LEL = 1.25% methane by volume = 12,500 parts per million by volume

If Breathing Zone methane concentration is **greater than 10%**, notify ARCADIS Project Manager.

If Breathing Zone methane concentration is **measured at 25% LEL**, immediately stop work, exit work area, and notify Project Manager.

Instrument Calibration Log

Project Name: _____ Date: _____

Project Number: _____

Calibrating Personnel: _____

Time of Calibration: _____

Weather Conditions: _____

Barometric Pressure: _____ inches H x 25.4 = _____ mm Hg

CALIBRANT	INSTRUMENT	INITIAL READING	VALUE ENTERED	FINAL READING	TIME	TEMP
pH 10.0			10.0			
pH 7.00			7.0			
pH 4.00			4.0			
Conductivity (mS/cm)						
Turbidity (NTU)						
DO (mg/L)						
DO%						
ORP (mV)						
_____						
_____						
_____						

Notes: _____

PIKA INTERNATIONAL, INC. — A JOINT VENTURE — MALCOLM PIRNIE

Groundwater Sample Log

Page ____ of ____

Project No. _____ Well ID _____ Date _____

Project Name/Location _____ Weather _____

Measuring Pt. Description _____ Screen Setting (ft-bmp) _____ Casing Diameter (in.) _____ Well Material ____ PVC ____ SS ____ Other

Total Depth (ft-bmp) _____ Static Water Level (ft-bmp) _____ Water Column in Well _____ Gallons in Well _____

Calc.Gallons Purged _____ Pump Intake (ft-bmp) _____ Purge Method: PDB _____ Sample Method _____

Gallons Purged _____ MP Elevation _____ Submersible _____

Disp. Bailer _____ Pump On/Off _____

Sample Time: Label _____ Replicate/ Code No. _____ Peristaltic _____

Sampled by _____

Time	Minutes Elapsed	Gallons Purged	Depth to Water (ft) TOC	Temp. (°C) (°F)	Cond. (µmhos) (mS/cm)	Dissolved Oxygen (mg/L)	pH	ORP (mV)	Appearance		Comments
									Color	Odor	

Constituents Sampled	**Container**	**Number**	**Preservative**

Well Information

Well Location: _____ Well Locked at Arrival: Yes / No

Condition of Well: _____ Well Locked at Departure: Yes / No

Well Completion: Flush Mount / Stick Up Key Number To Well: _____

NOTES: _____

Well Casing Volumes

Gallons/Foot					
	1" = 0.04	1.5" = 0.09	2.5" = 0.26	3.5" = 0.50	6" = 1.47
	1.25" = 0.06	2" = 0.16	3" = 0.37	4" = 0.65	

GROUNDWATER LEVEL MEASUREMENTS/ MONITORING SYSTEM INSPECTION FORM

Date: ____________________

Completed by: ________________________________

Well ID	Protective Casing Condition Good (G)/Damaged (D)	Locked Yes/No	Water Level Measurement (feet bMP)	Comments

PIKA INTERNATIONAL, INC. A JOINT VENTURE MALCOLM PIRNIE

Sample/Core Log

Boring/Well _____ Project/No. _____ Page _____ of _____

Site Location _____ Drilling Started _____ Drilling Completed _____

Total Depth Drilled _____ Feet Hole Diameter _____ inches Type of Sample/ Coring Device _____

Length and Diameter of Coring Device _____ Sampling Interval _____ feet

Land-Surface Elev. _____ feet ☐ Surveyed ☐ Estimated Datum _____

Drilling Fluid Used _____ Drilling Method _____

Drilling Contractor _____ Driller _____ Helper _____

Prepared By _____ Hammer Weight _____ Hammer Drop _____ ins.

Sample/Core Depth (feet below land surface) From	To	Core Recovery (inches)	PID Reading (ppm)	Sample/Core Description

Sample/Core Log (Cont.d)

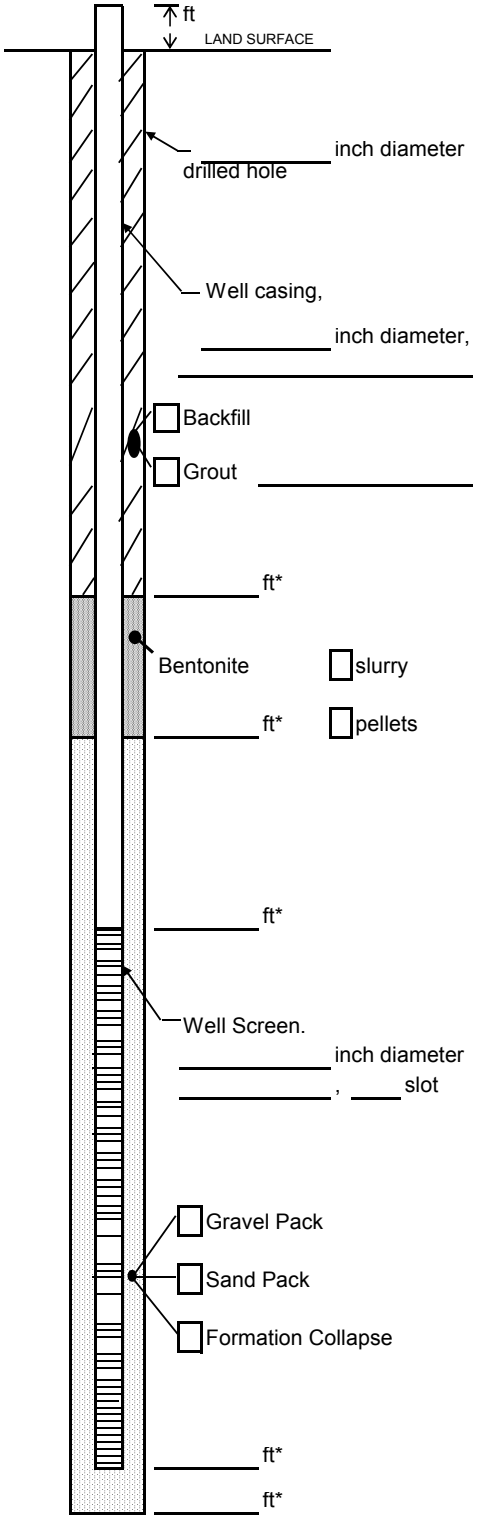
Boring/Well _____ Page _____ of _____

Prepared by _____

Sample/Core Depth (feet below land surface) From	To	Core Recovery (inches)	PID Reading (ppm)	Sample/Core Description

Transition Zone Well Construction Log

(Unconsolidated)



Measuring Point is
Top of Well Casing
Unless Otherwise Noted.

* Depth Below Land Surface

Project _____ Well _____

Town/City _____

County _____ State _____

Permit No. _____

Land-Surface Elevation and Datum:

_____ feet ☐ Surveyed ☐ Estimated

Installation Date(s) _____

Drilling Method _____

Drilling Contractor _____

Drilling Fluid _____

_____

Development Technique(s) and Date(s)

_____

_____

_____

Fluid Loss During Drilling _____ gallons

Water Removed During Development _____ gallons

Static Depth to Water _____ feet below M.P.

Pumping Depth to Water _____ feet below M.P.

Pumping Duration _____ hours

Yield _____ gpm Date _____

Specific Capacity _____ gpm/ft

Well Purpose _____

_____

_____

Remarks _____

_____

_____

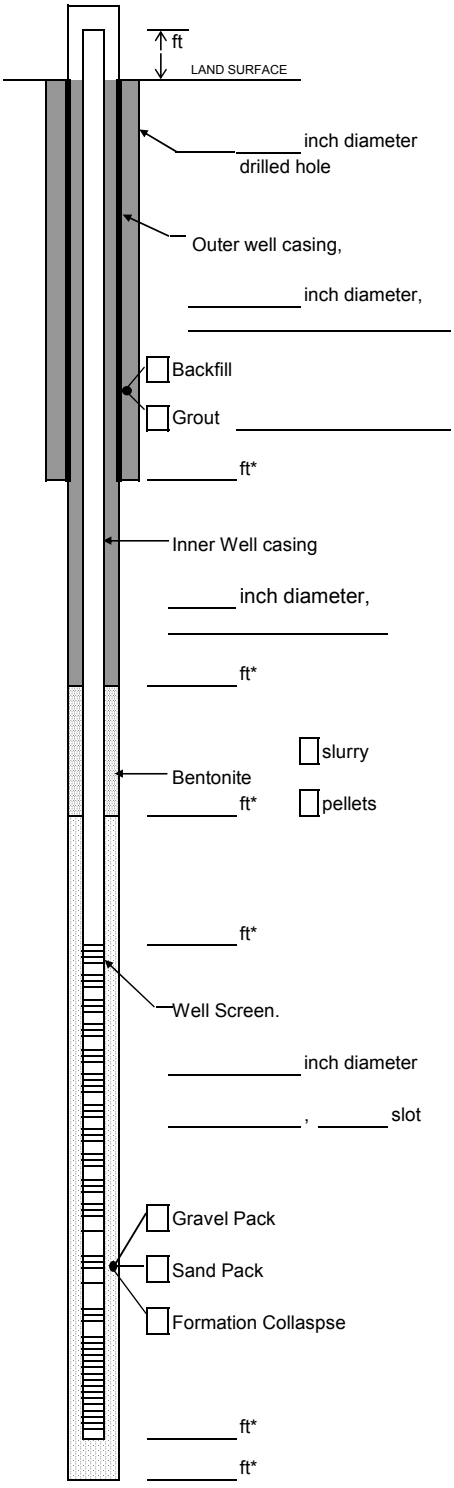
_____

_____

Prepared by _____

Well Completion Logs.xlsx.xls
Well Diag-TZ

Bedrock Well Construction Log



Measuring Point is
Top of Well Casing
Unless Otherwise Noted.

* Depth Below Land Surface

TBD = To be determined.

Project _____ Well _____

Town/City _____

County _____ State _____

Permit No. _____

Land-Surface Elevation and Datum:

_____ feet ☐ Surveyed

☐ Estimated

Installation Date(s) _____

Drilling Method _____

Drilling Contractor _____

Drilling Fluid _____

Development Technique(s) and Date(s)

Fluid Loss During Drilling _____ gallons

Water Removed During Development _____ gallons

Static Depth to Water _____ feet below M.P.

Pumping Depth to Water _____ feet below M.P.

Pumping Duration _____ hours

Yield _____ gpm Date _____

Specific Capacity _____ gpm/ft

Well Purpose _____

Remarks _____

Prepared by _____

Monitoring Well Development Log

Page _____ of _____

Project/No. _____ Well _____ Date _____

Total Depth _____ Casing Diameter (inches) _____ Purge Method

Water Level _____ Well Volume (gal) _____ Centrifugal _____

Water Column _____ Total Volume Purged _____ Submersible _____

Pump On _____ Pump Off _____ Other _____

Developed By _____

	Well Casing Volumes			
gallon/foot	1-¼" = 0.06	2" = 0.16	3" = 0.37	4" = 0.65
	1-½" = 0.09	2-½" = 0.26	3-½" = 0.50	6" = 1.47

Time	Minutes Elapsed	Rate (gpm) (mL/min)	DTW (ft)	Gallons Purged	pH	Specific Conductance (mS/cm)	Temp. (C) (F)	DO (mg/L)	OPR	Turbidity (NTU)

Investigation Derived Waste Log

Page ___ of ___

Project Name/Location _____ Date _____

Project/No. _____ Weather _____

Technician _____ Project Manager _____

Date	Container Number	Container Type (drum, rolloff, tank)	Container Capacity	Container Description (OT, CT, etc.)	Media (SO, GW)	Content Description	Source	Volume from Source	Storage Location

NOTES:

(C-1) A-12

Project Name:

City/State

PHOTO

Photo No.:

Date:

Time:

Direction:

Description:

PHOTO

Photographed By:

Photo No.:

Date:

Time:

Direction:

Description:

Photographed By:

Appendix B

Utilities and Structures Checklist

Utilities and Structures Checklist

Project: _____
Project Number: _____
Date: _____
Work locations applicable to this clearance checklist:

Pre-Field Work

One Call or "811" notified 48-72 hours in advance of work? ☐ Yes ☐ No
Utility companies notified during the One Call process ☐ See attached ticket

_____ _____ _____ _____
_____ _____ _____ _____
_____ _____ _____ _____

List any other utilities requiring notification: ☐ None

_____ _____ _____ _____

Client provided utility maps or "as built" drawings showing utilities? ☐ Yes ☐ No

Field Work

Markings present: ☐ Paint ☐ Pin flags/stakes ☐ Other ☐ None

Subsurface Utility Lines of Evidence Used (3 Minimum):

☐ One Call/"811"
☐ Client Provided Maps/Drawings
☐ Client Clearance
☐ Interviews: Name(s)/Affiliation(s) _____
_____
_____

Did persons interviewed indicate depths of any utilities in the subsurface?
☐ Yes, depths provided:

☐ Did not know or refused to answer

Comments:

☑ Site Inspection
☐ GPR
☐ Air-Knife
☐ Hydro-Knife
☐ Public Records/Maps
☐ Radiofrequency
☐ Metal Detector
☐ Handauger
☐ Potholing
☐ Probing
☐ Private Locator: Name and Company: _____
☐ Marine Locator: Name and Company: _____
☐ Other:

_____
_____

Tips for Successful Utility Location:
1. No excessive turning or downward force of handaugers/shovels, etc.
2. No hammering- no pickaxes-no digging bars-no hurrying or shortcutting
3. Select alternate/backup locations for clearance
4. Utilities may run directly under asphalt/concrete or be > 5 ft depth
5. Be on site when utilizing private utility locators

Site Inspection

During inspections look for the following ("YES" requires follow up investigation):

		Utility color codes		
a)	Natural gas line present (evidence of a gas meter)?	Yellow	☐ Yes	☐ No
b)	Evidence of subsurface electric lines :	Red		
	i) Conduits to ground from electric meter?		☐ Yes	☐ No
	ii) Overhead electric lines absent		☐ Yes	☐ No
	iii) Light poles, electric devices with no overhead lines?		☐ Yes	☐ No
c)	Evidence of water lines:	Blue		
	i) Water meter on site?		☐ Yes	☐ No
	ii) Fire hydrants in vicinity of work?		☐ Yes	☐ No
	iii) Irrigation systems?		☐ Yes	☐ No
d)	Evidence of sewers or storm drains:	Green		
	i) Restrooms or kitchen on site?		☐ Yes	☐ No
	ii) Gutter down spouts going into ground		☐ Yes	☐ No
	iii) Grates in ground in work area		☐ Yes	☐ No
e)	Evidence of telecommunication lines:	Orange		
	i) Fiber optic warning signs in areas?		☐ Yes	☐ No
	ii) Lines from cable boxes running into ground?		☐ Yes	☐ No
	iii) Conduits from power poles running into ground?		☐ Yes	☐ No
	iv) Aboveground boxes or housings in work area?		☐ Yes	☐ No
f)	Underground storage tanks:			
	i) Tank pit present?		☐ Yes	☐ No
	ii) Product lines running to dispensers/buildings?		☐ Yes	☐ No
	iii) Vent present away from tank pit?		☐ Yes	☐ No
g)	Proposed excavation markings in work area?	White	☐ Yes	☐ No
h)	Other:			
	i) Evidence of linear asphalt or concrete repair		☐ Yes	☐ No
	ii) Evidence of linear ground subsidence or change in vegetation?		☐ Yes	☐ No
	iii) Manholes or valve covers in work area?		☐ Yes	☐ No
	iv) Warning signs ("Call Before you Dig", etc) on or adjacent to site?		☐ Yes	☐ No
	v) Utility color markings not illustrated in this checklist?		☐ Yes	☐ No
i)	Aboveground lines in or near the work area:			
	i) < 50 kV within 10 ft of work area?		☐ Yes	☐ No
	ii) >50 - 200 kV within 15 ft of work area?		☐ Yes	☐ No
	iii) >200-350 kV within 20 ft of work area?		☐ Yes	☐ No
	iv) >350-500 kV within 25 ft of work area?		☐ Yes	☐ No
	v) >500-750 kV within 35 ft or work area?		☐ Yes	☐ No
	vi) >750-1000 kV within 45 ft of work area?		☐ Yes	☐ No

Comments:

Do not initiate intrusive work if utilities are suspected to be present in area and are not located, markings are over 14 days old, or if clearance methods provide incomplete or conflicting information. Do not perform intrusive work within 30 inches of a utility marking without hand clearing.

Name and signature of person completing the checklist:

Name: _____

Signature: _____

Date: _____

APPENDIX C-2
Quality Assurance Project Plan

Former Charlotte Naval Ammunition Depot
Charlotte, North Carolina

December 2011

DERP-FUDS Project No. I04NC080301
Contract No.:W912DY-10-D0025
Delivery Order No.: 0007

PREPARED FOR:

U.S. Army Corps of Engineers, Huntsville Center
U.S. Army Engineering and Support Center
4820 University Square
Huntsville, Alabama 33816-1822

Quality Assurance Project Plan

Former Charlotte Naval
Ammunition Depot
Charlotte, North Carolina

Ray Livermore
USACE Project Manager

Patrick Shirley
PIKA-PIRNIE JV Team Certified Project Manager

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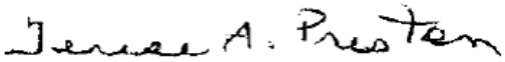

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Acronyms and Abbreviations

CNAD	Charlotte Naval Ammunition Depot
CFR	Code of Federal Regulations
COC	Chain of Custody
DMP	Data Management Plan
DoD	Department of Defense
DQO	Data Quality Objective
EDD	Electronic Data Deliverable
FSP	Field Sampling Plan
GC/MS	Gas Chromatography/Mass Spectrometry
H&S	Health and Safety
IDW	Investigation-Derived Waste
JV	Joint Venture
LCS	Laboratory Control Samples
LOQ	Level of Quantitation
MDL	Method Detection Limit
MNA	Monitored Natural Attenuation
MS	Matrix Spike
MS/MSD	Matrix Spike/Matrix Spike Duplicate
NCAC	North Carolina Administrative Code
NCDENR	North Carolina Department of Environment and Natural Resources
NEIC	National Enforcement Investigations Center
PIKA	PIKA International, Inc.

PIKA-PIRNIE JV Team	PIKA International, Inc./Malcolm Pirnie, Inc. Joint Venture LLC Team
Pirnie	Malcolm Pirnie, Inc.
QA	Quality Assurance
QAPP	Quality Assurance Project Plan
QA/QC	Quality Assurance/Quality Control
QC	Quality Control
QSM	Quality Systems Manual
RAO	Remedial Action Objective
RAWP	Remedial Action Work Plan
RPD	Relative Percent Difference
SAP	Sampling and Analysis Plan
SDG	Sample Delivery Group
SOP	Standard Operating Procedure
TCE	Trichloroethylene
TOC	Total Organic Carbon
USACE	United States Army Corps of Engineers, Huntsville Center
USEPA	United States Environmental Protection Agency
UST	Underground Storage Tank
VOC	Volatile Organic Compound

1. Introduction

The PIKA International, Inc. (PIKA)/Malcolm Pirnie, Inc. (Pirnie) Joint Venture (JV), LLC (the PIKA-PIRNIE JV Team), on behalf of the United States Army Corps of Engineers, Huntsville Center (USACE), has prepared this Quality Assurance Project Plan (QAPP) for the Former Charlotte Naval Ammunition Depot (CNAD) site located in Charlotte, North Carolina. This QAPP is being included as Appendix C-2 (Sampling and Analysis Plan [SAP]) of the Remedial Action Work Plan (RAWP).

This QAPP was prepared in a manner consistent with the following reference and guidance documents:

- United States Environmental Protection Agency (USEPA) guidance document entitled *EPA Requirements for Quality Assurance Project Plans*, EPA-QA/R-5 (USEPA 2001), which replaces QAMS-005/80, *Interim Guidance and Specifications for Preparing QA Project Plans.* (USEPA, 1980)

- USEPA *Guidance for Quality Assurance Project Plans,* EPA-QA/G-5. (USEPA, 2002)

- USACE. *Requirements for the Preparation of Sampling and Analysis Plans*. EM 200-1-3. (USACE, 2001)

- The National Enforcement Investigations Center (NEIC) *Policies and Procedures Manual.* (USEPA, 1991)

Information contained in this QAPP has been organized into the following sections:

Section	Content
1	Project Laboratory Organization and Responsibilities
2	Data Assessment Organization and Responsibilities
3	Data Quality Objectives
4	Sample Receipt, Handling, Custody and Holding Time Requirements
5	Analytical Procedures
6	Data Reduction/Calculation of Data Quality Indicators
7	Laboratory Operations Documentation
8	Data Assessment Procedures

Details on each of the subjects listed above are provided in the subsequent sections.

2. Project Organization

2.1 Project Organization

The activities to be completed under the RAWP will require integration of personnel from the organizations identified below, collectively referred to as the "Project Team." A detailed description of the responsibilities of each member of the Project Team is presented in Project Management Plan, which is included as Appendix D in the RAWP.

2.1.1 Project Management

The PIKA-PIRNIE JV Team will perform related sampling activities and will evaluate data and prepare the deliverables as specified in the Work Plans. Project direction will be provided by the USACE with lead regulatory oversight by the North Carolina Department of Environment and Natural Resources (NCDENR). A list of project management personnel related to laboratory data and assessment is provided below.

Company/Organization	Title	Name	Phone Number
PIKA-PIRNIE JV Team	Quality Assurance Coordinator	Dennis Capria	315.671.9299
	Database Manager	Maribel Vital	303.471.3425
Analytical Laboratory – TestAmerica Chicago	Project Manager	Marilyn Krueding	708.534.5200
	Quality Assurance Manager	Terese Preston	708.534.5200
Analytical Laboratory – Microseeps	Project Manager	Robbin Robl	412.826.5245
	Quality Assurance Manager	Patrick McLoughlin	412.826.5245

2.2 Team Member Responsibilities

The responsibilities of the above Project Team members are summarized below by organization.

2.2.1 Quality Assurance (QA) Coordinator

Responsibilities and duties for the QA Coordinator include:

- Oversee the review of laboratory data packages by data validation staff.
- Oversee and interface with the analytical laboratory.
- Coordinate field quality assurance/quality control (QA/QC) procedures with Task Managers, concentrating on field analytical measurements and practices to meet data quality objectives (DQOs).
- Perform and review audit reports.
- Prepare interim QA/QC compliance reports.
- Oversee the preparation of QA/QC report in accordance with USEPA guidelines, including an evaluation of laboratory data and data usability reports.

2.2.2 Analytical Laboratories

General responsibilities and duties of the analytical laboratories include:

- Perform sample analyses and associated laboratory QA/QC procedures.
- Supply sample bottles and shipping cartons.
- Maintain laboratory custody of sample.
- Strictly adhere to all protocols in the QAPP.

2.2.2.1 Laboratory Project Manager

Responsibilities and duties include:

- Serve as primary communication link between PIKA-PIRNIE JV Team and laboratory technical staff.

- Monitor workloads and maintain availability of resources.
- Oversee preparation of analytical reports.
- Supervise in-house chain-of-custody (COC).

2.2.2.2 QA Manager

Responsibilities and duties include:

- Supervise personnel reviewing and inspecting all project-related laboratory activities.
- Conduct audits of all laboratory activities.

2.3 Project Organization Chart

The project organization chart for the above personnel is presented below.

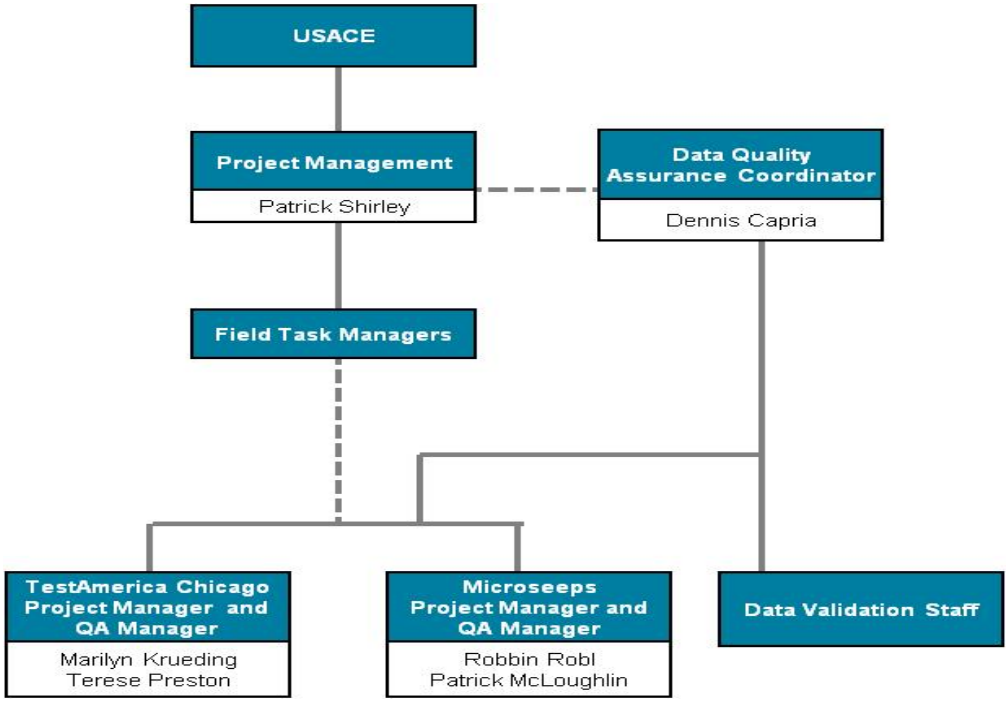

Figure 2-1: Project Organization Chart

3. Data Assessment Organization and Responsibilities

The Data Assessment Team is listed in the organization chart (**Figure 2-1**). The responsibilities of the above Data Assessment Team members are summarized below:

3.1 Data Validation Staff

Responsibilities and duties include:

- Review laboratory data packages.
- Interface with the analytical laboratory when questions arise with the data packages.
- Prepare a QA/QC report in accordance with USEPA guidelines, including an evaluation of laboratory data and data usability reports.
- Add validation qualifiers to the database.

4. DQOs

The remedial action objectives (RAOs) presented in the RAWP include:

- Address trichloroethylene (TCE) at concentrations exceeding 500 µg/L in groundwater through enhanced bioremediation, using dilute molasses solution injections in both the Transition and Bedrock Zones.

- Monitored Natural Attenuation (MNA) to reduce TCE concentrations to below the North Carolina Administrative Code (NCAC) 2L standard 2.8 µg/L.

The DQOs include obtaining data necessary to evaluate the effectiveness of meeting the RAOs and evaluating the progress of the Former CNAD site remediation in meeting the North Carolina groundwater quality standard for TCE and its daughter products, vinyl chloride and cis-1,2-dichloroethylene.

4.1 Data Use Background

4.1.1 Data Use

The data will be used to determine the effectiveness of the groundwater bioremediation.

4.1.2 Data Quantity

The sample quantities and quality control (QC) requirements are summarized in **Table 4-1**. Additional information regarding the choice of specific sample collection locations can be found in the RAWP.

4.1.3 Sampling and Analytical Methods

Sampling methods will be described in the RAWP. The analytical methods are as specified in **Table 4-1**. Level 2 reporting will be used for definitive data reporting (as defined below).

4.1.4 Measurement Performance Criteria

Precision and accuracy QC limits for chemical constituents used during data review to assess analytical performance are included in **Table 4-2**. Reporting limits are

presented in **Tables 4-3** and **4-4**. Data representativeness is addressed by the sample quantities and locations identified in the RAWP. Data comparability is intended to be achieved through the use of standard USEPA-approved methods. Data completeness will be assessed at the conclusion of the analytical activities.

Groundwater samples will be collected from monitoring wells as described in the Field Sampling Plan (FSP). The groundwater samples will be analyzed for:

- Volatile organic compounds (VOCs) by SW-846 8260
- Dissolved gases (methane, ethane, ethene) by Microseeps method AM20 GAX
- Total Organic Carbon (TOC) by SW-846 9060

In addition, MNA parameters will be collected for baseline sampling, and then annually.

- Total and dissolved iron and manganese by SW-846 6010
- Alkalinity by SM 2320B
- Nitrate and sulfate by SW-846 9056

Data categories have been defined to address various analytical data uses and the associated QA/QC effort and methods required to achieve the desired levels of quality. These categories are:

- **Screening Data:** Screening data afford a quick assessment of site characteristics or conditions. This DQO is applicable to data collection activities that involve rapid, non-rigorous methods of analysis and QA. This objective is generally applied to physical and/or chemical properties of samples, the degree of contamination relative to concentration differences, and preliminary health and safety (H&S) assessment.
- **Definitive Data:** Definitive data are generated using analytical methods, such as approved USEPA reference methods. Data are analyte-specific, with confirmation of analyte identity and concentration. Methods produce raw data (*e.g.,* chromatograms, spectra, and digital values) in the form of paper printouts or computer-generated electronic files.

It is anticipated that screening data and definitive data will be used during the remedial action. For this project the level of data reporting for definitive data has been defined as follows:

- **Level 2 – Modified Reporting:** Modified reporting is used for analyses that are performed following standard USEPA-approved methods and QA/QC protocols. Based on the intended data use, modified reporting may require some supporting documentation, but not full Contract Laboratory Program-type reporting. Examples of supporting documentation include, but may not be limited to, method blank results, laboratory control sample (LCS) recoveries, matrix spike (MS) recoveries and relative percent difference (RPD), and surrogate recoveries. Raw data is not required for Level 2 modified reporting.

The analytical analysis will be performed by TestAmerica located at Chicago, Illinois, and Microseeps of Pittsburgh, Pennsylvania. The analytical results will be reported by the laboratory in the electronic data deliverable (EDD) format outlined in EQuIS Laboratory Standard Operating Procedure (SOP) FSMP Rev. 5 (**Appendix A**). The EDD and Level 2 data packages from the laboratory will be due within 10 working days from date of sample receipt.

4.2 Measurement Quality Objectives for Chemical Data Measurement

The overall QA objective for this QAPP is to develop and implement procedures for sampling, COC, laboratory analysis, instrument calibration, data reduction and reporting, internal QC, audits, preventive maintenance, and corrective action, such that valid data will be generated. These procedures are presented or referenced in the following sections. Specific QC checks are discussed in Section 6.3.

QA indicators are generally defined in terms of six parameters:

1. Representativeness
2. Comparability
3. Completeness
4. Precision

5. Accuracy

6. Sensitivity

Each parameter is defined below. Specific objectives for the Former CNAD site are set forth in other sections of this QAPP, as referenced below.

4.2.1 Representativeness

Representativeness is the degree to which sampling data accurately and precisely represent site conditions and is dependent on sampling and analytical variability and the variability of environmental media at the site. The actions have been designed to assess the presence of the chemical constituents at the time of sampling. The RAWP presents the rationale for sample quantities and location. This QAPP presents field sampling and laboratory analytical methodologies. The use of the prescribed field and laboratory analytical methods, with associated holding times and preservation requirements, is intended to provide representative data.

4.2.2 Comparability

Comparability is the degree of confidence with which one data set can be compared to another. Comparability between phases of the actions (if additional phases are required) will be maintained through consistent use of the sampling and analytical methodologies set forth in this QAPP, established QA/QC procedures, and the utilization of appropriately trained personnel.

4.2.3 Completeness

Completeness is defined as a measure of the amount of valid data obtained from an event compared to the total amount that was obtained. As a general guideline, overall project completeness is expected to be at least 90 percent. The assessment of completeness will require professional judgment to determine data usability for intended purposes.

4.2.4 Precision

Precision is a measure of the reproducibility of sample results. The goal is to maintain a level of analytical precision consistent with the objectives of the action. To maximize precision, sampling and analytical procedures will be followed. All work for the

remedial action will adhere to established protocols presented in the QAPP. Checks for analytical precision will include the analysis of laboratory duplicates, and field duplicates. Checks for field measurement precision will include duplicate field measurements.

4.2.5 Accuracy

Accuracy is a measure of how close a measured result is to the true value. Both field and analytical accuracy will be monitored through initial and continuing calibration of instruments. In addition, reference standards, MSs, blank spikes, and surrogate standards will be used to assess the accuracy of the analytical data.

Laboratory accuracy will be assessed via the use of MSs, surrogate spikes, internal standards, and reference standards. Where available and appropriate, QA performance standards will be analyzed periodically to assess laboratory accuracy.

4.2.6 Sensitivity

Sensitivity is defined as the ability o f the method or instrument to detect the contaminant of concern and other target compounds at the level of interest. The method detection limit (MDL) is defined as the minimum concentration of a substance that can be identified, measured, and reported with a 99 percent confidence that the analyte concentration is greater than zero and is determined from repeated analysis of a sample in a given matrix containing the analyte. MDLs have been determined as required in Title 40 of the Code of Federal Regulation (CFR) Part 136B. The level of quantitation (LOQ) is greater than or equal to the lowest standard used to establish the calibration curve. The LOQs for this remedial action are generally at least two times greater than the MDL. Results greater than the MDL and less than the LOQ will be qualified estimated (J) by the laboratory.

Table 4-1
Sample Quantities and Quality Control Frequencies
Former Charlotte Naval Ammunition Depot - Charlotte North Carolina

Parameter	Estimated Env. Sample Quantity	Field QC Analyses						Laboratory QC Sample						Total
		Trip Blank		Rinse Blank		Field Duplicate		Matrix Spike		Matrix Spike Duplicate		Lab Duplicate		
		Freq.	No.	Freq.	No.	Freq.	No.	Freq.	No.	Freq.	No.	Freq.	No.	
Groundwater														
Volatile Organic Compounds (SW846 8260B)	32	1/cooler	3	1/day	3	1/10	3	1/10	3	1/10	3	NA	--	47
Dissolved Gases (AM20GAX)	32	NA	--	1/day	3	1/10	3	NA	--	NA	--	NA	--	38
Total Iron and Manganese (SW846 6010C)	32	NA	--	1/day	3	1/10	3	1/10	3	1/10	3	NA	--	44
Dissolved Iron and Manganese (SW846 6010C)	32	NA	--	1/day	3	1/10	3	1/10	3	1/10	3	NA	--	44
Total Organic Carbon (SW846 9060A)	32	NA	--	1/day	3	1/10	3	1/10	3	1/10	3	NA	--	44
Nitrate, Sulfate (SW846 9056A)	32	NA	--	1/day	3	1/10	3	1/10	3	1/10	3	NA	--	44
Alkalinity (SM 2320B)	32	NA	--	1/day	3	1/10	3	NA	--	NA	--	1/10	3	41
Adapative Design Sampling														
Volatile Organic Compounds (SW846 8260B)	10	1/cooler	10	NA	--	NA	--	NA	--	NA	--	NA	--	20
Investigation-Derived Waste (IDW)														
TCLP VOCs (SW846 1311/8260B)	TBD	1/cooler	1	NA	--	NA	--	NA	--	NA	--	NA	--	1
TCLP SVOCs (SW846 1311/8270C)	TBD	NA	--	NA	--	NA	--	NA	--	NA	--	NA	--	0
TCLP Metals (including Mercury) (SW846 1311/6010/7470A)	TBD	NA	--	NA	--	NA	--	NA	--	NA	--	NA	--	0

Notes:
Estimated Sample Quantity for Baseline Monitoring is 32 samples. Subsequent sampling rounds vary between 20 and 32 samples. When 20 samples are collected, the field QC Analyses will be 2 field duplicates, 2 Matrix Spike, and 2 Matrix Spike Duplicate.
Samples will be collected at the field engineers descretion in order to assit in injection transect layout.
Sample counts are an approximation.
1/day = One rinse blank per day or one per 10 samples, whichever is more frequent. Rinse blanks not required when dedicated sampling equipment is used.
Freq = Frequency
NA = Not Applicable
No. = Number
QC = Quality Control
SVOCs = Semi-volatile Organic Compounds
TBD = To Be Determined
TCLP = Toxicity Characteristic Leaching Procedure
VOCs = Volatile Organic Compounds

Table 4-2
Analytical Quality Control Limits
Former Charlotte Naval Ammunition Depot - Charlotte, North Carolina

Parameter	Accuracy - % Recovery			Precision - RPD		
	Surrogate	MS/MSD	LCS	MS/MSD	Lab Duplicate	Field Duplicate
Groundwater						
Volatile Organic Compounds						
Acetone	--	40-140	40-140	30	--	50
Benzene	--	80-120	80-120	30	--	50
Bromobenzene	--	75-125	75-125	30	--	50
Bromochloromethane	--	65-130	65-130	30	--	50
Bromodichloromethane	--	75-120	75-120	30	--	50
Bromoform	--	70-130	70-130	30	--	50
Bromomethane	--	30-145	30-145	30	--	50
2-Butanone (MEK)	--	30-150	30-150	30	--	50
n-Butylbenzene	--	70-135	70-135	30	--	50
sec-Butylbenzene	--	70-125	70-125	30	--	50
tert-Butylbenzene	--	70-130	70-130	30	--	50
Carbon disulfide	--	35-160	35-160	30	--	50
Carbon tetrachloride	--	65-140	65-140	30	--	50
Chlorobenzene	--	80-120	80-120	30	--	50
Chlorodibromomethane	--	60-135	60-135	30	--	50
Chloroethane	--	60-135	60-135	30	--	50
Chloroform	--	65-135	65-135	30	--	50
Chloromethane	--	40-125	40-125	30	--	50
2-Chlorotoluene	--	75-125	75-125	30	--	50
4-Chlorotoluene	--	75-130	75-130	30	--	50
1,2-Dibromo-3-Chloropropane	--	50-130	50-130	30	--	50
1,2-Dibromoethane	--	80-120	80-120	30	--	50
Dibromomethane	--	75-125	75-125	30	--	50
1,2-Dichlorobenzene	--	70-120	70-120	30	--	50
1,3-Dichlorobenzene	--	75-125	75-125	30	--	50
1,4-Dichlorobenzene	--	75-125	75-125	30	--	50
Dichlorodifluoromethane	--	30-155	30-155	30	--	50
1,1-Dichloroethane	--	70-135	70-135	30	--	50
1,2-Dichloroethane	--	70-130	70-130	30	--	50
1,1-Dichloroethene	--	70-130	70-130	30	--	50
cis-1,2-Dichloroethene	--	70-125	70-125	30	--	50
trans-1,2-Dichloroethene	--	60-140	60-140	30	--	50
1,2-Dichloropropane	--	75-125	75-125	30	--	50
1,3-Dichloropropane	--	75-125	75-125	30	--	50
2,2-Dichloropropane	--	70-135	70-135	30	--	50
1,1-Dichloropropene	--	75-130	75-130	30	--	50
cis-1,3-Dichloropropene	--	70-130	70-130	30	--	50
trans-1,3-Dichloropropene	--	55-140	55-140	30	--	50
Ethylbenzene	--	75-125	75-125	30	--	50
2-Hexanone	--	55-130	55-130	30	--	50
Hexachlorobutadiene	--	50-140	50-140	30	--	50
Isopropylbenzene	--	75-125	75-125	30	--	50
p-Isopropyltoluene	--	75-130	75-130	30	--	50
Methylene Chloride	--	55-140	55-140	30	--	50
4-Methyl-2-pentanone (MIBK)	--	60-135	60-135	30	--	50
Methyl tert-butyl ether	--	65-125	65-125	30	--	50
Naphthalene	--	55-140	55-140	30	--	50
N-Propylbenzene	--	70-130	70-130	30	--	50
Styrene	--	65-135	65-135	30	--	50
1,1,1,2-Tetrachloroethane	--	80-130	80-130	30	--	50
1,1,2,2-Tetrachloroethane	--	65-130	65-130	30	--	50
Tetrachloroethene	--	45-150	45-150	30	--	50
Toluene	--	75-120	75-120	30	--	50
1,2,3-Trichlorobenzene	--	55-140	55-140	30	--	50
1,2,4-Trichlorobenzene	--	65-135	65-135	30	--	50
1,1,1-Trichloroethane	--	65-130	65-130	30	--	50
1,1,2-Trichloroethane	--	75-125	75-125	30	--	50
Trichloroethene	--	70-125	70-125	30	--	50

Table 4-2
Analytical Quality Control Limits
Former Charlotte Naval Ammunition Depot - Charlotte, North Carolina

Parameter	Accuracy - % Recovery			Precision - RPD		
	Surrogate	MS/MSD	LCS	MS/MSD	Lab Duplicate	Field Duplicate
Trichlorofluoromethane	--	60-145	60-145	30	--	50
1,2,3-Trichloropropane	--	75-125	75-125	30	--	50
1,2,4-Trimethylbenzene	--	75-130	75-130	30	--	50
1,3,5-Trimethylbenzene	--	75-130	75-130	30	--	50
Vinyl chloride	--	50-145	50-145	30	--	50
o-Xylene	--	80-120	80-120	30	--	50
m&p-Xylene	--	75-130	75-130	30	--	50
Xylenes, Total	--	75-130	75-130	30	--	50
4-Bromofluorobenzene (Surr)	75-120	--	--	--	--	--
Dibromofluoromethane	85-115	--	--	--	--	--
1,2-Dichloroethane-d4 (Surr)	70-120	--	--	--	--	--
Toluene-d8 (Surr)	85-120	--	--	--	--	--
Dissolved Gases						
Methane	--	70-130	80-120	20	--	50
Ethane	--	70-130	80-120	20	--	50
Ethene	--	70-130	80-120	20	--	50
Total and Dissovled Metals						
Iron	--	80-120	80-120	20	--	50
Manganese	--	80-120	80-120	20	--	50
Wet Chemistry						
Total Organic Carbon	--	75-125	80-120	20	--	50
Nitrate	--	80-120	80-120	15	--	50
Sulfate	--	80-120	80-120	15	--	50
Alkalinity	--	--	80-120	20	20	50

Notes:
LCS = Laboratory Control Samples
MS/MSD = Matrix Spike/Matrix Spike Duplicate
RPD = Relative Percent Difference

Table 4-3
Parameters, Methods, and Target Quantitation Limits - Groundwater
Former Charlotte Naval Ammunition Depot - Charlotte, North Carolina

Analyte[1]	CAS Number	Groundwater		
		15A NCAC 2L.0202 Groundwater Standard	Laboratory	Laboratory
		(µg/L)	MDL (µg/L)	LOQ (µg/L)
Volatile Organic Compounds (Method 8260)				
Acetone	67-64-1	6000	1.9	5
Benzene	71-43-2	1	0.12	1
Bromobenzene	108-86-1	--	0.31	1
Bromochloromethane	74-97-5	--	0.5	1
Bromodichloromethane	75-27-4	0.6	0.23	1
Bromoform	75-25-2	4	0.45	1
Bromomethane	74-83-9	10	0.49	1
2-Butanone (MEK)	78-93-3	4000	1	5
n-Butylbenzene	104-51-8	70	0.21	1
sec-Butylbenzene	135-98-8	70	0.19	1
tert-Butylbenzene	98-06-6	70	0.24	1
Carbon disulfide	75-15-0	700	0.44	5
Carbon tetrachloride	56-23-5	0.3	0.28	1
Chlorobenzene	108-90-7	50	0.24	1
Chlorodibromomethane	124-48-1	0.4	0.25	1
Chloroethane	75-00-3	3000	0.33	1
Chloroform	67-66-3	70	0.25	1
Chloromethane	74-87-3	3	0.24	1
2-Chlorotoluene	95-49-8	100	0.21	1
4-Chlorotoluene	106-43-4	24	0.21	1
1,2-Dibromo-3-Chloropropane	**96-12-8**	**0.04**	**1.21**	**2**
1,2-Dibromoethane	**106-93-4**	**0.02**	**0.45**	**1**
Dibromomethane	74-95-3	70	0.39	1
1,2-Dichlorobenzene	95-50-1	20	0.21	1
1,3-Dichlorobenzene	541-73-1	200	0.26	1
1,4-Dichlorobenzene	106-46-7	6	0.24	1
Dichlorodifluoromethane	75-71-8	1000	0.26	1
1,1-Dichloroethane	75-34-3	6	0.24	1
1,2-Dichloroethane	107-06-2	0.4	0.28	1
1,1-Dichloroethene	75-35-4	--	0.29	1
cis-1,2-Dichloroethene	156-59-2	70	0.22	1
trans-1,2-Dichloroethene	156-60-5	100	0.27	1
1,2-Dichloropropane	78-87-5	1	0.36	1
1,3-Dichloropropane	142-28-9	--	0.27	1
2,2-Dichloropropane	594-20-7	--	0.31	1
1,1-Dichloropropene	563-58-6	--	0.25	1
cis-1,3-Dichloropropene	10061-01-5	0.4	0.28	1
trans-1,3-Dichloropropene	10061-02-6	0.4	0.35	1
Ethylbenzene	100-41-4	600	0.14	1
2-Hexanone	591-78-6	40	0.56	5
Hexachlorobutadiene	87-68-3	0.4	0.45	1
Isopropylbenzene	98-82-8	70	0.21	1
p-Isopropyltoluene	99-87-6	25	0.24	1
Methylene Chloride	75-09-2	5	0.63	2
4-Methyl-2-pentanone (MIBK)	108-10-1	100	0.79	5
Methyl tert-butyl ether	1634-04-4	20	0.28	1
Naphthalene	91-20-3	6	0.24	1
N-Propylbenzene	103-65-1	70	0.19	1
Styrene	100-42-5	70	0.26	1
1,1,1,2-Tetrachloroethane	630-20-6	1	0.31	1
1,1,2,2-Tetrachloroethane	**79-34-5**	**0.2**	**0.35**	**1**
Tetrachloroethene	127-18-4	0.7	0.22	1
Toluene	108-88-3	600	0.15	1
1,2,3-Trichlorobenzene	87-61-6	--	0.36	1
1,2,4-Trichlorobenzene	120-82-1	70	0.22	1
1,1,1-Trichloroethane	71-55-6	200	0.26	1
1,1,2-Trichloroethane	79-00-5	--	0.3	1
Trichloroethene	79-01-6	3	0.18	1
Trichlorofluoromethane	75-69-4	2000	0.22	1
1,2,3-Trichloropropane	**96-18-4**	**0.005**	**0.6**	**1**
1,2,4-Trimethylbenzene	95-63-6	400	0.22	1
1,3,5-Trimethylbenzene	108-67-8	400	0.23	1
Vinyl chloride	**75-01-4**	**0.03**	**0.13**	**1**
Xylenes, Total	1330-20-7	500	0.3	2

Table 4-3
Parameters, Methods, and Target Quantitation Limits - Groundwater
Former Charlotte Naval Ammunition Depot - Charlotte, North Carolina

Analyte[1]	CAS Number	Groundwater		
		15A NCAC 2L.0202 Groundwater Standard	Laboratory	Laboratory
		(µg/L)	MDL (µg/L)	LOQ (µg/L)
Dissolved Gases (Method Am20 GAx)				
Methane	74-82-8	--	0.023	0.1
Ethane	74-84-0	--	0.005	0.025
Ethene	74-85-1	--	0.008	0.025
Metals (SW846-6010)				
Iron, Total	7439-89-6	0.3	0.0279	0.2
Iron, Dissolved	7439-89-6	0.3	0.0279	0.2
Manganese, Total	7439-96-5	0.05	0.000944	0.01
Manganese, Dissolved	7439-96-5	0.05	0.000944	0.01
Wet Chemistry				
Total Organic Carbon (9060)	7440-44-0	--	0.36	1
Alkalinity (SM2320B)	NA	--	1.3	5
Nitrate as N (9056A)	14797-55-8	10	0.023	0.1
Sulfate (9056A)	14808-79-8	250	0.09	0.2

Notes:
1 USEPA. Office of Solid Waste and Emergency Response. *Test Methods for Evaluating Solid Waste. SW-846 3rd ed. Washington, D.C. 1996.*

Screening Criteria Source: North Carolina Division of Water Quality 15A NCAC 2L.0202 Groundwater Standards and Interim Maximum Allowable Concentrations

mg/L = milligrams per liter
µg/L = micrograms per liter
NA = Not Applicable

Table 4-4
Parameters, Methods, and Target Quantitation Limits - Investigation-Derived Waste
Former Charlotte Naval Ammunition Depot - Charlotte, North Carolina

Analyte	CAS Number	Leachate	
		Screening Criteria	Laboratory LOQ (mg/L)
Volatile Organic Compounds TCLP 1311/8260 [1]			
Benzene	71-43-2	0.5	0.02
Carbon tetrachloride	56-23-5	200	0.02
Chlorobenzene	108-90-7	0.5	0.02
Chloroform	67-66-3	100	0.02
1,2-Dichloroethane	107-06-2	6.0	0.02
1,1-Dichloroethene	75-35-4	0.5	0.02
2-Butanone (MEK)	78-93-3	0.7	0.1
Tetrachloroethene	127-18-4	0.7	0.02
Trichloroethene	79-01-6	0.5	0.02
Vinyl chloride	75-01-4	0.2	0.02
Semivolatile Organic Compounds TCLP 1311-8270 [1]			
2-Methylphenol	95-48-7	7.5	0.1
3 & 4 Methylphenol	15831-10-4	0.13	0.1
1,4-Dichlorobenzene	106-46-7	0.13	0.1
2,4-Dinitrotoluene	121-14-2	0.5	0.1
Hexachlorobenzene	118-74-1	3	0.1
Hexachlorobutadiene	87-68-3	200	0.1
Hexachloroethane	67-72-1	200	0.1
Nitrobenzene	98-95-3	200	0.1
Pentachlorophenol	87-86-5	2	0.5
Pyridine	110-86-1	100	0.2
2,4,5-Trichlorophenol	95-95-4	5	0.5
2,4,6-Trichlorophenol	88-06-2	400	0.1
TCLP-Metals 1311/6010/7470 [1]			
Arsenic	7440-38-2	5.0	0.05
Barium	7440-39-3	100	0.5
Cadmium	7440-43-9	1.0	0.005
Chromium	7440-47-3	5.0	0.025
Lead	7439-92-1	5.0	0.05
Selenium	7782-49-2	1.0	0.05
Silver	7440-22-4	5.0	0.025
Mercury	7439-97-6	0.2	0.002

Notes:

1 USEPA. Office of Solid Waste and Emergency Response. *Test Methods for Evaluating Solid Waste* SW-846 3rd ed. *Washington, D.C. 1996.*

5. Sample Receipt, Handling, Custody and Holding Time Requirements

5.1 Verification/Documentation of Cooler Receipt Condition

Appropriate sample containers, preservation methods, and laboratory holding times for the samples are shown in **Table 5-1**.

The analytical laboratory will supply appropriate sample containers and preservatives, as necessary. The bottles will be purchased pre-cleaned to USEPA Office of Solid Waste and Emergency Response Directive 9240.05A requirements (USEPA, 1992). The field personnel will be responsible for properly labeling containers and preserving samples (as appropriate). Sample handling and shipping procedures are described in the FSP. Sample labeling is included in the Data Management Plan (DMP).

Upon sample receipt, laboratory personnel will be responsible for sample custody. When shipping to the laboratory, the original field COC form will accompany all samples requiring laboratory analysis (**Appendix B**). The laboratory will use COC guidelines described in the USEPA guidance documents (USEPA, 2001). Samples will be kept secured in the laboratory until all stages of analysis are complete. All laboratory personnel having samples in their custody will be responsible for documenting and maintaining sample integrity.

5.1.1 Sample Receipt and Storage

Immediately upon sample receipt, the laboratory sample custodian will verify the integrity of the cooler seal, open the cooler, measure the temperature of the contents of the cooler, and compare the contents against the field COC. Laboratory personnel will be responsible for logging the samples in, assigning a unique laboratory identification number to each sample, labeling the sample bottle with the laboratory identification number, and moving the sample to an appropriate storage location to await analysis. The project name, field sample code, date sampled, date received, analysis required, storage location and date, and action for final disposition will be recorded in the laboratory tracking system. Relevant custody documentation will be placed in the project file.

5.2 Corrective Action for Incoming Samples

The samples are checked by the sample custodian as described above. Condition of the sample upon receipt must be documented.

The Project Manager, Task Manager and/or QA Coordinator must be notified when:

- A sample container is missing.
- A sample container is received broken.
- A sample is in an inappropriate container.
- A sample has not been preserved by appropriate means.
- Temperature blank upon receipt is greater than 6 degrees Celsius.

With the exception of VOCs, if the sample has been preserved correctly, but the proper pH was not achieved, appropriate preservative must be added to bring the sample to the proper pH. Any samples that do not meet the criteria and any adjustments made to samples must be documented in the case narrative of the analytical report.

Table 5-1
Sample Containers, Preservation, and Holding Times
Former Charlotte Naval Ammunition Depot - Charlotte, North Carolina

Parameter	Method	Bottle Type	Preservation	Holding Time
Groundwater				
Volatile Organic Compounds	8260B [1]	3 - 40 ml glass vials with Teflon®-lined lid with septum	Cool <4°C; pH <2 with HCl	14 days to analysis
Dissolved Gases	AM20GAX	2 x 40-ml glass vials	Trisodium Phosphate, Cool to ≤ 4°C	14 days to analysis
Total Iron and Manganese	6010C [1]	1-250 ml plastic	pH < 2 with HNO3	6 months to analysis
Dissolved Iron and Manganese	6010C [1]	1-250 ml plastic	Filter, then pH < 2 with HNO3	6 months to analysis
Total Organic Carbon	9060A [1]	2 - 40 ml glass vials with Teflon®-lined lid with septum	Cool <4°C; pH <2 with H_2SO_4	28 days to analysis
Nitrate	9056A [1]	1-250 ml plastic	Cool <4°C	48 hours to analysis
Sulfate				28 days to analysis
Alkalinity	SM 2320B	1-250 ml plastic	Cool <4°C	14 days to analysis
Investigation-Derived Waste				
TCLP VOCs	1311/8260B [1]	1 x 16 oz glass jar with Teflon®-lined lid with septum	Cool to <4°C	14 days to TCLP extraction 14 days to analysis
TCLP SVOCs	1311/8270 [1]		Cool <4°C	14 days to TCLP extraction, 7 days until extraction; 40 days to analysis
TCLP Metals	1311/6010C [1]		Cool <4°C	180 days to TCLP extraction 180 days to analysis

Notes:
[1] United States Environmental Protection Agency, Office of Solid Waste and Emergency Response. *Test Methods for Evaluating Solid Waste. SW-846 3rd ed. Update 4 Washington, D.C. 1996.*
All holding times are measured from the date of collection.
°C = degrees Celsius
HCl = hydrochloric acid
HNO3 = Nitric Acid
H2SO4 = Sulfuric Acid
ml = millileter
SVOCs = Semi-volatile Organic Compounds
TCLP = Toxicity Characteristic Leaching Procedure
VOCs = Volatile Organic Compounds

6. Analytical Procedures

The methods listed in Section 4.1 include the range of analyses expected to be performed. The associated laboratory SOPs can be found in **Appendix C (on CD)**. The QA Managers at each laboratory will be responsible for conducting and reporting corrective actions if problems arise during the course of laboratory analytical procedures.

Laboratory analytical requirements presented in the sub-sections below include a general summary of requirements, specifics related to each sample medium to be analyzed, and details of the methods to be used for this project. Current USEPA-approved and SW-846 methods will be used for all applicable parameters and sample media.

The tables listed below summarize the general analytical requirements:

Table	Title
4-1	Sample Quantities and Quality Control Frequencies
4-2	Analytical Quality Control Limits
4-3	Parameters, Methods, and Target Quantitation Limits – Groundwater
4-4	Parameters, Methods, and Target Quantitation Limits – Investigation-Derived Waste (IDW)
5-1	Sample Containers, Preservation, and Holding Times

The primary sources for methods used are provided in the following documents:

- Test Methods for Evaluating Solid Waste, SW-846 Third Edition, Update 4, USEPA, December 1996.
- Standard Methods for the Examination of Water and Wastewater, 20th Edition, American Public Health Association.
- Microseeps method AM20GAX

6.1 Preventive Maintenance

6.1.1 General

Laboratory instrument and equipment documentation procedures include details of any observed problems, corrective measure(s), routine maintenance, and instrument repair (including information regarding the repair and the individual who performed the repair).

Preventive maintenance of laboratory equipment generally will follow the guidelines recommended by the manufacturer. A malfunctioning instrument will be repaired immediately by in-house staff or through a service call from the manufacturer.

6.1.2 Instrument Maintenance

Maintenance schedules for laboratory equipment adhere to each manufacturer's recommendations. Records reflect the complete history of each instrument and specify the time frame for future maintenance. Major repairs or maintenance procedures are performed through service contracts with the manufacturer or qualified contractors. Paperwork associated with service calls and preventative maintenance calls will be kept on file by the laboratory.

Laboratory Systems Managers are responsible for the routine maintenance of instruments used in the particular laboratory. Any routine preventative maintenance carried out is logged into the appropriate logbooks. The frequency of routine maintenance is dictated by the nature of samples being analyzed, the requirements of the method used, and/or the judgment of the Laboratory Systems Manager.

All major instruments are backed up by comparable (if not equivalent) instrument systems in the event of unscheduled downtime. An inventory of spare parts is also available to minimize equipment/instrument downtime.

6.2 Calibration Procedures and Frequency

The calibration procedures and frequencies specified in the applicable method and Department of Defense (DoD) Quality Systems Manual (QSM) outlined in **Appendix D**, and/or included in the laboratory SOPs in **Appendix C**, will be followed (DoD, 2010). Records of calibrations will be filed and maintained by the laboratory. These records will be subject to QA audit. For all instruments, the laboratory will maintain trained repair staff with in-house spare parts or will maintain service contracts with vendors.

All standards used in the calibration of equipment are traceable, directly or indirectly, to National Institute of Standards and Technology. All standards received shall be logged into standard receipt logs maintained by the individual analytical groups. Each group will maintain a standards log that tracks the preparation of standards used for calibration and QC purposes.

6.3 Laboratory QC Procedures

6.3.1 Analytical Sequence QC

The sequence for analysis of QC checks and samples is required to insure the instrument calibration is maintained.

Typical analytical sequence for organic analyses:

Instrument tune – Gas Chromatography/Mass Spectrometry (GC/MS)
Initial calibration or Initial Continuing Calibration Verification
Method blank
LCS
Samples, MS/Matrix Spike Duplicate (MSD)
Continuing Calibration Standard every 12 hours

Typical analytical sequence for inorganic analyses:

Instrument tune (Inductively Coupled Plasma/Mass Spectrometry)
Initial calibration
Initial Calibration Verification
Initial Calibration Blank
Detection Limit Standard
Interference Check Sample A
Interference Check Sample AB
Samples, MS/MSD
Continuing Calibration Verification every 10 samples and at end of sequence

6.3.2 Batch/Matrix-Specific/Performance-Based QC

Internal laboratory QC checks will be used to monitor data integrity. These checks will include method blanks, LCSs, internal standards, surrogate samples and calibration standards. Project QC limits are identified in **Table 4-2**. Laboratory control charts will be used to determine long-term instrument trends.

6.3.3 Method Blanks

Sources of contamination in the analytical process, whether specific analyses or interferences, must be identified, isolated, and corrected. The method blank is useful in identifying possible sources of contamination within the analytical process. For this reason, it is necessary that the method blank be initiated at the beginning of the analytical process and encompasses all aspects of the analytical work. As such, the method blank would assist in accounting for any potential contamination attributable to glassware, reagents, instrumentation, or other sources that could affect sample analysis. One method blank will be analyzed with each analytical sequence following the instrument tune and initial calibration verification for VOCs. One method blank will be analyzed with each preparation batch of up to 20 samples for all other analyses.

6.3.4 MS/MSDs

MS/MSDs will be used to measure the accuracy of analyte recovery from the sample matrices and will be site specific. MS/MSD pairs will be analyzed at a 10 percent frequency (every 10 samples).

When MS recoveries are outside QC limits, associated control sample and surrogate spike recoveries will be evaluated, as applicable, to attempt to verify the reason for the deviation and determine the effect on the reported sample results. **Table 4-1** presents an estimated number of MS and MSD analyses for each applicable parameter. **Table 4-2** presents the laboratory control limits for MS/MSD recoveries and RPD.

6.3.5 LCS

LCS is a standard of known concentration and is independent in origin from the calibration standards. The intent of LCS analysis is to provide insight into the analytical proficiency within an analytical series. This includes preparation of calibration standards, validity of calibration, sample preparation, instrument set-up, and the premises inherent in quantitation. Reference standards will be analyzed at the frequencies specified within the analytical methods. **Table 4-2** presents the laboratory control limits for LCS recoveries.

6.3.6 Surrogate Spikes

Surrogates are compounds that are unlikely to occur under natural conditions, but that have properties similar to the analytes of interest. This type of control is primarily used for organic samples analyzed by GC/MS methods and is added to the samples prior to

purging or extraction. The surrogate spike is utilized to provide broader insight into the proficiency and efficiency of an analytical method on a sample-specific basis. This control reflects analytical conditions that may not be attributable to sample matrix.

If surrogate spike recoveries exceed specified QC limits, the analytical results must be evaluated thoroughly in conjunction with other control measures. In the absence of other control measures, the integrity of the data may not be verifiable, and re-analysis of the samples with additional control may be necessary.

Surrogate spike compounds will be selected utilizing the guidance provided in the analytical methods. **Table 4-2** presents the laboratory control limits for surrogate spike recoveries.

6.3.7 Laboratory Duplicates

Laboratory duplicates will be analyzed to assess laboratory precision. Laboratory duplicates are defined as a separate aliquot of an individual sample that is analyzed as a separate sample. **Table 4-1** presents an estimated number of laboratory duplicates for each applicable parameter. **Table 4-2** presents the laboratory control limits for laboratory duplicate RPD.

6.3.8 Calibration Standards

Calibration check standards analyzed within a particular analytical series provide insight regarding instrument stability. A calibration check standard will be analyzed at the beginning and end of an analytical series, or periodically throughout a series containing a large number of samples.

In general, calibration check standards will be analyzed after every 12 hours for organics analyses and every 10 samples for inorganic analyses, as specified in the applicable analytical method. If results of the calibration check standard exceed specified tolerances, samples analyzed since the last acceptable calibration check standard will be re-analyzed.

Laboratory instrument calibration standards will be selected utilizing the guidance provided in the analytical methods.

6.3.9 Internal Standards

Internal standard areas and retention times will be monitored for organic analyses performed by GC/MS methods. Method-specified internal standard compounds will be spiked into all field samples, calibration standards, and QC samples after preparation and prior to analysis. If internal standard areas in one or more samples exceed the specified tolerances, the cause will be investigated, the instrument will be recalibrated if necessary, and all affected samples may be re-analyzed.

The acceptability of internal standard performance will be determined using the guidance provided within the analytical methods.

6.4 Performance and System Audits

Internal laboratory audits are conducted by the Laboratory QA Manager. As part of the audit, the overall performance of the laboratory staff is evaluated and compared to the performance criteria outlined in the laboratory QA manual and SOPs. The results of the audits are summarized and issued to each department supervisor, the Laboratory Manager, and the Laboratory Director. A systems audit of each laboratory may be performed by the QA Manager to determine whether the procedures implemented by each laboratory are in compliance with the QA manual and SOPs.

As a participant in State and Federal certification programs, the laboratory is audited by representatives of the regulatory agency issuing certification, in addition to the laboratory's internal audits. Audits are usually conducted on an annual basis and focus on laboratory conformance to the specific program protocols for which the laboratory is seeking certification. The auditor reviews sample handling and tracking documentation, analytical methodologies, analytical supportive documentation, and final reports. The audit findings are formally documented and submitted to the laboratory for corrective action, if necessary.

6.5 Nonconformance/Corrective Actions

Corrective actions are required when analytical data are not within the objectives specified in this QAPP or the RAWP. Corrective actions include procedures to promptly investigate, document, evaluate, and correct data collection and/or analytical procedures. Laboratory corrective action procedures for the actions are described below.

6.5.1 Laboratory Procedures

In the laboratory, when a condition is noted to have an adverse effect on data quality, corrective action will be taken so as not to repeat this condition. Condition identification, cause, and corrective action taken will be documented and reported to the appropriate Project Manager and QA Coordinator.

Corrective action may be initiated, at a minimum, under the following conditions:

- Protocols as defined by this QAPP have not been followed.
- Predetermined data acceptance standards are not obtained.
- Equipment is not in proper working order or calibrated.
- Sample and test results are not completely traceable.
- QC requirements have not been met.
- Issues resulting from performance or systems audits have not been resolved.

Laboratory personnel will continuously monitor ongoing work performance in the normal course of daily responsibilities. Corrective action is initiated at the point where the problem has been identified. At whatever level this occurs (analyst, supervisor, data review, or QC), it is brought to the attention of the Laboratory QA Manager and, ultimately, the Laboratory Director. Final approval of any action deemed necessary is subject to the approval of the Laboratory Director. See Section 5.2 for corrective action for incoming samples.

Any corrective action deemed necessary based on system or performance audits, the analytical results of split samples, or the results of data review will be implemented. The corrective action may include sample re-extraction, re-preparation, re-analysis, cleanup, dilution, matrix modification, or other activities.

7. Data Reduction/Calculation of Data Quality Indicators

7.1 Laboratory Data Review

Data will be subject to multi-level review by the laboratory. Details of the multi-level review to be performed by TestAmerica Chicago are located in Section 20.14.4 of the laboratory's QA Manual. See Section 7.3 of Microseep's QSM for their data review procedures.

If discrepancies or deficiencies are present in the analytical results, corrective action will be taken, as discussed in Section 6.5. Deficiencies discovered as a result of internal data review, as well as the corrective actions to be used to rectify the situation, will be documented on a Corrective Action Form. This form will be submitted to the environmental Project Manager.

7.2 Precision

The precision of data will be measured by calculation of the RPD by the following equation:

$$RPD = \frac{(A-B)}{(A+B)/2} \times 100$$

Where:

A = Analytical result from one of two duplicate measurements
B = Analytical result from the second measurement

Precision objectives for duplicate analyses are identified in **Table 4-2**.

7.3 Bias

Bias (accuracy) will be calculated in terms of percent recovery as follows:

$$\% \text{ Recovery} = \frac{A-X}{B} \times 100$$

Where:

A = Value measured in spiked sample or standard
X = Value measured in original sample
B = True value of amount added to sample or true value of standard

This formula is derived under the assumption of constant accuracy between the original and spiked measurements. Accuracy objectives for MS recoveries are identified in **Table 4-2**.

7.4 Sample Quantitation/Reporting Limits (LOQ)

Due to the significant amount of error associated with results calculated at the MDL, and the fact the MDL may not be attainable within project matrices, the LOQ is established at approximately a factor of two times the MDL for the majority of target analytes.

The LOQ is set at the lowest standard used for the initial calibration curve (or low-level calibration verification standard) or higher for each target analyte. The lowest standard or low level calibration verification standard must be at least two times the MDL or greater. Target analyte values detected and reported below the LOQ must be flagged as an estimated quantity (*i.e.,* J-flag).

7.5 Laboratory Data Reduction

The calculations used for data reduction will be specified in each of the analytical methods referenced previously. Whenever possible, analytical data will be transferred directly from the instrument to a computerized data system. Raw data will be entered into permanently bound laboratory notebooks. The data entered must be sufficient to document all factors used to arrive at the reported value.

Concentration calculations for chromatographic analyses will be based on response factors. Quantitation for GC/MS methods will be performed using internal standards.

Unless otherwise specified, all values will be reported uncorrected for blank contamination.

7.6 Completeness

Completeness of laboratory data set will be calculated by comparing the number of valid sample results generated to the total number of results generated.

$$\text{Completeness} = \frac{\text{Number valid results}}{\text{Total number of results generated}} \times 100$$

As a general guideline, overall project completeness is expected to be at least 90 percent. The assessment of completeness will require professional judgment to determine data usability for intended purposes.

8. Laboratory Operations Documentation

8.1 Sample Management Records

8.1.1 Sample Storage

The laboratory shall provide an adequate, contamination-free, and well ventilated work space for the receipt of samples. All samples and their associated extracts shall be stored under conditions that will ensure their integrity and preservation and are demonstrated to be free from all potential contaminants. Sufficient refrigerator space shall be provided for the proper storage of all samples and their associated extracts. Samples shall not be stored with standards. Samples designated for volatile organics testing shall be segregated from other samples, while samples suspected to contain high levels of volatile organics (*e.g.,* underground storage tank [UST]soil samples) should be further isolated from other volatile organics samples.

Samples and their associated extracts shall be stored for a minimum of 60 days after receipt of the final data report for those samples. After that time, the laboratory is responsible for the disposal of the samples and their associated extracts in compliance with all Federal, State, and local regulations unless arrangements have been made for the return of any unused sample portions to the site. Disposal of samples will be documented.

8.1.2 Sample Security and Tracking

The laboratory shall maintain the integrity of the samples received, their associated extracts, and the data generated. Limited and controlled access to all laboratory areas shall be maintained.

8.1.3 Sample Holding Times

Extraction/digestion holding times shall be defined from the date/time of sample collection in the field to the date/time when the sample is first exposed to the extraction/digestion solvent. Analysis holding times shall be defined from the date/time of sample extraction to the date/time of sample analysis. It is required that laboratories maintain documentation that clearly show the dates (and times when applicable) for all sample handling/manipulation processes. Samples should be analyzed as soon as possible after sample collection. Published holding times are generally considered

maximum times that samples may be held before analysis and still be considered compliant with method guidelines. Sufficient time should be allowed for the re-preparation or re-analysis of samples within holding times should calibration, method, or QC failures occur.

8.1.4 Sample Analysis

Analysis of an acceptable sample will be initiated by worksheets that contain all pertinent information for analysis. The analyst will sign and date the laboratory COC form when removing the samples from storage.

Samples will be organized into sample delivery groups (SDGs) by the laboratory. An SDG may contain up to 20 field samples (field duplicates, trip blanks, and rinse blanks are considered field samples for the purposes of SDG assignment). All field samples assigned to a single SDG must be processed through the laboratory (preparation, analysis, and reporting) as a group.

8.2 Data Reporting Procedures

8.2.1 Data Package Format and Contents

The laboratory is responsible for preparing Level 2 data packages for all samples. Data reports for all parameters will include, at a minimum, the following items:

Narrative: Summary of activities that took place during the course of sample analysis, including the following information:

- Laboratory name and address
- Date of sample receipt
- Cross reference of laboratory identification number to sample identification
- Analytical methods used
- Deviations from specified protocol
- Corrective actions taken

Included with the narrative will be any sample handling documents, including field and internal COC forms, air bills, and shipping tags.

Analytical Results: These will be reported according to analysis type and include the following information, as applicable:

- Sample identification
- Laboratory identification
- Date of collection
- Date of receipt
- Date of extraction/digestion
- Date of analysis
- Dilution factor
- Detection limits
- Concentration units

Sample results on the report forms will be corrected for dilutions. Unless otherwise specified, all results will be reported uncorrected for blank contamination.

The Level 2 data packages (defined in Section 4.1) from the laboratory will be due within 10 working days from date of receipt.

8.2.2 Electronic Deliverables

The analytical results will be reported by the laboratory in the EDD format outlined in EQuIS SOP in **Appendix A**.

8.3 Data Management Procedures

The purpose of the data management is to provide for the accuracy and ready accessibility of all of the necessary data to meet the analytical and reporting objectives of the project.

The data management program established for the project includes field documentation and sample QA/QC procedures, methods for tracking and managing the data, and a system for filing all site-related information. More specifically, data management procedures will be employed to efficiently process the information collected such that the data are readily accessible and accurate. These procedures are described in detail in the DMP.

8.3.1 Laboratory Turnaround Time

The Form 1s (results sheets) in a PDF or electronic spreadsheet format will be due within 10 working days from date of receipt.

8.3.2 Data Archival/Retention Requirements

The laboratory will establish a file for pertinent data. The file will include correspondence, faxed information, phone logs, COC forms, and laboratory receipt documentation. The laboratory will retain project files, all electronic files and deliverables for no less than five years; hard copy data packages (or electronic copies) will also be retained for no less than five years.

9. Data Assessment Procedures

9.1 Data QC Review

The following laboratory QC data will be reviewed: holding times, methods utilized, the results for method blanks, LCS/LCS duplicates, MS/MSDs, matrix duplicates, and surrogates. The findings and potential affect on the data will be submitted in the data validation report described below in Section 9.2.

9.2 Data Verification/Validation

All data generated will be subjected to the data validation and verification procedures. Data generated for screening or disposal purposes will not be reviewed.

Analytical results will be provided by the laboratory in both digital and a hard copy or pdf format. The data packages will be examined by the QA Coordinator or designee to confirm that the correct analyses were performed for each sample submitted and that all of the analyses requested on the COC form were performed. If discrepancies are noted, the QA Coordinator or designee will promptly follow up with the laboratory to resolve any issues.

Each data package will be validated in accordance with the procedures presented herein. Any data that do not meet the specified standards will be flagged pending resolution of the issue. The flag will not be removed from the data until the issue associated with the sample results is resolved. Although flags may remain for certain data, the use of those data may not necessarily be restricted.

Data validation entails a review of the QC data and the raw data to verify that the laboratory was operating within required limits; the analytical results were correctly transcribed from the instrument read-outs; and which, if any, environmental samples were related to out-of-control QC samples. The objective of data validation is to identify any questionable or invalid laboratory measurements.

All data generated will be validated using the most recent versions of the USEPA's National Functional Guidelines (USEPA, 1999; 2004) available at the time of project initiation, where appropriate. These procedures and criteria may be modified, as necessary, to address project-specific and method-specific criteria, control limits, and procedures. Data validation will consist of data screening, checking, reviewing, and

editing to document analytical data quality and to determine whether the quality is sufficient to meet the DQOs.

The Data Validator will verify that reduction of laboratory measurements and laboratory reporting of analytical parameters is in accordance with the procedures specified for each analytical method and/or as specified in this QAPP. Any deviations from the analytical method or any special reporting requirements apart from those specified in this QAPP will be detailed on COC forms.

Upon receipt of laboratory data, the following procedures will be executed by the Data Validator:

- Evaluate completeness of data package.
- Verify that field COC forms were completed and that samples were handled properly.
- Verify that holding times were met for each parameter. Holding time exceedances, should they occur, will be documented. Data for all samples exceeding holding time requirements will be flagged as either estimated or rejected. The decision as to which qualifier is more appropriate will be made on a case-by-case basis.
- Verify that parameters were analyzed according to the methods specified.
- Review QA/QC data (*i.e.,* confirm that duplicates, blanks, and LCSs were analyzed on the required number of samples, as specified in the method and verify that duplicate RPD are acceptable).
- Investigate anomalies identified during review. When anomalies are identified, they will be discussed with the Project Manager and/or Laboratory Manager, as appropriate.

Deficiencies discovered as a result of the data review, as well as the corrective actions implemented in response, will be documented and submitted in the form of a written report addressing the following topics, as applicable to each method:

- Assessment of the data package.

- Description of any protocol deviations.
- Failures to reconcile reported and/or raw data.
- Assessment of any compromised data.
- Overall appraisal of the analytical data.
- Table of site name, sample quantities, matrix, and fractions analyzed.

It should be noted that qualified results do not necessarily invalidate data. The goal to produce the best possible data does not necessarily mean that data must be produced without QC qualifiers. Qualified data can provide useful information.

During the review process, laboratory qualified and unqualified data are verified against the supporting documentation. Based on this evaluation, qualifier codes may be added, deleted, or modified by the data reviewer. Results will be qualified with the following codes in accordance with National Functional Guidelines:

Concentration (C) qualifiers

U The analyte/compound was analyzed for but not detected. The associated value is the compound quantitation limit.

J The compound was positively identified; however, the associated numerical value is an estimated concentration only.

Q One or more quality control criteria failed *(e.g.,* LCS recovery, surrogate spike recovery or continuing calibration verification).

Quantitation (Q) qualifiers

Inorganics:

B The compound has been found in the sample as well as its associated blank, its presence in the sample may be suspect.

E The reported value is estimated due to the presence of interference.

N Spiked sample recovery not within control limits.

* Duplicate analysis not within control limits.

Organics:

B The compound has been found in the sample as well as its associated blank, its presence in the sample may be suspect.

N The analysis indicates the presence of a compound for which there is presumptive evidence to make a tentative identification.

JN The analysis indicates the presence of a compound for which there is presumptive evidence to make a tentative identification. The associated numerical value is an estimated concentration only.

E The compound was quantitated above the calibration range.

D Concentration is based on a diluted sample analysis.

C Identification confirmed by GC/MS.

Validation qualifiers

UJ The compound was not detected above the reported sample quantitation limit. However, the reported limit is approximate and may or may not represent the actual limit of quantitation.

J The compound was positively identified; however, the associated numerical value is an estimated concentration only.

UB Compound considered non-detect at the listed value due to associated blank contamination.

R The sample results are rejected.

Two facts will be noted to all data users. First, the "R" flag means that the associated value is unusable. In other words, due to significant QC problems, the analysis is invalid and provides no information as to whether the compound is present or not. "R"

values should not appear on data tables because they cannot be relied upon, even as a last resort. The second fact is that no compound concentration, even if it has passed all QC tests, is guaranteed to be accurate. Strict QC serves to increase confidence in data but any value potentially contains error.

Resolution of any issues regarding laboratory performance or deliverables will be handled between the laboratory and the Data Validator. Suggestions for re-analysis may be made by the QA Coordinator at this point.

Data validation reports will be kept in electronic format (PDF) at the PIKA-PIRNIE JV Team's office.

9.3 DQO Reconciliation

The data results will be examined to determine the performance that was achieved for each data usability criterion. The performance will then be compared with the project objectives and DQOs. Deviations from objectives will be noted. Additional action may be warranted when performance does not meet performance objectives for critical data. Options for corrective action relating to incomplete information, questionable results, or inconsistent data may include any or all of the following:

- Retrieval of missing information.
- Request for additional explanation or clarification.
- Reanalysis of sample from extract (when appropriate).
- Recalculation or reinterpretation of results by the laboratory.

These actions may improve the data quality, reduce uncertainty, and eliminate the need to qualify or reject data.

If these actions do not improve the data quality to an acceptable level, the following additional actions may be taken:

- Extrapolation of missing data from existing data points.
- Use of historical data.

- Evaluation of the critical/non-critical nature of the sample.

If the data gap cannot be resolved by these actions, an evaluation of the data bias and potential for false negatives and positives can be performed. If the resultant uncertainty level is unacceptable, additional sample collection and analysis may be required.

9.4 Project Completeness Assessment

Project completeness will be a determination of the completeness of the data packages, and of the percent of chemical measurements that met project DQOs. All data packages will be examined to insure all documentation *(i.e.,* COC), analytical results for all samples, and the required QC checks for a Level 2 data package, as described in Section 4.1, are included.

10. References

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USACE, 2001. *Requirements for the Preparation of Sampling and Analysis Plans*. EM 200-1-3. February.

USEPA, 1980. *Interim Guidance and Specifications for Preparing Quality Assurance Project Plans*. QAMS-005/80. Office of Research and Development. December.

USEPA, 1991. NEIC Policies and Procedures Manual. EPA-330/9-78-001R. National Enforcement Investigations Center. May 1978, revised August 1991.

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USEPA, 1999. *Contract Laboratory Program National Functional Guidelines for Organic Data Review*. EPA-540/R-99-008. October.

USEPA, 2001. *EPA Requirements for Quality Assurance Project Plans for Environmental Operations*. EPA-QA/R-5. Office of Environmental Information. March.

USEPA, 2002. *Guidance for Quality Assurance Project Plans*. EPA-QA/G-5. Office of Environmental Information. December.

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Appendix A

EQuIS Laboratory SOP

INTRODUCTION

PIKA-PIRNIE JV Team manages and verifies/validates analytical data generated by commercial analytical laboratories in the EQuIS database (product of Earthsoft, Inc.). All laboratories contracted by PIKA-PIRNIE JV Team or their clients, on a site-by-site basis, may be required to submit electronic data deliverables (EDDs) in addition to the hard copy report. This Standard Operating Procedure (SOP) describes the structure, format, and submission requirements for electronic data deliverables (EDDs) in the EQuIS EFWEDD (Sample, Test, Result, Batch) format.

This document is a general guidance for preparation of the required electronic data and associated quality control information. The structure of the EDD as defined in this document will remain constant unless Earthsoft modifies the database structure. Reference values and requirements for population of additional fields with specific information will not change from project to project.

Modification to reference value lists may NOT be made by the laboratory without authorization from PIKA-PIRNIE JV Team.

Section I provides PIKA-PIRNIE JV Team contact information and the procedure to submit electronic deliverables directly via e-mail. However, all EDDs will be required to be submitted in a final CD compilation for each specific sampling event or as directed by the PIKA-PIRNIE JV Team Project Manager (PM).

Section II outlines the table structures and general requirements of the EDDs. The EDD structure is based on EarthSoft's EFWEDD EDD format. EarthSoft's EDD format has not been changed; however, some 'optional' fields identified in the EarthSoft EDD have been modified to be 'required' in this EDD format. Additional information regarding the EarthSoft products can be found at http://www.earthsoft.com/.

Section III presents some additional explanation and requirements for populating the table structure and population set forth in Section II.

Section IV summarizes the use of the EDP. Each laboratory **MUST** use EDP to check each EDD file set prior to submission to PIKA-PIRNIE JV Team. The EDP Error Report must be submitted with the EDD. ***All errors identified by the EDP routine must be corrected prior to forwarding the files for entry into the EQuIS database. Or approval for submittal with errors must be authorized by PIKA-PIRNIE JV Team.***

I. CONTACT INFORMATION

Laboratories should contact the PIKA-PIRNIE JV Team National Program Lab Managers with questions regarding this document. The contact info is as follows:

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ELECTRONIC LABORATORY DATA CHECKER EDP

Prior to submitting an EDD to PIKA-PIRNIE JV Team, the EarthSoft EDP must be run to check and verify the EDD structure, format and reference value compliance. The EDP report must be submitted for each file with each EDD set. The Data Checker error report, which demonstrates that the EDD files were successfully checked, must be electronically submitted with the four EDD files to PIKA-PIRNIE JV Team.

REFERENCE VALUES

A specific set of values is required to be utilized in populating certain key fields of the EDD. The Reference Value Lists for the EDP will be provided for each PIKA-PIRNIE JV Team subcontracted laboratory. The Reference Value Lists must be utilized as provided. Alterations or additions to the Reference Values are **NOT** allowed **without prior** written authorization by the PIKA-PIRNIE JV Team Data Manager. Electronic mail may be considered written authorization.

ELECTRONIC DATA DELIVERABLE (EDD) SUBMISSION

Prior to submission to PIKA-PIRNIE JV Team, each data file must also be reviewed by the laboratory to ensure that the sample IDs, dates, times and other inter-related information is consistent between all four (4) files and the EDD is complete. All parameters that are subcontracted to other laboratories must be included in the EDD for a specific SDG or Laboratory Project Number. It is not acceptable to submit separate EDDs for subcontract parameters. Manual review of the files may be necessary to complete this review.

It is **IMPERATIVE** that the EDD results match the hard copy results. If the results do not match the lab will correct the error ASAP at no additional charge. This includes issues involving various rounding routines for different electronic data management programs within the laboratory (i.e. LIMS vs. EPA CLP). Significant figures must also match hard copy and be consistent from one sampling event to the next. Reporting limits must be consistent between events as well and must be in compliance with the Laboratory Task Order or Project Statement of Work. There may be instances where diluted surrogates and unrecovered spike compounds will require population of the EDD with numeric values in lieu of data flags in the hard copy report. The PIKA-PIRNIE JV Team Data Manager will provide project specific guidance for these conditions. Adherence to the SOP requirements for population of spike/surrogate recovery and RPD fields is required to allow electronic validation of the data.

The EDP Reports for each file must be submitted with the 4 files of the actual EDD.

Laboratories must submit EDDs via e-mail for verification of compatibility and completeness to the assigned PIKA-PIRNIE JV Team Data Manager for the project.

The subject line of this e-mail must include the following text:

[Facility-Code] [Laboratory Project/Log/SDG Number] - EDD Submission

The e-mail should also include the laboratory contact name and phone number.

EDDs must be submitted via e-mail prior to or at the same time the final hard copy document is delivered. PIKA-PIRNIE JV Team may review the EDDs prior to requesting final submittal on CD. EDDs will be returned to the laboratory for modifications until the files can be successfully imported into the EQuIS Project Database and Electronic Data Validation can be performed without field population errors. Any revisions to the EDD will be required within 24 hours of notification to the laboratory regarding observed problems with the EDD. When the EDD is acceptable to the PIKA-PIRNIE JV Team Data Manager and Project Manager, a CD containing all final versions of the EDD should be submitted to PIKA-PIRNIE JV Team for archiving.

Invoices for analytical work will not be approved for payment until the final EDD revisions are acceptable.

II. ELECTRONIC DELIVERABLE DATA FORMAT

This section identifies the structure and format requirements for EQuIS EFWEDD EDDs submitted by all laboratories to PIKA-PIRNIE JV Team. Specific field definitions are presented for each of the four files. Laboratories should review the unique requirements for these fields. The format population and adherence to the criteria are mandatory. Data are electronically validated and errors are quickly identifiable if the EDD is incorrect.

GENERAL FORMAT REQUIREMENTS

All laboratory data must be saved as an ASCII file format using the following standard format. Each subcontracting laboratory's data must be incorporated into the primary laboratory's EDD.

Each data field must be either separated by tabs or enclosed in double quotes (") and separated by commas. Data fields that do not contain information may be represented by two commas. Maximum length of text fields is indicated in the parentheses. If the input information is less than the maximum field length, **DO NOT ADD** spaces to account for the difference.

Each record must be terminated with a carriage return/line feed (i.e., standard DOS text file). The file can be produced using any software with the capability to create ASCII files.

THE LABORATORY SHALL LEAVE THE HEADERS IN EACH ASCII FILE TO ASSIST IN REVIEW AND RESOLUTION OF ERRORS.

Four files are required for each SDG or Laboratory Project Number: one each for samples, tests, results, and batches. Each file must be saved as a Tab Delimited or Comma Separated file.

Enterprise EDD File Naming Conventions
EDD packages must be named using a specific naming convention. An EDD Package consists of a .zip file containing the text (.txt) EDDs and a User Certificate. The zip file and text file names must contain the specific elements listed below under file naming conventions, separated by a period. A User Certificate file will be supplied to the lab by PIKA-PIRNIE JV Team for inclusion in the zip file. Please include in the subject line of emailed EDD submissions the facility code and Sample Delivery Group (SDG) number.

File Naming Conventions:
ZIP File Name = Unique ID.Facility Code.Format Name.zip
Text File EDDs Name = Unique ID.EDD Section Name.txt

Unique ID = SDG number.
Facility Code = The facility code (i.e., Site Name from ENFOS)
Format Name = The EQuIS EDD format name (e.g., ESBasic, EFWEDD, etc.).
EDD Section Name = The name of the section within the EDD (e.g. EFW2FSample, EFW2LabTST, etc.).

For example, ZIP File Name = "2009001.BP-99999.EFWEDD.zip" will contain the following files: "2009001.EFW2FSample.txt", "2009001.EFW2LabTST.txt", “2009001.EFW2LabRES.txt", '2009001.EFW2LabBCH.txt' and "pfoos.usr".

Package re-submittal
In order to re-submit corrected EDDs, the .zip file and text (.txt) EDDs must each be renamed. If the example EDD package above were to be re-submitted it would have ZIP File Name = "2009001B.BP-99999.EFWEDD.zip" containing "2009001B.EFW2FSample.txt", "2009001B.EFW2LabTST.txt", “2009001B.EFW2LabRES.txt", '2009001B.EFW2LabBCH.txt' and "pfoos.usr". Note that a “B” has been appended to the SDG name in both the zip file name and each of the text file names. A subsequent re-submittal of the same SDG would require that a C be appended and so on.

Referential integrity is enforced between tables (e.g. sys_sample_code present in the result, batch, and test tables must also be present in the sample table). For example, a data record with a specific sys_sample_code found in the result table, but not in the sample table, will cause and error in the Data Import Module and the file will not be allowed to be entered into the database. Dates and times associated with each test must match in the “Test” and “Result” files or the database will not allow entry of the entire file.

Reference values must be adhered to for a variety of fields as identified in the Reference Value list and described in the following table format requirements.

FORMAT DETAILS

The following four sections provide a detailed summary and the specific layout for each field required in each of the four (4) tables of the EDD. The PIKA-PIRNIE JV Team EDD has been derived from the EarthSoft EFWEDD EDD.

Date is reported as MM/DD/YY (month/day/year) and time as HH:MM (hour:minute). Time must be reported in 24-hour (military) format (3:30 p.m. = 15:30 and 8:30 AM = 08:30 not 8:30). **NOTE:** Make certain that the LIMS systems format the date and time the same way for all files.

The columns in the following 4 tables relate to:

“Number” Column in Tables = Column of EDD table

“Attribute Name” = Column Name
PK after attribute indicates this is a primary key within Access for the table.

"Column Data" Type = Text or Numeric values required. Parenthetical number indicates total allowable number of characters in the field.

"Required" Column:

The column titled 'Required' will contain the text 'Yes' if the field is required to be populated by the laboratory. In addition, a "condition" is added to indicate additional information applying to population of the associated field. The first number of the condition relates to the table in which the condition applies, i.e. 1 is the Sample File, 2 is the Test File, 3 is the Result File, and 4 is the Batch File. Conditions apply as follows:

Condition	Table	Description
0	ALL	Field always required
1-1	SAMPLE	Field required for field samples only not required for laboratory samples
1-2	SAMPLE	Field required (parent_sample_code) for **laboratory** QC samples that have 'parents'
1-3	SAMPLE	Field not required for field samples
2-1	TEST	Field required if applicable for specific test
3-1	RESULT	Field required (result_value) for detected analytes only (TRG or TICs). Must be NULL if non-detect or surrogates, internal standards or spiked compounds
3-2	RESULT	Field required if available or appropriate for result
3-3	RESULT	Field required for matrix spikes or matrix spike duplicates (NOT required for surrogate compounds or LCS samples where the original concentration is assumed to be zero).
3-4	RESULT	Field required for surrogate compounds, LCS, Blank Spikes, Matrix Spikes, and Internal Standards.
3-5	RESULT	Field required for LCS duplicates, Blank Spike Duplicates, Matrix Spike Duplicates, Lab Replicates
3-6	RESULT	Field required for LCSD, BSD, MSD, and Lab duplicate samples
3-7	RESULT	Field required for surrogates and spike compounds
4-1	BATCH	Field required if available or appropriate for result

"REQUIRED":

"YES" = Required data if applicable

"NO" = Optional information unless otherwise directed by PIKA-PIRNIE JV Team Data Manager or preferred for insertion by lab except where lab is specifically directed to leave the field Null.

Parent Sample Definition

Parent Samples are base samples for duplicates or spikes. i.e. original field samples used for matrix spikes or field sample used for Lab Duplicate/Replicate. A Matrix Spike is not the Parent Sample of the Matrix Spike Duplicate.

POPULATING SPIKE FIELDS

SURROGATES: surrogate recoveries are to be populated in qc_spike_added, qc_spike_measure, and qc_spike_recovery fields. Surrogates are analyte type = SUR. Control limits for surrogate recoveries must also be populated.

INTERNAL STANDARDS: internal standard values are to be populated in qc_spike_added, qc_spike_measure, and qc_spike_recovery fields. Internal Standards are analyte type = IS.

LCS, BS, and MS COMPOUNDS: recoveries are to be populated in qc_spike_added, qc_spike_measured, and qc_spike_recovery fields. Compounds spiked to evaluate method accuracy are analyte type = SC. Control limits for spike recoveries must also be populated.

LCSD, BD, AND MSD COMPOUNDS: recoveries are to be populated in qc_dup_spike_added, qc_dup_spike_measured, and qc_dup_spike_recovery fields. The Compounds spiked to evaluate method accuracy are analyte type = SC. Control limits for spike recoveries must also be populated. Additionally, the qc_rpd and qc_rpd_cl fields must be populated for these samples.

LAB REPLICATE SAMPLE DATA: values for lab duplicates/replicates are to be populated in qc_dup_spike_measured field. The qc_rpd and qc_rpd_cl fields must be populated for these samples.

III. ADDITIONAL REQUIREMENTS

SAMPLE TABLE				
Num	**Attribute Name**	**Column Data Type**	**Required**	**Attribute Definition**
1	sys_sample_code	Text(40)	Yes (0)	Unique sample identifier (COC Sample ID). Each sample must have a unique value, including spikes and duplicates. Unique sample identifiers **throughout** the database are an **ABSOLUTE** restriction enforced by EQuIS Chemistry. This unique identifier also carries through to each subsequent sampling event where the samples IDs must be unique for EVERY event of the project (continuing years). Laboratory QC samples must also have unique identifiers between sampling event and from 1 year to the next and between laboratories in the event subcontractors are used. For Matrix Spike, Matrix Spike Duplicate, and Laboratory Duplicates of Field Samples, add the suffix **MS, MSD, and LR**, respectively to create unique identifiers for these types of Lab QC samples.
2	sample_name	Text(30)	No	Additional sample identification information as necessary. Is not required to be unique (i.e., duplicates are OK).

SAMPLE TABLE				
Num	**Attribute Name**	**Column Data Type**	**Required**	**Attribute Definition**
3	sample_matrix_code	Text(10)	Yes (0)	Code, which distinguishes between different types of sample matrix. **Examples**: Soil samples =“SO” , groundwater samples = “WG”. Field Blanks, Trip Blanks, and Rinsate Blanks = “WQ”. Water Method Blanks and liquid matrix spikes = “WQ” Soil Method Blanks and soil/sludge/sediment matrix spikes = “SQ’ This field refers to the sample matrix not the matrix after preparation or extraction. See rt_matrix for the list of valid values.
4	sample_type_code	Text(10)	Yes (0)	Code that distinguishes between different types of samples. **For example**, normal field samples = “N” and laboratory method blank =“LB”. Field QC sample types are Field Duplicates = “FD”, Field Blanks = “FB”, Trip Blanks = “TB”. Lab QC sample types are LCS or Blank Spikes = “BS”, LCSD or BS Duplicates = “BD” and Matrix Spikes = “MS” and Matrix Spike Duplicates = “SD”. See rt_sample_type in Reference Values list of valid values.
5	sample_source	Text(10)	Yes (0)	Must be either "Field" for field samples **or** "Lab" for laboratory QC samples. No other values are allowed. Matrix spikes and lab duplicate/replicate are "Lab" samples, even though the parent is a "Field" and the base sample came from the field. The spiking or splitting for duplication is done in the lab. Field duplicates as submitted to the lab by field sampling teams are “Field”
6	parent_sample_code	Text(40)	Yes (1-2)	The value in the "sys_sample_code" that identifies the sample that was the source of this sample. *For example*, the Matrix Spike and the Matrix Spike Duplicate or Lab Replicates parent_sample_code is the sys_sample_code for the originating field sample that is spiked to generate the MS/MSD or split by the lab for use as the laboratory duplicate. This field is only required in the EDD for laboratory "clone" samples (e.g., matrix spikes and duplicates). Field duplicates are submitted blind to the laboratory, so this field cannot be completed by the laboratory. This field must be blank for samples that have no parent (e.g., normal field samples, method blanks, etc.).
7	sample_delivery_group	Text(10)	Yes (0)	Sample delivery group or laboratory Project/Log Number. All deliverables must reference the SDG or Lab Log-in Number. **This field MUST BE POPULATED**
8	sample_date	Date	Yes (1-1)	Date of sample collection in **MM/DD/YY** format including trip blanks. Must be blank for laboratory samples.
9	sample_time	Time	Yes (1-1)	Time of sample collection in 24-hour (military) **HH:MM** format. 8:45 AM = 08:45 and 3:30 PM = 15:30. Must be blank for laboratory samples.

SAMPLE TABLE				
Num	**Attribute Name**	**Column Data Type**	**Required**	**Attribute Definition**
10	sys_loc_code	Text(20)	No	Sample collection location. To be populated by PIKA-PIRNIE JV Team unless otherwise directed at project initiation.
11	start_depth	Double	No	Beginning depth (top) of soil sample. To be populated by PIKA-PIRNIE JV Team unless otherwise directed at project initiation.
12	end_depth	Double	No	Ending depth (bottom) of soil sample. To be populated by PIKA-PIRNIE JV Team unless otherwise directed at project initiation.
13	depth_unit	Text(15)	No	Unit of measurement for the sample begin and end depths. IRPIMS-style unit of measurement codes (see table X03) are recognized by Chem; other codes may be allowed by the Chem project manager. To be populated by PIKA-PIRNIE JV Team unless otherwise directed at project initiation.
14	chain_of_custody	Text(15)	Yes (1-1)	Chain of custody identifier or number. A single sample may be assigned to only one chain of custody. The COC identifier will be provided by the field sampling team based on conventions established for a specific project.
15	sent_to_lab_date	Date	No	Date sample was sent to lab (in MM/DD/YY format for EDD).
16	sample_receipt_date	Date	Yes (1-1)	Date that sample was received at laboratory in **MM/DD/YY** format. Must be blank for laboratory samples.
17	sampler	Text(30)	No	Name or initials of sampler.
18	sampling_company_ code	Text(10)	Yes (1-1)	Name or initials of sampling company (no controlled vocabulary). "PIKA-PIRNIE JV Team" should be entered into this field unless otherwise directed at project initiation.
19	sampling_reason	Text(30)	No	Optional reason for sampling. No controlled vocabulary is enforced.
20	sampling_technique	Text(40)	No (1-1)	To be populated by PIKA-PIRNIE JV Team unless otherwise directed at project initiation. Sampling technique. **For example**, low flow, bailing, MIP, etc… Must be blank for laboratory samples.
21	task_code	Text(10)	No	Code used to identify the task under which the field sample was retrieved.
22	collection_quarter	Text(5)	No	Quarter of the year sample was collected (e.g., "1Q96")
23	composite_yn	Text(1)	No	Boolean field used to indicate whether a sample is a composite sample.
24	composite_desc	Text(255)	No	Description of composite sample (if composite_yn is YES).

SAMPLE TABLE				
Num	**Attribute Name**	**Column Data Type**	**Required**	**Attribute Definition**
25	sample_class	Text(10)	No	Navy sample class code.
26	custom_field_1	Text(255)	No	Custom sample field
27	custom_field_2	Text(255)	No	Custom sample field
28	custom_field_3	Text(255)	No	Custom sample field
29	comment	Text(255)	Yes (0)	Field required to contain the full sample ID code.
30	sample_receipt_time	Text(5)	Yes (1-1)	Time of sample receipt by laboratory in 24-hour (military) **HH:MM** format. 8:45 AM = 08:45 and 3:30 PM = 15:30

TEST TABLE				
Num	**Attribute Name**	**Column Data Type**	**Required**	**Attribute Definition**
1	sys_sample_code (PK)	Text (40)	Yes (0)	SAME AS #1 IN SAMPLE TABLE. This value is used in enforcing referential integrity between tables. Must match sys_sample_code in Sample Table.
2	lab_anl_method_name (PK)	Text (35)	Yes (0)	Laboratory analytic method name or description. See rt_analytic_method in reference value tables for list of valid values.
3	analysis_date (PK)	Date/ Time	Yes (0)	Date of sample analysis in MM/DD/YY format. Refers to initiation of the analysis not prep method date.
4	analysis_time (PK)	Text (5)	Yes (0)	Time of sample analysis in 24-hour (military) HH:MM format. Note that this field, combined with the "analysis_date" field is used to distinguish between reextractions, reanalyses, and dilutions. Please ensure that retests have "analysis_date" and/or analysis_time" different from the original test event (and complete test_type field as appropriate).
5	total_or_dissolved (PK)	Text (1)	Yes (0)	"T" for total metal organic carbon concentration, "D" for dissolved or filtered metal or organic carbon concentration ONLY. USE "N" for organic (or other) constituents for which neither "total" nor "dissolved" is applicable including TDS.
6	column_number (PK)	Text (2)	Yes (2-1)	Applicable for GC or HPLC methods. "1C" for first column analyses, "2C" for second column analyses, or "NA" for analyses where not applicable. If any "2C" tests are listed, then there must be corresponding "1C" tests present also. Laboratories must indicate which of the two columns is to be considered "primary" by entering “Y” in the "reportable_result" field of the result table for the result presented in hard copy reports. It is NOT acceptable to identify both “1C” and “2C” reportable_result as “Y:; one must be “N” if” “1C” and “2C” are provided in the EDD.

TEST TABLE				
Num	**Attribute Name**	**Column Data Type**	**Required**	**Attribute Definition**
7	test_type (PK)	Text (10)	Yes (0)	Type of test. Valid values include "initial", "reextract", and "reanalysis", "dilution" are acceptable. See rt_test_type for al valid values.
8	lab_matrix_code	Text (10)	Yes (0)	Code that distinguishes between different types of matrix analyzed. Soil = "SO"; groundwater = "GW" and TCLP = TCLP as a lab matrix. See rt_matrix for valid values
9	analysis_location	Text (2)	Yes (0)	"LB" for fixed-based laboratory analysis, "FI" for field instrument, "FL" for mobile field laboratory analysis, or.
10	basis	Text (10)	Yes (0)	"Wet" for wet-weight basis; or "Dry" for dry-weight basis. For tests for which this distinction is not applicable use Wet
11	container_id	Text (30)	No	Sample container identifier.
12	dilution_factor	Single	Yes (0)	Test or analytical run dilution factor. **Must** be "1'" if no dilution.
13	Prep_method	Text (35)	Yes (2-1)	Laboratory sample preparation method name. See rt_std_prep_method for valid values.
14	prep_date	Date/ Time	Yes (2-1)	Date of sample preparation in MM/DD/YY format.
15	prep_time	Text (5)	Yes (2-1)	Time of sample preparation in 24-hour (military) HH:MM format
16	leachate_method	Text (15)	Yes (2-1)	Method name, e.g., SW1311 or SW1312. See rt_analytic_method for valid values.
17	leachate_date	Date/ Time	Yes (2-1)	Date of leachate preparation in MM/DD/YY format.
18	leachate_time	Text (5)	Yes (2-1)	Time of leachate preparation in 24-hour (military) HH:MM format.
19	lab_name_code	Text (10)	Yes (0)	Unique identifier of the laboratory reporting results. See rt_subcontractor for valid values.
20	qc_level	Text (10)	NO	Not populated by Lab.
21	lab_sample_ id	Text (20)	Yes (0)	Laboratory sample identifier. A field sample may have more than one laboratory lab_sample_id; however it is limited to only ONE lab_sample_id per method).
22	percent_moisture	Text (5)	Yes (2-1)	Percent moisture of the sample portion used in the specific lab_anl_methd_name test; this value may vary from test to test for any sample. The value must be NUMERIC as "NN.MM", e.g., 70.1% could be reported as "70.1" but not as 70.1%". The database assumes that the number is a "%" and units of measure are not necessary. **NOTE: This field MUST be populated for all soil, sludge, and sediment samples whether or not the value is reported in the hard copy. Use "0" for lab soil QC samples.**
23	subsample_amount	Text (14)	Yes 0)	Amount of sample used for the test. THIS FIELD MUST BE POPULATED
24	subsample_amount_u nit	Text (15)	Yes (0)	Unit of measurement for subsample amount. See rt_unit for valid values.

TEST TABLE				
Num	**Attribute Name**	**Column Data Type**	**Required**	**Attribute Definition**
25	analyst_name	Text (30)	Yes (0)	Name or initials of laboratory analyst.
26	instrument_lab	Text (50)	Yes (0)	Instrument identifier.
27	comment	Text (255)	NO	Comments about the test as necessary (Optional).
28	preservative	Text (50)	Yes (2-1)	Indicate preservative or leave blank, if none. **THIS FIELD MUST BE POPULATED IF A PRESERVATIVE WAS IN THE SAMPLE AS RECEIVED FROM THE FIELD OR IF THE SAMPLE WAS PRESERVED BY THE LABORATORY BEFORE PREPARATION AND ANALYSIS.**
29	final_volume	Text (15)	Yes (2-1)	Final amount of extract or digestate.
30	final_volume_unit	Text (15)	Yes (2-1)	Unit of measure for final_volume. See rt_unit for valid values.

RESULT TABLE				
Num	**Attribute Name**	**Column Data Type**	**Required**	**Attribute Definition**
1	sys_sample_code (PK)	Text (40)	Yes (0)	SAME AS #1 IN SAMPLE & TEST TABLES. This value is used in enforcing referential integrity between tables.
2	lab_anl_method_name (PK)	Text (35)	Yes (0)	Laboratory analytic method name. Must be same as lab_anl_method_name in Test File. See rt _analytic_method for valid values.
3	analysis_date (PK)	Date/Time	Yes (0)	Must be the SAME AS #3 IN THE TEST TABLE. This value is used in enforcing referential integrity between tables. Date of sample analysis in MM/DD/YY format.
4	analysis_time (PK)	Text (5)	Yes (0)	Must be the SAME AS #4 IN THE TEST TABLE. This value is used in enforcing referential integrity between tables.
5	total_or_dissolved_ (PK)	Text (1)	Yes (0)	Must be the SAME AS #5 IN THE TEST FILE.
6	column_number (PK)	Text (2)	Yes (3-2)	Must be the SAME AS #6 IN THE TEST FILE
7	test_type (PK)	Text (10)	Yes (0)	Must be the SAME AS #7 IN THE TEST FILE
8	cas_rn (PK)	Text (15)	Yes (0)	Chemical Abstracts Number for the parameter if available. This must be the true CAS # and "not made up". Where CAS #s are not available, i.e. wet chem. Parameters, identifiers will be provided by PIKA-PIRNIE JV Team project requirements. See notes at end of section for TIC management. See rt_analyte for valid values. The lab is not authorized to add internally developed "CAS #s" for general chemistry parameters, surrogates, internal standards, TICs. CAS#s used for TICs must be available through an outside source such as "Chemfinder".
9	chemical_name	Text (60)	Yes (0)	Chemical name associated with CAS # in #8. The cas_rn field is the only chemical identifier information actually imported in EQuIS Chemistry.

RESULT TABLE				
Num	**Attribute Name**	**Column Data Type**	**Required**	**Attribute Definition**
10	result_value	Text (20)	Yes (3-1)	Analytical result reported for **"TRG" or "TIC"** result_type **ONLY**. Appropriate and consistent number of significant digits must be entered. **MUST BE BLANK FOR NON-DETECTS.** "SUR", "IS", and "SC" results do **NOT** populate this field (populate the QC fields).
11	result_error_delta	Text (20)	Yes (3-2) [Radiochem)	Error range applicable to the result value for radiochemistry results.
12	result_type_code	Text (10)	Yes (0)	Must be either "TRG" for a target or regular results, "TIC" for tentatively identified compounds, "SUR" for surrogates, "IS" for internal standards, or "SC" for spiked compounds.[**LCS, LCSD, MS, MSD, BS, BSD]**
13	reportable_result	Text (10)	Yes (0)	Must be either "**Yes**" for results, which are considered to be reportable, **or** "**No**" for other results. Used to distinguish between multiple results where a sample is retested after dilution or to indicate which of the first or second column result should be considered primary. For re-analyses and dilutions all results must be entered into the database if hard copy data is provided **BUT ONLY ONE RESULT FOR EACH COMPOUND/ANALYTE MAY BE FLAGGED AS REPORTABLE**.
14	detect_flag	Text (2)	Yes (0)	Either "Y" for detected analytes or "N" for non-detects**. MUST be "N" for NON-DETECTS**.
15	lab_qualifiers	Text (7)	Yes (3-2)	Qualifier flags assigned by the laboratory. See rt_qualifier for valid qualifiers that may be used.
16	Organic_ yn	Yes/No	Yes (0)	Must be either "Y" for organic constituents or "N" for inorganic constituents.
17	method_detection_ limit	Text (20)	Yes (0)	Laboratory determined MDL per 40 CFR Part 136, adjusted for dilutions and percent moisture (if it applies).
18	reporting_detection_ limit	Text (20)	Yes (0)	Detection limit that reflects sample analysis conditions including analysis volumes and dilution factors. This should be the laboratory PQL or standard reporting limits
19	quantitation_limit	Text (20)	No	NOT Currently used unless specifically defined for the project.
20	Result_unit	Text (15)	Yes (0)	Units of measure relates to **ALL** results including result_value, qc_original_concentration, qc_spike added, qc_spike_measured, qc_dup_orginal_conc, qc_dup_spike_added, qc_dup_spike_measured. See rt_unit for valid values.
21	detection_limit_unit	Text (15)	Yes (0)	Units of measure for detection limit(s). See rt_unit for valid values.
22	tic_retention_time	Text (8)	Yes (3-2)	Retention time in minutes for tentatively identified compounds (TICs). Populated only for TIC result_type
23	result_comment	Text (255)	NO	MUST BE LEFT BLANK BY THE LAB

RESULT TABLE				
Num	**Attribute Name**	**Column Data Type**	**Required**	**Attribute Definition**
24	qc_original_conc	Text (14)	Yes (3-3)	The concentration of the analyte in the original (unspiked) sample. Populated for matrix spike samples. Not populated where original concentration is assumed to be zero, i.e. LCS or BS samples.
25	qc_spike_added	Text (14)	Yes (3-4)	The concentration of the analyte added to the original sample. Populated for **ALL** Surrogates, and LCS, BS, and MS samples
26	qc_spike_measured	Text (14)	Yes (3-4)	The measured concentration of the analyte. Use zero for spiked compounds that were not detected in the sample. **MUST BE NUMBERIC** even if diluted out or not recovered (use “0” if diluted, matrix interference, elevated concentrations of target compounds, etc.) Populated for **ALL** Surrogates, and LCS, BS, and MS samples
27	qc_spike_recovery	Text (14)	Yes (3-4)	The percent recovery for “SUR” and “SC” results. **MUST BE NUMERIC** even if diluted out or not recovered (use “0” if diluted, matrix interference, elevated concentrations of target compounds, etc.) Report as percentage (e.g., report "120%" as "120"); DO NOT include “%” sign in field. Populated for **ALL** Surrogates, and LCS, BS, and MS samples
28	qc_dup_original conc	Text (14)	Yes (3-5)	The concentration of the analyte in the original (unspiked) sample. Populated for matrix spike duplicate samples. Not populated where original concentration is assumed to be zero, i.e. LCSD or BSD samples.
29	qc_dup_spike_added	Text (14)	Yes (3-5)	The concentration of the analyte added to the original sample. Populated for **ALL** LCSD, BSD, and MSD samples.
30	qc_dup_spike_measured	Text (14)	Yes (3-5)	The measured concentration of the analyte in the duplicate. Populated for **ALL** LCSD, BSD, and MSD samples. MUST be NUMERIC. Use zero for spiked compounds that were not recovered due to dilution, matrix interference, elevated concentrations of target compounds, etc..
31	qc_dup_spike_recovery	Text (14)	Yes (3-5)	The duplicate percent recovery. Populated for **ALL** LCSD, BSD, and MSD samples. **MUST be NUMERIC.** Use zero for spiked compounds that were not recovered due to dilution, matrix interference, elevated concentrations of target compounds, etc Report as percentage (e.g., report "120%" as "120").
32	qc_rpd	Text (8)	Yes (3-6)	The relative percent difference between MS and MSD, LCS and LCSD, BS and BSD, & primary field sample result and Lab Replicate. Populated for **ALL** LCSD, BSD, MSD, and LR samples. **MUST be NUMERIC**. Use zero for RPDs that were not calculated due to elevated concentrations of target compounds, dilution, matrix interference, etc Report as percentage (e.g., report "120%" as 120").
33	qc_spike_lcl	Text (8)	Yes (3-7)	Lower control limit for spike recovery. Required for spikes, spike duplicates, surrogate compounds, LCS and any spiked sample. Report as

RESULT TABLE				
Num	**Attribute Name**	**Column Data Type**	**Required**	**Attribute Definition**
				percentage (e.g., report "120%" as "120").
34	qc_spike_ucl	Text (8)	Yes (3-7)	Upper control limit for spike recovery. Required for spikes, spike duplicates, surrogate compounds, LCS and any spiked sample. Report as percentage (e.g., report "120%" as "120").
35	qc_rpd_cl	Text (8)	Yes (3-6)	Relative percent difference control limit. Required for any duplicated sample. Report as percentage (e.g., report "120%" as "120").
36	qc_spike_status	Text (10)	Yes (3-4)	Used to indicate whether the spike recovery was within control limits. Use the "+" character to indicate failure, otherwise leave blank.
37	qc_dup_spike_status	Text (10)	Yes (3-5)	Used to indicate whether the duplicate spike recovery was within control limits. Use the "+" character to indicate failure, otherwise leave blank.
38	qc_rpd_status	Text (10)	Yes (3-6)	Used to indicate whether the relative percent difference was within control limits. Use the "+" character to indicate failure, otherwise leave blank. Required for any duplicated sample.

BATCH TABLE				
Num	**Attribute Name**	**Column Datatype**	**Required**	**Attribute Definition**
1	sys_sample_code (PK)	Text (40)	Yes (0)	SAME AS #1 IN SAMPLE , TEST TABLE. This value is used in enforcing referential integrity between tables.
2	lab_anl_method_name (PK)	Text (35)	Yes (0)	SAME AS #2 IN TEST TABLE. See rt _analytic_method for valid values.
3	analysis_date (PK)	Date	Yes (0)	SAME AS #3 IN TEST TABLE. This value is used in enforcing referential integrity between tables. Date of sample analysis in MM/DD/YY format. May refer to either beginning or end of the analysis as required by EQuIS Chemistry project manager.
4	analysis_time (PK)	Text (5)	Yes (0)	SAME AS #4 IN TEST, AND RESULT TABLES. This value is used in enforcing referential integrity between tables.
5	total_or_dissolved (PK)	Text (1)	Yes (0)	SAME AS #5 IN TEST TABLE. This value is used in enforcing referential integrity between tables.
6	column_number (PK)	Text (2)	Yes (4-1)	SAME AS #6 IN TEST TABLE. This value is used in enforcing referential integrity between tables.
7	test_type (PK)	Text (10)	Yes (0)	SAME AS #7 IN TEST TABLE. This value is used in enforcing referential integrity between tables.
8	test_batch_type (PK)	Text (10)	Yes (0)	Lab batch type. Valid values include "Prep", "Analysis", and "Leach". Additional valid values may optionally be provided by the EQuIS Chemistry project manager. This is a required field for all batches.
9	test_batch_id	Text (20)	Yes (0)	Unique identifier for all and each lab batches. Must be unique within EQuIS Chemistry database. For example, the same identifier cannot be used for a prep batch and an analysis batch and the values must be different from one sampling event to another. THIS IDENTIFIER CANNOT BE USED FROM ONE YEAR TO THE NEXT.

ADDITIONAL INFORMATION FOR PREPARING THE 4-FILE EDD

SAMPLE FILE AND SYS_SAMPLE_CODE

1. The sys_sample_code is the unique sample ID as supplied on the Chain of Custody form with the same spacing as identified on the COC or on a supplemental Sample ID list submitted to the laboratory with the Laboratory Task Order or prior to submission of samples.

2. In order to uniquely identify MS/MSD, laboratory duplicates, TCLP, and SPLP samples, the laboratory shall add a suffix to the original sample ID listed on the chain of custody:

   Matrix Spike Sample = xxxxx MS
   Matrix Spike Duplicate Sample = xxxxx MSD
   Lab Duplicate/Replicate = xxxxx LR
   TCLP Extract Sample = xxxxx TCLP
   SPLP Extract Sample = xxxxx SPLP

   These are the only characters that are allowed to be amended to ANY sample ID as listed on the COC or the sample ID list referred to above.
   The parent_sample_code shall be entered into the parent_sample_code field of the Sample File.

3. If the sample_name field is provided it must contain the full sample ID from the chain of custody.

4. Sample_Type_Code must be appropriately applied as follows:
   "N" = normal field samples
   "FD" = field duplicates samples submitted blind to the laboratory
   "TB" = trip blanks
   "FB" = field blanks
   "EB" = rinsate or equipment blanks
   "BS" = laboratory control samples or blank spikes
   "BD" = laboratory control sample duplicates or blank spike duplicates
   "MS" = matrix spikes
   "SD" = matrix spike duplicates
   "LR" = laboratory duplicates or laboratory replicates

5. The following "**matrix_type**" codes must be used ("**SQ**" = soil QC sample and "**WQ"** = water QC sample):
   Method Blank = "SQ" or "WQ"
   MS/MSDs = "SQ" or "WQ"
   LCS/LCSDs = "SQ" or "WQ"
   BS/BSDs = "SQ" or "WQ"

6. SDG Numbers or laboratory Log Numbers (per PIKA-PIRNIE JV Team PM direction) **MUST** be populated in "**sample_delivery_group**" field of the **Sample File.**

QUALITY CONTROL SAMPLES AND DATA

7. The source of Lab Duplicates, Lab Replicates, Matrix Spikes, and Matrix Spike Duplicates is the Lab not the Field even if the MS/MSD are identified on the COC by the field sampling team. The samples are spiked in the laboratory not in the field.

8. Laboratory QC data, which span more than one SDG may be submitted with each appropriate SDG.

9. Laboratory LCS and LCSD should be reported as two separate samples.

10. **Matrix Spike and Matrix Spike Duplicate recoveries must be reported as "0" if the value is not calculated due to concentrations of the spiked analyte in the sample at concentrations above the 4X factor.**

11. **All laboratory method performance site-specific and batch Quality Control sample results (i.e. Method Blanks, LCS/LCSDs, Blank Spikes, Leachate Blanks as method appropriate) must be included in the EDD.** For most projects, this does **NOT** include **non-site-specific** matrix spikes and laboratory duplicates/replicates.

12. Laboratory batch sample duplicate/replicate and MS/MSD results from **non-project specific** samples (i.e. batch QC samples) shall **NOT** be included in the EDD.

13. Surrogates populate the qc_spike fields not qc_dup_spike fields or the result_value field even if the surrogates are reported for MSD, BSD, or LCSD samples.

14. QC_Spike_Added values for Spike, IS and Surrogate compounds are REQUIRED.

15. QC_Spike_Measured values for Spike, IS and Surrogate compounds are REQUIRED.

16. RPDs for LCSDs, BSDs, MSDs, and Laboratory Duplicates must be populated in the "**qc_rpd"** field. A value of "0" or "100" must be reported, as appropriate, if the RPD is not calculated due to excessive concentrations or interference present in the sample. The "**qc_rpd**" must be a numeric entry.

17. The RPD control limit must be listed in the "**rpd_cl"** field for all parameters where an RPD is reported. This includes lab duplicate/replicate samples.

SAMPLE FILE

18. The following "**matrix_type**" codes must be used for QC samples ("**SQ**" = soil QC sample and "**WQ"** = water QC sample):

    Method Blank = "SQ" of "WQ"
    MS/MSDs = "SQ" or "WQ"
    LCS/LCSDs = "SQ" or "WQ"
    BS/BSDs = "SQ" or "WQ"

19. SDG or Laboratory Project numbers must be populated in "sample_delivery_group" field.

TEST FILE

20. Percent moisture must be reported in the "**percent_moisture**" field in the **Test File** for all solid samples (i.e., soil, sediment, and sludge).

21. Subsample weights and final volumes must be listed for all parameters as appropriate.

RESULTS FILE

22. Result_value is only populated with data for "TRG" and "TIC" detections. All other data is entered in the "qc_" fields. The field must be "NULL" for non-detects and other analyte_types. The Reporting Limit must not be entered in this field.

23. Non-detected data shall have a lab_qualifier of "U" in addition to other qualifiers deemed applicable. The Detect_Flag shall be "N" and the Result_value field shall be blank.

24. The Reporting Limit must be provided for all parameters. The RL MUST be adjusted for dilutions made during analysis.

25. Surrogate recoveries MUST BE REPORTED in the qc_spike_measured and qc_spike_recovery fields, even if the surrogate had been diluted out. List “0” as the measured and recovered amount. Control Limits must also be entered for surrogates. Surrogates are “SUR” analyte_type not “TRG”.

26. Surrogate, LCS, LCSD, BS, BSD, MS, and MSD detected concentrations, and percent recoveries must be populated with a numeric value. A value of “0” **must** be entered if the Spiked Compound is diluted out or not recovered. An “+” is unacceptable as this is a numeric field.

27. “**QC_original_concentration**” must be populated for matrix spikes and matrix spike duplicates

28. Valid entries for the reportable_result field are "Yes" or "No” only.

29. ONLY report compounds of interest for any method blank, sample, and sample duplicate, trip blank.

30. Laboratory Qualifier designation must be consistent. For an estimated concentration with blank contamination “BJ” must be used. Note that “JB”, “B J” or “J B” cannot be used.

31. Explanation of Duplicate Qualifiers:

B	Analyte found in associated blank	Organic Analysis
B	<CRDL but >= Instrument Detection Limit	Inorganic Analysis
N	Presumptive evidence of a compound	Organic Analysis
N	Sample recovery not within control limits	Inorganic Analysis

It is preferred by PIKA-PIRNIE JV Team that the laboratory not qualifiers with multiple explanations. **Any qualifiers utilized in the hard copy report or the electronic report must be defined in the hard copy report. There is no exception to this requirement for explanation of qualifiers applied to electronic data.**

32. Nomenclature for tentatively identified compounds (TIC):

Use the CAS # if it is available and **REAL** **(outside verifiable source)** for TICs and enter the chemical name in the chemical_name field.

For UNKNOWN TICs follow the following protocol:

cas_rn for unkown VOA TIC = VTIC 1 through VTIC 10
cas_rn for unkown SVOA TIC = SVTIC 1 through SVTIC 20

Enter “UNKNOWN”, “UNKNOWN Hydrocarbon”, “UNKNOWN Aliphatic”, or other identifier as appropriate or applicable in “chemical_name” field.

TICs will produce errors in the ELDC/EDDP that cannot be corrected by the laboratory. These are the only acceptable errors in the data checker report unless otherwise authorized by PIKA-PIRNIE JV Team.

33. TCLP or SPLP results must be submitted in units of mg/L or appropriate liquid units. **(Make sure that moisture correction is not automatically enforced).**

BATCH FILE

34. The laboratory must use unique Batch File Names for each analytical department/method and for continuing years. Electronic validation utilizes Batch IDs to link field samples with quality control data. Overlapping Batch IDs are not acceptable.

GENERAL ISSUES

35. Incomplete chain-of-custody (C-O-C) forms must be immediately communicated to the project manager. Some of the C-O-C information is used for completion of the Sample_Matrix_Code and Sample_Delivery_Group. These discrepancies must be rectified upon receipt of samples at the laboratory prior to log in.

36. **Duplicate sample IDs are not acceptable within the EQuIS database. It is imperative that samples including field blanks, trip blanks, equipment blanks, field duplicates have unique sample IDs for projects including ongoing sampling events such as quarterly groundwater monitoring.**

SUBCONTRACTED PARAMETERS

37. The EDD must be populated with **ALL** appropriate and applicable fields, including **ALL** QC data for any subcontracted parameters.

PLEASE CONTACT THE PIKA-PIRNIE JV Team PROJECT CHEMIST, DATA MANAGER or PROJECT MANAGER IF THERE ARE ANY QUESTIONS REGARDING PREPARATION OR GENERATION OF THE EDD.

EXAMPLE EDD REPORTS

The following subsections provide examples of how the EQuIS EDD should be populated for QC data.

RESULT FILE FIELDS FOR A NORMAL FIELD SAMPLE, TRG AND TIC RESULTS

The table below shows some of the fields in the Result File for a normal field sample (i.e., Sample_type_code = N, TB, FD, etc.) and "TRG" or "TIC" analyte_type_code. NOTE: all QC fields are blank.

cas_rn	result value	qc original conc	qc spike added	qc spike measured	qc spike recovery	qc dup. original conc	qc dupl. spike added	qc dup. spike measured	qc dup. spike recovery
93-76-5 94-75-7 94-82-6	3.17 1.56 2.31								

RESULT FILE FIELDS FOR A NORMAL FIELD SAMPLE WITH SURROGATES

The following table shows some of the fields in the result file for a normal field sample (i.e., Sample_type_code = N, TB, etc.). Note that QC fields are blank except on surrogate Rows.

cas_rn	result value	result unit	result type code	qc original conc	qc spike added	qc spike measured	qc spike recovery
93-76-5	1.56	mg/L	TRG				
94-75-7	3.17	mg/L	TRG				
PHEN2F		mg/L	SUR		12.5	12.9	103

RESULT FILE FIELDS FOR A MATRIX SPIKE

The following table shows some of the fields in the result file for a matrix spike sample (i.e., Sample_type_code = MS). Note that all "dup" QC fields are blank, and that the result_value field is NULL. Also, the qc_rpd field would be blank for these rows. The parent_sample_code must contain the contents of the sys_sample_code of the original (parent) sample.

cas_rn	result value	qc original conc	qc spike added	qc spike measured	qc spike recovery	qc dup. original conc	qc dupl. Spike added	qc dup. spike measured	qc dup. spike recovery
93-76-5		1.56	4.18	5.36	90.9				
94-75-7		3.17	4.18	7.15	95.2				
94-82-6		2.31	4.22	5.66	79.3				

RESULT FILE FIELDS FOR A MATRIX SPIKE DUPLICATE

The following table shows some of the fields in the result file for a matrix spike/matrix spike duplicate considered as a single sample (i.e., Sample_type_code = MSD). Note that all QC fields are completed, and that the result_value field is not needed. Also, the qc_rpd field would be completed for these rows. The parent_sample_code must contain the contents of the sys_sample_code of the original (parent) sample.

cas_rn	result value	qc original conc	qc spike added	qc spike measured	qc spike recovery	qc dup original conc	qc dup. spike added	qc dup spike measured	qc dup spike recovery
93-76-5						1.56	4.23	5.70	97.8
94-75-7						3.17	4.23	7.62	105
94-82-6						2.31	4.13	5.33	73.1

**RESULT FILE FIELDS FOR A LCS or BS **

The following table shows some of the fields in the result file for an LCS sample (i.e., laboratory control sample, blank spike, Sample_type_code = BS). The qc_rpd field is left blank for these rows.

cas_rn	result value	qc original conc	qc spike added	qc spike measured	qc spike recovery	qc dup original conc	qc dup spike added	qc dup spike measured	qc dup spike recovery
93-76-5		1.5	5.00	5.26	105				
94-75-7		10.2	1.00	1.02	102				
94-82-6		3.4	12.5	12.9	103				

RESULT FILE FIELDS FOR A LCS DUPLICATE OR BS DUPLICATE

The following table shows some of the fields in the result file for a laboratory control sample duplicate (i.e., Sample_type_code = BD). Note that the result_value field is not required. Also, the qc_rpd field must be completed for these rows.

cas_rn	result value	qc original conc	qc spike added	qc spike measured	qc spike recovery	qc dup original conc	qc dup spike added	qc dup spike measured	qc dup spike recovery	qc_rpd
93-76-5							5.00	4.92	98	2.0
94-75-7							1.00	0.95	95	6.6
94-82-6							12.5	11.8	94	12.3

REANALYSES, REEXTRACTIONS, DILUTIONS

The following table shows how to report retests for three different circumstances. The first example, the sample was retested (for 75-25-2) because the initial result required reanalysis due to QC failure. For the second example, the initial sample result (for 95-95-4) required dilution. The third example (for 67-66-3) required both reanalysis and dilution (reanalysis supercedes dilution). The fourth example (87-86-5) shows an initial result that require re-extraction due to QC failure or elevated concentrations that could not be diluted based on the original extraction. The other results are "turned off" by setting the reportable_result field to "No".

test_type	cas_rn	result_value	reportable_result
initial	75-25-2	1.2	No
reanalysis	75-25-2	1.1	Yes
initial	95-95-4	250E	No
dilution	95-95-4	328	Yes
initial	67-66-3	3.4	No
reanalysis	67-66-3	3.3	Yes
initial	87-86-5	980E	No
reextraction	87-86-5	1500	Yes

ANALYSES REQUIRING SECOND COLUMN CONFIRMATION

Analyte identification requiring confirmation by a second analytical technique is required by certain gas chromatography (GC) methods. A common technique used to confirm the identity of an analyte is to analyze the sample using a second GC column that is dissimilar from the GC column used for the first analysis. This confirmation technique is used routinely when analyzing samples for pesticides, herbicides, and certain volatile organic compounds (e.g., BTEX), and the two analyses often are performed simultaneously using an instrument equipped with dual GC columns connected to common injection port.

The method for reporting data from dual column GC analyses is not standard throughout the environmental laboratory industry. PIKA-PIRNIE JV Team recommends that laboratories use the method described in SW-846 Method 8000B, unless project-specific requirements or the method used for analysis dictate otherwise. The following table illustrates the proper format to be used to report first and second column results. The results for the first and third constituents (75-25-2 and 95-95-4) are being reported from column 1, and the result for the second constituent (67-66-3) is being reported from column 2. The other results are "turned off" by setting the reportable_result field to "No".

column_number	cas_rn	result_value	reportable_result
1C	75-25-2	6.2	Yes
1C	67-66-3	3.4	No
1C	95-95-4	5.6	Yes
2C	75-25-2	1.3	No
2C	67-66-3	33.7	Yes
2C	95-95-4	5.4	No

REFERENCE TABLES

A number of fields in each of the EDD files must be entered to correspond exactly with reference values standardized by PIKA-PIRNIE JV Team. These reference values will be updated from time to time. Each laboratory will be supplied a copy of the updated document. It is the laboratory's responsibility to submit EDDs using the most current reference tables as defined by a specific project.

The following table summarizes the EDD fields where standard reference values must be used:

EDD File	EDD Field	Reference Table
Sample	sample_type_code	rt_sample_type
	sample_matrix_code	rt_matrix
Test	lab_anl_method_name	rt_anl_mthd
	lab_matrix_code	rt_matrix
	prep_method	rt_std_prep_mthd
	subsample_amount_unit	rt_unit
	final_volume_unit	rt_unit
Result	lab_anl_method_name	rt_anl_mthd
	cas_rn	rt_analyte
	chemical_name	rt_analyte
	result_type_code	rt_result_type
	lab_qualifier	rt_qualifier
	result_unit	rt_unit
	detection_limit_unit	rt_unit
Batch	lab_anl_method_name	rt_anl_mthd

IV. EDP

The EDP data checker assists the **LABORATORY** in checking EDD files to ensure that they are error-free prior to submission to PIKA-PIRNIE JV Team. All laboratories providing data to PIKA-PIRNIE JV Team **must use** the EDP program to verify that EDDs are without error. The EDP error reports for each file **must be** submitted with each EDD.

The use of the EDDP helps to solve common data population problems including duplicate data, incorrectly populated fields, and incorrect methods, CAS #s, and other acceptable reference values. If an EDD is received by PIKA-PIRNIE JV Team containing errors it will be rejected until the EDD report is acceptable for import into the EQuIS database. Invoice payment will not be made until the EDD is acceptable.

PIKA-PIRNIE JV Team will provide laboratories with the most recent version of the EDP.

Appendix B

COC Form

PIKA MALCOLM PIRNIE

ID#:

CHAIN OF CUSTODY & LABORATORY ANALYSIS REQUEST FORM

Page ___ of ___

Lab Work Order #

Send Results to:

Contact & Company Name:	Telephone:
Address:	Fax:
City State Zip	E-mail Address:
Project Name/Location (City, State):	Project #:
Sampler's Printed Name:	Sampler's Signature:

Preservative								
Filtered (✓)								
# of Containers								
Container Information								

PARAMETER ANALYSIS & METHOD

Keys

Preservation Key:
A. H_2SO_4
B. HCL
C. HNO_3
D. NaOH
E. None
F. Other: _____
G. Other: _____
H. Other: _____

Container Information Key:
1. 40 ml Vial
2. 1 L Amber
3. 250 ml Plastic
4. 500 ml Plastic
5. Encore
6. 2 oz. Glass
7. 4 oz. Glass
8. 8 oz. Glass
9. Other: _____
10. Other: _____

Matrix Key:
SO - Soil | SE - Sediment | NL - NAPL/Oil
W - Water | SL - Sludge | SW - Sample Wipe
T - Tissue | A - Air | Other: _____

Sample ID	Collection Date	Collection Time	Type (✓) Comp	Type (✓) Grab	Matrix								REMARKS

Special Instructions/Comments:

☐ **Special QA/QC Instructions(✓):**

Laboratory Information and Receipt		Relinquished By	Received By	Relinquished By	Laboratory Received By
Lab Name:	Cooler Custody Seal (✓)	Printed Name:	Printed Name:	Printed Name:	Printed Name:
☐ Cooler packed with ice (✓)	☐ Intact ☐ Not Intact	Signature:	Signature:	Signature:	Signature:
Specify Turnaround Requirements:	Sample Receipt:	Firm:	Firm/Courier:	Firm/Courier:	Firm:
Shipping Tracking #:	Condition/Cooler Temp: _____	Date/Time:	Date/Time:	Date/Time:	Date/Time:

Appendix C

Laboratory QA Manuals and SOPs

CERTIFICATE OF ACCREDITATION

ANSI-ASQ National Accreditation Board/ACLASS
500 Montgomery Street, Suite 625, Alexandria, VA 22314, 877-344-3044

This is to certify that

TestAmerica Chicago
2417 Bond Street
University Park, IL 60484

has been assessed by ACLASS
and meets the requirements of

DoD-ELAP

while demonstrating technical competence in the field(s) of

TESTING

Refer to the accompanying Scope(s) of Accreditation for information regarding the types of tests to which this accreditation applies.

ADE-1429
Certificate Number

ACLASS Approval
Certificate Valid: 01/06/2010-01/06/2012
Version No. 001

SCOPE OF DoD-ELAP ACCREDITATION

TestAmerica Chicago
2417 Bond Street, University Park, IL 60484
Terese A. Preston Phone: 708-534-5200

TESTING

Valid to: January 6, 2012 Certificate Number: ADE- 1429

I. Environmental

MATRIX	SPECIFIC TEST or GROUP OF ANALYTES	SPECIFICATION OR STANDARD METHOD (all SW846 unless specified)	* KEY EQUIPMENT OR TECHNOLOGY USED
Water	Acid digestion for metals analysis	3010A	
Water	Acid digestion for metals analysis	3005A	
Water	Mercury digestion for water analysis	7470A	
Solid	Acid digestion for metals analysis	3050B	
Solid	Mercury digestion for soil analysis	7471A / 7471B	
Water	Purge and trap for aqueous samples	5030B / 5030C	
Solid	Closed-system purge and trap extraction for VOA analysis	5035 / 5035A	
Water	Separatory extraction for semivolatile and non-volatile organics	3510C	
Solid	Soxhlet extraction for semivolatile and non-volatile organics	3541	
Water	Herbicide extraction for water analysis	8151A	
Solid	Herbicide extraction for soil analysis	8151A	

MATRIX	SPECIFIC TEST or GROUP OF ANALYTES	SPECIFICATION OR STANDARD METHOD (all SW846 unless specified)	* KEY EQUIPMENT OR TECHNOLOGY USED
Water	Aqueous salting out extraction for explosive analysis	8330 / 8330A	
Solid	Sonication extraction for explosive analysis	8330 / 8330A	
Solid	Hexavalent chromium digestion for solid analysis	3060A	
Water	Cyanide extraction for water analysis	9010B / 9010C	
Solid	Cyanide extraction for soil and oil analysis	9013 / 9013A	
Solid	Extraction procedure for acid extractable sulfide analysis	9030B	
Water/Solid	Volatile analysis	8260B	GC/MS
Water/Solid	Semivolatile analysis	8270C / 8270D	GC/MS
Water/Solid	Pesticide analysis	8081A / 8081B	GC / ECD
Water/Solid	Herbicide analysis	8151A	GC / ECD
Water/Solid	PCB analysis	8082 / 8082A	GC / ECD
Water/Solid	GRO volatile analysis	8015B_GRO / 8015C_GRO	GC / FID
Water/Solid	DRO semivolatile analysis	8015B_DRO / 8015C_DRO	GC / FID
Water/Solid	PAH analysis	8310	HPLC / Fluorescence / Variable Wavelength UV Detector
Water/Solid	Explosive analysis	8330 / 8330A	HPLC / Variable Wavelength UV Detector

MATRIX	SPECIFIC TEST or GROUP OF ANALYTES	SPECIFICATION OR STANDARD METHOD (all SW846 unless specified)	* KEY EQUIPMENT OR TECHNOLOGY USED
Water/Solid	Metals ICPMS analysis	6020 / 6020A	ICP-MS
Water/Solid	Metals ICPAES analysis	6010B / 6010C	ICP-AES
Water	Mercury CVAA analysis	7470A	CVAA
Solid	Mercury CVAA analysis	7471A / 7471B	CVAA
Water/Solid	Hexavalent chromium analysis	7196A	Spectrophotometer
Water/ Solid	Cyanide analysis	9014	Spectrophotometer
Water	Anion analysis	9056 / 9056A / 300.0	Ion Chromatography
Solid	Anion analysis	9056 / 9056A /	Ion Chromatography
Water	Total organic carbon analysis	9060	UV Persulfate
Solid	Total organic carbon analysis	Lloyd Kahn	Combustion / Oxidation
Water/Solid	Total phenolics analysis	9066	Discrete Analyzer
Water	n-Hexane Extractable Material (HEM and SGT-HEM) analysis	EPA 1664A	Automated SPE – Gravimetric
Solid	n-Hexane Extractable Material (HEM and SGT-HEM) analysis	9071B	Soxtherm – Gravimetric
Water/Solid	Acid extractable sulfide analysis	9034	Titrimetric

Notes:

1. ** = As Applicable*
2. *This scope is part of and must be included with the Certificate of Accreditation No. ADE-1429*

Vice President

DoD ELAP -- PT Performance Summary Review					
Lab Name :	**TestAmerica, Chicago**				
City/State :	University Park/IL				
PT provider used :	Wibby				
PartName	Matrix	EPA Method#	AnalyteName	PT results - Pass / Acceptable	
Oil & Grease	HW	1664A	HEM (Oil & Grease)	**Pass**	
Minerals	HW	300.0	Bromide	**Pass**	
Minerals	HW	300.0	Chloride	**Pass**	
Minerals	HW	300.0	Fluoride	**Pass**	
Anions (1)	HW	300.0	Nitrate and Nitrite as N	**Pass**	
Nutrients (1) Amm, Nitra, O-Phos	HW	300.0	Nitrate as N	**Pass**	
Nutrients (3) Nitrite as N	HW	300.0	Nitrite as N	**Pass**	
Nutrients (1) Amm, Nitra, O-Phos	HW	300.0	Orthophosphate as P	**Pass**	
Minerals	HW	300.0	Sulfate	**Pass**	
Trace Metals	Soil/HW	6010B	Aluminum	**Pass**	
Trace Metals	Soil/HW	6010B	Antimony	**Pass**	
Trace Metals	Soil/HW	6010B	Arsenic	**Pass**	
Trace Metals	Soil/HW	6010B	Barium	**Pass**	
Trace Metals	Soil/HW	6010B	Beryllium	**Pass**	
Trace Metals	Soil/HW	6010B	Boron	**Pass**	
Trace Metals	Soil/HW	6010B	Cadmium	**Pass**	
Minerals	Soil/HW	6010B	Calcium	**Pass**	
Trace Metals	Soil/HW	6010B	Chromium	**Pass**	
Trace Metals	Soil/HW	6010B	Cobalt	**Pass**	
Trace Metals	Soil/HW	6010B	Copper	**Pass**	
Trace Metals	Soil/HW	6010B	Iron	**Pass**	
Trace Metals	Soil/HW	6010B	Lead	**Pass**	
Minerals	Soil/HW	6010B	Magnesium	**Pass**	
Trace Metals	Soil/HW	6010B	Manganese	**Pass**	
Trace Metals	Soil/HW	6010B	Molybdenum	**Pass**	
Trace Metals	Soil/HW	6010B	Nickel	**Pass**	
Minerals	Soil/HW	6010B	Potassium	**Pass**	
Trace Metals	Soil/HW	6010B	Selenium	**Pass**	
Trace Metals	Soil/HW	6010B	Silver	**Pass**	
Silica	HW	6010B	SiO2, Silica	**Pass**	
Minerals	Soil/HW	6010B	Sodium	**Pass**	
Trace Metals	Soil/HW	6010B	Strontium	**Pass**	
Trace Metals	Soil/HW	6010B	Thallium	**Pass**	
Tin & Titanium	Soil/HW	6010B	Tin	**Pass**	
Tin & Titanium	Soil/HW	6010B	Titanium	**Pass**	
Trace Metals	Soil/HW	6010B	Vanadium	**Pass**	
Trace Metals	Soil/HW	6010B	Zinc	**Pass**	
Trace Metals	Soil/HW	6010C	Aluminum	**Pass**	
Trace Metals	Soil/HW	6010C	Antimony	**Pass**	
Trace Metals	Soil/HW	6010C	Arsenic	**Pass**	
Trace Metals	Soil/HW	6010C	Barium	**Pass**	
Trace Metals	Soil/HW	6010C	Beryllium	**Pass**	
Trace Metals	Soil/HW	6010C	Boron	**Pass**	
Trace Metals	Soil/HW	6010C	Cadmium	**Pass**	
Trace Metals	Soil/HW	6010C	Calcium	**Pass**	
Trace Metals	Soil/HW	6010C	Chromium	**Pass**	
Trace Metals	Soil/HW	6010C	Cobalt	**Pass**	
Trace Metals	Soil/HW	6010C	Copper	**Pass**	
Trace Metals	Soil/HW	6010C	Iron	**Pass**	

Trace Metals	Soil/HW	6010C	Lead	**Pass**	
Trace Metals	Soil/HW	6010C	Magnesium	**Pass**	
Trace Metals	Soil/HW	6010C	Manganese	**Pass**	
Trace Metals	Soil/HW	6010C	Molybdenum	**Pass**	
Trace Metals	Soil/HW	6010C	Nickel	**Pass**	
Trace Metals	Soil/HW	6010C	Potassium	**Pass**	
Trace Metals	Soil/HW	6010C	Selenium	**Pass**	
Trace Metals	Soil/HW	6010C	Silver	**Pass**	
Trace Metals	Soil/HW	6010C	Sodium	**Pass**	
Trace Metals	Soil/HW	6010C	Strontium	**Pass**	
Trace Metals	Soil/HW	6010C	Thallium	**Pass**	
Trace Metals	Soil/HW	6010C	Tin	**Pass**	
Trace Metals	Soil/HW	6010C	Titanium	**Pass**	
Trace Metals	Soil/HW	6010C	Vanadium	**Pass**	
Trace Metals	Soil/HW	6010C	Zinc	**Pass**	
Trace Metals	Soil/HW	6020	Aluminum	**Pass**	
Trace Metals	Soil/HW	6020	Antimony	**Pass**	
Trace Metals	Soil/HW	6020	Arsenic	**Pass**	
Trace Metals	Soil/HW	6020	Barium	**Pass**	
Trace Metals	Soil/HW	6020	Beryllium	**Pass**	
Trace Metals	Soil/HW	6020	Boron	**Pass**	
Trace Metals	Soil/HW	6020	Cadmium	**Pass**	
Minerals	Soil/HW	6020	Calcium	**Pass**	
Trace Metals	Soil/HW	6020	Chromium	**Pass**	
Trace Metals	Soil/HW	6020	Cobalt	**Pass**	
Trace Metals	Soil/HW	6020	Copper	**Pass**	
Trace Metals	Soil/HW	6020	Iron	**Pass**	
Trace Metals	Soil/HW	6020	Lead	**Pass**	
Minerals	Soil/HW	6020	Magnesium	**Pass**	
Trace Metals	Soil/HW	6020	Manganese	**Pass**	
Trace Metals	Soil/HW	6020	Molybdenum	**Pass**	
Trace Metals	Soil/HW	6020	Nickel	**Pass**	
Minerals	Soil/HW	6020	Potassium	**Pass**	
Trace Metals	Soil/HW	6020	Selenium	**Pass**	
Trace Metals	Soil/HW	6020	Silver	**Pass**	
Minerals	Soil/HW	6020	Sodium	**Pass**	
Trace Metals	Soil/HW	6020	Thallium	**Pass**	
Trace Metals	Soil/HW	6020	Vanadium	**Pass**	
Trace Metals	Soil/HW	6020	Zinc	**Pass**	
Chromium VI	Soil/HW	7196A	Chromium, hexavalent	**Pass**	
Mercury	Soil/HW	7470A	Mercury	**Pass**	
DRO	Soil/HW	8015B	Diesel Range Organics [C10-C28]	**Pass**	
GRO	Soil/HW	8015B	Gasoline Range Organics (C6-C9)	**Pass**	
Pesticides	Soil/HW	8081A	4,4'-DDD	**Pass**	
Pesticides	Soil/HW	8081A	4,4'-DDE	**Pass**	
Pesticides	Soil/HW	8081A	4,4'-DDT	**Pass**	
Pesticides	Soil/HW	8081A	Aldrin	**Pass**	
Pesticides	Soil/HW	8081A	alpha-BHC	**Pass**	
Pesticides	Soil/HW	8081A	alpha-Chlordane	**Pass**	
Pesticides	Soil/HW	8081A	beta-BHC	**Pass**	
Chlordane	Soil/HW	8081A	Chlordane (technical)	**Pass**	
Pesticides	Soil/HW	8081A	delta-BHC	**Pass**	
Pesticides	Soil/HW	8081A	Dieldrin	**Pass**	
Pesticides	Soil/HW	8081A	Endosulfan I	**Pass**	
Pesticides	Soil/HW	8081A	Endosulfan II	**Pass**	
Pesticides	Soil/HW	8081A	Endosulfan sulfate	**Pass**	

Pesticides	Soil/HW	8081A	Endrin	**Pass**	
Pesticides	Soil/HW	8081A	Endrin aldehyde	**Pass**	
Pesticides	Soil/HW	8081A	Endrin ketone	**Pass**	
Pesticides	Soil/HW	8081A	gamma-BHC (Lindane)	**Pass**	
Pesticides	Soil/HW	8081A	gamma-Chlordane	**Pass**	
Pesticides	Soil/HW	8081A	Heptachlor	**Pass**	
Pesticides	Soil/HW	8081A	Heptachlor epoxide	**Pass**	
Pesticides	Soil/HW	8081A	Methoxychlor	**Pass**	
Toxaphene	Soil/HW	8081A	Toxaphene	**Pass**	
PCBs in Water	Soil/HW	8082	PCB-1016	**Pass**	
PCBs in Water	Soil/HW	8082	PCB-1221	**Pass**	
PCBs in Water	Soil/HW	8082	PCB-1232	**Pass**	
PCBs in Water	Soil/HW	8082	PCB-1242	**Pass**	
PCBs in Water	Soil/HW	8082	PCB-1248	**Pass**	
PCBs in Water	Soil/HW	8082	PCB-1254	**Pass**	
PCBs in Water	Soil/HW	8082	PCB-1260	**Pass**	
Herbicides	Soil/HW	8151A	2,4,5-T	**Pass**	
Herbicides	Soil/HW	8151A	2,4-D	**Pass**	
Herbicides	Soil/HW	8151A	2,4-DB	**Pass**	
Herbicides 4np&pcp- Acid std	Soil/HW	8151A	4-Nitrophenol	**Pass**	
Herbicides	Soil/HW	8151A	4-Nitrophenol	**Pass**	
Herbicides	Soil/HW	8151A	Dalapon	**Pass**	
Herbicides	Soil/HW	8151A	Dicamba	**Pass**	
Herbicides	Soil/HW	8151A	Dichlorprop	**Pass**	
Herbicides	Soil/HW	8151A	Dinoseb	**Pass**	
Herbicides 4np&pcp- Acid std	Soil/HW	8151A	Pentachlorophenol	**Pass**	
Herbicides	Soil/HW	8151A	Pentachlorophenol	**Pass**	
Herbicides	Soil/HW	8151A	Picloram	**Pass**	
Herbicides	Soil/HW	8151A	Silvex (2,4,5-TP)	**Pass**	
Volatiles (8260B)	HW	8260B	1,1,1,2-Tetrachloroethane	**Pass**	
Volatiles (8260B)	HW	8260B	1,1,1-Trichloroethane	**Pass**	
Volatiles (8260B)	HW	8260B	1,1,2,2-Tetrachloroethane	**Pass**	
Volatiles (8260B)	HW	8260B	1,1,2-Trichloroethane	**Pass**	
Volatiles (8260B)	HW	8260B	1,1-Dichloroethane	**Pass**	
Volatiles (8260B)	HW	8260B	1,1-Dichloroethene	**Pass**	
Volatiles (8260B)	HW	8260B	1,1-Dichloropropene	**Pass**	
Volatiles (8260B)	HW	8260B	1,2,3-Trichlorobenzene	**Pass**	
Volatiles (8260B)	HW	8260B	1,2,3-Trichloropropane	**Pass**	
Volatiles (8260B)	HW	8260B	1,2,4-Trichlorobenzene	**Pass**	
Volatiles (8260B)	ust/HW	8260B	1,2,4-Trimethylbenzene	**Pass**	
Volatiles (8260B)	HW	8260B	1,2-Dibromo-3-Chloropropane	**Pass**	
Volatiles (8260B)	HW	8260B	1,2-Dibromoethane	**Pass**	
Volatiles (8260B)	HW	8260B	1,2-Dichlorobenzene	**Pass**	
Volatiles (8260B)	HW	8260B	1,2-Dichloroethane	**Pass**	
Volatiles (8260B)	HW	8260B	1,2-Dichloropropane	**Pass**	
Volatiles (8260B)	ust/HW	8260B	1,3,5-Trimethylbenzene	**Pass**	
Volatiles (8260B)	HW	8260B	1,3-Dichlorobenzene	**Pass**	
Volatiles (8260B)	HW	8260B	1,4-Dichlorobenzene	**Pass**	
Volatiles (8260B)	HW	8260B	2-Butanone (MEK)	**Pass**	
Volatiles (8260B)	HW	8260B	2-Chloroethyl vinyl ether	**Pass**	
Volatiles (8260B)	HW	8260B	2-Chlorotoluene	**Pass**	
Volatiles (8260B)	HW	8260B	2-Hexanone	**Pass**	
Volatiles (8260B)	HW	8260B	4-Methyl-2-pentanone (MIBK)	**Pass**	
Volatiles (8260B)	HW	8260B	Acetone	**Pass**	
Volatiles (8260B)	HW	8260B	Acetonitrile	**Pass**	
Volatiles (8260B)	HW	8260B	Acrolein	**Pass**	

Volatiles (8260B)	HW	8260B	Acrylonitrile	**Pass**	
Volatiles (8260B)	ust/HW	8260B	Benzene	**Pass**	
Volatiles GC (BTEX&MTBE)	HW	8260B	Benzene	**Pass**	
WI-GRO/PVOC (WI-GRO,8260B)	HW	8260B	Benzene	**Pass**	
Volatiles (8260B)	HW	8260B	Bromochloromethane	**Pass**	
Volatiles (8260B)	HW	8260B	Bromodichloromethane	**Pass**	
Volatiles (8260B)	HW	8260B	Bromoform	**Pass**	
Volatiles (8260B)	HW	8260B	Bromomethane	**Pass**	
Volatiles (8260B)	HW	8260B	Carbon disulfide	**Pass**	
Volatiles (8260B)	HW	8260B	Carbon tetrachloride	**Pass**	
Volatiles (8260B)	HW	8260B	Chlorobenzene	**Pass**	
Volatiles (8260B)	HW	8260B	Chloroethane	**Pass**	
Volatiles (8260B)	HW	8260B	Chloroform	**Pass**	
Volatiles (8260B)	HW	8260B	Chloromethane	**Pass**	
Volatiles (8260B)	HW	8260B	cis-1,2-Dichloroethene	**Pass**	
Volatiles (8260B)	HW	8260B	cis-1,3-Dichloropropene	**Pass**	
Volatiles (8260B)	HW	8260B	Dibromochloromethane	**Pass**	
Volatiles (8260B)	HW	8260B	Dibromomethane	**Pass**	
Volatiles (8260B)	HW	8260B	Dichlorodifluoromethane	**Pass**	
Volatiles (8260B)	ust/HW	8260B	Ethylbenzene	**Pass**	
Volatiles GC (BTEX&MTBE)	HW	8260B	Ethylbenzene	**Pass**	
WI-GRO/PVOC (WI-GRO,8260B)	HW	8260B	Ethylbenzene	**Pass**	
Volatiles (8260B)	HW	8260B	Hexachlorobutadiene	**Pass**	
Volatiles (8260B)	HW	8260B	m&p-Xylene	**Pass**	
BTEX&MTBE (8260B,OA-1MS)	HW	8260B	m&p-Xylene	**Pass**	
WI-GRO/PVOC (WI-GRO,8260B)	HW	8260B	m&p-Xylene	**Pass**	
Volatiles (8260B)	ust/HW	8260B	Methyl tert-butyl ether	**Pass**	
Volatiles GC (BTEX&MTBE)	HW	8260B	Methyl tert-butyl ether	**Pass**	
WI-GRO/PVOC (WI-GRO,8260B)	HW	8260B	Methyl tert-butyl ether	**Pass**	
Volatiles (8260B)	HW	8260B	Methylene Chloride	**Pass**	
Volatiles (8260B)	HW	8260B	Naphthalene	**Pass**	
Volatiles (8260B)	HW	8260B	o-Xylene	**Pass**	
BTEX&MTBE (8260B,OA-1MS)	HW	8260B	o-Xylene	**Pass**	
WI-GRO/PVOC (WI-GRO,8260B)	HW	8260B	o-Xylene	**Pass**	
Volatiles (8260B)	HW	8260B	Styrene	**Pass**	
Volatiles (8260B)	HW	8260B	Tetrachloroethene	**Pass**	
Volatiles (8260B)	ust/HW	8260B	Toluene	**Pass**	
Volatiles GC (BTEX&MTBE)	HW	8260B	Toluene	**Pass**	
WI-GRO/PVOC (WI-GRO,8260B)	HW	8260B	Toluene	**Pass**	
Volatiles (8260B)	HW	8260B	trans-1,2-Dichloroethene	**Pass**	
Volatiles (8260B)	HW	8260B	trans-1,3-Dichloropropene	**Pass**	
Volatiles (8260B)	HW	8260B	Trichloroethene	**Pass**	
Volatiles (8260B)	HW	8260B	Trichlorofluoromethane	**Pass**	
Volatiles (8260B)	HW	8260B	Vinyl acetate	**Pass**	
Volatiles (8260B)	HW	8260B	Vinyl chloride	**Pass**	
Volatiles (8260B)	ust/HW	8260B	Xylenes, Total	**Pass**	
Volatiles GC (BTEX&MTBE)	HW	8260B	Xylenes, Total	**Pass**	
WI-GRO/PVOC (WI-GRO,8260B)	HW	8260B	Xylenes, Total	**Pass**	
BaseNeutrals (625, 8270C)	Soil/HW	8270C	1,2,4,5-Tetrachlorobenzene	**Pass**	
BaseNeutrals (625, 8270C)	Soil/HW	8270C	1,2,4-Trichlorobenzene	**Pass**	
BaseNeutrals (625, 8270C)	Soil/HW	8270C	1,2-Dichlorobenzene	**Pass**	
BaseNeutrals (625, 8270C)	Soil/HW	8270C	1,3-Dichlorobenzene	**Pass**	
Explosives in Soil	Soil/HW	8270C	1,3-Dinitrobenzene	**Pass**	
BaseNeutrals (625, 8270C)	Soil/HW	8270C	1,4-Dichlorobenzene	**Pass**	
BaseNeutrals (625, 8270C)	Soil/HW	8270C	1-Chloronaphthalene	**Pass**	
BaseNeutrals (625, 8270C)	Soil/HW	8270C	2,2'-oxybis[1-chloropropane]	**Pass**	

Acids (625, 8270C)	Soil/HW	8270C	2,3,4,6-Tetrachlorophenol	**Pass**	
Acids (625, 8270C)	Soil/HW	8270C	2,4,5-Trichlorophenol	**Pass**	
Acids (625, 8270C)	Soil/HW	8270C	2,4,6-Trichlorophenol	**Pass**	
Acids (625, 8270C)	Soil/HW	8270C	2,4-Dichlorophenol	**Pass**	
Acids (625, 8270C)	Soil/HW	8270C	2,4-Dimethylphenol	**Pass**	
Acids (625, 8270C)	Soil/HW	8270C	2,4-Dinitrophenol	**Pass**	
BaseNeutrals (625, 8270C)	Soil/HW	8270C	2,4-Dinitrotoluene	**Pass**	
Acids (625, 8270C)	Soil/HW	8270C	2,6-Dichlorophenol	**Pass**	
BaseNeutrals (625, 8270C)	Soil/HW	8270C	2,6-Dinitrotoluene	**Pass**	
BaseNeutrals (625, 8270C)	Soil/HW	8270C	2-Chloronaphthalene	**Pass**	
Acids (625, 8270C)	Soil/HW	8270C	2-Chlorophenol	**Pass**	
BaseNeutrals (625, 8270C)	Soil/HW	8270C	2-Methylnaphthalene	**Pass**	
Acids (625, 8270C)	Soil/HW	8270C	2-Methylphenol	**Pass**	
BaseNeutrals (625, 8270C)	Soil/HW	8270C	2-Nitroaniline	**Pass**	
Acids (625, 8270C)	Soil/HW	8270C	2-Nitrophenol	**Pass**	
Acids (625, 8270C)	Soil/HW	8270C	3 & 4 Methylphenol	**Pass**	
BaseNeutrals (625, 8270C)	Soil/HW	8270C	3,3'-Dichlorobenzidine	**Pass**	
BaseNeutrals (625, 8270C)	Soil/HW	8270C	3-Nitroaniline	**Pass**	
Acids (625, 8270C)	Soil/HW	8270C	4,6-Dinitro-2-methylphenol	**Pass**	
BaseNeutrals (625, 8270C)	Soil/HW	8270C	4-Bromophenyl phenyl ether	**Pass**	
Acids (625, 8270C)	Soil/HW	8270C	4-Chloro-3-methylphenol	**Pass**	
BaseNeutrals (625, 8270C)	Soil/HW	8270C	4-Chloroaniline	**Pass**	
BaseNeutrals (625, 8270C)	Soil/HW	8270C	4-Chlorophenyl phenyl ether	**Pass**	
BaseNeutrals (625, 8270C)	Soil/HW	8270C	4-Nitroaniline	**Pass**	
Acids (625, 8270C)	Soil/HW	8270C	4-Nitrophenol	**Pass**	
BaseNeutrals (625, 8270C)	Soil/HW	8270C	Acenaphthene	**Pass**	
BaseNeutrals (625, 8270C)	Soil/HW	8270C	Acenaphthylene	**Pass**	
BaseNeutrals (625, 8270C)	Soil/HW	8270C	Aniline	**Pass**	
BaseNeutrals (625, 8270C)	Soil/HW	8270C	Anthracene	**Pass**	
BaseNeutrals (625, 8270C)	Soil/HW	8270C	Benzidine	**Pass**	
BaseNeutrals (625, 8270C)	Soil/HW	8270C	Benzo[a]anthracene	**Pass**	
BaseNeutrals (625, 8270C)	Soil/HW	8270C	Benzo[a]pyrene	**Pass**	
BaseNeutrals (625, 8270C)	Soil/HW	8270C	Benzo[b]fluoranthene	**Pass**	
BaseNeutrals (625, 8270C)	Soil/HW	8270C	Benzo[g,h,i]perylene	**Pass**	
BaseNeutrals (625, 8270C)	Soil/HW	8270C	Benzo[k]fluoranthene	**Pass**	
Acids (625, 8270C)	Soil/HW	8270C	Benzoic acid	**Pass**	
BaseNeutrals (625, 8270C)	Soil/HW	8270C	Benzyl alcohol	**Pass**	
BaseNeutrals (625, 8270C)	Soil/HW	8270C	Bis(2-chloroethoxy)methane	**Pass**	
BaseNeutrals (625, 8270C)	Soil/HW	8270C	Bis(2-chloroethyl)ether	**Pass**	
BaseNeutrals (625, 8270C)	Soil/HW	8270C	Bis(2-ethylhexyl) phthalate	**Pass**	
BaseNeutrals (625, 8270C)	Soil/HW	8270C	Butyl benzyl phthalate	**Pass**	
BaseNeutrals (625, 8270C)	Soil/HW	8270C	Carbazole	**Pass**	
BaseNeutrals (625, 8270C)	Soil/HW	8270C	Chrysene	**Pass**	
BaseNeutrals (625, 8270C)	Soil/HW	8270C	Dibenz(a,h)anthracene	**Pass**	
BaseNeutrals (625, 8270C)	Soil/HW	8270C	Dibenzofuran	**Pass**	
BaseNeutrals (625, 8270C)	Soil/HW	8270C	Diethyl phthalate	**Pass**	
BaseNeutrals (625, 8270C)	Soil/HW	8270C	Dimethyl phthalate	**Pass**	
BaseNeutrals (625, 8270C)	Soil/HW	8270C	Di-n-butyl phthalate	**Pass**	
BaseNeutrals (625, 8270C)	Soil/HW	8270C	Di-n-octyl phthalate	**Pass**	
Herbicides in Soil	Soil/HW	8270C	Dinoseb	**Pass**	
BaseNeutrals (625, 8270C)	Soil/HW	8270C	Fluoranthene	**Pass**	
BaseNeutrals (625, 8270C)	Soil/HW	8270C	Fluorene	**Pass**	
BaseNeutrals (625, 8270C)	Soil/HW	8270C	Hexachlorobenzene	**Pass**	
BaseNeutrals (625, 8270C)	Soil/HW	8270C	Hexachlorobutadiene	**Pass**	
BaseNeutrals (625, 8270C)	Soil/HW	8270C	Hexachlorocyclopentadiene	**Pass**	
BaseNeutrals (625, 8270C)	Soil/HW	8270C	Hexachloroethane	**Pass**	

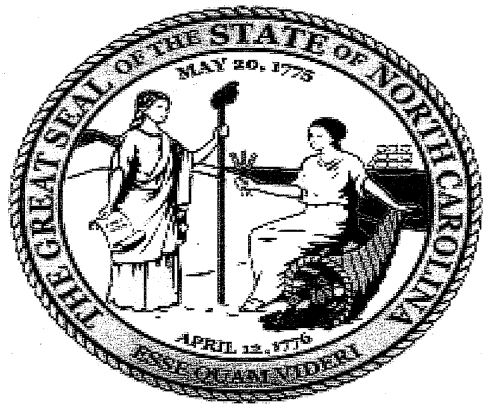
BaseNeutrals (625, 8270C)	Soil/HW	8270C	Indeno[1,2,3-cd]pyrene	**Pass**	
BaseNeutrals (625, 8270C)	Soil/HW	8270C	Isophorone	**Pass**	
BaseNeutrals (625, 8270C)	Soil/HW	8270C	Naphthalene	**Pass**	
BaseNeutrals (625, 8270C)	Soil/HW	8270C	Nitrobenzene	**Pass**	
BaseNeutrals (625, 8270C)	Soil/HW	8270C	N-Nitrosodiethylamine	**Pass**	
BaseNeutrals (625, 8270C)	Soil/HW	8270C	N-Nitrosodimethylamine	**Pass**	
BaseNeutrals (625, 8270C)	Soil/HW	8270C	N-Nitrosodi-n-propylamine	**Pass**	
BaseNeutrals (625, 8270C)	Soil/HW	8270C	N-Nitrosodiphenylamine	**Pass**	
BaseNeutrals (625, 8270C)	Soil/HW	8270C	o-Toluidine	**Pass**	
BaseNeutrals (625, 8270C)	Soil/HW	8270C	Pentachlorobenzene	**Pass**	
Acids (625, 8270C)	Soil/HW	8270C	Pentachlorophenol	**Pass**	
BaseNeutrals (625, 8270C)	Soil/HW	8270C	Phenanthrene	**Pass**	
Acids (625, 8270C)	Soil/HW	8270C	Phenol	**Pass**	
BaseNeutrals (625, 8270C)	Soil/HW	8270C	Pyrene	**Pass**	
BaseNeutrals (625, 8270C)	Soil/HW	8270C	Pyridine	**Pass**	
PAHs - HPLC	Soil/HW	8310	Acenaphthene	**Pass**	
PAHs - HPLC	Soil/HW	8310	Acenaphthylene	**Pass**	
PAHs - HPLC	Soil/HW	8310	Anthracene	**Pass**	
PAHs - HPLC	Soil/HW	8310	Benzo[a]anthracene	**Pass**	
PAHs - HPLC	Soil/HW	8310	Benzo[a]pyrene	**Pass**	
PAHs - HPLC	Soil/HW	8310	Benzo[b]fluoranthene	**Pass**	
PAHs - HPLC	Soil/HW	8310	Benzo[g,h,i]perylene	**Pass**	
PAHs - HPLC	Soil/HW	8310	Benzo[k]fluoranthene	**Pass**	
PAHs - HPLC	Soil/HW	8310	Chrysene	**Pass**	
PAHs - HPLC	Soil/HW	8310	Dibenz(a,h)anthracene	**Pass**	
PAHs - HPLC	Soil/HW	8310	Fluoranthene	**Pass**	
PAHs - HPLC	Soil/HW	8310	Fluorene	**Pass**	
PAHs - HPLC	Soil/HW	8310	Indeno[1,2,3-cd]pyrene	**Pass**	
PAHs - HPLC	Soil/HW	8310	Naphthalene	**Pass**	
PAHs - HPLC	Soil/HW	8310	Phenanthrene	**Pass**	
PAHs - HPLC	Soil/HW	8310	Pyrene	**Pass**	
Explosives	Soil/HW	8330	1,3,5-Trinitrobenzene	**Pass**	
Explosives	Soil/HW	8330	1,3-Dinitrobenzene	**Pass**	
Explosives	Soil/HW	8330	2,4,6-Trinitrotoluene	**Pass**	
Explosives	Soil/HW	8330	2,4-Dinitrotoluene	**Pass**	
Explosives	Soil/HW	8330	2,6-Dinitrotoluene	**Pass**	
Explosives	Soil/HW	8330	2-Amino-4,6-dinitrotoluene	**Pass**	
Explosives	Soil/HW	8330	2-Nitrotoluene	**Pass**	
Explosives	Soil/HW	8330	3-Nitrotoluene	**Pass**	
Explosives	Soil/HW	8330	4-Amino-2,6-dinitrotoluene	**Pass**	
Explosives	Soil/HW	8330	4-Nitrotoluene	**Pass**	
Explosives	Soil/HW	8330	Cyclotrimethylenetrinitramine	**Pass**	
Explosives	Soil/HW	8330	Methyl-2,4,6-trinitrophenylnitramine	**Pass**	
Explosives	Soil/HW	8330	Nitrobenzene	**Pass**	
Explosives	Soil/HW	8330	Octahydro-1,3,5,7-tetranitro-1,3,5,7-	**Pass**	
Total Cyanide	Soil/HW	9014	Cyanide, Total	**Pass**	
Sulfide	HW	9034	Sulfide	**Pass**	
Minerals	Soil/HW	9056	Bromide	**Pass**	
Minerals	Soil/HW	9056	Chloride	**Pass**	
Minerals	Soil/HW	9056	Fluoride	**Pass**	
Nutrients (1) Amm, Nitra, O-Phos	Soil/HW	9056	Nitrate as N	**Pass**	
Nutrients (3) Nitrite as N	Soil/HW	9056	Nitrite as N	**Pass**	
Nutrients (1) Amm, Nitra, O-Phos	Soil/HW	9056	Orthophosphate as P	**Pass**	
Minerals	Soil/HW	9056	Sulfate	**Pass**	
Demand	Soil/HW	9060	TOC Dup	**Pass**	
Phenolics	Soil/HW	9066	Phenolics, Total Recoverable	**Pass**	

Oil & Grease	ust / soil	9071B	HEM (Oil & Grease)	**Pass**	
Nutrients (1) Amm, Nitra, O-Phos	Soil/HW	SM 4500 P E	Orthophosphate as P	**Pass**	
Nutrients (2) TKN, Tot Phos	Soil/HW	SM 4500 P E	Phosphorus as P	**Pass**	
Sulfide	Soil/HW	SM 4500 S2 F	Sulfide	**Pass**	

STATE OF NORTH CAROLINA DEPARTMENT OF THE
ENVIRONMENT AND NATURAL RESOURCES

DIVISION OF WATER QUALITY
LABORATORY CERTIFICATION PROGRAM

In accordance with the provisions of N.C.G.S. 143-215.3 (a) (1), 143-215.3 (a)(10) and NCAC 2H.0800:



2011

TESTAMERICA CHICAGO

Is hereby certified to perform environmental analysis as listed on Attachment I and report monitoring data to DWQ for compliance with NPDES effluent, surface water, groundwater, and pretreatment regulations.

By reference 15A NCAC 2H .0800 is made a part of this certificate.

This certificate does not guarantee validity of data generated, but indicates the methodology, equipment, quality control procedures, records, and proficiency of the laboratory have been examined and found to be acceptable.

This certificate shall be valid until December 31, 2011

Certificate No 291

J. Kent Wiggins

J. Kent Wiggins

(C-2) C-12

Attachment I

North Carolina Wastewater/Groundwater Laboratory Certification

Certified Parameters Listing

Lab Name:	TestAmerica Chicago	Certificate Number:	291
Address:	2417 Bond Street University Park Chicago, IL 60484-	Effective Date:	01/01/2011
		Expiration Date:	12/31/2011
		Date of Last Amendment:	09/19/2011

The above named laboratory, having duly met the requirements of 15A NCAC 2H.0800, is hereby certified for the measurement of the parameters listed below.

CERTIFIED PARAMETERS

INORGANICS

ALKALINITY
- Std Method 2320B

BOD
- Std Method 5210B

COD
- Std Method 5220C

CHLORIDE
- Std Method 4500 Cl E
- EPA Method 300
- SW846 Method 9056A

CYANIDE
- Std Method 4500 CN E (Total)
- SW846 Method 9014 (Colorimetric)

FLUORIDE
- Std Method 4500 F C
- EPA Method 300
- SW846 Method 9056A

HARDNESS TOTAL
- Std Method 2340B
- EPA Method 200.7

AMMONIA NITROGEN
- Std Method 4500 NH3 C
- Std Method 4500 NH3 G 20th Ed
- EPA Method 350.1

TOTAL KJELDAHL NITROGEN
- Std Method 4500 NH3 C
- EPA Method 351.1

NO2 + NO3 NITROGEN
- Std Method 4500 NO3 F
- EPA Method 353.2

NITRATE NITROGEN
- SW846 Method 9056A
- EPA Method 300
- Nitrate-nitrite N minus Nitrite N

NITRITE NITROGEN
- Std Method 4500 NO2 B
- EPA Method 300
- SW846 Method 9056A

TOTAL PHOSPHORUS
- Std Method 4500 P E
- Std Method 4500 P F
- EPA Method 365.1

ORTHOPHOSPHATE
- EPA Method 300
- SW846 Method 9056A

OIL & GREASE
- EPA Method 1664 Rev A

pH
- Std Method 4500 H B
- SW846 Method 9040C

INORGANIC PHENOLS
- EPA Method 420.4
- SW846 Method 9066

RESIDUE TOTAL
- Std Method 2540B
- Std Method 2540G

RESIDUE DISSOLVED 180 C
- Std Method 2540C

RESIDUE SUSPENDED
- Std Method 2540D

SULFATE
- EPA Method 300
- SW846 Method 9056A
- SW846 Method 9038

SULFIDE
- Std Method 4500 S F
- SW846 Method 9034

TOTAL ORGANIC CARBON
- Std Method 5310C
- SW846 Method 9060A

METALS

ALUMINUM
- EPA Method 200.7
- EPA Method 200.8
- SW846 Method 6010C
- SW846 Method 6020A

ANTIMONY
- EPA Method 200.7
- EPA Method 200.8
- SW846 Method 6010C
- SW846 Method 6020A

ARSENIC
- EPA Method 200.7
- EPA Method 200.8
- SW846 Method 6010C
- SW846 Method 6020A

BARIUM
- EPA Method 200.7
- EPA Method 200.8
- SW846 Method 6010C
- SW846 Method 6020A

BERYLLIUM
- EPA Method 200.7
- EPA Method 200.8
- SW846 Method 6010C
- SW846 Method 6020A

CADMIUM
- EPA Method 200.7
- EPA Method 200.8
- SW846 Method 6010C
- SW846 Method 6020A

CALCIUM
- EPA Method 200.7
- EPA Method 200.8
- SW846 Method 6010C
- SW846 Method 6020A

CHROMIUM TOTAL
- EPA Method 200.7
- EPA Method 200.8
- SW846 Method 6010C
- SW846 Method 6020A

COBALT
- EPA Method 200.7
- EPA Method 200.8
- SW846 Method 6010C
- SW846 Method 6020A

COPPER
- EPA Method 200.7
- EPA Method 200.8
- SW846 Method 6010C
- SW846 Method 6020A

IRON
- EPA Method 200.7
- SW846 Method 6010C
- SW846 Method 6020A
- EPA Method 200.8

LEAD
- EPA Method 200.7
- EPA Method 200.8
- SW846 Method 6010C
- SW846 Method 6020A

MAGNESIUM
- EPA Method 200.7
- EPA Method 200.8
- SW846 Method 6010C
- SW846 Method 6020A

MANGANESE
- EPA Method 200.7
- EPA Method 200.8
- SW846 Method 6010C
- SW846 Method 6020A

MERCURY
- EPA Method 245.1

This certification requires maintance of an acceptable quality assurance program, use of approved methodology, and satisfactory performance on evaluation samples. Laboratories are subject to civil penalties and/or decertification for infractions as set forth in 15A NCAC 2H.0807.

Attachment I

North Carolina Wastewater/Groundwater Laboratory Certification

Certified Parameters Listing

Lab Name:	TestAmerica Chicago	Certificate Number:	291
Address:	2417 Bond Street University Park Chicago, IL 60484-	Effective Date:	01/01/2011
		Expiration Date:	12/31/2011
		Date of Last Amendment:	09/19/2011

The above named laboratory, having duly met the requirements of 15A NCAC 2H.0800, is hereby certified for the measurement of the parameters listed below.

CERTIFIED PARAMETERS

SW846 Method 7470A

MOLYBDENUM
- EPA Method 200.7
- EPA Method 200.8
- SW846 Method 6010C
- SW846 Method 6020A

NICKEL
- EPA Method 200.7
- EPA Method 200.8
- SW846 Method 6010C
- SW846 Method 6020A

POTASSIUM
- EPA Method 200.7
- SW846 Method 6010C
- EPA Method 200.8
- SW846 Method 6020A

SELENIUM
- EPA Method 200.7
- EPA Method 200.8
- SW846 Method 6010C
- SW846 Method 6020A

SILVER
- EPA Method 200.7
- EPA Method 200.8
- SW846 Method 6010C
- SW846 Method 6020A

SODIUM
- EPA Method 200.7
- SW846 Method 6010C
- EPA Method 200.8
- SW846 Method 6020A

THALLIUM
- EPA Method 200.7
- EPA Method 200.8
- SW846 Method 6010C
- SW846 Method 6020A

VANADIUM
- EPA Method 200.7
- EPA Method 200.8
- SW846 Method 6010C
- SW846 Method 6020A

ZINC
- EPA Method 200.7
- EPA Method 200.8
- SW846 Method 6010C
- SW846 Method 6020A

TCLP METALS
- SW846 Method 1311

SPLP METALS
- SW846 Method 1312

ORGANICS

ORGANOCHLORINE PESTICIDES & PCBs
- EPA Method 608

ORGANOCHLORINE PESTICIDES
- SW846 Method 8081B

POLYCHLORINATED BIPHENYLS (PCB's)
- SW846 Method 8082A

POLYNUCLEAR AROMATIC HYDROCARBONS
- EPA Method 610
- SW846 Method 8310

PURGEABLE ORGANICS
- EPA Method 624
- SW846 Method 8260B

BASE NEUTRAL/ACID ORGANICS
- EPA Method 625
- SW846 Method 8270D

CHLORINATED ACID HERBICIDES
- SW846 Method 8151A

TPH DIESEL RANGE ORGANICS
- SW846 Method 8015C

TPH GASOLINE RANGE ORGANICS
- SW846 Method 8015C

TOTAL ORGANIC HALIDES
- SW846 Method 9020B

TCLP ORGANICS
- SW846 Method 1311

SPLP ORGANICS
- SW846 Method 1312

This certification requires maintance of an acceptable quality assurance program, use of approved methodology, and satisfactory performance on evaluation samples. Laboratories are subject to civil penalties and/or decertification for infractions as set forth in 15A NCAC 2H.0807.

North Carolina Department of Environment and Natural Resources
Division of Water Quality
Coleen H. Sullins
Director

Beverly Eaves Perdue
Governor

Dee Freeman
Secretary

October 6, 2011

RECEIVED

OCT - 6 2011

TESTAMERICA LABORATORIES
CHICAGO

291
Ms. Nadine Jernberg
TestAmerica Chicago
2417 Bond Street
University Park
Chicago, IL 60484

SUBJECT: Additional Parameter Certification
Ammonia Nitrogen – EPA Method 350.1 and Std Method, 20th Edition, 4500 NH_3 G
TKN – EPA Method 351.1
Total Phosphorus - EPA Method 365.1 and Std Method, 20th Edition, 4500-P F

Dear Ms. Jernberg:

The Department of Environment and Natural Resources, in accordance with the provisions of 15A NCAC 2H .0800, is pleased to certify your laboratory to perform additional analytical parameter(s). This change to your certification is effective September 19, 2011.

Enclosed is an amended certificate attachment that includes the new parameter(s). The same requirements applying to your present certification are applicable to the new parameter addition(s). Please review this attachment to insure that your laboratory is certified for all parameters required to properly meet your certification needs.

Contact your assigned auditor, Todd Crawford, at (919) 733-3908 ext. 251, if you have questions or need additional information.

Sincerely,

J. Kent Wiggins
Laboratory Section Chief
Laboratory Section

Enclosure
cc: Todd Crawford
Dana Satterwhite

DENR DWQ Laboratory Section NC Wastewater/Groundwater Laboratory Certification Branch
1623 Mail Service Center, Raleigh, North Carolina 27699-1623
Location: 4405 Reedy Creek Road. Raleigh, North Carolina 27607-6445
Phone: 919-733-3908 \ FAX: 919-733-6241
Internet: www.dwqlab.org

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

Quality Assurance Manual

TestAmerica Chicago
2417 Bond Street
University Park, IL 60484
Phone: 708-534-5200
Fax: 708-534-5211

www.testamericainc.com

Quality Assurance Manual Approval Signatures

Michael J Healy 2/28/11 Laboratory Director – Michael J. Healy Date	Gary Rynkar 2/28/11 Supervisor, GC, GC/MS Semivolatiles, HPLC – Gary Rynkar Date
Terese A. Preston 2/28/11 Quality Manager - Terese A. Preston Date	JoAnn Retruszak Kmetty 2-28-11 Supervisor, GC, GC/MS Volatiles JoAnn Retruszak Kmetty Date
Diane L Harper 2/28/11 Inorganics Manager – Diane L. Harper Date	Carla Bonner 2/28/11 Supervisor, General Chemistry – Carla Bonner Date
Jodi Gromala 2/28/11 Organics Manager – Jodi Gromala Date	Debbie Johnson 2/28/11 Supervisor, Metals – Debbie Johnson Date
2/28/11 Customer Service Manager – Eric Lang Date	2/28/11 EH&S Coordinator – John D. Nagel Date
Karen LeClair 2/28/11 Report Production Manager - Karen LeClair Date	2/28/11 Supervisor, Sample Receipt & Container Management Jeff James Date

Facility Distribution No. Uncontrolled **Distributed To:**_____

SECTION 2

TABLE OF CONTENTS

LIST OF TABLES

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REFERENCED CORPORATE SOPs AND POLICIES

SOP/Policy Reference	Title	Cited Section No(s)
CA-C-S-001	Work Sharing Process	8.1; 8.2
CA-I-P-002	Electronic Reporting and Signature Policy	26.2.25
CA-L-P-001	Ethics Policy	5.2; 18.4; Appendix I
CA-L-P-002	Contract Compliance Policy	7.2
CA-L-S-001	Internal Investigation of Potential Data Discrepancies and Determination for Data Recall	5.2; 12.2; 12.3; 13.4; Table 13.1; 17.1; 17.3
CA-L-S-002	Subcontracting Procedures	8.1, 8.2; 26.5
CA-Q-M-002	Corporate Quality Management Plan (CQMP)	3.1; 3.4.1; 4.1; 4.2.1; 5.3.1
CA-Q-S-001	Solvent and Acid Lot Testing and Approval	9.3; 22.4
CA-Q-S-002	Acceptable Manual Integration Practices	20.14.2; 20.14.4; 20.14.5
CA-Q-S-003	Management of Change Procedure	14.2
CA-Q-S-004	Method Compliance & Data Authenticity Audits	16.1
CA-Q-S-005	Calibration Curves (General)	21.4.2.1; 21.4.2.3; 21.4.2.7
CA-Q-S-006	Detection Limits	20.7
CA-Q-S-008	Management Systems Review	17.2
CA-Q-WI-020	Management Review	17.2
CA-T-P-001	Qualified Products List	9.4
CW-E-M-001	Corporate Environmental Health & Safety Manual	9.3.4
CW-F-P-002	Authorization Matrix Policy	9.1
CW-F-P-004	Procurement & Contracts Policy	9.1; 9.6
CW-F-S-007	Controlled Purchases Policy/Procedure	9.1
CW-F-S-018	Vendor Selection	9.6
CW-F-WI-007	J.D. Edwards Vendor Add Request Form	9.6.1
CW-F-WI-009	Vendor Performance Report	8.2.3; 9.6

SOP/Policy Reference	Title	Cited Section No(s)
CW-L-P-001	Record Retention	15.1.1; 15.5.6.3
CW-Q-S-001	Corporate Document Control and Archiving	6.1; 6.3
CW-Q-S-002	Writing a Standard Operating Procedure (SOPs)	6.3; 20.2

REFERENCED LABORATORY SOPs

SOP Reference	Title	Cited Section No(s)
UP-DM-002	Data Management: Record Retention & Purging	6.4; 15.0; 15.1; 15.1.3; 15.1.4
UP-FS-001	Field Services; Groundwater Sampling – Bailing Method	23.1
UP-IS-014	Proc/Processes Entry, Storage, Backup/Retrieval, Mgmt Bench Data	15.0; 15.1; 15.1.4; 20.14.1
UP-QA-003	Balance Calibration, Care and Use	21.3.1
UP-QA-006	Document Control	3.4.1; 6.1; 6.3; 20.2
UP-QA-014	Training Program: Mechanisms and Documentation Processes Defined by Operational Assessment	18.3
UP-QA-017	Method Detection Limits	20.7; 20.7.5; 20.7.6; 20.9.1
UP-QA-022	Refrigerated Storage Monitoring Volatile Samples	24.4
UP-QA-032	SOP Change Protocol	3.4.1; 6.3; 20.2
UP-QA-034	Thermometer Calibrations	21.3.3
UP-QA-039	Sample Homogenization Sub Sampling Procedures	23.6
UP-QA-040	Quality Assurance – Measurement Uncertainty	20.12.4
UP-SR-001	Sample Receipt: Handling and Processing Procedures	24.2; 24.2.1.8; 24.2.2; Figure 24-5
UP-WM-001	Laboratory Waste Disposal Procedures	24.7

SECTION 3

INTRODUCTION *(NELAC 5.1 - 5.3)*

3.1 INTRODUCTION AND COMPLIANCE REFERENCES *(ISO17025 4.1.2; 4.2.4)*

TestAmerica Chicago's Quality Assurance Manual (QAM) is a document prepared to define the overall policies, organization objectives and functional responsibilities for achieving TestAmerica's data quality goals. The laboratory maintains a local perspective in its scope of services and client relations and maintains a national perspective in terms of quality.

The QAM has been prepared to assure compliance with the 2003 National Environmental Laboratory Accreditation Conference (NELAC) standards and International ANS/ISO/IEC Standard 17025:2005. In addition, the policies and procedures outlined in this manual are compliant with TestAmerica's Corporate Quality Management Plan (CQMP) and the various accreditation and certification programs listed in Appendix 5. The CQMP provides a summary of TestAmerica's quality and data integrity system. It contains requirements and general guidelines under which all TestAmerica facilities shall conduct their operations. The relevant NELAC section is included in the heading of each QAM section.

The QAM has been prepared to be consistent with the requirements of the following documents:

- EPA 600/4-88/039, *Methods for the Determination of Organic Compounds in Drinking Water,* EPA, Revised July 1991.
- EPA 600/R-95/131, *Methods for the Determination of Organic Compounds in Drinking Water,* Supplement III, EPA, August 1995.
- EPA 600/4-79-019, *Handbook for Analytical Quality Control in Water and Wastewater Laboratories,* EPA, March 1979.
- Test Methods for Evaluating Solid Waste Physical/Chemical Methods (SW846), Third Edition, September 1986, Final Update I, July 1992, Final Update IIA, August 1993, Final Update II, September 1994; Final Update IIB, January 1995; Final Update III, December 1996; Final Update IV, January 2008.
- Federal Register, 40 CFR Parts 136, 141, 172, 173, 178, 179 and 261.
- Statement of Work for Inorganics Analysis, SOM and ISM, current versions, USEPA Contract Laboratory Program Multi-media, Multi-concentration.
- APHA, *Standard Methods for the Examination of Water and Wastewater,* 18th Edition, 19th, 20th and 21st Edition.
- U.S. Department of Energy Order 414.1C, Quality Assurance, June 17, 2005.
- *U.S. Department of Energy, Quality Systems for Analytical Services,* Revision 2.4, October 28, 2008.
- U.S. Department of Defense, *Quality Systems Manual for Environmental Laboratories,* Final Version 4.2, October 2010.
- U.S. Department of Defense, *Air Force Center for Environmental Excellence Quality Assurance Project Plan (QAPP),* Version 4.0.02, May 2006.
- National Environmental Laboratory Accreditation Conference, Constitution, Bylaws, and Standards, EPA 600/R-04/003, US EPA Office of Research and Development, June 2003
- Toxic Substances Control Act (TSCA).

3.2 TERMS AND DEFINITIONS *(ISO17025 4.2.4)*

A Quality Assurance Program is a company-wide system designed to ensure that data produced by the laboratory conforms to the standards set by state and/or federal regulations. The program functions at the management level through company goals and management policies, and at the analytical level through Standard Operating Procedures (SOPs) and quality control. The TestAmerica program is designed to minimize systematic error, encourage constructive, documented problem solving, and provide a framework for continuous improvement within the organization.

Refer to Appendix 4 for the Glossary/Acronyms.

3.3 SCOPE / FIELDS OF TESTING *(ISO17025 4.1.2; 4.2.4)*

The laboratory analyzes a broad range of environmental and industrial samples every month. Sample matrices vary among drinking water, surface water, groundwater, effluent water, leachates, wastewater, soil, sediment, sludge, ash, paint chips, filters, wipes, waste and tissue. The Quality Assurance Program contains specific procedures and methods to test samples of differing matrices for chemical, physical and biological parameters. The Program also contains guidelines on maintaining documentation of analytical process, reviewing results, servicing clients and tracking samples through the laboratory. The technical and service requirements of all requests to provide analyses are thoroughly evaluated before commitments are made to accept the work. Measurements are made using published reference methods or methods developed and validated by the laboratory.

The methods covered by this manual include the most frequently requested methodologies needed to provide analytical services in the United States and its territories. The specific list of test methods used by the laboratory can be found in Appendix 7. The approach of this manual is to define the minimum level of quality assurance and quality control necessary to meet requirements. All methods performed by the laboratory shall meet these criteria as appropriate. In some instances, quality assurance project plans (QAPPs), project specific data quality objectives (DQOs) or local regulations may require criteria other than those contained in this manual. In these cases, the laboratory will abide by the requested criteria following review and acceptance of the requirements by the Laboratory Director, Project Manager and the Quality Assurance (QA) Manager. In some cases, QAPPs and DQOs may specify less stringent requirements. The Laboratory Director and the QA Manager must determine if it is in the lab's best interest to follow the less stringent requirements.

3.4 MANAGEMENT OF THE MANUAL

3.4.1 Review Process *(ISO17025 4.2.1; 4.2.7; 4.3.3.2; 4.3.3.3)*

This manual is reviewed annually by senior laboratory management to assure that it reflects current practices and meets the requirements of the laboratory's clients and regulators as well as the CQMP. Occasionally, the manual may need changes in order to meet new or changing regulations and operations. The QA Manager will review the changes in the normal course of business and incorporate changes into revised sections of the document. All updates will be reviewed by the senior laboratory management staff. The laboratory updates and approves such changes according to our Document Control & Updating procedures (refer to SOP No. UP-QA-006). Laboratory-specific QAM and SOP changes are approved and documented as detailed in the following SOP: *UP-QA-032*_SOP Change Protocol.

Figure 3-1.
Example - Policy/Procedure Memorandum *(ISO17025 4.3.3.2; 4.3.3.3)*

TestAmerica Chicago
STANDARD OPERATING PRACTICE (SOP) CHANGE FORM

Original SOP Number/Revision #: _____ Last Mod ID (circle): NA / ____
SOP Title:_____
Affected SOP Section Number(s):_____
Effective Date:_____

CONTROLLED DISTRIBUTION

COPY # :

ISSUED TO :

Full Signature Approvals Are Kept on File with TestAmerica Chicago Laboratories Standard Practice Records

Revision Number with Mod ID:

The following SOP change is in effect as of the stated date. This form will remain attached to the referenced SOP until such a time that the SOP is updated, approved, and redistributed, at which time it will become part of the historical SOP record. **Append this form to the front of the SOP copy.**

1. Reason for SOP Change:_____

2. Summary of Procedure Change (circle to indicate if there are attachments to this form: No / Yes: # pages attached = ___)

_____ Initiated/Reviewed By: Name/Date

_____ Approval Signature/Date: Section Manager

_____ Initiated/Reviewed By: Name/Date

_____ Approval Signature/Date: QA Manager or Designee

SECTION 4

ORGANIZATION AND MANAGEMENT *(NELAC 5.4.1)*

4.1 OVERVIEW *(ISO17025 4.1.1; 4.1.3; 4.1.5; 4.2.6)*

TestAmerica Chicago is a local operating unit of TestAmerica Laboratories, Inc.. The organizational structure, responsibilities and authorities of the corporate staff of TestAmerica Laboratories, Inc. are presented in the CQMP. The laboratory has day-to-day independent operational authority overseen by corporate officers (e.g., President, Chief Operating Officer, Corporate Quality Assurance, etc.) The laboratory operational and support staff work under the direction of the Laboratory Director. The organizational structure for both Corporate & TestAmerica Chicago is presented in figure 4-1 and 4-2 respectively.

4.2 ROLES AND RESPONSIBILITIES *(ISO17025 4.1.3; 4.1.5; 4.2.6; 5.2.4)*

In order for the Quality Assurance Program to function properly, all members of the staff must clearly understand and meet their individual responsibilities as they relate to the quality program. The following descriptions define each role in its relationship to the Quality Assurance Program.

4.2.1 Quality Assurance Program

The responsibility for quality lies with every employee of the laboratory. All employees have access to the QAM, are trained to this manual, and are responsible for upholding the standards therein. Each person carries out his/her daily tasks in a manner consistent with the goals and in accordance with the procedures in this manual and the laboratory's SOPs. Role descriptions for Corporate personnel are defined in the CQMP. This manual is specific to the operations of the TestAmerica Chicago laboratory.

4.2.2 General Manager (GM)

Each GM reports directly to the COO. Each GM has full responsibility for the overall administrative and operational management of their respective laboratories. The GM's responsibilities include allocation of personnel and resources, long-term planning, setting goals, and achieving the financial, business, and quality objectives of TestAmerica. The GM ensures timely compliance with corporate management directives, policies, and management systems reviews. The GM is also responsible for restricting any laboratory from performing analyses that cannot be consistently and successfully performed to meet the standards set forth in this manual and in the Corporate Quality Management Plan.

4.2.3 Laboratory Director *(ISO17025 4.1.6; 4.2.6)*

TestAmerica Chicago's Laboratory Director is responsible for the overall quality, safety, financial, technical, human resource and service performance of the whole laboratory and reports to their respective GM. The Laboratory Director provides the resources necessary to implement and maintain an effective and comprehensive Quality Assurance and Data Integrity Program.

The Laboratory Director, with the assistance of the Quality Assurance Manager, has the overall responsibility for establishing policies that ensure the quality of analytical services meet our client's expectations. These policies are defined in this QAM.

Specific responsibilities include, but are not limited to:

- Ensures that all analysts and supervisors have the appropriate education and training to properly carry out the duties assigned to them and ensures that this training has been documented.
- Ensures that personnel are free from any commercial, financial and other undue pressures which might adversely affect the quality of their work.
- Supports the Human Resources function to ensure that all policies and programs are applied consistently throughout the laboratory.
- Ensures that sufficient numbers of qualified personnel are employed to supervise and perform the work of the laboratory.
- Ensures that appropriate corrective actions are taken to address analyses identified as requiring such actions by internal and external performance or procedural audits. Procedures that do not meet the standards set forth in the QAM or laboratory SOPs may be temporarily suspended by the Laboratory Director.
- Reviews and approves Quality Assurance SOPs for the facility and ensures their implementation so that the facility is operated in a compliant manner which allows it to produce defensible data.
- Interfaces with Project Management and Customer Service to forecast receipts, provide quality analytical data to clients and meet on-time delivery dates.
- Communicates facility-specific goals and objectives to employees. Communicates and implements company initiatives designed to foster teamwork and communication.
- Ensures that the facility has appropriate Information Technology resources and that they are being used effectively to support operational requirements.
- Actively participates in the process of sharing and adopting best practices within TestAmerica. Provides technical assistance to other TestAmerica laboratories as needed to improve productivity and customer service.
- Prioritizes the activities of the operations groups to ensure key goals are achieved and customer service needs are addressed.
- Capital forecasting and instrument life cycle planning for second generation methods and instruments.

4.2.4 Quality Assurance (QA) Manager *(ISO17025 4.1.5; 4.1.6; 4.2.1; 4.2.6)*

The QA Manager has responsibility and authority to ensure the continuous implementation of the quality system based on ISO 17025:2005.

The QA Manager reports directly to the Laboratory Director and has access to Corporate QA for advice and resources. This position is able to evaluate data objectively and perform assessments without outside (i.e., managerial) influence. Corporate QA may be used as a

resource in dealing with regulatory requirements, certifications and other quality assurance related items. The QA Manager directs the activities of the QA officers to accomplish specific responsibilities, which include, but are not limited to:

- Ensuring communication and monitoring standards of performance to ensure that systems are in place to produce the level of quality as defined in this document.
- Notifying laboratory management of deficiencies in the quality system and ensuring corrective action is taken. Procedures that do not meet the standards set forth in the QAM or laboratory SOPs are temporarily suspended following the procedures outlined in Section 12.
- Evaluation of the thoroughness and effectiveness of training.
- Compliance with the ISO 17025:2005 Standard.
- Having functions independent from laboratory operations for which he/she has quality assurance oversight.
- Maintaining and updating the QAM.
- Monitoring laboratory certifications and scheduling of proficiency testing samples.
- Monitor and communicate regulatory changes that may affect the laboratory to the management staff.
- Training and advising the laboratory staff on quality assurance/quality control procedures that are pertinent to their daily activities.
- Training documentation review and maintenance.
- Having a general knowledge of the analytical test methods for which data audit/review is performed (and/or having the means of getting this information when needed).
- Arranging for or conducting internal audits on quality systems and the technical operation.
- The laboratory QA Manager will maintain records of all ethics-related training, including the type and proof of attendance.
- Maintain, improve, and evaluate the corrective action database and the corrective and preventive action systems.
- Notifying laboratory management of deficiencies in the quality system and ensuring corrective action is taken. Procedures that do not meet the standards set forth in the QAM or laboratory SOPs are temporarily suspended following the procedures outlined in Section 13.
- Monitoring standards of performance to ensure that systems are in place to produce the level of quality control as defined in the QAM.
- Coordinating of document control of SOPs, MDLs, control limits, and miscellaneous forms and information.
- Review and approval of IDL/MDL studies.
- Review and approval of Method Validation studies (IDOCs/CDOCs).
- Review and approve yearly statistical control limit evaluations.
- Review a percentage of all final data reports for internal consistency. Review of Chain of Custody (COC), correspondence with the analytical request, batch QC status, completeness

of any corrective action statements, 5% of calculations, format, holding time, sensibility and completeness of the project file contents.

- Review of external audit reports and data validation requests.
- Follow-up with audits to ensure client QAPP requirements are met.
- Establishment of reporting schedule and preparation of various quality reports for the Laboratory Director, clients and/or Corporate QA.
- Development of suggestions and recommendations to improve quality systems.
- Research of current state and federal requirements and guidelines.
- QA Manager assists in the preparation, compilation, and submittal of quality assurance plans; reviews program plans for consistency with organizational and contractual requirements (advises appropriate personnel of deficiencies).
- TALs LIMs method development, validation, verification and maintenance LIMs method reference limits
- The QA Manager shall have the final authority to accept or reject data, and to stop work in progress in the event that procedures or practices compromise the validity and integrity of analytical data.
- The QA Manager is available to any employee at the facility to resolve data quality or ethical issues.
- The QA Manager must address any data integrity issue identified internally or externally, establish a corrective action plan and resolve the issue to the client's satisfaction. Issues that involve data recall must be discussed with the Corporate Quality Director Verl Preston.
- Asset Inventory Management

4.2.5 Quality Assurance Specialist *(ISO17025 4.1.5; 4.1.6; 4.2.1)*

The QA Specialist is responsible for QA documentation and involvement in the following activities:

- Assist the QA Manager in performing the annual internal laboratory audits, compiling the evaluation, and coordinating the development of an action plan to address any deficiency identified.
- Facilitate external audits, coordinating with the QA Manager and Laboratory Staff to address any deficiencies noted at the time of the audit and subsequently presented in the final audit report.
- Assist the QA Manager in the preparation of new SOP's and in the maintenance of existing SOPs, coordinating annual reviews and updates.
- Manages the performance testing (PT) studies, coordinates follow up studies for failed analytes and works with QA Manager and Laboratory Staff to complete needed corrective action reports.
- Personnel training records review and maintenance.

- Document control maintenance.
- Assists the Quality Manager and Project Management Group in the review of program plans for consistency with organizational and contractual requirements. Summarize and convey to appropriate personnel anomalies or inconsistencies observed in the review process.
- Manages certifications and accreditations.
- Monitors for compliance the following QA Metrics: Temperature Monitoring of refrigeration units and incubators; thermometer calibrations; balance calibrations; eppendorf/pipette calibrations; and proper standard/reagent storage.
- Periodic checks on the proper use and review of instrument logs.
- Initiate the Mint-miner data file review process for organic instrumentation. Maintain tracking sheet of activity.
- Initiate the annual Instrument review.
- Assist in the technical review of data packages which require QA review.

4.2.6 Technical Director *(ISO17025 4.1.5; 4.1.6; 4.2.1)*

At TestAmerica Chicago, the responsibilities of the Technical Director are divided among the supervisory, managerial, and QA staff. These individuals are responsible for compliance with the ISO 17025 Standard.

4.2.7 LIMS Administrator

The LIMS Administrator reports directly to the Regional IT Supervisor.

The overall role of the Information Technology (IT) Manager is to enhance laboratory productivity through improved information access, flow, and security. For information to be of greatest value, it must be readily accessible and reliable. It is the responsibility of the IT Manager to provide software tools that allow quick and user friendly access to that information, while at the same time controlling access to that information to those that have the need and proper authority.

Information flow can be enhanced through automation. Automation is the minimization of human intervention in a process. Reduction in human intervention can result in significant error reductions and time savings. The IT Manager assists the laboratory in automation by providing hardware and software solutions to help minimize human intervention in data collection, processing, and storage.

The IT Manager is responsible for providing data security by controlling access, as mentioned above, and for providing for disaster recovery. Data stored on the central Laboratory Information Management System (LIMS) is the direct responsibility of the IT Manager. No fewer than two copies of all data should exist at any time so that lost or destroyed data can always be retrieved from an alternate source. These copies may consist of data within the system and on magnetic tape in the case of live data, or two copies on magnetic tape for archived data. Data stored

electronically in other departments is the direct responsibility of those departments. However, the IT Manager is responsible for providing procedures and training to all laboratory operations, as appropriate, to assist in making backup copies of local data within the respective operating unit.

In the pursuit of his duties, he:

- Maintains the laboratory information system (LIMS) for tracking all samples in the laboratory.
- Maintains historical files of software, software operating procedures (manuals), software changes/modifications (Change Log) and software version numbers.
- Maintains log of repairs and service performed on LIMS hardware.
- Verifies security practices to assure the integrity of LIMS data. Identifies threats, potential threats, and future threats.
- Maintains awareness of any environmental conditions of the facility housing the LIMS that may compromise LIMS raw data and informs management.
- LIMS database back-up once daily.

4.2.8 Department Manager (s) *(ISO17025 4.1.5; 4.1.6; 4.2.1)*

The Department Managers manage and direct the analytical production sections of the laboratory, and report directly to the Laboratory Director. More specifically, he/she:

- Is responsible for compliance with health, safety and quality assurance programs among the reporting laboratory groups.
- Continuously evaluates production capacity and improves capacity utilization.
- Continuously evaluates turnaround time and addresses any problems that may hinder meeting the required and committed turnaround time from the various departments.
- Develops and improves the training of all analysts in cooperation with the reporting Supervisors, QA department, and in compliance with regulatory requirements.
- Works with the reporting Supervisors to ensure that scheduled instrument maintenance is completed.
- Works with the Laboratory Director and reporting Supervisors to evaluate and maintain appropriate staffing levels within reporting sections.
- Is responsible for efficient utilization of supplies.
- Constantly monitors and modifies the processing of samples through the departments.
- Fully supports the quality system and, if called upon in the absence of the QA Manager, serves as his substitute in the interim.
- Assists with the review and development of standard operating procedures and offers recommendations as the need arises for new or revised analytical methods.
- Monitors the validity of the analyses performed and data generated in the laboratory, by analyzing the frequency and content of internal non-conformances to identify root cause issues and implementing the resulting corrective and preventive actions, facilitating the data

review process (training, development, and accountability at the bench), and providing technical and troubleshooting expertise on routine and unusual or complex problems.

- Enhancing efficiency and improving quality through technical advances and improved LIMS utilization.
- Captains department supervisors to communicate quality, technical, personnel, and instrumental issues for a consistent team approach.
- Coordinates audit responses with reporting Supervisors and QA Manager.
- Participates in the selection, development of performance objectives and standards of performance, appraisal (measurement of objectives), counseling, discipline, and motivation of the reporting Supervisors and documents these activities in accordance with systems developed by the QA and Personnel Departments.
- Coordinating, writing, and reviewing preparation of all test methods, i. e., SOPs, with regard to quality, integrity, regulatory and optimum and efficient production techniques, and subsequent analyst training and interpretation of the SOPs for implementation and unusual project samples. He/she insures that the SOPs are properly managed and adhered to at the bench.
- Ensure timely completion of all internal method audits and corrective actions.
- Providing training and development programs to applicable laboratory staff as new hires and, subsequently, on a scheduled basis. Training includes instruction on calculations, instrumentation management to include troubleshooting and preventive maintenance. Note: This responsibility is often shared with the Department Supervisors.

4.2.9 Data Management Supervisor

The Data Management Supervisor is responsible for coordinating receipt of all data from the various analytical groups within the laboratory, and ensuring that data are reported in a timely manner and in the proper format.

4.2.10 Hazardous Waste Coordinator

The Hazardous Waste Coordinator reports directly to the Laboratory Director. The duties consist of:

- Staying current with the hazardous waste regulations.
- Continuing training on hazardous waste issues.
- Reviewing and updating annually the Hazardous Waste Contingency Plan in the Environmental Health & Safety Manual.
- Auditing the staff with regard to compliance with the Hazardous Waste Contingency Plan.
- Contacting the hazardous waste subcontractors for review of procedures and opportunities for minimization of waste.

4.2.11 Supervisors *(ISO17025 4.1.5; 4.1.6; 4.2.1)*

Supervisors report to the Department Manager. Each one is responsible to:

- Ensure that analysts in their department adhere to applicable SOPs and the QA Manual. Perform frequent SOP and QA Manual review to determine if analysts are in compliance and if new, modified, and optimized measures are feasible and should be added to these documents.
- Participate in the selection, training (including familiarization with SOP, QC, safety, and computer systems), development of performance objectives and standards of performance, appraisal (measurement of objectives), scheduling, counseling, discipline, and motivation of analysts. Ensure the documentation of these activities in accordance with systems developed by the QA and Personnel Departments.
- Work with the Department Manager and Laboratory Director to evaluate staffing sufficiency and overtime needs.
- Encourage the reporting analysts to become self-supervising and to function as a departmental team member by facilitating cross-training in various methods and/or the operation of multiple instruments efficiently.
- Provide guidance to analysts in resolving problems encountered daily during sample prep/analysis in conjunction with the Department Manager, and/or QA Manager.
- Ensure the 200% review cycle is complete for all analysis, that non-conformance issues are completely documented and all corrective action taken in a timely manner and reported to the QA Manager, Department Manager, Project Manager and/or Laboratory Director, as appropriate. Be pro-active when possible.
- Ensure the on-time delivery of client sample results, as well as results for performance evaluation samples and IDLs/MDLs, and pro-actively report any late work to the Department Manager and/or Project Manager.
- Ensure all logbooks are completely and correctly maintained, current, and properly archived.
- Ensure that preventive maintenance is performed on instrumentation as detailed in the QA Manual, the instrument operations manual, or the SOPs. Assist in the timely resolution of instrument problems by helping with non-scheduled instrument repairs and trouble-shooting when possible, and /or facilitating technical support from manufacturers.
- Coordinate the staff in order to achieve optimum turnaround time on analyses and compliance with holding times.
- Provide written responses to external and internal audit issues and performance evaluation sample issues, as requested by the Department Manager or QA department.

4.2.12 Laboratory Analysts *(ISO17025 4.1.5)*

Laboratory analysts are responsible for conducting analysis and performing all tasks assigned to them by the group leader or supervisor. The responsibilities of the analysts are listed below:

- Perform analyses by adhering to analytical and quality control protocols prescribed by current SOPs, this QA Manual, and project-specific plans honestly, accurately, timely, safely, and in the most cost-effective manner.

- Document standard and sample preparation, instrument calibration and maintenance, data calculations, sample matrix effects, and any observed non-conformance on checklists, in lab data books and/or in the Non-Conformance Database by means of Non-Conformance Memos (NCMs).
- Pro-actively report all non-conformance situations, instrument problems, matrix problems and QC failures, which might affect the reliability of the data, to the Supervisor, the Department Manager, Project Manager and/or the QA Manager or member of QA staff.
- Perform 100% review of the data generated and document the review in the raw data and on the review checklist prior to entering and submitting for secondary level review.
- Perform the analytical work critically, and suggest improvements to the Supervisor, the Department Manager, and the QA Manager. These improvements, if within the constraints of the reference material and approved, are encouraged and will be incorporated.
- Work cohesively as a team with others in the department to achieve the goals of accurate results, optimum turnaround time, cost effectiveness, cleanliness, complete documentation, and personal knowledge of environmental analysis.
- Adhere to all safety protocols and attend safety meetings as required.
- Attend and participate in staff meetings.
- Maintain adequate and valid inventory of reagents, standards, spare parts, and other relevant resources required to perform daily analysis.
- Perform annual reviews of assigned SOPs to ensure that methods are followed and the SOP agrees with actual practices. SOPs must also be reviewed and revised any time a change has been made at the bench.
- Adhere to maintenance schedules outlined in instrument operations manuals and SOPs. Keep instruments and working areas clean with respect to both chemical contamination and clutter.
- Adhere to the ethics policies of TestAmerica Laboratories at all times and during all laboratory activities.
- Work to achieve optimal turn-around-times and adherence to all EPA-regulated holding times.

4.2.13 Safety Officer

The Safety Officer reports to the Laboratory Director and ensures that systems are maintained for the safe operation of the laboratory.

The Environmental Health and Safety Officer is responsible for the safety and well-being of all employees while at the laboratory. This includes, but is not limited to, administering the Corporate Safety Manual that complies with federal regulations, MSDS training and review, conducting laboratory safety orientation and tours for all new employees, providing instructions on safety equipment, cleaning up laboratory spills, and instructing personnel of laboratory procedures for emergency situations. The Health and Safety Coordinator is on-call 24-hours a day, 7-days a week for all laboratory situations.

The Environmental Health and Safety Officer responsibilities additionally include waste management of laboratory generated hazardous waste in accordance with appropriate regulations. This includes maintenance of required documentation, such as waste manifests, segregation of waste in accordance with requirements, and training of personnel in proper segregation of waste.

- Conduct ongoing, necessary safety training and conduct new employee safety orientation.
- Assist in developing and maintaining the Chemical Hygiene/Safety Manual.
- Administer dispersal of all Material Safety Data Sheet (MSDS) information.
- Perform regular chemical hygiene and housekeeping instruction.
- Give instruction on proper labeling and practice.
- Serve as chairman of the laboratory safety committee.
- Provide and train personnel on protective equipment.
- Oversee the inspection and maintenance of general safety equipment – fire extinguishers, safety showers, eyewash fountains, etc. and ensure prompt repairs as needed.
- Supervise and schedule fire drills and emergency evacuation drills.
- Determine what initial and subsequent exposure monitoring, if necessary to determine potential employee exposure to chemicals used in the laboratory.
- When determined necessary, conduct exposure monitoring assessments.
- Determine when a complaint of possible over-exposure is "reasonable" and should be referred for medical consultation.
- Assist in the internal and external coordination of the medical consultation/monitoring program conducted by TestAmerica's medical consultants.

4.2.14 Log-in Manager

The Log-in Manager reports to the Director of Project Management. The responsibilities are outlined below:

- Direct the logging of incoming samples into the LIMS.
- Ensure the verification of data entry from login.
- Coordinating sample management from "cradle to grave," insuring that no time is lost in locating samples.

4.2.15 Director of Project Management

The Director of Project Management reports to the Laboratory Director and serves as the interface between the laboratory's technical departments and the laboratory's clients. The staff consists of the Project Management team. With the overall goal of total client satisfaction, the functions of this position are outlined below:

The laboratory recognizes the importance of efficient project management. The laboratory Project Managers (PM) are responsible for preparing quotes, preparing the Project Technical

Profile which summarizes QA/QC requirements for the project, maintaining the laboratory schedule, ensuring that technical requirements are understood by the laboratory, and advising the Laboratory, QA and Technical Managers of all variances. The laboratory Project Manager will provide technical guidance and the necessary laboratory-related information to the preparer of project-specific QAPPs and provide peer review of the final document to ensure accuracy of the laboratory information.

The Project Manager is designated as the Sample Management Coordinator for any work subcontracted under their management. The Project Manager verifies each subcontracting request to ensure that special client restrictions are not jeopardized (e.g., samples must be analyzed by the receiving affiliated or network laboratory and must maintain specific certification(s)). The Project Manager is also responsible for verifying the credentials; establishing the service agreement; ensuring data review; and invoicing of all laboratory subcontractors. The Project Manager discusses any deficiencies or anomalies with the subcontractor prior to reporting any data to the client.

- Responsible for providing quotes for new opportunities.
- Technical training and growth of the Project Management team.
- Technical liaison for the Project Management team.
- Human resource management of the Project Management team.
- Responsible to ensure that clients receive the proper sampling supplies.
- Accountable for response to client inquiries concerning sample status.
- Responsible for assistance to clients regarding the resolution of problems concerning COC.
- Ensuring that client specifications, when known, are met by communicating project and quality assurance requirements to the laboratory.
- Notifying the supervisors of incoming projects and sample delivery schedules.
- Forecasting revenue and sample loading.
- Accountable to clients for communicating sample progress with agreed-upon due dates.
- Responsible for discussing with client any project-related problems, resolving service issues, and coordinating technical details with the laboratory staff.
- Responsible for staff familiarization with specific quotes, sample log-in review, and final report completeness.
- Monitor the status of all data package projects in-house to ensure timely and accurate delivery of reports.
- Inform clients of data package-related problems and resolve service issues.
- Coordinate requests for sample containers and other services (data packages).

4.2.16 Shipping Manager/Sample Archiving

The Shipping/Sample Login Manager reports directly to the Laboratory Director. He is responsible for:

- Supervising the timely and correct shipment of sample containers, including proper preservatives and instructions, to clients.
- Maintaining accurate records of sample container shipments
- The organized storage and appropriate climate control of samples

4.2.17 Facilities Maintenance/Sample Disposal

The Facilities Maintenance Manager reports directly to the Laboratory Director. He is responsible for the following:

- Managing facility maintenance.
- Supervising the disposal of samples in accordance with the Waste Disposal SOP, the Hazardous Waste Contingency Plan in the Chemical Hygiene/Safety Manual, and the U. S. Department of Agriculture requirements.

4.3 DEPUTIES *(ISO17025 4.1.5; 4.2.7)*

The following table defines who assumes the responsibilities of key personnel in their absence:

Key Personnel	Deputy	Comment
Laboratory Director – Mike Healy	Eric Lang Karen LeClair John D. Nagel	Client / Quote Purchasing / CapEx. Facility
Customer Service Manager – Eric Lang	Dick Wright Bonnie Stadelmann Marilyn Krueding Donna Ingersoll Jim Knapp Cindy Pritchard	PM PM PM CSM – Decatur Service Center CSM – Chicago Service Center Proposal Coordinator
QA Manager – Terese Preston	Nadine Jernberg	QA Specialist
Organic Department Manager / Technical Director – Jodi Gromala	JoAnn Petruszak Kmetty Gary Rynkar Dan Knieriemen	Supervisor – GC VOA / MS VOA Supervisor – GC/ HPLC / GCMS SVOA Supervisor – Organic Extractions
Metals Department Manager / Technical Director - Diane Harper	Debbie Johnson	Supervisor – Metals
Wet Chemistry Technical Director – Diane Harper	Carla Bonner	Supervisor – Wet Chemistry
EH&S Coordinator – John D. Nagel	Jeff James	Supervisor – Sample Management
Data Management – Karen LeClair	Paula Buckley	Specialist – Data Management
Sample Management – Jeff James	Jeff Lunt Lisa Piunti	Login Bottle Prep

Figure 4-1.

Corporate Organization Chart *(ISO17025 4.1.3; 4.1.5; 4.2.6)*

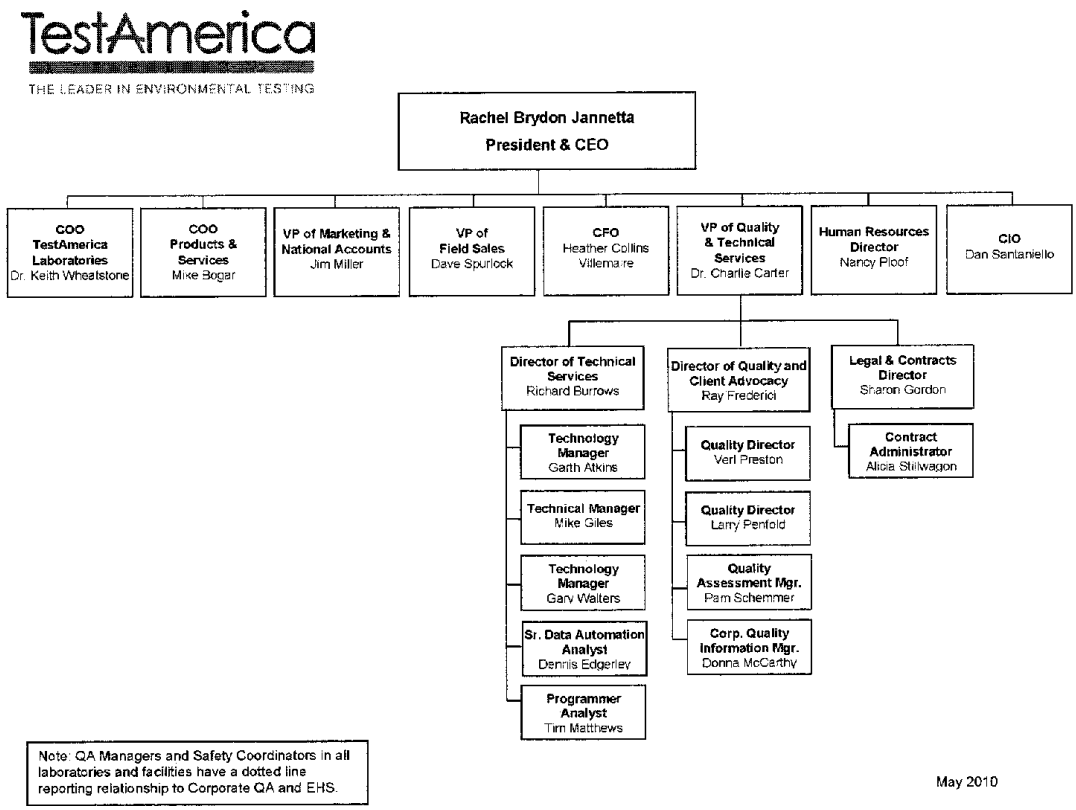
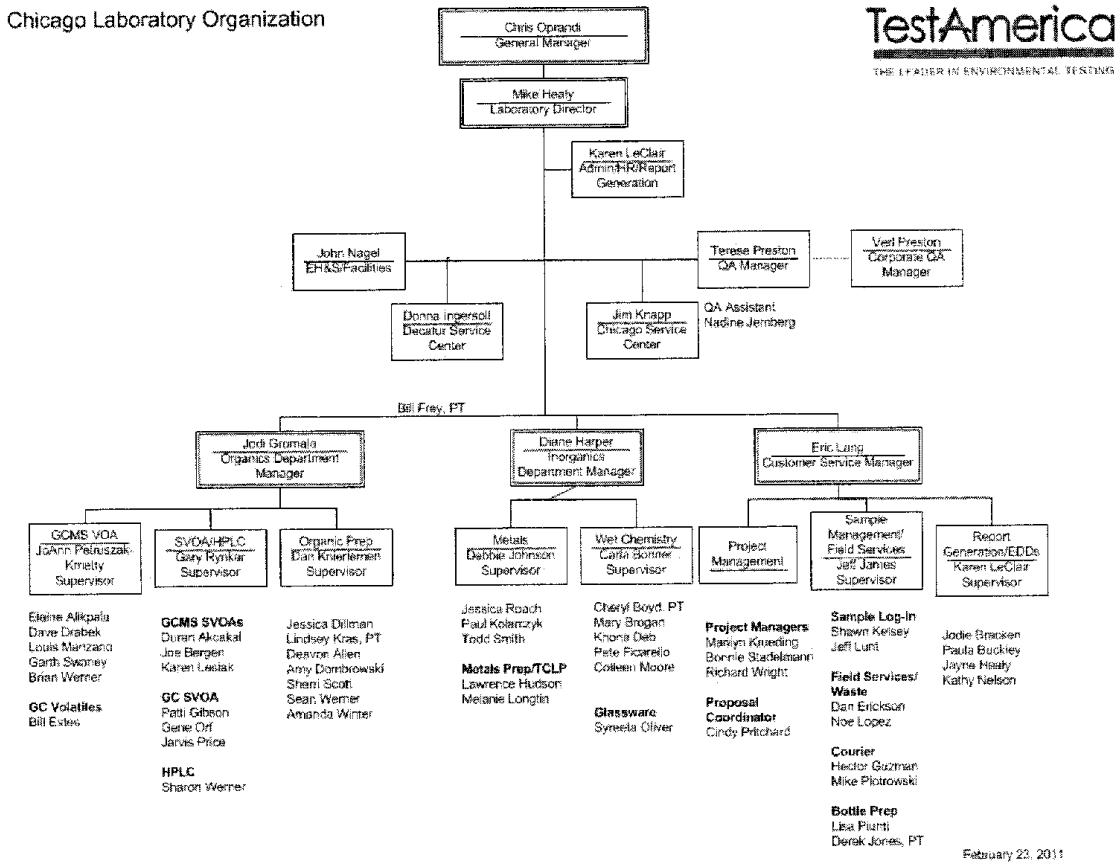


Figure 4-2.

TestAmerica Chicago Organization Chart *(ISO17025 4.1.3; 4.1.5; 4.2.6)*



SECTION 5

QUALITY SYSTEM *(NELAC 5.4.2)*

5.1 QUALITY POLICY STATEMENT *(ISO17025 4.1.5; 4.2.2; 4.2.3)*

It is TestAmerica's Policy to:

- Provide data of known quality to its clients by adhering to approved methodologies, regulatory requirements and the QA/QC protocols.
- Effectively manage all aspects of the laboratory and business operations by the highest ethical standards.
- Continually improve systems and provide support to quality improvement efforts in laboratory, administrative and managerial activities. TestAmerica recognizes that the implementation of a quality assurance program requires management's commitment and support as well as the involvement of the entire staff.
- Provide clients with the highest level of professionalism and the best service practices in the industry.
- The laboratory management staff is committed to comply with International ANS/ISO/IEC Standard 17025:2005 and to continually improve the effectiveness of the management system.

Every staff member at the laboratory plays an integral part in quality assurance and is held responsible and accountable for the quality of their work. It is, therefore, required that all laboratory personnel are trained and agree to comply with applicable procedures and requirements established by this document.

5.2 ETHICS AND DATA INTEGRITY *(ISO17025 4.1.5; 4.2.2)*

TestAmerica is committed to ensuring the integrity of its data and meeting the quality needs of its clients. The elements of TestAmerica's Ethics and Data Integrity Program include:

- An Ethics Policy (Corporate Policy No. CA-L-P-001) and Employee Ethics Statements (Appendix 1).
- Ethics and Compliance Officer (ECOs).
- A Training Program.
- Self-governance through disciplinary action for violations.
- A confidential mechanism for anonymously reporting alleged misconduct and a means for conducting internal investigations of all alleged misconduct. (Corporate SOP No. CA-L-S-001)
- Procedures and guidance for recalling data if necessary (Corporate SOP No. CA-L-S-001).
- Effective external and internal monitoring system that includes procedures for internal audits (Section 16).

- Produce results, which are accurate and include QA/QC information that meets client pre-defined Data Quality Objective (DQO's).
- Present services in a confidential, honest and forthright manner.
- Provide employees with guidelines and an understanding of the Ethical and Quality Standards of our Industry.
- Operate our facilities in a manner that protects the environment and the health and safety of employees and the public.
- Obey all pertinent federal, state and local laws and regulations and encourage other members of our industry to do the same.
- Educate clients as to the extent and kinds of services available.
- Assert competency only for work for which adequate personnel and equipment are available and for which adequate preparation has been made.
- Promote the status of environmental laboratories, their employees, and the value of services rendered by them.

5.3 QUALITY SYSTEM SUPPORTING DOCUMENTATION *(ISO17025 4.2.2; 4.2.5)*

The laboratory's Quality System is communicated through a variety of documents.

- Quality Assurance Manual – Each laboratory has a lab specific quality assurance manual.
- Corporate SOPs and Policies - Corporate SOPs and Policies are developed for use by all relevant laboratories. They are incorporated into the laboratory's normal SOP distribution, training and tracking system. Corporate SOPs may be general or technical.
- Work Instructions - A subset of procedural steps, tasks or forms associated with an operation of a management system (e.g., checklists, preformatted bench sheets, forms).
- Laboratory SOPs – General and Technical
- Corporate Quality Policy Memorandums
- Laboratory QA/QC Policy Memorandums

5.3.1 Order of Precedence *(ISO17025 4.2.5)*

In the event of a conflict or discrepancy between policies, the order of precedence is as follows:

- Corporate Quality Policy Memorandum
- Corporate Quality Management Plan (CQMP)
- Corporate SOPs and Policies
- Laboratory QA/QC Policy Memorandum
- Laboratory Quality Assurance Manual (QAM)
- Laboratory SOPs and Policies
- Other (Work Instructions (WI), memos, flow charts, etc.)

Note: The laboratory has the responsibility and authority to operate in compliance with regulatory requirements of the jurisdiction in which the work is performed. Where the CQMP conflicts with those regulatory requirements, the regulatory requirements of the jurisdiction shall hold primacy. The laboratory's QAM shall take precedence over the CQMP in those cases.

5.4 QA/QC OBJECTIVES FOR THE MEASUREMENT OF DATA *(ISO17025 4.1.5; 4.2.2)*

Quality Assurance (QA) and Quality Control (QC) are activities undertaken to achieve the goal of producing data that accurately characterize the sites or materials that have been sampled. Quality Assurance is generally understood to be more comprehensive than Quality Control. Quality Assurance can be defined as the integrated system of activities that ensures that a product or service meets defined standards.

Quality Control is generally understood to be limited to the analyses of samples and to be synonymous with the term *"analytical quality control"*. QC refers to the routine application of statistically based procedures to evaluate and control the accuracy of results from analytical measurements. The QC program includes procedures for estimating and controlling precision and bias and for determining reporting limits.

Request for Proposals (RFPs) and Quality Assurance Project Plans (QAPP) provide a mechanism for the client and the laboratory to discuss the data quality objectives in order to ensure that analytical services closely correspond to client needs. The client is responsible for developing the QAPP. In order to ensure the ability of the laboratory to meet the Data Quality Objectives (DQOs) specified in the QAPP, clients are advised to allow time for the laboratory to review the QAPP before being finalized. Additionally, the laboratory will provide support to the client for developing the sections of the QAPP that concern laboratory activities.

Historically, laboratories have described their QC objectives in terms of precision, accuracy, representativeness, comparability, completeness, selectivity and sensitivity (PARCCSS).

5.4.1 Precision

The laboratory objective for precision is to meet the performance for precision demonstrated for the methods on similar samples and to meet data quality objectives of the EPA and/or other regulatory programs. Precision is defined as the degree of reproducibility of measurements under a given set of analytical conditions (exclusive of field sampling variability). Precision is documented on the basis of replicate analysis, usually duplicate or matrix spike (MS) duplicate samples. The calculation of precision is described in Section 25.5.3.2.

5.4.2 Accuracy

The laboratory objective for accuracy is to meet the performance for accuracy demonstrated for the methods on similar samples and to meet data quality objectives of the EPA and/or other regulatory programs. Accuracy is defined as the degree of bias in a measurement system. Accuracy may be documented through the use of laboratory control samples (LCS) and/or MS. A statement of accuracy is expressed as an interval of acceptance recovery about the mean recovery. The calculation of accuracy is described in Section 25.4.1.7.

5.4.3 Representativeness

The laboratory objective for representativeness is to provide data which is representative of the sampled medium. Representativeness is defined as the degree to which data represent a characteristic of a population or set of samples and is a measurement of both analytical and field sampling precision. The representativeness of the analytical data is a function of the procedures used in procuring and processing the samples. The representativeness can be documented by the relative percent difference between separately procured, but otherwise identical samples or sample aliquots.

The representativeness of the data from the sampling sites depends on both the sampling procedures and the analytical procedures. The laboratory may provide guidance to the client regarding proper sampling and handling methods in order to assure the integrity of the samples.

5.4.4 Comparability

The comparability objective is to provide analytical data for which the accuracy, precision, representativeness and reporting limit statistics are similar to these quality indicators generated by other laboratories for similar samples, and data generated by the laboratory over time.

The comparability objective is documented by inter-laboratory studies carried out by regulatory agencies or carried out for specific projects or contracts, by comparison of periodically generated statements of accuracy, precision and reporting limits with those of other laboratories.

5.4.5 Completeness

The completeness objective for data is 90% (or as specified by a particular project), expressed as the ratio of the valid data to the total data over the course of the project. Data will be considered valid if they are adequate for their intended use. Data usability will be defined in a QAPP, project scope or regulatory requirement. Data validation is the process for reviewing data to determine its usability and completeness. If the completeness objective is not met, actions will be taken internally and with the data user to improve performance. This may take the form of an audit to evaluate the methodology and procedures as possible sources for the difficulty or may result in a recommendation to use a different method.

5.4.6 Selectivity

Selectivity is defined as: The capability of a test method or instrument to respond to a target substance or constituent in the presence of non-target substances. Target analytes are separated from non-target constituents and subsequently identified/detected through one or more of the following, depending on the analytical method: extractions (separation), digestions (separation), interelement corrections (separation), use of matrix modifiers (separation), specific retention times (separation and identification), confirmations with different columns or detectors (separation and identification), specific wavelengths (identification), specific mass spectra (identification), specific electrodes (separation and identification), etc..

5.4.7 Sensitivity

Sensitivity refers to the amount of analyte necessary to produce a detector response that can be reliably detected (Method Detection Limit or DL) or quantified (Reporting Limit or LOQ).

5.5 CRITERIA FOR QUALITY INDICATORS

The laboratory maintains a Quality Control Limit Summary that contains tables that summarize the precision and accuracy acceptability limits for performed analyses. This summary includes an effective date, is updated each time new limits are generated and is managed by the laboratory's QA department. The summary is located on the LAN in the W:/QC/LIMITS/STATS Directory. The excel spreadsheets enable a historical review of control limits that have been generated and applied to the methods. The current limits are updated in the TALs LIMs system for use in the evaluation of data in LIMs. This information is readily available to project managers at the quote, project and job level. Control limit summaries are available upon request. Unless otherwise noted, limits within these tables are laboratory generated. Some acceptability limits are derived from US EPA methods when they are required. Where US EPA method limits are not required, the laboratory has developed limits from evaluation of data from similar matrices. Criteria for the development of control limits are contained in Section 25.6.

5.6 STATISTICAL QUALITY CONTROL

Statistically-derived precision and accuracy limits are required by selected methods (such as SW-846) and programs [such as the Ohio Voluntary Action Plan (VAP)]. The laboratory routinely utilizes statistically-derived limits to evaluate method performance and determine when corrective action is appropriate. The analysts are instructed to use the current limits in the laboratory (dated and approved by the Department Manager/Supervisor and QA Manager) and entered into the Laboratory Information Management System (LIMS). The Quality Assurance department maintains an archive of all limits used within the laboratory on the LAN in the W:/QC/LIMITS/STAT directory. If a method defines the QC limits, the method limits are used.

If a method requires the generation of historical limits, the lab develops such limits from recent data in the QC database of the LIMS following the guidelines described in Section 25.6. All calculations and limits are documented and dated when approved and effective. On occasion, a client requests contract-specified limits for a specific project.

Surrogate recoveries are determined for a specific time period as defined above. The resulting ranges are entered in LIMS.

Current QC limits are entered and maintained in the LIMS analyte database. As sample results and the related QC are entered into LIMS, the sample QC values are compared with the limits in LIMS to determine if they are within the acceptable range. The analyst then evaluates if the sample needs to be rerun or re-extracted/rerun or if a comment should be added to the report explaining the reason for the QC outlier.

5.6.1 QC Charts

Statistical control limits and control charts are used to establish method performance of a given analysis and to monitor trends of QC results graphically over time. Once a data base of the laboratory results for a method/matrix/QC analyte combination is established, the acceptability of a given analysis of that QC parameter (and of the analytical batch to which it belongs) can be evaluated in light of the laboratory's normal performance. This is intended to help identify problems before they might affect data. Often, patterns of response that are not at all evident in sets of numbers are very distinct when the same values are viewed as a chronological graph.

The charting program in the TALs LIMs system at TestAmerica Chicago automatically applies an outlier test to the queried data and identifies the data points that fail the test. The program also has 'Warning Triggers' for each of the chart types that are set as follows:
"Warn if number of consecutive points outside warning limit" (currently set at 3 points).
"Warn if number of consecutive points on either side of mean" (currently set at 5 points).

Establishment of Limits
The purpose of using statistical control limits is to define, for each analyte in a given method/matrix/QC type combination, a range of expected values. This range encompasses the random variation that occurs normally in the laboratory and allows one to evaluate control samples in that context, rather than according to an arbitrary or external set of values. Limits for accuracy and precision are defined below:

Accuracy
As recoveries of a QC analyte in a given matrix are tabulated over time, a mean value for recovery is established, as is the standard deviation (s) of those recoveries. If the analysis is in statistical control (e.g., if the set of QC recoveries over time show random variation about the mean) approximately 99.7% of all recoveries for that QC will fall within three standard deviations (3s) of the mean. Thus, assuming that the mean itself is an acceptable level of recovery, the values corresponding to 3s above and 3s below the mean are defined as the Control Limits. Any single recovery outside these values is assumed to have resulted from some circumstance other than normal variation and shall be investigated.

Roughly 95% of points should fall within 2s of the mean. The values +2s and -2s are the Warning Limits. Any normal result has approximately a 1/20 chance of being between 2s and 3s from the mean, so a result in this region doesn't necessarily warrant corrective action, but attention should be paid to such points.

Precision
Precision is used to indicate matrix variability so that appropriate decisions can be made by the client when repeated analyses vary significantly. The coefficient of variation, expressed as a percentage (e.g., the %RSD) for the data set used to calculate accuracy control limits defines the control limit for precision. Duplicate analyses of the QC samples, such as duplicates or MS/MSD, should have an RPD less than or equal to this established precision control limit to be considered free of matrix interferences.

The laboratory calculates statistical control limits on an annual basis or more frequently if instrument conditions or method procedure warrants the re-calculation of control limits. The QA Manager and Department Managers/Supervisors are responsible for the review and approval of the limits for use.

5.7 QUALITY SYSTEM METRICS

In addition to the QC parameters discussed above, the entire Quality System is evaluated on a monthly basis through the use of specific metrics (refer to Section 17). These metrics are used to drive continuous improvement in the laboratory's Quality System.

SECTION 6

DOCUMENT CONTROL *(NELAC 5.4.3)*

(ISO17025 4.2.7; 4.3.1; 4.3.2.2; 4.3.3.3; 4.3.3.4)

6.1 OVERVIEW

The QA Department is responsible for the control of documents used in the laboratory to ensure that approved, up-to-date documents are in circulation and out-of-date (obsolete) documents are archived or destroyed. The following documents, at a minimum, must be controlled:

- Laboratory Quality Assurance Manual (QAM)
- Laboratory Standard Operating Procedures (SOP)
- Laboratory Policies
- Work Instructions and Forms
- Corporate Policies and Procedures distributed outside the intranet

The Corporate Quality staff posts Corporate Manuals, SOPs, Policies, Work Instructions, White Papers and Training Materials on the company intranet site. These corporate documents are only considered controlled when they are read on the intranet site. Printed copies are considered uncontrolled unless the laboratory physically distributes them as controlled documents. A detailed description of the procedure for issuing, authorizing, controlling, distributing, and archiving Corporate documents is found in Corporate SOP No. CW-Q-S-001, Corporate Document Control and Archiving and laboratory SOP *UP-QA-006*, Document Control.

The laboratory QA Department also maintains access to various references and document sources integral to the operation of the laboratory. This includes reference methods and regulations. Instrument manuals (hard or electronic copies) are also maintained by the laboratory.

The laboratory maintains control of records for raw analytical data and supporting records such as audit reports and responses, logbooks, standard logs, training files, MDL studies, Proficiency Testing (PT) studies, certifications and related correspondence, and corrective action reports. Raw analytical data consists of bound logbooks, instrument printouts, any other notes, magnetic media, electronic data and final reports. Discussion on records control is described in Section 15.

6.2 DOCUMENT APPROVAL AND ISSUE *(ISO17025 4.3.2.1; 4.3.2.2; 4.3.2.3; 4.3.3.1)*

The pertinent elements of a document control system for each document include a unique document title and number, the number of pages of the item, the effective date, revision number and the laboratory's name. The Quality Assurance personnel are responsible for the maintenance of this system.

Controlled documents are authorized by the QA Department. In order to develop a new document, a department manager or supervisor submits an electronic draft to the QA Department for suggestions and approval before use. Once approved, the document is issued a document control number and is added to the tracking spreadsheet located on the LAN at W:/QC/List/SOP List. Upon approval, QA personnel add the identifying version information to

the document and retain the official document on file. The official document is provided to all applicable operational units (may include electronic access). Controlled documents are identified as such and records of their distribution are kept by the QA Department. Document control may be achieved by either electronic or hardcopy distribution.

The QA Department maintains a list of the official versions of controlled documents.

Quality System Policies and Procedures will be reviewed at a minimum of yearly and revised as appropriate. Changes to documents occur when a procedural change warrants a revision of the document.

6.3 PROCEDURES FOR DOCUMENT CONTROL POLICY *(ISO17025 4.3.2.1; 4.3.2.2; 4.3.3.1)*

For changes to the QA Manual, refer to laboratory SOP *UP-QA-032*, SOP Change Protocol and the Corporate Document control SOP CW-Q-S-001. Uncontrolled copies must not be used within the laboratory. Previous revisions and back-up data are stored by the QA department. Hardcopies are filed within the QA department or in are archived in the secure data storage room and electronic copies are stored on the LAN in the W:/QC/SOP department directories for the applicable revision.

For changes to SOPs, refer to the SOP No. *CW-Q-S-002*, Writing a Standard Operating Procedure SOP and the laboratory SOP UP-QA-032. The SOPs identified above also defines the process of changes to SOPs.

Forms, worksheets, work instructions and information are organized by department in the QA office and tracked on the LAN in the W:/QC/List/SOP List excel spreadsheet. Electronic versions are kept on the LAN in the appropriate W:/QC directory; hard copies are kept in QA files. The procedure for the care of these documents is in the laboratory SOP *UP-QA-006*, Document Control.

6.4 OBSOLETE DOCUMENTS *(ISO17025 4.3.2.1; 4.3.2.2)*

All invalid or obsolete documents are removed, or otherwise prevented from unintended use. The laboratory has specific procedures as described above to accomplish this. In general, obsolete documents are collected from employees according to distribution lists and are marked obsolete on the cover or destroyed. At least one copy of the obsolete document is archived as described in Section 15 and according to SOP UP-DM-002, Data Management: Record Retention & Purging.

SECTION 7

SERVICE TO THE CLIENT (NELAC 5.4.7)

(ISO17025 4.4.1; 4.4.2; 4.4.3; 4.4.4)

7.1 OVERVIEW *(ISO17025 4.4.5; 5.7.1)*

The laboratory has established procedures for the review of work requests and contracts, oral or written. The procedures include evaluation of the laboratory's capability and resources to meet the contract's requirements within the requested time period. All requirements, including the methods to be used, must be adequately defined, documented and understood. For many environmental sampling and analysis programs, testing design is site or program specific and does not necessarily "fit" into a standard laboratory service or product. It is the laboratory's intent to provide both standard and customized environmental laboratory services to our clients.

A thorough review of technical and QC requirements contained in contracts is performed to ensure project success. The appropriateness of requested methods, and the lab's capability to perform them must be established. Projects, proposals and contracts are reviewed for adequately defined requirements and the laboratory's capability to meet those requirements. Alternate test methods that are capable of meeting the clients' requirements may be proposed by the lab. A review of the lab's capability to analyze non-routine analytes is also part of this review process.

All projects, proposals and contracts are reviewed for the client's requirements in terms of compound lists, test methodology requested, sensitivity (detection and reporting levels), accuracy, and precision requirements (% Recovery and RPD). The reviewer ensures that the laboratory's test methods are suitable to achieve these regulatory and client requirements and that the laboratory holds the appropriate certifications and approvals to perform the work. The laboratory and any potential subcontract laboratories must be certified, as required, for all proposed tests.

The laboratory must determine if it has the necessary physical, personnel and information resources to meet the contract, and if the personnel have the expertise needed to perform the testing requested. Each proposal is checked for its impact on the capacity of the laboratory's equipment and personnel. As part of the review, the proposed turnaround time will be checked for feasibility.

Electronic or hard copy deliverable requirements are evaluated against the laboratory's capacity for production of the documentation.

If the laboratory cannot provide all services but intends to subcontract such services, whether to another TestAmerica facility or to an outside firm, this will be documented and discussed with the client prior to contract approval. (Refer to Section 8 for Subcontracting Procedures.)

The laboratory informs the client of the results of the review if it indicates any potential conflict, deficiency, lack of accreditation, or inability of the lab to complete the work satisfactorily. Any discrepancy between the client's requirements and the laboratory's capability to meet those requirements is resolved in writing before acceptance of the contract. It is necessary that the contract be acceptable to both the laboratory and the client. Amendments initiated by the client and/or TestAmerica, are documented in writing.

All contracts, QAPPs, Sampling and Analysis Plans (SAPs), contract amendments, and documented communications become part of the project record.

The same contract review process used for the initial review is repeated when there are amendments to the original contract by the client, and the participating personnel are informed of the changes.

7.2 REVIEW SEQUENCE AND KEY PERSONNEL *(ISO17025 4.4.5)*

Appropriate personnel will review the work request at each stage of evaluation.

For routine projects and other simple tasks, a review by the Project Manager (PM) is considered adequate. The PM confirms that the laboratory has any required certifications, that it can meet the clients' data quality and reporting requirements and that the lab has the capacity to meet the clients turn around needs. It is recommended that, where there is a sales person assigned to the account, an attempt should be made to contact that sales person to inform them of the incoming samples.

For new, complex or large projects, the proposed contract is given to the National Account Director, who will decide which lab will receive the work based on the scope of work and other requirements, including certification, testing methodology, and available capacity to perform the work. The contract review process is outlined in TestAmerica's Corporate SOP No. CA-L-P-002, Contract Compliance Policy.

This review encompasses all facets of the operation. The scope of work is distributed to the appropriate personnel, as needed based on scope of contract, to evaluate all of the requirements shown above (not necessarily in the order below):

- Legal & Contracts Director
- General Manager
- The Laboratory Project Management Director
- The Laboratory Operations Manager
- Laboratory and/or Corporate Technical Directors
- Laboratory and/or Corporate Information Technology Managers/Directors
- Regional and/or National Account representatives
- Laboratory and/or Corporate Quality
- Laboratory and/or Corporate Environmental Health and Safety Managers/Directors
- The Laboratory Director reviews the formal laboratory quote and makes final acceptance for their facility.

The National Account Director, Legal Contracts Director, or local account representative then submits the final proposal to the client.

In the event that one of the above personnel is not available to review the contract, his or her back-up will fulfill the review requirements.

The Legal & Contracts Director maintains copies of all signed contracts. The laboratory's Proposal Coordinator, Cindy Pritchard also maintains a copy.

7.3 DOCUMENTATION

Appropriate records are maintained for every contract or work request. All stages of the contract review process are documented and include records of any significant changes.

The contract will be distributed to and maintained by the appropriate sales/marketing personnel and the Regional Account Manager. A copy of the contract and formal quote will be filed with the laboratory PM, Lab Director or local account representative/proposal coordinator.

Records are maintained of pertinent discussions with a client relating to the client's requirements or the results of the work during the period of execution of the contract. The PM keeps a phone log of conversations with the client.

7.3.1 Project-Specific Quality Planning *(ISO17025 5.7.1)*

Communication of contract specific technical and QC criteria is an essential activity in ensuring the success of site specific testing programs. To achieve this goal, the laboratory assigns a PM to each client. The PM is the first point of contact for the client. It is the PM's responsibility to ensure that project specific technical and QC requirements are effectively evaluated and communicated to the laboratory personnel before and during the project. QA department involvement may be needed to assist in the evaluation of custom QC requirements.

PM's are the primary client contact and they ensure resources are available to meet project requirements. Although PM's do not have direct reports or staff in production, they coordinate opportunities and work with laboratory management and supervisory staff to ensure that resources are sufficient to perform work for the client's project. Project management is positioned between the client and laboratory resources.

Prior to work on a new project, the dissemination of project information and/or project opening meetings may occur to discuss schedules and unique aspects of the project. Items to be discussed may include the project technical profile, turnaround times, holding times, methods, analyte lists, reporting limits, deliverables, sample hazards, or other special requirements. The PM introduces new projects to the laboratory staff through project kick-off meetings or to the supervisory staff during production meetings. These meetings provide direction to the laboratory staff in order to maximize production and client satisfaction, while maintaining quality. In addition, project notes may be associated with each sample batch as a reminder upon sample receipt and analytical processing.

During the project, any change that may occur within an active project is agreed upon between the client/regulatory agency and the PM/laboratory. These changes (e.g., use of a non-standard method or modification of a method) and approvals must be documented prior to implementation. Documentation pertains to any document, e.g., letter, e-mail, variance, contract addendum, which has been signed by both parties.

Such changes are also communicated to the laboratory during production meetings. Such changes are updated to the project notes and are introduced to the managers at these meetings. The laboratory staff is then introduced to the modified requirements via the PM or the individual laboratory Department Manager/Supervisor. After the modification is implemented into the laboratory process, documentation of the modification is retained as an attached document to the project or job in LIMs or as an NCM in the LIMs system which can then be added to the case narrative of the data report(s).

The laboratory strongly encourages client visits to the laboratory and for formal/informal information sharing session with employees in order to effectively communicate ongoing client needs as well as project specific details for customized testing programs.

7.4 SPECIAL SERVICES *(ISO17025 4.7.1; 4.7.2)*

The laboratory cooperates with clients and their representatives to monitor the laboratory's performance in relation to work performed for the client. It is the laboratory's goal to meet all client requirements in addition to statutory and regulatory requirements. The laboratory has procedures to ensure confidentiality to clients (Section 16.2.2 and 26.6)

Note: ISO 17025:2005 / NELAC 2003 states that a laboratory "shall afford clients or their representatives cooperation to clarify the client's request". This topic is discussed in Section 7.

The laboratory's standard procedures for reporting data are described in Section 26. Special services are also available and provided upon request. These services include:

- Reasonable access for our clients or their representatives to the relavant areas of the laboratory for the witnessing of tests performed for the client.
- Assist client specified third party data validators as specified in the client's contract.
- Supplemental information pertaining to the analysis of their samples. Note: An additional charge may apply for additional data/information that was not requested prior to the time of sample analysis or previously agreed upon.

7.5 CLIENT COMMUNICATION *(ISO17025 4.7.1; 4.7.2)*

Project managers are the primary communication link to the clients. They shall inform their clients of any delays in project completion as well as any con-conformances in either sample receipt or sample analysis. Project management will maintain ongoing client communication throughout the entire client project.

Technical directors are available to discuss any technical questions or concerns that the client may have.

7.6 REPORTING *(ISO17025 4.7.1; 4.7.2)*

The laboratory works with our clients to produce any special communication reports required by the contract.

7.7 CLIENT SURVEYS *(ISO17025 4.7.1; 4.7.2)*

The laboratory assesses both positive and negative client feedback. The results are used to improve overall laboratory quality and client services.

TestAmerica's Client Advocacy team has developed a lab/client specific survey to assess client satisfaction. With each report emailed to a client, a link to a Report/Project Feedback Survey is attached with the invitation to the client to provide feedback regarding the service they have received. (Figure 7-1)

Figure 7-1

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

Report/Project Feedback

Please let us know if we met your expectations by rating the service you received from TestAmerica on this project.

Which TestAmerica Laboratory location performed this work?
(Select One from the drop down box)

--Click Here--

If "other" was selected, please specify the location

Please enter the TestAmerica report #

How would you rate the overall performance of this laboratory location?

Provide a score ranging from 1 poor to 10 for excellent

Poor 1 2 3 4 5 6 7 8 9 10 Excellent

Are there any comments that you would like to share?

Please enter your company name

Please enter your name: (optional)

If you would like to be contacted to discuss and issue, please indicate your (phone# or E-mail address

If you need further assistance, please contact your TestAmerica Project Manager. If you would prefer an alternative contact, send an e-mail to **ClientFirst2@TestAmericainc.com** and it will be answered promptly by Tim O'Shields or Ray Frederici our Directors of Client Advocacy.

SECTION 8

SUBCONTRACTING OF TESTS *(NELAC 5.4.5)*

(ISO17025 4.4.3; 4.5.4)

8.1 OVERVIEW *(ISO17025 4.5.1; 4.5.2; 4.5.3; 5.3.1)*

For the purpose of this quality manual, the phrase subcontract laboratory refers to a laboratory external to the TestAmerica laboratories. The phrase "work sharing" refers to internal transfers of samples between the TestAmerica laboratories. The term outsourcing refers to the act of subcontracting tests.

When contracting with our clients, the laboratory makes commitments regarding the services to be performed and the data quality for the results to be generated. When the need arises to outsource testing for our clients because project scope, changes in laboratory capabilities, capacity or unforeseen circumstances, we must be assured that the subcontractors or work sharing laboratories understand the requirements and will meet the same commitments we have made to the client. Refer to TestAmerica's Corporate SOP's on Subcontracting Procedures (CA-L-S-002) and the Work Sharing Process SOP (CA-C-S-001).

When outsourcing analytical services, the laboratory will assure, to the extent necessary, that the subcontract or work sharing laboratory maintains a program consistent with the requirements of this document, the requirements specified in NELAC/ISO 17025 and/or the client's Quality Assurance Project Plan (QAPP). All QC guidelines specific to the client's analytical program are transmitted to the subcontractor and agreed upon before sending the samples to the subcontract facility. Additionally, work requiring accreditation will be placed with an appropriately accredited laboratory. The laboratory performing the subcontracted work will be identified in the final report, as will non-NELAC accredited work where required.

For DOD projects the subcontractor laboratories used must have an established and documented laboratory quality system that complies with DoD QSM requirements. If a lab meeting those criteria is not available, the client must pre-approve use of the subcontract laboratory. The subcontractor laboratories are evaluated following the procedures outlined below and as seen in Figure 8-1. The subcontractor laboratory must receive project-specific approval from the DoD client before any samples are analyzed.

The QSM has 5 specific requirements for subcontracting:

1. Subcontractor laboratories must have an established laboratory quality system that complies with the QSM.
2. Subcontractor laboratories must be approved by the specific DoD Component laboratory approval process.
3. Subcontractor laboratories must demonstrate the ability to generate acceptable results from the analysis of PT samples, subject to availability, using each applicable method, in the specified matrix, and provide appropriate documentation to the DoD client.
4. Subcontractor laboratories must receive project-specific approval from the DoD client before any samples are analyzed.
5. Subcontractor laboratories are subject to project-specific, on-site assessments by the DoD client or their designated representatives.

Project Managers (PMs) or Customer Service Managers (CSM), or Regional Account Executives (RAE) for the Export Lab are responsible for obtaining client approval prior to outsourcing any samples. The laboratory will advise the client of a subcontract or work sharing arrangement in writing and when possible approval from the client shall be retained in the project folder.

Note: In addition to the client, some regulating agencies, such as the US Army Corps of Engineers and the USDA, require notification prior to placing such work.

8.2 QUALIFYING AND MONITORING SUBCONTRACTORS *(ISO17025 4.5.1; 4.5.2; 4.5.3)*

Whenever a PM, Customer Service Manager (CSM) or Regional Account Executives (RAE) becomes aware of a client requirement or laboratory need where samples must be outsourced to another laboratory, the other laboratory(s) shall be selected based on the following:

- The first priority is to attempt to place the work in a qualified TestAmerica laboratory;
- Firms specified by the client for the task (Documentation that a subcontractor was designated by the client must be maintained with the project file. This documentation can be as simple as placing a copy of an e-mail from the client in the project folder);
- Firms listed as pre-qualified and currently under a subcontract with TestAmerica: A listing of all approved subcontracting laboratories and supporting documentation is available on the TestAmerica intranet site. Verify necessary accreditation, where applicable, (e.g., on the subcontractors NELAC, A2LA accreditation or State Certification) for the requested tests prior to sending samples.
- Firms identified in accordance with the company's Small Business Subcontracting program as small, women-owned, veteran-owned and/or minority-owned businesses;
- NELAC or A2LA accredited laboratories.
- In addition, the firm must hold the appropriate certification to perform the work required.

All TestAmerica laboratories are pre-qualified for work sharing provided they hold the appropriate accreditations, can adhere to the project/program requirements, and the client approved sending samples to that laboratory. The client must provide acknowledgement that the samples can be sent to that facility (an e-mail is sufficient documentation or if acknowledgement is verbal, the date, time, and name of person providing acknowledgement must be documented). The originating laboratory is responsible for communicating all technical, quality, and deliverable requirements as well as other contract needs. Refer to Corporate SOP No. CA-C-S-001, Work Sharing Process.

When the potential sub-contract laboratory has not been previously approved, Account Executives or PMs may nominate a laboratory as a subcontractor based on need. The decision to nominate a laboratory must be approved by the Laboratory Director. The Laboratory Director requests that the QA Manager begin the process of approving the subcontract laboratory as outlined in Corporate SOP no. CA-L-S-002, Subcontracting Procedures. The client must provide acknowledgement that the samples can be sent to that facility (an e-mail is sufficient documentation or if acknowledgement is verbal, the date, time, and name of person providing acknowledgement must be documented).

8.2.1 Once the appropriate accreditation and legal information is received by the laboratory, it is evaluated for acceptability (where applicable) and forwarded to Corporate Contracts for formal contracting with the laboratory. They will add the lab to the approved list on the intranet site along with the associate documentation and notify the finance group for JD Edwards.

**USDA permit is required if soils less than three feet deep from New York, North Carolina, South Carolina, Georgia, Florida, Tennessee, Alabama, Mississippi, Louisiana, Arkansas, Texas, Oklahoma, New Mexico, Arizona, California, Hawaii, or outside the continental U. S. are to be analyzed. These samples require special shipping measures; check with the EHS Department. It may be necessary to heat-treat the samples before shipping if the subcontract laboratory does not have a USDA permit; however, some analytes/tests may be irrelevant after heat treatment.

8.2.2 The client will assume responsibility for the quality of the data generated from the use of a subcontractor they have requested the lab to use. The qualified subcontractors on the intranet site are known to meet minimal standards. TestAmerica does not certify laboratories. The subcontractor is on our approved list and can only be recommended to the extent that we would use them.

8.2.3 The status and performance of qualified subcontractors will be monitored periodically by the Corporate Contracts and/or Quality Departments. Any problems identified will be brought to the attention of TestAmerica's Corporate Finance or Corporate Quality personnel.

- Complaints shall be investigated. Documentation of the complaint, investigation and corrective action will be maintained in the subcontractor's file on the intranet site. Complaints must be posted using the Vendor Performance Report (Form No. CW-F-WI-009).
- Information must be updated on the intranet when new information is received from the subcontracted laboratories.
- Subcontractors in good standing will be retained on the intranet listing. The QA Manager will notify all TestAmerica laboratories, Corporate Quality and Corporate Contracts if any laboratory requires removal from the intranet site. This notification will be posted on the intranet site and e-mailed to all Lab Directors, QA Managers and Sales Personnel.

8.3 OVERSIGHT AND REPORTING

The PM must request that the selected subcontractor be presented with a subcontract, if one is not already executed between the laboratory and the subcontractor. The subcontract must include terms which flow down the requirements of our clients, either in the subcontract itself or through the mechanism of work orders relating to individual projects. A standard subcontract and the Lab Subcontractor Vendor Package (posted on the intranet) can be used to accomplish this, and the Legal & Contracts Director can tailor the document or assist with negotiations, if needed. The PM or CSM responsible for the project must advise and obtain client consent to the subcontract as appropriate, and provide the scope of work to ensure that the proper requirements are made a part of the subcontract and are made known to the subcontractor.

Prior to sending samples to the subcontracted laboratory, the PM confirms their certification status to determine if it's current and scope-inclusive. The information is documented on a Subcontracted Sample Form (Workshare Agreement Figure 8-2) and the form is retained in the project folder. For TestAmerica laboratories, certifications can be viewed on the company's TotalAccess Database.

The Sample Control department is responsible for ensuring compliance with QA requirements and applicable shipping regulations when shipping samples to a subcontracted laboratory.

All subcontracted samples must be accompanied by a Chain of Custody (COC). A copy of the original COC sent by the client must be included with all samples subbed within TestAmerica.

Through communication with the subcontracted laboratory, the PM monitors the status of the subcontracted analyses, facilities successful execution of the work, and ensures the timeliness and completeness of the analytical report.

Non-NELAC accredited work must be identified in the subcontractor's report as appropriate. If NELAC accreditation is not required, the report does not need to include this information.

Reports submitted from subcontractor laboratories are not altered and are included in their original form in the final project report. This clearly identifies the data as being produced by a subcontractor facility. If subcontract laboratory data is incorporated into the laboratories EDD (i.e., imported), the report must explicitly indicate which lab produced the data for which methods and samples.

Note: The results submitted by a TestAmerica work sharing laboratory may be transferred electronically and the results reported by the TestAmerica work sharing lab are identified on the final report. The report must explicitly indicate which lab produced the data for which methods and samples. The final report must include a copy of the completed COC for all work sharing reports.

8.4 CONTINGENCY PLANNING

The Laboratory Director may waive the full qualification of a subcontractor process temporarily to meet emergency needs. In the event this provision is utilized, the QA Manager will be required to verify certifications. The comprehensive approval process must then be initiated within 30 calendar days of subcontracting.

Figure 8-1.
Example - Subcontracting Laboratory Approval Form (Initial / Renewal)

SUBCONTRACTING LABORATORY APPROVAL

Date: _____
Laboratory: _____
Address: _____
_____
Contact and e-mail address: _____
Phone: Direct _____ Fax _____

Requested Item[3]	Date Received	Reviewed/ Accepted	Date
1. QA Manual[3]			
2. Copy of State Certification[1]			
3. State Audit with Corrective Action Response (or NELAC or A2LA Audit)[3]			
4. Most Recent (and relevant) 2 Sets of WP/WS Reports with Corrective Action Response[1,3]			
5. SOQ or Summary list of Technical Staff and Qualifications [3]			
6. SOPs for Methods to Be Loadshifted[2,3]			
7. USDA Soil Permit			
8. Insurance Certificate			
9. Sample Report[3]			
10. For DoD Work: Statement that Lab quality system complies with QSM.			
11. For DoD Work: Approved by specific DoD Component laboratory approval process.			
11. Description of Ethics Program[3]			

1 - Required when emergency procedures are implemented.
2 - Some labs may not submit copies due to internal policies. In these cases, a copy of the first page and signature page of the SOP is acceptable. This requirement may also be fulfilled by supplying a table of SOPs with effective dates.
3 – If the laboratory has NELAC accreditation, Item #s 4 through 10 are not required.

On Site Audit Planned: YES NO If yes, Date Completed: _____ By Whom: _____

Comments:

Lab Acceptable for Subcontracting Work: YES NO Limitations: _____

QA Manager: _____ Date: _____
(Printed Name)

☐ Forwarded to Contract Coordinator, by: _____ Date: _____

CHI-22-23-0012/A-02/11

Figure 8-2
Example - Subcontracted Sample Form (Workshare Agreement)

TestAmerica	Work Sharing Agreement (include with every sample shipment)		Exporting Lab / Project Name	TAL Chicago
Import Lab Information			**Export Lab Information**	
Lab Name	TAL Chicago		PM Contact Name	
PM Contact Name			Backup Contact Name	
Backup Contact Name			Agreement Date	
Pricing Information			**Project Information**	
QA/QC (i.e. MS/MSD) Billable?	No		Client Company Name	
Raw Data Surcharge	%		Date First Samples to Arrive	01/06/11
EDD Surcharge	%		Est. Duration of Sampling Event	
TAT Surcharges	%			
Penalty Terms	None			
Other Charges Not In Unit Price? (i.e. canisters, Level IV/III, shipping, bottles)	None		Quote or Contract Reference ID	
Project Details				
Non-Standard Work Product	No			
Quality Assurance Plan	No			
List Certifications Required				
Analyte/Cmpd. List with RLs Attached	No			
Results Dry-Weight Corrected	No			
Special Method Holding Times	None			
Internal Chain of Custody Required	No			
Known Hazards/High Analyte Level	No			
Saturday/Special Delivery Options	None			
Special Instructions	None			
Reporting Limit Value Convention	Report to MDL with "J" Values up to	"Non-Detect" Presentation Convention		U

Deliverable Requirements		Transmittal medium	Format Column	Date Due to Export Lab	Import and Export Lab Agreement
Preliminary Report:	No				*Import lab must acknowledge receipt of Agreement and samples via E-Mail*
Final Report:	No				
EDD:	No		Type Format Name		
Total Access Reporting:	No	NA	NA		
Custom Forms:	No	NA	Type Format Name		
"Other" Deliverable Definitions or Notes:					

Analysis	Method	Matrix	# of Samples	Import Lab's Unit Price	Unit Price w/Surcharges	Extended Price
					$ -	$ -
					$ -	$ -
					$ -	$ -
					$ -	$ -
					$ -	$ -
					$ -	$ -
					$ -	$ -
					$ -	$ -
					$ -	$ -
					$ -	$ -
					Approximate Total Project Value	$ -

Work Instruction No. CA-WI-010/C-01/11

SECTION 9

PURCHASING SERVICES AND SUPPLIES *(NELAC 5.4.6)*
(ISO17025 4.6.1)

9.1 OVERVIEW *(ISO17025 4.6.2; 4.6.3; 4.6.4)*

Evaluation and selection of suppliers and vendors is performed, in part, on the basis of the quality of their products, their ability to meet the demand for their products on a continuous and short term basis, the overall quality of their services, their past history, and competitive pricing. This is achieved through evaluation of objective evidence of quality furnished by the supplier, which can include certificates of analysis, recommendations, and proof of historical compliance with similar programs for other clients. To ensure that quality critical consumables and equipment conform to specified requirements, which may affect quality, all purchases from specific vendors are approved by a member of the supervisory or management staff. Capital expenditures are made in accordance with the TestAmerica's Corporate Controlled Purchases Procedure, SOP No. CW-F-S-007.

Contracts will be signed in accordance with TestAmerica's Corporate Authorization Matrix Policy, Policy No. CW-F-P-002. Request for Proposals (RFP's) will be issued where more information is required from the potential vendors than just price. Process details are available in TestAmerica's Corporate Procurement and Contracts (Policy No. CW-F-P-004). RFP's allow TestAmerica to determine if a vendor is capable of meeting requirements such as supplying all of the TestAmerica facilities, meeting required quality standards and adhering to necessary ethical and environmental standards. The RFP process also allows potential vendors to outline any additional capabilities they may offer.

9.2 GLASSWARE

Glassware used for volumetric measurements must be Class A or verified for accuracy according to laboratory procedure. Pyrex (or equivalent) glass should be used where possible. For safety purposes, thick-wall glassware should be used where available.

9.3 REAGENTS, STANDARDS & SUPPLIES *(ISO17025 4.6.2; 4.6.3; 4.6.4)*

Purchasing guidelines for equipment and reagents must meet with the requirements of the specific method and testing procedures for which they are being purchased. Solvents and acids are pre-tested in accordance with TestAmerica's Corporate SOP on Solvent & Acid Lot Testing & Approval, SOP No. CA-Q-S-001.

9.3.1 Purchasing

Chemical reagents, solvents, glassware, and general supplies are ordered as needed to maintain sufficient quantities on hand. Materials used in the analytical process must be of a known quality. The wide variety of materials and reagents available makes it advisable to specify recommendations for the name, brand, and grade of materials to be used in any determination. This information is contained in the method SOP. The analyst should completes the Material Request Sheet (Figure 9-1) when requesting reagents, standards, or supplies. The analyst may check the item out of the on-site consignment system that contains items approved for laboratory use.

The analyst must provide the item number, item description, package size, catalogue page number, and the quantity needed. If an item being ordered is not the exact item requested, approval must be obtained from the department manager or supervisor prior to placing the order. The laboratory's purchasing manager (Karen LeClair) or designee enters the order into the JDE system. The order is approved by the LD and the Corporate Purchasing Department places the order with the vendor.

9.3.2 Receiving

It is the responsibility of the facility manager and sample login staff or designee to receive the shipment and document date received. It is the responsibility of the analyst who ordered the materials to date the material when opened for use and inspect the item. Once the ordered reagents or materials are received, the analyst compares the information on the label or packaging to the original order to ensure that the purchase meets the quality level specified. Material Safety Data Sheets (MSDSs) are available in the facility/safety manager's office on file and online through the Company's intranet website. Anyone may review these for relevant information on the safe handling and emergency precautions of on-site chemicals.

9.3.3 Specifications

All methods in use in the laboratory specify the grade of reagent that must be used in the procedure. If the quality of the reagent is not specified, it may be assumed that it is not significant in that procedure and, therefore, any grade reagent may be used. It is the responsibility of the analyst to check the procedure carefully for the suitability of grade of reagent.

Chemicals must not be used past the manufacturer's expiration date and must not be used past the expiration time noted in a method SOP. If expiration dates are not provided, the laboratory may contact the manufacturer to determine an expiration date.

The laboratory assumes a five year expiration date on inorganic dry chemicals unless noted otherwise by the manufacturer or by the reference source method. Chemicals should not be used past the manufacturer's or SOP expiration date unless 'verified' (refer to item 3 listed below).

- An expiration date can not be extended if the dry chemical is discolored or appears otherwise physically degraded. The dry chemical must then be discarded.
- Expiration dates can be extended if the dry chemical is found to be satisfactory based on acceptable performance of quality control samples (Continuing Calibration Verification (CCV), Blanks, Laboratory Control Sample (LCS), etc.).
- If the dry chemical is used for the preparation of standards, the expiration dates can be extended 6 months if the dry chemical is compared to an unexpired independent source in performing the method and the performance of the dry chemical is found to be satisfactory. The comparison must show that the dry chemical meets CCV limits. The comparison studies are maintained within the department where the study was performed, documented in the data book of the applicable method that was performed.

Wherever possible, standards must be traceable to national or international standards of measurement or to national or international reference materials. Records to that effect are available to the user.

Compressed gases in use are checked for pressure and secure positioning daily. The minimum total pressure must be 500 psig or the tank must be replaced. The quality of the gases must meet method or manufacturer specification or be of a grade that does not cause any analytical interference.

Water used in the preparation of standards or reagents must have a specific conductivity of less than 1- umho/cm (or specific resistivity of greater than 1.0 megaohm-cm) at 25°C. The specific conductivity is checked and recorded daily. If the water's specific conductivity is less than the specified limit, the Facility Manager and appropriate Department Managers/Supervisors must be notified immediately in order to notify all departments, decide on cessation (based on intended use) of activities, and make arrangements for correction.

The laboratory may purchase reagent grade (or other similar quality) water for use in the laboratory. This water must be certified "clean" by the supplier for all target analytes or otherwise verified by the laboratory prior to use. This verification is documented.

Standard lots are verified before first time use if the laboratory switches manufacturers or has historically had a problem with the type of standard.

Purchased VOA vials must be certified clean and the certificates must be maintained. If uncertified VOA vials are purchased, all lots must be verified clean prior to use. This verification must be maintained.

Records of manufacturer's certification and traceability statements are maintained in files or binders in each laboratory section. These records include date of receipt, lot number (when applicable), and expiration date (when applicable). Incorporation of the item into the record indicates that the analyst has compared the new certificate with the previous one for the same purpose and that no difference is noted, unless approved and so documented by the Technical Director or QA Manager.

9.3.4 Storage

Reagent and chemical storage is important from the aspects of both integrity and safety. Light-sensitive reagents may be stored in brown-glass containers. Storage conditions are per the Corporate Environmental Health & Safety Manual (Corp. Doc. No. CW-E-M-001) and method SOPs or manufacturer instructions. Table 9-1 details specific storage instructions for reagents and chemicals. Section 22 discusses conditions for standard storage.

9.4 PURCHASE OF EQUIPMENT/INSTRUMENTS/SOFTWARE

When a new piece of equipment is needed, either for additional capacity or for replacing inoperable equipment, the analyst or supervisor makes a supply request to the Technical Director and/or the Laboratory Director. If they agree with the request the procedures outlined in TestAmerica's Corporate Policy No. CA-T-P-001, Qualified Products List are followed. A decision is made as to which piece of equipment can best satisfy the requirements. The

appropriate written requests are completed and purchasing places the order. The quote and justification are forwarded to the controller to start the capex process.

Upon receipt of a new or used piece of equipment, it is given an equipment tag number and is assigned a unique instrument ID such as ICPMS2 that will be used to identify the equipment in logbooks and in LIMs. The equipment list described in Section 21 that is maintained by the QA Department will be updated and IT will be notified so that can be set up in LIMs for data import (if applicable) and to synchronize the instrument for back-ups. Its capability is assessed to determine if it is adequate or not for the specific application. For instruments, a calibration curve is generated, followed by MDLs, Demonstration of Capabilities (DOCs), and other relevant criteria (refer to Section 20). For software, its operation must be deemed reliable and evidence of instrument verification must be retained by the IT Department or QA Department as specified in the laboratory's procedure for software verification. Software certificates supplied by the vendors are filed with the LIMS Administrator. The manufacturer's operation manual is retained at the bench with the instrument.

9.5 SERVICES

Service to analytical instruments (except analytical balances) is performed on an as needed basis. Routine preventative maintenance is discussed in Section 21. The need for service is determined by analysts and/or Department Managers. The service providers that perform the services are approved by the Department Managers/Lab Director.

9.6 SUPPLIERS

TestAmerica selects vendors through a competitive proposal / bid process, strategic business alliances or negotiated vendor partnerships (contracts). This process is defined in the Corporate Finance documents on Vendor Selection (SOP No. CW-F-S-018) and Procurement & Contracts Policy (Policy No. CW-F-P-004). The level of control used in the selection process is dependent on the anticipated spending amount and the potential impact on TestAmerica business. Vendors that provide test and measuring equipment, solvents, standards, certified containers, instrument related service contracts or subcontract laboratory services shall be subject to more rigorous controls than vendors that provide off-the-shelf items of defined quality that meet the end use requirements. The JD Edwards purchasing system includes all suppliers/vendors that have been approved for use.

Evaluation of suppliers is accomplished by ensuring the supplier ships the product or material ordered and that the material is of the appropriate quality. This is documented by signing off on packing slips or other supply receipt documents. The purchasing documents contain the data that adequately describe the services and supplies ordered.

Any issues of vendor performance are to be reported immediately by the laboratory staff to the Corporate Purchasing Group by completing a Vendor Performance Report (CW-F-WI-009).

The Corporate Purchasing Group will work through the appropriate channels to gather the information required to clearly identify the problem and will contact the vendor to report the problem and to make any necessary arrangements for exchange, return authorization, credit, etc.

As deemed appropriate, the Vendor Performance Reports will be summarized and reviewed to determine corrective action necessary, or service improvements required by vendors

The laboratory has access to a listing of all approved suppliers of critical consumables, supplies and services. This information is provided through the JD Edwards purchasing system.

9.6.1 New Vendor Procedure

TestAmerica employees who wish to request the addition of a new vendor must complete a J.D. Edwards Vendor Add Request Form (CW-F-WI-007 – refer to Figure 9-2).

New vendors are evaluated based upon criteria appropriate to the products or services provided as well as their ability to provide those products and services at a competitive cost. Vendors are also evaluated to determine if there are ethical reasons or potential conflicts of interest with TestAmerica employees that would make it prohibitive to do business with them as well as their financial stability. The QA Department, Technical Director and/or the purchasing manager are consulted with vendor and product selection that have an impact on quality.

Figure 9-1.
Materials Request Sheet

PURCHASE REQUISITION
TestAmerica CHICAGO

Date of Order: _____

SHIP TO:
TestAmerica Chicago
2417 Bond Street
University Park, IL 60484

Purchase Order # _____

Purchase Req. # _____

VENDOR: _____
_____
_____

Vendor Phone: _____
Contact: _____

QUANTITY	UOM	PART NUMBER	DESCRIPTION	UNIT PRICE	TOTAL	ACCOUNT #

SUBTOTAL: _____
TAX: _____
SHIP: _____
TOTAL: _____

DATE REQUIRED: _____
REQUISITIONED BY: _____
SUPERVISOR: _____

Table 9-1.

Storage of Reagents and Chemicals

Chemical	Storage Requirements
Concentrated Acids and Bases	Stored in the original containers at room temperature. All organic acids must be stored separately from inorganic acids. Acids should not be stored with bases.
Bulk Dry Chemicals	Stored in the original containers at room temperature. All organic acids must be stored separately from inorganic acids. Acids should not be stored with bases.
Working Solutions containing Organic Compounds	Stored as per method recommendation/ requirement. They are generally stored refrigerated at 4°C± 2°C (greater than freezing and < 6°C).
Working Solutions containing only Inorganics	Stored at room temperature; refrigeration is optional.
Flammable Solvents	Stored in solvent cabinets at room temperature.
Non-Flammable Solvents	Stored separately from the flammable solvents in cabinets at room temperature.

Note: Exceptions to the above detailed storage requirements may be made.

Figure 9-2
Example – JD Edwards Vendor Add Request Form

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

JD EDWARDS VENDOR ADD REQUEST FORM

Instructions: Please complete form electronically if possible. Use the "TAB" key to advance to the next field.
Email to NCPurchasing@testamericainc.com

All sections to be completed by the Requestor

Requestor and Facility:			
Vendor Business Name:			
Vendor Address: (Remit To)			
City, State, Zip			
Vendor Phone:		Vendor Fax:	
Contact Name:		Product / Service Provided:	
Vendor E-mail Address:			

Reason for Vendor Addition: Check all reasons that apply

☐	Cost Reduction	Estimated Annual Savings $	
☐	Replace Current Vendor	Vendor Being Replaced:	
	Reason Being Replaced?		
☐	New Product or Service	Describe:	
☐	ISO Approved (Required for EMLab Only)		

Small Business:

Does this vendor help us to meet our small business objectives?	☐	YES	☐ NO

If yes, check all that apply:

☐	**HUB Zone Business**	☐	**Small Disadvantaged Business**	☐	**Woman Owned Business**
☐	**Veteran Owned Business**	☐	**Disabled Veteran Owned Business**	☐	**Small Woman Owned Business**

Personal and Ethical Considerations:

Is there any personal conflict of interest with a TestAmerica employee and the vendor listed above?	☐	YES	☐	NO
Have ethical considerations been taken into account in your evaluation of this vendor?	☐	YES	☐	NO

Can this product be sourced from another TestAmerica facility?	☐	YES	☐	NO

I approve the addition of this vendor:

Patrick Eckman – Director of Procurement

Leslie Bowers – Corporate Controller

Form No. CW-F-WI-007

SECTION 10

SERVICE TO THE CLIENT *(NELAC 5.4.7)*

10.1 OVERVIEW

Refer to Sections 7.4 through 7.7 for details regading client services.

SECTION 11

COMPLAINTS *(NELAC 5.4.8)*
(ISO17025 4.8)

11.1 OVERVIEW

The laboratory considers an effective client complaint handling process to be of significant business and strategic value. Listening to and documenting client concerns captures 'client knowledge' that enables our operations to continually improve processes and client satisfaction. An effective client complaint handling process also provides assurance to the data user that the laboratory will stand behind its data, service obligations and products.

A client complaint is any expression of dissatisfaction with any aspect of our business services, (e.g., communications, responsiveness, data, reports, invoicing and other functions) expressed by any party, whether received verbally or in written form. Client inquiries, complaints or noted discrepancies are documented, communicated to management, and addressed promptly and thoroughly.

The laboratory has procedures for addressing with both external and internal complaints with the goal of providing satisfactory resolution to complaints in a timely and professional manner.

The nature of the complaint is identified, documented and investigated, and an appropriate action is determined and taken. In cases where a client complaint indicates that an established policy or procedure was not followed, the QA Department must evaluate whether a special audit must be conducted to assist in resolving the issue. A written confirmation or letter to the client, outlining the issue and response taken is recommended as part of the overall action taken.

The process of complaint resolution and documentation utilizes the procedures outlined in Section 13 (Corrective Actions) and is documented following the *TestAmerica Chicago – Complaint Handling Process* work instruction (CHI-22-23-002). It is the laboratory's goal to provide a satisfactory resolution to complaints in a timely and professional manner.

11.2 EXTERNAL COMPLAINTS

An employee that receives a complaint initiates the complaint resolution process by first documenting the complaint according to the *TestAmerica Chicago – Complaint Handling Process* work instruction (CHI-22-23-002).

Complaints fall into two categories: correctable and non-correctable. An example of a correctable complaint would be one where a report re-issue would resolve the complaint. An example of a non-correctable complaint would be one where a client complains that their data was repeatedly late. Non-correctable complaints should be reviewed for preventive action measures to reduce the likelyhood of future occurrence and mitigation of client impact.

The general steps in the complaint handling process are:

- Receiving and Documenting Complaints
- Complaint Investigation and Service Recovery
- Process Improvement

The laboratory shall inform the initiator of the complaint of the results of the investigation and the corrective action taken, if any.

11.3 INTERNAL COMPLAINTS

Internal complaints include, but are not limited to: errors and non-conformances, training issues, internal audit findings, and deviations from methods. Corrective actions may be initiated by any staff member who observes a nonconformance and shall follow the procedures outlined in Section 13. In addition, Corporate Management, Sales and Marketing and Information Technology (IT) may initiate a complaint by contacting the laboratory or through the corrective action system described in Section 13.

11.4 MANAGEMENT REVIEW

The number and nature of client complaints is reported by the QA Manager to the laboratory and QA Director in the QA Monthly report. Monitoring and addressing the overall level and nature of client complaints and the effectiveness of the solutions is part of the Annual Management Review (Section 17)

SECTION 12

CONTROL OF NON-CONFORMING WORK *(NELAC 5.4.9)*

(ISO17025 4.9.1; 5.10.5)

12.1 OVERVIEW *(ISO17025 4.9.1; 4.11.3; 4.11.5)*

When data discrepancies are discovered or deviations and departures from laboratory SOPs, policies and/or client requests have occurred, corrective action is taken immediately. First, the laboratory evaluates the significance of the nonconforming work. Then, a corrective action plan is initiated based on the outcome of the evaluation. If it is determined that the nonconforming work is an isolated incident, the plan could be as simple as adding a qualifier to the final results and/or making a notation in the case narrative. If it is determined that the nonconforming work is a systematic or improper practices issue, the corrective action plan could include a more in depth investigation and a possible suspension of an analytical method. In all cases, the actions taken are documented using the laboratory's corrective action system (refer to Section 13).

Due to the frequently unique nature of environmental samples, sometimes departures from documented policies and procedures are needed. When an analyst encounters such a situation, the problem is presented to the supervisor for resolution. The supervisor may elect to discuss it with the Project Manager, QA Manager or have a representative contact the client to decide on a logical course of action. Once an approach is agreed upon, the analyst documents it using the laboratories corrective action system described in Section 13. This information can then be supplied to the client in the case narrative with the report.

Project Management may encounter situations where a client may request that a special procedure be applied to a sample that is not standard lab practice. Based on a technical evaluation, the lab may accept or opt to reject the request based on technical or ethical merit. An example might be the need to report a compound that the lab does not normally report. The lab would not have validated the method for this compound following the procedures in Section 20. The client may request that the compound be reported based only on the calibration. Such a request would need to be approved by the Department Manager/Supervisor and QA Manager. Documentation detailing the method deviation must be included in the project folder. Deviations **must** also be noted on the final report with a statement that the compound is not reported in compliance with NELAC (or the analytical method) requirements and the reason. (The laboratory will use a Non-Conformance Memo (NCM) in LIMs which will be designated to print on the final report in the case narrative). Data being reported to a non-NELAC state would need to note the change made to how the method is normally run.

12.2 RESPONSIBILITIES AND AUTHORITIES *(ISO17025 4.9.1; 4.11.3; 4.11.5)*

TestAmerica's Corporate SOP entitled *Internal Investigation of Potential Data Discrepancies and Determination for Data Recall* (SOP No. CA-L-S-001) outlines the general procedures for the reporting and investigation of data discrepancies and alleged incidents of misconduct or violations of TestAmerica's data integrity policies as well as the policies and procedures related to the determination of the potential need to recall data.

Under certain circumstances the Laboratory Director, a Department Manager/Supervisor, or a member of the QA team may exceptionally authorize departures from documented procedures

or policies. The departures may be a result of procedural changes due to the nature of the sample; a one-time procedure for a client; QC failures with insufficient sample to reanalyze, etc. In most cases, the client will be informed of the departure prior to the reporting of the data. Any departures must be well documented using the laboratory's corrective action procedures described in Section 13. This information may also need to be documented in logbooks and/or data review checklists and on a Non-Conformance Memo (NCM) in TALs LIMs as appropriate. Any impacted data must be referenced in a case narrative and/or flagged with an appropriate data qualifier.

Any misrepresentation or possible misrepresentation of analytical data discovered by any laboratory staff member must be reported to facility Senior Management within 24-hours. The Senior Management staff is comprised of the Laboratory Director, the QA Manager, the Department Managers and Department Supervisors. The reporting of issues involving alleged violations of the company's Data Integrity or Manual Integration procedures must be conveyed to an Ethics and Compliance Officer (ECO), Director of Quality & Client Advocacy and the laboratory's Quality Director within 24 hours of discovery.

Whether an inaccurate result was reported due to calculation or quantitation errors, data entry errors, improper practices, or failure to follow SOPs, the data must be evaluated to determine the possible effect.

The Laboratory Director, QA Manager, ECOs, Corporate Quality, the COO, General Managers and the Quality Directors have the authority and responsibility to halt work, withhold final reports, or suspend an analysis for due cause as well as authorize the resumption of work.

12.3 EVALUATION OF SIGNIFICANCE AND ACTIONS TAKEN *(ISO17025 4.9.1; 4.11.3; 4.11.5)*

For each nonconforming issue reported, an evaluation of its significance and the level of management involvement needed is made. This includes reviewing its impact on the final data, whether or not it is an isolated or systematic issue, and how it relates to any special client requirements.

TestAmerica's Corporate Data Investigation and Recall Procedure (SOP No. CA-L-S-001) distinguishes between situations when it would be appropriate for laboratory management to make the decision on the need for client notification (written or verbal) and data recall (report revision) and when the decision must be made with the assistance of the ECO's and Corporate Management. Laboratory level decisions are documented and approved using the laboratory's standard nonconformance/corrective action reporting (Section 13) in lieu of the data recall determination form contained in TestAmerica's Corporate SOP No. CA-L-S-001.

12.4 PREVENTION OF NONCONFORMING WORK *(ISO17025 4.9.2; 4.11.2)*

If it is determined that the nonconforming work could recur, further corrective actions must be made following the laboratory's corrective action system (Section 13). On a monthly basis, the QA Department evaluates non-conformances to determine if any nonconforming work has been repeated multiple times. If so, the laboratory's corrective action process may be followed.

12.5 METHOD SUSPENSION/RESTRICTION (STOP WORK PROCEDURES)

(ISO17025 4.9.1; 4.9.2; 4.11.5)

In some cases it may be necessary to suspend/restrict the use of a method or target compound which constitutes significant risk and/or liability to the laboratory. Suspension/restriction procedures can be initiated by any of the persons noted in Section 12.2, Paragraph 5 above.

Prior to suspension/restriction, confidentiality will be respected, and the problem with the required corrective and preventive action will be stated in writing and presented to the Laboratory Director.

The Laboratory Director shall arrange for the appropriate personnel to meet with the QA Manager as needed. This meeting shall be held to confirm that there is a problem, that suspension/restriction of the method is required and will be concluded with a discussion of the steps necessary to bring the method/target or test fully back on line. In some cases that may not be necessary if all appropriate personnel have already agreed there is a problem and there is agreement on the steps needed to bring the method, target or test fully back on line.

The QA Manager will also initiate a corrective action report as described in Section 13 if one has not already been started. A copy of any meeting notes and agreed upon steps should be faxed or e-mailed by the laboratory to the appropriate General Manager and member of Corporate QA. This fax/e-mail acts as notification of the incident.

After suspension/restriction, the lab will hold all reports to clients pending review. No faxing, mailing or distributing through electronic means may occur. The report must not be posted for viewing on the internet. It is the responsibility of the Laboratory Director to hold all reporting and to notify all relevant laboratory personnel regarding the suspension/restriction (i.e., Project Management, Log-in, etc...). Clients will NOT generally be notified at this time. Analysis may proceed in some instances depending on the non-conformance issue.

Within 72 hours, the QA Manager will determine if compliance is now met and reports can be released, OR determine the plan of action to bring work into compliance, and release work. A team, with all principals involved (Laboratory Director, QA Manager, Department Manager, Department Supervisor) can devise a start-up plan to cover all steps from client notification through compliance and release of reports. Project Management and the Director of Client Services and Sales and Marketing **must** be notified if clients must be notified or if the suspension/restriction affects the laboratory's ability to accept work. The QA Manager must approve start-up or elimination of any restrictions after all corrective action is complete. This approval is given by final signature on the completed corrective action report as described in Section 13.

SECTION 13

CORRECTIVE ACTION *(NELAC 5.4.10)*

13.1 OVERVIEW *(ISO17025 4.9.2; 4.11.1; 4.11.2)*

A major component of TestAmerica's Quality Assurance (QA) Program is the problem investigation and feedback mechanism designed to keep the laboratory staff informed on quality related issues and to provide insight to problem resolution. When nonconforming work or departures from policies and procedures in the quality system or technical operations are identified, the corrective action procedure provides a systematic approach to assess the issues, restore the laboratory's system integrity, and prevent reoccurrence. Corrective actions are documented using Non-Conformance Memos (NCMs) (refer to Figure 13-1).

13.2 GENERAL *(ISO17025 4.11.2; 4.11.3)*

Problems within the quality system or within analytical operations may be discovered in a variety of ways, such as QC sample failures, internal or external audits, proficiency testing (PT) performance, client complaints, staff observation, etc.

The purpose of a corrective action system is to:

- Identify non-conformance events and assign responsibility(s) for investigating.
- Resolve non-conformance events and assign responsibility for any required corrective action.
- Identify Systematic Problems before they become serious.
- Identify and track Client complaints and provide resolution (see more on client complaints in Section 11).

13.2.1 Non-Conformance Memo (NCM) - is used to document the following types of corrective actions:

- Deviations from an established procedure or SOP
- QC outside of limits (non matrix related)
- Missed sample Holding Times
- Isolated Reporting / Calculation Errors
- Client report based complaints and corrections.
- Discrepancies in materials / good received vs. manufacturer packing slips.

13.2.2 Corrective Action Report (CAR) – is used to document the following types of corrective actions:

- Questionable trends that are found in the monthly review of NCMs.
- Issues found while reviewing NCMs that warrant further investigation.
- Internal and external audit findings.
- Failed or unacceptable PT results.
- Corrective actions that cross multiple departments in the laboratory.
- Systematic Reporting / Calculation Errors

13.3 CLOSED LOOP CORRECTIVE ACTION PROCESS *(ISO17025 4.11.2; 4.11.3; 4.12.2)*

Any employee in the company can initiate a corrective action. There are four main components to a closed-loop corrective action process once an issue has been identified: Cause Analysis, Selection and Implementation of Corrective Actions (both short and long term), Monitoring of the Corrective Actions, and Follow-up.

13.3.1 Cause Analysis

- Upon discovery of a non-conformance event, the event must be defined and documented. An NCM or corrective action report must be initiated, someone is assigned to investigate the issue and the event is investigated for cause. Table 13-1, Appendix 3 (Default) and Appendix 8 (DoD QSM 4.2) tables provide some general guidelines on determining responsibility for assessment.
- The cause analysis step is the key to the process as a long term corrective action cannot be determined until the cause is determined.
- If the cause is not readily obvious, the Supervisor, Lab Director, Project Manager or QA Manager (or QA designee) is consulted.

13.3.2 Selection and Implementation of Corrective Actions

- Where corrective action is needed, the laboratory shall identify potential corrective actions. The action(s) most likely to eliminate the problem and prevent recurrence are selected and implemented. Responsibility for implementation is assigned.
- Corrective actions shall be to a degree appropriate to the magnitude of the problem identified through the cause analysis.
- Whatever corrective action is determined to be appropriate, the laboratory shall document and implement the changes. The NCM is used for this documentation.

13.3.3 Root Cause Analysis

Root Cause Analysis is a class of problem solving (investigative) methods aimed at identifying the basic or causal factor(s) that underlie variation in performance or the occurrence of a significant failure. The root cause may be buried under seemingly innocuous events, many steps preceding the perceived failure. At first glance, the immediate response is typically directed at a symptom and not the cause. Typically, root cause analysis would be best with three or more incidents to triangulate a weakness.

Systematically analyze and document the Root Causes of the more significant problems that are reported. Identify, track, and implement the corrective actions required to reduce the likelihood of recurrence of significant incidents. Trend the Root Cause data from these incidents to identify Root Causes that, when corrected, can lead to dramatic improvements in performance by eliminating entire classes of problems.

Identify the one event associated with problem and ask why this event occurred. Brainstorm the root causes of failures by asking why events occurred or conditions existed; and then why the cause occurred 5 consecutive times until you get to the root cause. For each of these sub events or causes, ask why it occurred. Repeat the process for the other events associated with the incident.

Root cause analysis does not mean the investigation is over. Look at technique, or other systems outside the normal indicators. Often creative thinking will find root causes that ordinarily would be missed, and continue to plague the laboratory or operation.

13.3.4 Monitoring of the Corrective Actions *(ISO17025 4.11.4)*

- The Department Manager/Supervisor and QA Manager are responsible to ensure that the corrective action taken was effective.
- Ineffective actions are documented and re-evaluated until acceptable resolution is achieved. Department Managers are accountable to the Laboratory Director to ensure final acceptable resolution is achieved and documented appropriately.
- Each NCM is entered into LIMs for tracking purposes and a monthly summary of all corrective actions is printed out for review to aid in ensuring that the corrective actions have taken effect.
- The QA Manager reviews monthly NCMs for trends. Highlights are included in the QA monthly report (refer to Section 17). If a significant trend develops that adversely affects quality, an audit of the area is performed and corrective action implemented.
- Any out-of-control situations that are not addressed acceptably at the laboratory level may be reported to the Corporate Quality Director by the QA Manager, indicating the nature of the out-of-control situation and problems encountered in solving the situation.

13.3.5.1 Follow-up Audits *(ISO17025 4.11.4)*

- Follow-up audits may be initiated by the QA Manager and shall be performed as soon as possible when the identification of a nonconformance casts doubt on the laboratory's compliance with its own policies and procedures, or on its compliance with state or federal requirements. (Section 16 includes additional information regarding internal audit procedures.)
- These audits often follow the implementation of the corrective actions to verify effectiveness. An additional audit would only be necessary when a critical issue or risk to business is discovered.

(Also refer to Section 16.1.4, Special Audits.)

13.4 TECHNICAL CORRECTIVE ACTIONS

In addition to providing acceptance criteria and specific protocols for technical corrective actions in the method specific SOPs, Appendix 3 (Default), and in Appendix 8 (DoD QSM 4.2), the laboratory has general procedures to be followed to determine when departures from the documented policies and procedures and quality control have occurred (refer to Section 12 for information regarding the control of non-conforming work). The documentation of these procedures is through the use of an NCM.

Table 13-1 includes examples of general technical corrective actions. For specific criteria and corrective actions refer to the analytical methods or specific method SOPs. Appendix 3 of this QAM also summarizes QC requirements and corrective actions for the methods run in the lab.

Table 13-1 provides some general guidelines for identifying the individual(s) responsible for assessing each QC type and initiating corrective action. The table also provides general guidance on how a data set should be treated if associated QC measurements are unacceptable. Specific procedures are included in Method specific SOPs, Work Instructions, Appendix 3 (Default) and Appendix 8 (DoD QSM 4.2) of this QAM. QAM Sections 20 and 21, and SOP CA-L-S-001 (Internal Investigation of Potential Data Discrepancies and Determination for Data Recall). All corrective actions are reviewed at a minimum monthly by the QA Manager and highlights are included in the QA monthly report.

To the extent possible, samples shall be reported only if all quality control measures are acceptable. If the deficiency does not impair the usability of the results, data will be reported with an appropriate data qualifier and/or the deficiency will be noted in the case narrative. Where sample results may be impaired, the Project Manager is notified by a written NCM and appropriate corrective action (e.g., reanalysis) is taken and documented.

13.5 BASIC CORRECTIONS *(ISO17025 4.11.1; 4.13.2.3)*

When mistakes occur in records, each mistake shall be crossed-out, [not obliterated (e.g. no white-out)], and the correct value entered alongside. All such corrections shall be initialed (or signed) and dated by the person making the correction. In the case of records stored electronically, the original "uncorrected" file must be maintained intact and a second "corrected" file is created.

This same process applies to adding additional information to a record. All additions made later than the initial must also be initialed (or signed) and dated.

When corrections are due to reasons other than obvious transcription errors, the reason for the corrections (or additions) shall also be documented.

Figure 13-1.
Example – Non-conformance Memo *(ISO17025 4.11.2 ; 4.13.2.3)*

Non-Conformance Memo

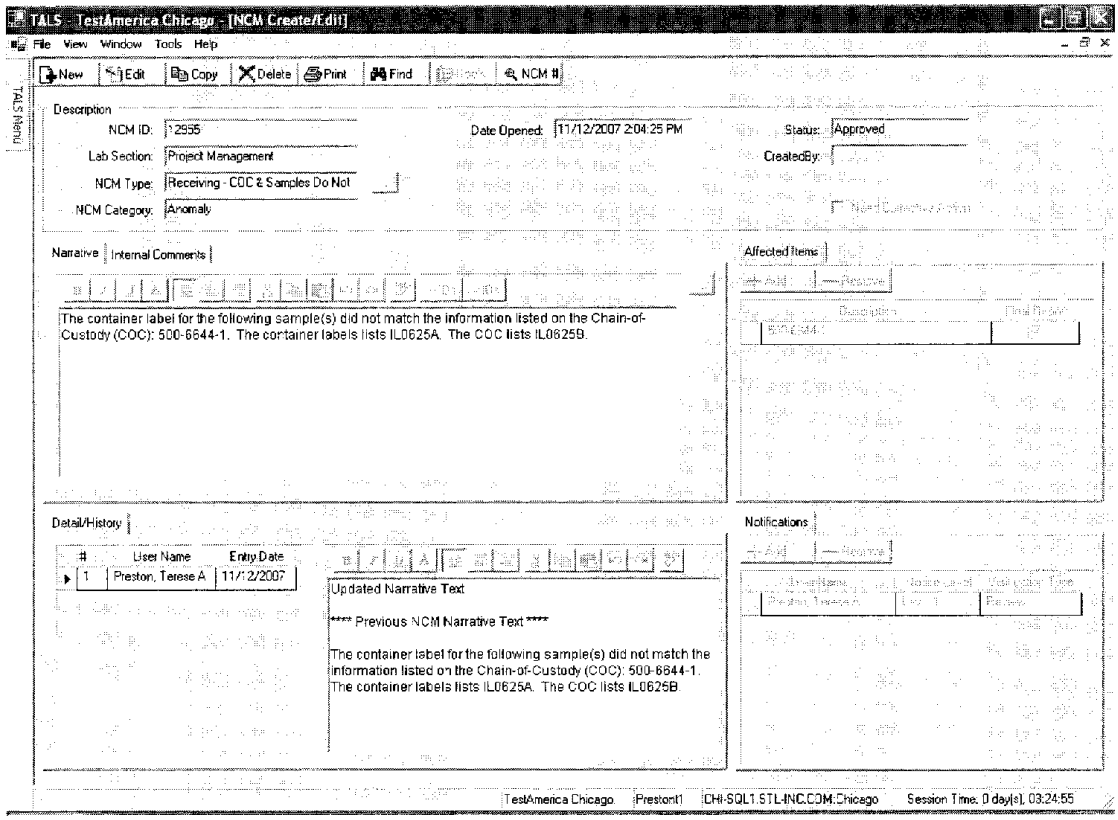


Table 13-1.

Example – General Corrective Action Procedures *(ISO17025 4.11.2)*

QC Activity ***(Individual Responsible for Initiation/Assessment)***	**Acceptance Criteria**	**Recommended Corrective Action**
Proficiency Testing (PT) Samples *(QA Manager, QA Specialist, Department Manager/Supervisor)*	- Criteria supplied by PT Supplier.	- Any failures or warnings must be investigated for cause. Failures may result in the need to repeat a PT sample to show the problem is corrected.
Internal / External Audits *(QA Manager, QA Specialist, Department Manager/Supervisor, Laboratory Director)*	- Defined in Quality System documentation such as SOPs, QAM, etc.	- Non-conformances must be investigated through CAR system and necessary corrections must be made. The determination of the root cause of the non-conformance must also be evaluated where applicable.
Reporting / Calculation Errors (Depends on issue – possible individuals include: Analysts, Data Reviewers, Project Managers, Department Manager/ Supervisor, QA Manager, Corporate QA, Corporate Management)	- SOP CA-L-S-001, Internal Investigation of Potential Data Discrepancies and Determination for Data Recall.	- Corrective action is determined by type of error. Follow the procedures in SOP CA-L-S-001.
Client Complaints *(Project Managers, Lab Director, Sales and Marketing, QA Manager, Department Supervisor/ Manager)*	-	- Corrective action is determined by the type of complaint. For example, a complaint regarding an incorrect address on a report will result in the report being corrected and then follow-up must be performed on the reasons the address was incorrect (e.g., database needs to be updated).
QA Monthly Report (Refer to Section 17 for an example) (QA Manager, Lab Director)	- QAM, SOPs.	- Corrective action is determined by the type of issue. For example, NCMs for the month are reviewed and possible trends are investigated.
Health and Safety Violation (Safety Officer, Lab Director, Department Supervisor/Manager)	- Environmental Health and Safety (EHS) Manual.	- Non-conformance is investigated and corrected through CAR system.

SECTION 14.0

PREVENTIVE ACTION (NELAC 5.4.11)

(ISO17025 4.10; 4.12.1; 4.12.2)

14.1 OVERVIEW *(ISO17025 4.15.1; 4.15.2)*

The laboratory's preventive action programs improve, or eliminate potential causes of nonconforming product and/or nonconformance to the quality system. This preventive action process is a proactive continuous process improvement activity that can be initiated through feedback from clients, employees, business providers, and affiliates. The QA Department has the overall responsibility to ensure that the preventive action process is in place, and that relevant information on actions is submitted for management review.

Dedicating resources to an effective preventive action system emphasizes the laboratory's commitment to its Quality Program. It is beneficial to identify and address negative trends before they develop into complaints, problems and corrective actions. Additionally, customer service and satisfaction can be improved through continuous improvements to laboratory systems.

Opportunities for improvement may be discovered during management reviews, the QA Metrics Report, internal or external audits, proficiency testing performance, client complaints, staff observation, etc..

The monthly QA Metrics Report shows performance indicators in all areas of the quality system. These areas include revised reports, corrective actions, audit findings, internal auditing and data authenticity audits, client complaints, PT samples, holding time violations, SOPs, ethics training, etc. These metrics are used to help evaluate quality system performance on an ongoing basis and provide a tool for identifying areas for improvement.

The laboratory's corrective action process (Section 13) is integral to implementation of preventive actions. A critical piece of the corrective action process is the implementation of actions to prevent further occurrence of a non-compliance event. Historical review of corrective action provides a valuable mechanism for identifying preventive action opportunities.

14.1.1 The following elements are part of a preventive action system:

- Identification of an opportunity for preventive action.
- Process for the preventive action.
- Define the measurements of the effectiveness of the process once undertaken.
- Execution of the preventive action.
- Evaluation of the plan using the defined measurements.
- Verification of the effectiveness of the preventive action.
- Close-Out by documenting any permanent changes to the Quality System as a result of the Preventive Action. Documentation of Preventive Action is incorporated into the monthly QA reports, corrective action process and management review.

Note: There may be varying levels of formality and documentation during the preventive action process due to the simplicity/complexity of the action taken.

14.1.2 Any Preventive Actions undertaken or attempted shall be taken into account during the Annual Management Review (Section 17.2). A highly detailed recap is not required; a simple recount of success and failure within the preventive action program will provide management a measure for evaluation.

14.2 MANAGEMENT OF CHANGE

The Management of Change process is designed to manage significant events and changes that occur within the laboratory. Through these various tracking indicators, the potential risks inherent with a new event or change are identified and evaluated. The risks are minimized or eliminated through pre-planning and the development of preventive measures. The types of indicators monitored under this collective system include:

- SOP Tracking
  - Current Revisions w/ Effective Dates
  - Required Annual Revisions w/ Due Date
- Proficiency Testing (PT) Sample Tracking
  - Pass / Fail – most current 2 out of 3 studies.
- Instrument / Equipment List
  - Current / Location
- Accreditations
  - New / Expiring
- Method Capabilities
  - Current Listing by program (e.g., Potable Water, Soils, etc.)
- Key Personnel
  - Technical Managers, Department Supervisors, etc..

These items are maintained on TestAmerica's Intranet (Proposal Library) or on our internal database (TotalAccess) which uploads to our company internet site.

This process is discussed in further detail in SOP CA-Q-S-003, Management of Change.

SECTION 15.0

CONTROL OF RECORDS (NELAC 5.4.12)

(ISO17025 4.2.7; 4.13.1.1)

The laboratory maintains a record system appropriate to its needs and that complies with applicable standards or regulations as required. The system produces unequivocal, accurate records that document all laboratory activities. The laboratory retains all original observations, calculations and derived data, calibration records and a copy of the analytical report for a minimum of five years after it has been issued. Refer to *SOP UP-DM-002* Data Management: Record Retention & Purging and *SOP UP-IS-014* Proc/Processes Entry, Storage, Backup/Retrieval, Mgmt Bench Data for further details.

15.1 OVERVIEW *(ISO17025 4.13.1.1; 4.13.1.2; 4.13.2.2; 4.13.2.3)*

The laboratory has established procedures for identification, collection, indexing, access, filing, storage, maintenance and disposal of quality and technical records. A record index is listed in Table 15-1. Quality records are maintained by the QA department on the local area network (LAN) in the W:/QC directory of data files which is backed up as part of the regular laboratory backup. Records are of two types; either electronic or hard copy paper formats depending on whether the record is computer or hand generated (some records may be in both formats). Technical records are maintained in accordance with the SOP UP-DM-002 *Data Management: Record Retention and Purging and* SOP UP-IS-014 *Proc/Processes Entry, Storage, Backup/Retrieval, Mgmt Bench Data.*

Table 15-1. Record Index[1] *(ISO17025 4.13.1.1)*

Record Type[1]		Retention Time:
Technical Records	- Raw Data - Logbooks - Standards - Certificates - Analytical Records - Lab Reports	5 Years from analytical report issue*
Official Documents	- Quality Assurance Manual (QAM) - Work Instructions - Policies - SOPs - Policy Memorandums - Manuals	5 Years from document retirement date*

	Record Type[1]	Retention Time:
QA Records	Internal & External Audits/Responses Certifications PTs Corrective/Preventive Actions/NCMs Management Reviews Review Checklists MDL/IDL/IDOC Studies Statistical Evaluations Laboratory Training Records Method & Software Validation / Verification Data Data Investigation	5 Years from archival* Data Investigation: 5 years or the life of the affected raw data storage whichever is greater (beyond 5 years if ongoing project or pending investigation)
Project Records	Sample Receipt & COC Documentation Contracts & Amendments Correspondence QAPP SAP Telephone Logs E-mail correspondence Lab Reports Electronic Data (EDD)	5 Years from analytical report issue*
Administrative Records	Finance & Accounting	5 Years
	EH&S Manual, Permits, Disposal Records	7 Years
	Employee Handbook	Indefinitely
	Personnel files, Employee Signature & Initials, Administrative Training Records (e.g., Ethics)	7 Years (HR Personnel Files must be maintained Indefinitely)
	Administrative Policies Technical Training Records	7 Years

[1] Record Types encompass hardcopy and electronic records.
[2] Examples of Logbook types: Maintenance, Instrument Run, Preparation (standard and samples), Standard and Reagent Receipt, Archiving, Balance Calibration, Temperature (hardcopy or electronic records).
* Exceptions listed in Table 15-2.

15.1.1 *(ISO17025 4.13.1.3)* All records are legible and stored and retained in such a way that they are secure and readily retrievable at the laboratory facility or the offsite location that provides a suitable environment to prevent damage or deterioration and to prevent loss. Backup data tapes are stored at the File Center. Retention of records is maintained on-site at the laboratory for at least 1 year after their generation and moved offsite for the remainder of the required storage time. All records shall be protected against fire, theft, loss, environmental deterioration, and vermin. In the case of electronic records, electronic or magnetic sources, storage media are protected from deterioration caused by magnetic fields and/or electronic deterioration.

Access to the data is limited to laboratory and company employees. Records archived off-site are stored in a secure location where a record is maintained of any entry into the storage facility. Whether on-site or off-site storage is used, logs are maintained in each storage box to note removal and return of records. Retention of records are maintained on-site at the laboratory for at least 1 year after their generation and moved off-site for the remainder of the required storage time. Records are maintained for a minimum of five years unless otherwise specified by a client or regulatory requirement.

For raw data and project records, record retention shall be calculated from the date the project report is issued. For other records, such as Controlled Documents, QA, or Administrative Records, the retention time is calculated from the date the record is formally retired. Records related to the programs listed in Table 15-2 have lengthier retention requirements and are subject to the requirements in Section 15.1.2. Policy CW-L-P-001 (Record Retention) provides additional information on record retention requirements.

15.1.2 Programs with Longer Retention Requirements

Some regulatory programs have longer record retention requirements than the standard record retention time. These are detailed in Table 15-2 with their retention requirements. In these cases, the longer retention requirement is enacted. If special instructions exist such that client data cannot be destroyed prior to notification of the client, the container or box containing that data is marked as to who to contact for authorization prior to destroying the data.

Table 15-2. Example: Special Record Retention Requirements

Program	[1]Retention Requirement
Drinking Water – All States	10 years (project records)
Drinking Water Lead and Copper Rule	12 years (project records)
Commonwealth of MA – All environmental data 310 CMR 42.14	10 years
FIFRA – 40 CFR Part 160	Retain for life of research or marketing permit for pesticides regulated by EPA
Housing and Urban Development (HUD) Environmental Lead Testing	10 years
Alaska	10 years
Louisiana – All	10 years
Michigan Department of Environmental Quality – all environmental data	10 years
Navy Facilities Engineering Service Center (NFESC)	10 years
NY Potable Water NYCRR Part 55-2	10 years
Ohio VAP	10 years and State contacted prior to disposal
TSCA - 40 CFR Part 792	10 years after publication of final test rule or negotiated test agreement

[1]Note: Extended retention requirements must be noted with the archive documents or addressed in facility-specific records retention procedures.

15.1.3 *(ISO17025 4.13.1.4)* The laboratory has procedures to protect and back-up records stored electronically and to prevent unauthorized access to or amendment of these records. All analytical data is maintained as hard copy or in a secure readable electronic format. For analytical reports that are maintained as copies in PDF format, refer to Section 20.14 for more information. Refer to SOP *UP-DM-002 - Data Management: Record Retention and Purging* for the discussion on electronic archiving and the maintenance of electronic records.

15.1.4 *(ISO17025 4.13.2.1)* The record keeping system allows for historical reconstruction of all laboratory activities that produced the analytical data, as well as rapid recovery of historical data (Records stored off site should be accessible within 2 days of a request for such records). The history of the sample from when the laboratory took possession of the samples must be readily understood through the documentation. This shall include inter-laboratory transfers of samples and/or extracts.

- The records include the identity of personnel involved in sampling, sample receipt, preparation, or testing. All analytical work contains the initials (at least) of the personnel involved. The laboratory's copy of the COC is stored with the invoice and the work order sheet generated by the LIMS within the TALs LIMs system and can be accessed via the Project Management Desktop. The chain of custody would indicate the name of the sampler. If any sampling notes are provided with a work order, they are kept with this package. The original COC is filed separately by Job number in the report generation and data management department. A copy of the COC is included as part of every data report.
- All information relating to the laboratory facilities equipment, analytical test methods, and related laboratory activities, such as sample receipt, sample preparation, or data verification are documented.
- The record keeping system facilitates the retrieval of all working files and archived records for inspection and verification purposes (e.g., set format for naming electronic files, set format for what is included with a given analytical data set refer to laboratory SOP UP-IS-014, *Proc/Processes Entry, Storage, Backup/Retrieval, Mgmt Bench Data* for further details. Instrument data is stored sequentially by instrument. A given day's analyses are maintained in the order of the analysis. Run logs are maintained for each instrument or method; a copy of each day's run log or instrument sequence is stored with the data to aid in re-constructing an analytical sequence. Where an analysis is performed without an instrument, bound logbooks or bench sheets are used to record and file data. Standard and reagent information is recorded in logbooks or entered into the LIMS for each method as required.
- Changes to hardcopy records shall follow the procedures outlined in Section 13 and 20. Changes to electronic records in LIMS or instrument data are recorded in audit trails.
- The reason for a signature or initials on a document is clearly indicated in the records such as "sampled by," "prepared by," "reviewed by", or "Analyzed by".
- All generated data except those that are generated by automated data collection systems, are recorded directly, promptly and legibly in permanent dark ink.
- Hard copy data may be scanned into PDF format for record storage as long as the scanning process can be verified in order to ensure that no data is lost and the data files and storage media must be tested to verify the laboratory's ability to retrieve the information prior to the destruction of the hard copy that was scanned. The procedure for this verification can be found in SOP *UP-DM-002 – Data Management: Record Retention and Purging.*
- Also refer to Section 20.14.1 'Computer and Electronic Data Related Requirements'.

15.2 TECHNICAL AND ANALYTICAL RECORDS *(ISO17025 4.13.2.2; 4.13.2.3)*

15.2.1 The laboratory retains records of original observations, derived data and sufficient information to establish an audit trail, calibration records, staff records and a copy of each analytical report issued, for a minimum of five years unless otherwise specified by a client or regulatory requirement (refer to Section 15.1). The records for each analysis shall contain sufficient information to enable the analysis to be repeated under conditions as close as possible to the original. The records shall include the identity of laboratory personnel responsible for the preparation, performance of each analysis and reviewing results.

15.2.2 Observations, data and calculations are recorded real-time and are identifiable to the specific task.

15.2.3 Changes to hardcopy records shall follow the procedures outlined in Section 13 and 20. Changes to electronic records in LIMS or instrument data are recorded in audit trails.
The essential information to be associated with analysis, such as strip charts, tabular printouts, computer data files, analytical notebooks, and run logs, include (previous discussions relate where most of this information is maintained – specifics may be added below):

- laboratory sample ID code;
- Date of analysis and Time of Analysis is also required if the holding time is seventy-two (72) hours or less, or when time critical steps are included in the analysis (e.g., drying times, incubations, etc.); instrumental analyses have the date and time of analysis recorded as part of their general operations. Where a time critical step exists in an analysis, location for such a time is included as part of the documentation in a specific logbook and in the LIMs.
- Instrumentation identification and instrument operating conditions/parameters. Operating conditions/parameters are typically recorded in instrument maintenance logs where available.
- analysis type;
- all manual calculations and manual integrations;
- analyst's or operator's initials/signature;
- sample preparation including cleanup, separation protocols, incubation periods or subculture, ID codes, volumes, weights, instrument printouts, meter readings, calculations, reagents;
- test results;
- standard and reagent origin, receipt, preparation, and use;
- calibration criteria, frequency and acceptance criteria;
- data and statistical calculations, review, confirmation, interpretation, assessment and reporting conventions;
- quality control protocols and assessment;
- electronic data security, software documentation and verification, software and hardware audits, backups, and records of any changes to automated data entries; and
- Method performance criteria including expected quality control requirements. These are indicated both in the LIMS and on specific analytical report formats.

15.3 LABORATORY SUPPORT ACTIVITIES

In addition to documenting all the above-mentioned activities, the following are retained QA records and project records (previous discussions in this section relate where and how these data are stored):

- all original raw data, whether hard copy or electronic, for calibrations, samples and quality control measures, including analysts' work sheets and data output records (chromatograms, strip charts, and other instrument response readout records);
- a written description or reference to the specific test method used which includes a description of the specific computational steps used to translate parametric observations into a reportable analytical value;
- copies of final reports;
- archived SOPs;
- correspondence relating to laboratory activities for a specific project;
- all corrective action reports, audits and audit responses;
- proficiency test results and raw data; and
- results of data review, verification, and crosschecking procedures

15.3.1 Sample Handling Records

Sample handling and tracking is discussed in Section 24. Records of all procedures to which a sample is subjected while in the possession of the laboratory are maintained. These include but are not limited to records pertaining to:

- sample preservation including appropriateness of sample container and compliance with holding time requirement;
- sample identification, receipt, acceptance or rejection and login;
- sample storage and tracking including shipping receipts, sample transmittal / COC forms; and
- procedures for the receipt and retention of samples, including all provisions necessary to protect the integrity of samples.

15.4 ADMINISTRATIVE RECORDS

The laboratory also maintains the administrative records in either electronic or hard copy form. See Table 15-1.

15.5 RECORDS MANAGEMENT, STORAGE AND DISPOSAL

15.5.1 All records (including those pertaining to test equipment), certificates and reports are safely stored, held secure and in confidence to the client. Certification related records are available to the accrediting body upon request.

15.5.2 All information necessary for the historical reconstruction of data is maintained by the laboratory. Records that are stored only on electronic media must be supported by the hardware and software necessary for their retrieval.

15.5.3 Records that are stored or generated by computers or personal computers have hard copy, write-protected backup copies, or an electronic audit trail controlling access.

15.5.4 The laboratory has a record management system (a.k.a., document control) for control of laboratory notebooks, instrument logbooks, standards logbooks, and records for data reduction, validation, storage and reporting. Laboratory bound notebooks are issued on a per analysis basis, and pages are numbered sequentially within a given logbook. All data are recorded sequentially within a series of sequential notebooks. Bench sheets, when used, are filed with any subsequent raw data by Department and sequentially by the TALs LIMs Batch number. Standards are maintained in the LIMS – logbooks are used in several departments in conjunction with the TALs LIMs record. Records are considered archived when noted as such in the records management system (a.k.a., document control).

15.5.5 Transfer of Ownership

In the event that the laboratory transfers ownership or goes out of business, the laboratory shall ensure that the records are maintained or transferred according to client's instructions. Upon ownership transfer, record retention requirements shall be addressed in the ownership transfer agreement and the responsibility for maintaining archives is clearly established. In addition, in cases of bankruptcy, appropriate regulatory and state legal requirements concerning laboratory records must be followed. In the event of the closure of the laboratory, all records will revert to the control of the corporate headquarters. Should the entire company cease to exist, as much notice as possible will be given to clients and the accrediting bodies who have worked with the laboratory during the previous 5 years of such action.

15.5.6 Records Disposal

15.5.6.1 Records are removed from the archive and destroyed after 5 years unless otherwise specified by a client or regulatory requirement. On a project specific or program basis, clients may need to be notified prior to record destruction. Records are destroyed in a manner that ensures their confidentiality such as shredding, mutilation or incineration (Refer to Tables 15-1 and 15-2).

15.5.6.2 Electronic copies of records must be destroyed by erasure or physically damaging off-line storage media so no records can be read.

15.5.6.3 If a third party records management company is hired to dispose of records, a "Certificate of Destruction" is required. [Refer to Policy No. CW-L-P-001 (Records Retention).]

SECTION 16

AUDITS *(NELAC 5.4.13)*

16.1 INTERNAL AUDITS *(ISO17025 4.14.1; 4.14.2; 4.14.3; 5.9.1)*

Internal audits are performed to verify that laboratory operations comply with the requirements of the lab's quality system and with the external quality programs under which the laboratory operates. Audits are planned and organized by the QA staff. Personnel conducting the audits should be independent of the area being evaluated. Auditors will have sufficient authority, access to work areas, and organizational freedom necessary to observe all activities affecting quality and to report the assessments to laboratory management and when requested to corporate management.

Audits are conducted and documented as described in the TestAmerica Corporate SOP on performing Internal Audits, SOP No. CA-Q-S-004. The types and frequency of routine internal audits are shown in Table 16-1. Special or ad hoc assessments may be conducted as needed under the direction of the QA staff.

Table 16-1. Types of Internal Audits and Frequency

Description	Performed by	Frequency
Quality Systems	QA Department or Designee	All areas of the laboratory annually
QA Technical Audits - Evaluate raw data versus final reports - Analyst integrity - Data authenticity	QA Department or Designee	All methods within a 2-year period, with at least 15% of methods every quarter
SOP Method Compliance	Technical Director / Department Supervisor	- All SOPs within a 2-year period - All new analysts or new analyst/methods within 3 months of IDOC
Special	QA Department or Designee	Surveillance or spot checks performed as needed
Performance Testing	Analysts with QA oversight	Two successful per year for each NELAC field of testing or as dictated by regulatory requirements

16.1.1 Annual Quality Systems Audit

An annual quality systems audit is required to ensure compliance to analytical methods and SOPs, the laboratory's Data Integrity and Ethics Policies, NELAC quality systems, client and state requirements, and the effectiveness of the internal controls of the analytical process, including but not limited to data review, quality controls, preventive action and corrective action. The completeness of earlier corrective actions is assessed. The audit is divided into modules for each operating or support area of the lab, and each module is comprehensive for a given area. The area audits may be done on a rotating schedule throughout the year to ensure adequate coverage of all areas. This schedule may change as situations in the laboratory warrant.

16.1.2 QA Technical Audits

QA technical audits are based on client projects, associated sample delivery groups, and the methods performed. Reported results are compared to raw data to verify the authenticity of results. The validity of calibrations and QC results are compared to data qualifiers, footnotes, and case narratives. Documentation is assessed by examining run logs and records of manual integrations. Manual calculations are checked. Where possible, MintMiner is used to identify unusual manipulations of the data deserving closer scrutiny. QA technical audits will include all methods within a two-year period.

16.1.3 SOP Method Compliance

Compliance of all SOPs with the source methods and compliance of the operational groups with the SOPs will be assessed by the Technical Director at least every two years. The work of each newly hired analyst is assessed within 3 months of working independently, (e.g., completion of method IDOC). In addition, as analysts add methods to their capabilities, (new IDOC) reviews of the analyst work products will be performed within 3 months of completing the documented training.

16.1.4 Special Audits

Special audits are conducted on an as needed basis, generally as a follow up to specific issues such as client complaints, corrective actions, PT results, data audits, system audits, validation comments, regulatory audits or suspected ethical improprieties. Special audits are focused on a specific issue, and report format, distribution, and timeframes are designed to address the nature of the issue.

16.1.5 Performance Testing *(ISO17025 5.9.1; 5.9.2)*

The laboratory participates semi-annually in performance audits conducted through the analysis of PT samples provided by a third party. The laboratory generally participates in the following types of PT studies: Drinking Water (WS), Nonpotable Water (WP), Soil (SW) and Sediment.

It is TestAmerica's policy that PT samples be treated as typical samples in the production process. Furthermore, where PT samples present special or unique problems, in the regular production process they may need to be treated differently, as would any special or unique request submitted by any client. The QA Manager must be consulted and in agreement with any decisions made to treat a PT sample differently due to some special circumstance.

Written responses to unacceptable PT results are required. In some cases it may be necessary for blind QC samples to be submitted to the laboratory to show a return to control.

16.2 EXTERNAL AUDITS *(ISO17025 4.14.2; 4.14.3; 4.14.4)*

External audits are performed when certifying agencies or clients conduct on-site inspections or submit performance testing samples for analysis. It is TestAmerica's policy to cooperate fully with regulatory authorities and clients. The laboratory makes every effort to provide the auditors with access to personnel, documentation, and assistance. Laboratory supervisors are responsible for providing corrective actions to the QA Manager who coordinates the response for any deficiencies discovered during an external audit. Audit responses are due in the time allotted by the client or agency performing the audit. A copy of the audit report and the labs corrective action plan will be forwarded to Corporate Quality.

The laboratory cooperates with clients and their representatives to monitor the laboratory's performance in relation to work performed for the client. The client may only view data and systems related directly to the client's work. All efforts are made to keep other client information confidential.

16.2.2 Confidential Business Information (CBI) Considerations

During on-site audits, on-site auditors may come into possession of information claimed as business confidential. A business confidentiality claim is defined as "a claim or allegation that business information is entitled to confidential treatment for reasons of business confidentiality or a request for a determination that such information is entitled to such treatment." When information is claimed as business confidential, the laboratory must place on (or attach to) the information at the time it is submitted to the auditor, a cover sheet, stamped or typed legend or other suitable form of notice, employing language such as "trade secret", "proprietary" or "company confidential". Confidential portions of documents otherwise non-confidential must be clearly identified. CBI may be purged of references to client identity by the responsible laboratory official at the time of removal from the laboratory. However, sample identifiers may not be obscured from the information. Additional information regarding CBI can be found in within the 2003 NELAC standards.

16.3 AUDIT FINDINGS

Audit findings are documented using the corrective action process and database (refer to Section 13). The laboratory's corrective action responses for both types of audits may include action plans that could not be completed within a predefined timeframe. In these instances, a completion date must be set and agreed to by operations management and the QA Manager.

Developing and implementing corrective actions to findings is the responsibility of the Department Manager and Department Supervisor where the finding originated. Findings that are not corrected by specified due dates are reported monthly to management in the QA monthly report. When requested a copy of the audit report and the labs corrective action plan will be forwarded to Corporate Quality.

If any audit finding casts doubt on the effectiveness of the operations or on the correctness or validity of the laboratory's test results, the laboratory shall take timely corrective action, and shall notify clients in writing if the investigations show that the laboratory results have been affected. Once corrective action is implemented, a follow-up audit is scheduled to ensure that the problem has been corrected.

Clients must be notified promptly in writing, of any event such as the identification of defective measuring or test equipment that casts doubt on the validity of results given in any test report or amendment to a test report. The investigation must begin within 24-hours of discovery of the problem and all efforts are made to notify the client within two weeks after the completion of the investigation.

Figure 16-1.

Example - Internal Audit Workbook *(ISO17025 4.14.1; 4.14.3)*

TestAmerica Chicago

Internal Audit Schedule 2011

Department Audited	Date Scheduled	Date Audited	# Findings	# Findings Open	Date Corrective Action Due	Date Audit Closed	Comments
Administrative:							
Container Management							
Sample Management							
Project Management Data Reporting & Archive							
Quality Assurance							
Sample Preparation:							
Metals Digestion / TCLP & SPLP							
Organic Extractions							
Inorganics :							
Metals Analysis							
Inorganic Chemistry							
Organics:							
GC/HPLC Extractables							
GC Volatiles							
GCMS Volatiles							
GCMS Semivolatiles							
Total	0	0	0	0		0	

Figure 16-2.

Example – Internal Audit System Checklist: Container Management *(ISO17025 4.14.3)*

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

Container Management
Quality System Audit w/DOD
December 2010
Revision 2

Location: _____

Audit Date:	
Auditor:	**Personnel Interviewed:**
Most Current SOP Title: Revision No., Effective Date (NELAC Ref. 5.5.8.1):	

System Status:
0 = No system or system not defined
1 = Informal system or system being developed
2 = System initiated, documented, implemented; but failures or lapses occur
3 = System developed, documented, implemented and is functional
E = System in place, exceeds minimum expectations; evidence of continuous quality improvement (CQI)

'Follow Up' column indicates that:
a) the listed requirement needs to be audited in an alternate dept.; or
b) was / can be audited via the Internal Audit Workbook for Metrology Items (CA-Q-WI-011).

			System Status							
Item	**Requirement**	**Ref.**	**0**	**1**	**2**	**3**	**E**	**NA**	**Evidence/Comments**	**Follow Up**
1	***General***									
2	Is a current/controlled SOP available within this department (electronic or hardcopy?	5.5.4.1.1, TNI 4.2.8.5, ISO 4.3.1, 5.4.1	0	1	2	3	E	NA	Auditor: Personnel should be able to readily locate. No uncontrolled copies of SOPs in use or in area.	
3	Do all posted Work Instructions or Flowcharts have a document control number?	5.4.3.1, TNI 4.2.8.5, ISO 4.3.1	0	1	2	3	E	NA	Auditor: Examples include preservatives, container types and critical volumes.	
4	***Supplies***									
5	Are sample containers purchased precleaned? Is ESS the vendor?	Info.	0	1	2	3	E	NA		
6	Are the containers certified to a level of cleanliness ≤ ½ LOQ for all DoD analytes?	Box 11 & QSM F Tables	0	1	2	3	E	NA		

SECTION 17

MANAGEMENT REVIEWS *(NELAC 5.4.14)*

(ISO17025 4.1.6; 4.15.1; 4.15.2)

17.1 QUALITY ASSURANCE REPORT

A comprehensive QA Report shall be prepared each month by the laboratory's QA Department and forwarded to the Laboratory Director, Customer Service Manager (CSM), Organics Manager, Inorganics Manager, their Quality Director as well as the General Manager. All aspects of the QA system are reviewed to evaluate the suitability of policies and procedures. During the course of the year, the Laboratory Director, General Manager or Corporate QA may request that additional information be added to the report.

The TestAmerica QA Report template is comprised of a discussion of three key QA issues facing the laboratory and ten specific sections (Figure 17-1):

- **Metrics:** Describe actions or improvement activities underway to address any outlying quality metrics that have been reported in the monthly Quality System Metrics Table.
- **SOPs:** Report SOPs that have been finalized and report status of any outstanding SOP reviews.
- **Corrective Actions:** Describe highlights and the most frequent cause for report revisions and corrective/preventive action measures underway. Include a discussion of any recalls handled at the lab level as per Section 6.2.2 in the Investigation/Recall SOP (SOP: CA-L-S-001). Include a section for client feedback and complaints. Include both positive and negative feedback. Describe the most serious client complaints and resolutions in progress.
- **MDLs and Control Limits:** Report which MDLs/ MDL verifications are due. Report the same for Control Limits.
- **Audits:** Report Internal and External Audits that were conducted. Include all relevant information such as which methods, by whom, corrective actions needed by when and discuss unresolved audit findings.
- **Performance Testing (PT) Samples:** Report the PT tests that are currently being tested with their due dates, report recent PT results by study, acceptable, total reported and the month and year.
- **Certifications:** Report on any certification programs being worked on by due date, packages completed. Describe any issues, lapses, or potential revocations.
- **Regulatory Updates:** Include information on new state or federal regulations that may impact the laboratory. Report new methods that require new instrumentation, deletion of methods, changes in sampling requirements and frequencies etc…
- **Miscellaneous:** Include any issues that may impact quality within the laboratory. This section may also be used to communicate the status on any Management of Change Request Forms (CRFs) that have missed targeted due dates
- **Next Month:** Report on plans for the upcoming month.

- **Lab Director Comments Section:** This section gives the Laboratory Director the opportunity to comment on issues discussed in the report and to document plans to resolve these issues. Unresolved issues that reappear in subsequent monthly reports must be commented on by the Laboratory Director.
- **Quality System Metrics Table:** The report also includes statistical results that are used to assess the effectiveness of the quality system. Effective quality systems are the responsibility of the entire laboratory staff. Each laboratory provides their results in a template provided by Corporate QA (Figure 17-2).

On a monthly basis, Corporate QA compiles information from all the monthly laboratory reports. The Corporate Quality Directors prepare a report that includes a compilation of all metrics and notable information and concerns regarding the QA programs within the laboratories. The report also includes a listing of new regulations that may potentially impact the laboratories. This report is presented to the Senior Management Team and General Managers.

17.2 ANNUAL MANAGEMENT REVIEW *(ISO17025 4.2.2)*

The senior lab management team (Laboratory Director, QA Manager, Department Managers and Customer Service Manager) conducts a review annually of its quality systems and LIMS to ensure its continuing suitability and effectiveness in meeting client and regulatory requirements and to introduce any necessary changes or improvements. It will also provide a platform for defining quality goals & objectives. Corporate Operations and Corporate QA personnel may be included in this meeting at the discretion of the Laboratory Director. The LIMS review consists of examining any audits, complaints or concerns that have been raised through the year that are related to the LIMS. The laboratory will summarize any critical findings that can not be solved by the lab and report them to Corporate IT.

This management systems review as outlined in the Corporate SOP CA-Q-S-008, *Management Systems Review,* references the Corporate Work Instruction No. CA-Q-WI-020. Part 1 of the Checklist uses information generated during the preceding year to assess the "big picture" by ensuring that routine quality actions taken and reviewed on a monthly basis are not components of larger systematic concerns. The monthly review (refer to Section 17.1) should keep the quality systems current and effective, therefore, the annual review is a formal senior management process to review specific existing documentation. Significant issues from the following documentation are compiled or summarized by the QA Manager prior to the review meeting:

- Matters arising from the previous annual review.
- Prior Monthly QA Reports issues.
- Laboratory QA Metrics.
- Review of report reissue requests.
- Review of client feedback and complaints.
- Issues arising from any prior management or staff meetings.
- Minutes from prior senior lab management team meetings. Issues that may be raised from these meetings include:

- Adequacy of staff, equipment and facility resources.
- Adequacy of policies and procedures.
- Future plans for resources and testing capability and capacity.

- The annual internal double blind PT program sample performance (if performed),
- Compliance to the Ethics Policy and Data Integrity Plan. Including any evidence/incidents of inappropriate actions or vulnerabilities related to data Integrity.

A report is generated by the QA Manager and senior management staff in the form of the management systems review summary and an objectives and goals summary (Part 2 and Part 3 of CA-Q-WI-020). The report is distributed to the appropriate General Manager and the Quality Director. The report includes, but is not limited to:

- The date of the review and the names and titles of participants.
- A reference to the existing data quality related documents and topics that were reviewed.
- Quality system or operational changes or improvements that will be made as a result of the review [e.g., an implementation schedule including assigned responsibilities for the changes (Action Table)].

Changes to the quality systems requiring update to the laboratory QA Manual shall be included in the next revision of the QA Manual.

17.3 POTENTIAL INTEGRITY RELATED MANAGERIAL REVIEWS

Potential integrity issues (data or business related) must be handled and reviewed in a confidential manner until such time as a follow-up evaluation, full investigation, or other appropriate actions have been completed and issues clarified. TestAmerica's Corporate Data Investigation/ Recall SOP shall be followed (SOP No. CA-L-S-001). All investigations that result in finding of inappropriate activity are documented and include any disciplinary actions involved, corrective actions taken, and all appropriate notifications of clients.

TestAmerica's COO, VP of Client & Technical Services, General Managers and Quality Directors receive a monthly report from the Director of Quality & Client Advocacy summarizing any current data integrity or data recall investigations. The General Manager's are also made aware of progress on these issues for their specific labs.

Figure 17-1.

Example - QA Monthly Report to Management *(ISO17025 4.15.1)*

TestAmerica Chicago *Name of preparer (QAM)* *Date report submitted to corporate* *GM, LD, additional staff as warranted*	**January 2011** **Prepared by:** **Report Date:** **Distributed To:**
THREE KEY ISSUES FOR MONTH: *Include a discussion of three key issues that were focused on this month.* **1** **2** **3**	-
METRICS CONCERNS *For any quality metrics that fall in 'red' or yellow', describe actions or improvement activities underway.* **Revised reports** **Data Recall** **Audits** **Performance Testing** **Corrective Action** **SOPs**	-
CORRECTIVE ACTIONS **Highlights:** *Provide discussion of any major investigations or closures.* **Revised Reports:** *Describe the most frequent cause for report revisions and corrective/preventive action measures underway.* **Data Investigations/Recalls:** *Provide a short summary or update of any events handled at the lab level as per Section 6.2.2 in the Investigation/Recall SOP. Include detailed explanation on the 'Data Recall' tab.* **Client Complaints:** *Describe the most serious client complaints and associated corrective actions in progress. Include client company names.* **Client Compliments:** *Describe any compliments received - include client company names.*	-
AUDITS **Internal Audits (QA Technical, Departmental Quality Systems and Lab Requested Investigations):** *Discuss any outstanding issues or concerns (may attach summary).* **External Audits:** *Discuss any outstanding issues or concerns (may attach summary). Include schedule for upcoming audits.*	-

PROFICIENCY TESTING STUDIES **PT Corrective Actions:** *Include discussion of any outstanding issues.*	-
SOPs **SOPs:** *Provide a discussion of any concerns. If SOPs are greater than 6 months overdue, provide a corrective action plan. Include an SOP Tracking Tab in your monthly report.* **SOPs have not been developed for the following methods or processes** (include list).	-
MDLs , TRAINING DOCUMENTATION AND CONTROL LIMITS **MDLs Due (or attach summary):** *Include discussion of any concerns* **Control Limits Due (or attach summary):** *Include discussion of any concerns* **Training Documentation:** *Describe any concerns or system failures*	-
HOLD TIME VIOLATIONS *Describe major HT violation events and associated corrective actions in progress.*	-
CERTIFICATIONS *Describe any issues, lapses or potential revocations:* **Certification Packages Being Worked On** (Include Due Date):	-
REGULATORY UPDATE *Include information on new state or federal regulations that may impact the laboratory – new methods that require new instrumentation, deletion of methods, changes in sampling requirements or frequencies, …*	-
MISCELLANEOUS *Include any issues that may impact quality within the laboratory.*	-
TRAINING or MEETINGS *Include information on participation in any industry or regulatory meetings or any training sessions being scheduled.*	-
NEXT MONTH (Items planned for next month)	-
LAB DIRECTOR COMMENTS AND PLANNED CORRECTIVE ACTIONS: Date: Comments: Signature (Name):	-

Figure 17-2. Example - Laboratory QS Metrics Categories *(ISO17025 4.15.1)*

Category	Objective	Period	Metrics
Revised Reports		This month	# Reports for month
		This month	# Reports revised due to lab error
	<2.5%	This month	**% Reports Revised due to lab error (1.0)**
	Indicator	This month	# Reports Reviewed by QA
Data Recall	<1	Jan-to-date	**# Data Recall Investigations Past Due (1.0)** (data recall summary)
	Indicator	From Corp.	Cumulative # Days Data Recalls Open
Client	>3	This month	**# Client Complaints (1.0)**
	>3	This month	# Client Compliments
Method Audits			# of Method the Lab Performs *(enter total for 100% of all methods)*
		50% by end Dec	# of QA Technical Method Audits performed Jan11-Dec11
	4% per month	Jan11 to Dec11	**% Method Audits Performed (2.0)**
Department QS Audits		Planned Jan-Dec	# of Planned Department Quality Systems Audits (enter total # planned for 2011)
		Jan-to-date	# of Planned Department Quality Systems Audits Complete
		Planner	% Quality Systems Audit Complete *(all depts within 12 months of corp audit)*
	1 year after Corp. Audit	2011	Date Quality Systems Audit Complete*(all departments audited - date)*
	13 months after Corp Audit	2011	**Date Lab QS Audit Report Submitted to Corp** *(<13 months from Corp QS Audit)*
Corporate Internal Quality Systems Audits		Current	Date of Corporate Internal Audit *(2009, 2010 & 2011 - Use date of Debrief Meeting)*
		Current	**% Audit Findings Closed (from Corporate)**
	<6 months	Current	Date Audit Closed *(2009, 2010 or 2011 closure notification from Corp)*
Audits Findings		Jan-to-date	Total Number of Audit Findings (Int. & Ext.) (include summary tabs)
		Current	# Audit Findings with Corrective Action Implementation Date Past Due (internal & external)
	Indicator	Jan-to-date	Total Number Repeat Audit Findings *(internal & external) (cumulative - not scored in 2011)*
	Indicator	Opened in 2010	2010 Open Audit Findings *(Corrective Actions not Implemented)*
Proficiency Testing		Past 12 months	# of PT analytes participated and received scores (12 month total)
		Past 12 months	# of PT analytes not acceptable (12 month total)
	>97%	Past 12 months	**% PT Cumulative Score (2.0)** (rolling 12 month average)
	No more than 1 per 200 results	Jan-to-date	**# PT Repeat Analyte Failures Cumulative (1.0)** (analyte failed more than once in 4 consecutive studies by PT Type) (cumulative for 2011 - do not drop off when corrected)
Corrective Action		Jan-to-date	Total # Corrective Action Items (audit findings, PT failures, lab investigations)
		Current	# Corrective Actions Not Implemented and Past Due
	<15%	Current	**% Corrective Action Items Past Due (1.0)**
SOPs / Method Compliance		Current	# SOPs
		Current	# SOP with Procedure Compliance Review/Revision Past Due
		Current	# Methods or Administrative procedures without approved SOPs
	100%	Current	**% SOP Complete (1.0)**
Ethics		Current	Date of Last Comprehensive Ethics Training Session

Training	0	Current	**# Staff >90 Days from Hire Date AND have not received Comprehensive Ethics Training (1.0)**
Operations Method & Personnel	Good	Current	Total # MDLs/MDLVs Required
		Current	# MDLs/MDLVs Past Due
	100%	Current	**% MDLs/MDLVs Complete (1.0)**
	Good	Current	Training Documentation Records (Good>90%, Fair 70-90%, or Poor <70%)
Enter 2010 Average	2010 Average	This month	Hold Time Violations due to Lab Error
Accreditation	<3 months	Current	Total Access Update Status - Date Last Updated
	Good	Current	Total Access Certifications current (Good, Fair, Poor)
	Good	Current	Total Access Methods current (Good, Fair, Poor)
	<1	Jan-to-date	**Method certification Losses (performance or audit issues) (1.0)** (can be removed after certification is reinstated by agency)
Quality System Review		Current	Last NELAC Audit Date (Where applicable - informational)
		Current	QAM Effective Date
	<12 months	Current	**Last Management QS Review Date (1.0)**
		Current	**Metrics Report Submitted to Corp On Time (1.0)**
			Good >90%, Needs Work between 75-90%, Poor <75%

SECTION 18

PERSONNEL *(NELAC 5.5.2)*
(ISO17025 5.2.1)

18.1 OVERVIEW *(SIO17025 5.2.2; 5.2.3; 5.2.5)*

The laboratory's management believes that its highly qualified and professional staff is the single most important aspect in assuring a high level of data quality and service. The staff consists of professionals and support personnel as outlined in the laboratory's organization chart in Figure 4-2.

All personnel must demonstrate competence in the areas where they have responsibility. Any staff that is undergoing training shall have appropriate supervision until they have demonstrated their ability to perform their job function on their own. Staff shall be qualified for their tasks based on appropriate education, training, experience and/or demonstrated skills as required.

The laboratory employs sufficient personnel with the necessary education, training, technical knowledge and experience for their assigned responsibilities.

All personnel are responsible for complying with all QA/QC requirements that pertain to the laboratory and their area of responsibility. Each staff member must have a combination of experience and education to adequately demonstrate a specific knowledge of their particular area of responsibility. Technical staff must also have a general knowledge of lab operations, test methods, QA/QC procedures and records management.

Laboratory management is responsible for formulating goals for lab staff with respect to education, training and skills and ensuring that the laboratory has a policy and procedures for identifying training needs and providing training of personnel. The training shall be relevant to the present and anticipated responsibilities of the lab staff.

The laboratory only uses personnel that are employed by or under contract to, the laboratory. Contracted personnel, when used, must meet competency standards of the laboratory and work in accordance to the laboratory's quality system.

18.2 EDUCATION AND EXPERIENCE REQUIREMENTS FOR TECHNICAL PERSONNEL *(ISO17025 5.2.1; 5.2.3; 5.2.4)*

The laboratory makes every effort to hire analytical staff that possesses a college degree (AA, BA, BS) in an applied science with some chemistry in the curriculum. Exceptions can be made based upon the individual's experience and ability to learn. There are competent analysts and technicians in the industry who have not earned a college degree. Selection of qualified candidates for laboratory employment begins with documentation of minimum education, training, and experience prerequisites needed to perform the prescribed task. Minimum education and training requirements for TestAmerica employees are outlined in job descriptions and are generally summarized for analytical staff in the table below.

The laboratory maintains job descriptions for all personnel who manage, perform or verify work affecting the quality of the environmental testing the laboratory performs. Job Descriptions are located on the TestAmerica intranet site's Human Resources web-page (Also see Section 4 for position descriptions/responsibilities).

Experience and specialized training are occasionally accepted in lieu of a college degree (basic lab skills such as using a balance, colony counting, aseptic or quantitation techniques, etc. are also considered).

As a general rule for analytical staff:

Specialty	Education	Experience
Extractions, Digestions, some electrode methods (pH, DO etc.), or Titrimetric and Gravimetric Analyses	H.S. Diploma	On the job training (OJT)
CVAA, Single component or short list Chromatography (e.g. BTEX-GC, IC)	A college degree in an applied science or 2 years of college and at least 1 year of college chemistry	or 2 years prior analytical experience is required
ICP, ICPMS, Long List or complex GC Chromatography (e.g., Pesticides, PCB, Herbicides, Fuels, HPLC, etc.), GCMS	A college degree in an applied science or 2 years of college chemistry	or 5 years of prior analytical experience
Spectra Interpretation	A college degree in an applied science or 2 years of college chemistry	And 2 years relevant experience or 5 years of prior analytical experience
Technical Directors/ Department Managers/Supervisors	Bachelors Degree in an applied science or engineering with 24 semester hours in chemistry An advanced (MS, PhD.) degree may substitute for one year of experience	And 2 years experience in environmental analysis of representative analytes for which they will oversee

When an analyst does not meet these requirements, they can perform a task under the direct supervision of a qualified analyst, peer reviewer or Department Supervisor/Manager, and are considered an analyst in training. The person supervising an analyst in training is accountable for the quality of the analytical data and must review and approve data and associated corrective actions.

18.3 TRAINING *(ISO17025 5.2.5)*

The laboratory is committed to furthering the professional and technical development of employees at all levels.

Orientation to the laboratory's policies and procedures, in-house method training, and employee attendance at outside training courses and conferences all contribute toward employee proficiency. Below are examples of various areas of required employee training:

Required Training	Time Frame*	Employee Type
Environmental Health & Safety	Prior to lab work	All
Ethics – New Hires	1 week of hire	All
Ethics – Comprehensive	90 days of hire	All
Data Integrity	30 days of hire	Technical and PMs
Quality Assurance	90 days of hire	Technical and PMs
Ethics – Comprehensive Refresher	Annually	All
Initial Demonstration of Capability (IDOC)	Prior to unsupervised method performance	Technical

The laboratory maintains records of relevant authorization/competence, education, professional qualifications, training, skills and experience of technical personnel (including contracted personnel) as well as the date that approval/authorization was given. These records are kept on file at the laboratory. Also refer to "Demonstration of Capability" in Section 20.

The training of technical staff is kept up to date by:

- Each employee must have documentation in their training file that they have read, understood and agreed to follow the most recent version of the laboratory QA Manual and SOPs in their area of responsibility. This documentation is updated as SOPs are updated.
- Documentation from any training courses or workshops on specific equipment, analytical techniques or other relevant topics are maintained in their training file.
- Documentation of proficiency (refer to Section 20).
- An Ethics Agreement signed by each staff member (renewed each year) and evidence of annual ethics training.
- A Confidentiality Agreement signed by each staff member signed at the time of employment.
- Human Resources maintains documentation and attestation forms on employment status & records; benefit programs; timekeeping/payroll; and employee conduct (e.g., ethics). This information is maintained in the employee's secured personnel file.

Evidence of successful training could include such items as:

- Adequate documentation of training within operational areas, including one-on-one technical training for individual technologies, and particularly for people cross-trained.
- Analysts knowledge to refer to QA Manual for quality issues.
- Analysts following SOPs, i.e., practice matches SOPs.
- Analysts regularly communicate to supervisors and QA if SOPs need revision, rather than waiting for auditors to find problems.

For additional details on the laboratory's training program refer to the laboratory SOP *UP-QA-014*, Training Program: Mechanisms and Documentation Processes Defined by Operational Assessment.

18.4 DATA INTEGRITY AND ETHICS TRAINING PROGRAM

Establishing and maintaining a high ethical standard is an important element of a Quality System. Ethics and data integrity training is integral to the success of TestAmerica and is provided for each employee at TestAmerica. It is a formal part of the initial employee orientation within 1 week of hire followed by technical data integrity training within 30 days, comprehensive training within 90 days, and an annual refresher for all employees. Senior management at each facility performs the ethics training for their staff.

In order to ensure that all personnel understand the importance TestAmerica places on maintaining high ethical standards at all times; TestAmerica has established a Corporate Ethics Policy No. CA-L-P-001 and an Ethics Statement/Agreement (Appendix 1). All initial and annual training is documented by signature on the signed Ethics Policy and Code of Ethical Conduct demonstrating that the employee has participated in the training and understands their obligations related to ethical behavior and data integrity.

Violations of this Ethics Policy will not be tolerated. Employees who violate this policy will be subject to disciplinary actions up to and including termination. Criminal violations may also be referred to the Government for prosecution. In addition, such actions could jeopardize TestAmerica's ability to do work on Government contracts, and for that reason, TestAmerica has a Zero Tolerance approach to such violations.

Employees are trained as to the legal and environmental repercussions that result from data misrepresentation. Key topics covered in the presentation include:

- Organizational mission and its relationship to the critical need for honesty and full disclosure in all analytical reporting.
- Ethics Policy (Appendix 1)
- How and when to report ethical/data integrity issues. Confidential reporting.
- Record keeping.
- Discussion regarding data integrity procedures.
- Specific examples of breaches of ethical behavior (e.g. peak shaving, altering data or computer clocks, improper macros, etc., accepting/offering kickbacks, illegal accounting practices, unfair competition/collusion)
- Internal monitoring. Investigations and data recalls.
- Consequences for infractions including potential for immediate termination, debarment, or criminal prosecution.
- Importance of proper written narration / data qualification by the analyst and project manager with respect to those cases where the data may still be usable but are in one sense or another partially deficient.

Additionally, a data integrity hotline (1-800-736-9407) is maintained by TestAmerica and administered by the Corporate Quality Department.

SECTION 19

ACCOMMODATIONS AND ENVIRONMENTAL CONDITIONS *(NELAC 5.5.3)*

19.1 OVERVIEW *(ISO17025 5.3.1; 5.3.3; 5.3.4; 5.3.5)*

TestAmerica Chicago is a 28,000 ft^2 secure laboratory facility with controlled access and designed to accommodate an efficient workflow and to provide a safe and comfortable work environment for employees. All visitors sign in and are escorted by laboratory personnel. Access is controlled by various measures.

The laboratory is equipped with structural safety features. Each employee is familiar with the location, use, and capabilities of general and specialized safety features associated with their workplace. The laboratory provides and requires the use of protective equipment including safety glasses, protective clothing, gloves, etc. OSHA and other regulatory agency guidelines regarding required amounts of bench and fume hood space, lighting, ventilation (temperature and humidity controlled), access, and safety equipment are met or exceeded.

Traffic flow through sample preparation and analysis areas is minimized to reduce the likelihood of contamination. Adequate floor space and bench top area is provided to allow unencumbered sample preparation and analysis space. Sufficient space is also provided for storage of reagents and media, glassware, and portable equipment. Ample space is also provided for refrigerated sample storage before analysis and archival storage of samples after analysis. Laboratory HVAC and deionized water systems are designed to minimize potential trace contaminants.

The laboratory is separated into specific areas for sample receiving, sample storage, sample disposal, organic glassware cleaning, inorganic glassware cleaning, organic sample preparation, metals sample preparation, GC and GC/MS Volatile sample analysis, GC Semivolatile & HPLC sample analysis, GC/MS Semivolatile sample analysis, General Chemistry sample analysis, Metals sample analysis, and administrative functions.

19.2 ENVIRONMENT *(ISO17025 5.3.1; 5.3.2; 5.3.3; 5.3.4; 5.3.5)*

Laboratory accommodation, test areas, energy sources, lighting are adequate to facilitate proper performance of tests. The facility is equipped with heating, ventilation, and air conditioning (HVAC) systems appropriate to the needs of environmental testing performed at this laboratory.

The environment in which these activities are undertaken does not invalidate the results or adversely affect the required accuracy of any measurements.

The laboratory provides for the effective monitoring, control and recording of environmental conditions that may affect the results of environmental tests as required by the relevant specifications, methods, and procedures. Such environmental conditions include humidity, voltage and temperature in the laboratory

When any of the method or regulatory required environmental conditions change to a point where they may adversely affect test results, analytical testing will be discontinued until the environmental conditions are returned to the required levels (refer to Section 12).

Environmental conditions of the facility housing the computer network and LIMS are regulated to protect against raw data loss.

19.3 WORK AREAS *(ISO17025 5.3.3; 5.3.4; 5.3.5)*

There is effective separation between neighboring areas when the activities therein are incompatible with each other. Examples include:

- Volatile organic chemical handling areas, including sample preparation and waste disposal, and volatile organic chemical analysis areas.

Access to and use of all areas affecting the quality of analytical testing is defined and controlled by secure access to the laboratory building as described below in the Building Security section.

Adequate measures are taken to ensure good housekeeping in the laboratory and to ensure that any contamination does not adversely affect data quality. These measures include regular cleaning to control dirt and dust within the laboratory.

Work areas are available to ensure an unencumbered work area. Work areas include:

- Access and entryways to the laboratory.
- Sample receipt areas.
- Sample storage areas.
- Chemical and waste storage areas.
- Data handling and storage areas.
- Sample processing areas.
- Sample analysis areas.

19.4 FLOOR PLAN

A laboratory floor plan can be found in Appendix 2.

19.5 BUILDING SECURITY *(ISO17025 5.3.4)*

Building keycards are issued to every employee, keys and alarm codes are distributed to several employees as necessary.

Visitors to the laboratory sign in and out in a visitor's logbook. A visitor is defined as any person who visits the laboratory who is not an employee of the laboratory. In addition to signing into the laboratory, the Environmental, Health and Safety Manual contains requirements for visitors and vendors. There are specific safety forms that must be reviewed and signed.

Visitors (with the exception of company employees) are escorted by laboratory personnel at all times, or the location of the visitor is noted in the visitor's logbook.

SECTION 20.0

TEST METHODS AND METHOD VALIDATION *(NELAC 5.5.4)*

(ISO17025 5.4.1)

20.1 OVERVIEW *(ISO17025 5.4.1; 5.4.5.1)*

The laboratory uses methods that are appropriate to meet our clients' requirements and that are within the scope of the laboratory's capabilities. These include sampling, handling, transport, storage and preparation of samples, and, where appropriate, an estimation of the measurement of uncertainty as well as statistical techniques for analysis of environmental data.

Instructions are available in the laboratory for the operation of equipment as well as for the handling and preparation of samples. All instructions, Standard Operating Procedures (SOPs), reference methods and manuals relevant to the working of the laboratory are readily available to all staff. Deviations from published methods are documented (with justification) in the laboratory's approved SOPs. SOPs are submitted to clients for review at their request. Significant deviations from published methods require client approval and regulatory approval where applicable.

20.2 STANDARD OPERATING PROCEDURES (SOPs) *(ISO17025 4.3.3.1; 5.4.2)*

The laboratory maintains SOPs that accurately reflect all phases of the laboratory such as assessing data integrity, corrective actions, handling customer complaints as well as all analytical methods and sampling procedures. The method SOPs are derived from the most recently promulgated/approved, published methods and are specifically adapted to the laboratory facility. Modifications or clarifications to published methods are clearly noted in the SOPs. All SOPs are controlled in the laboratory (refer to Section 6 on Document Control):

- All SOPs contain a revision number, effective date, and appropriate approval signatures. Controlled copies are available to all staff.
- Procedures for preparation, review, revision and control are incorporated by reference to TestAmerica's Corporate SOP entitled 'Writing a Standard Operating Procedure', No. CW-Q-S-002 and the laboratory SOPs *UP-QA-032*, SOP Change Protocol and SOP *UP-QA-006*, Document Control.
- SOPs are reviewed at a minimum of every 2 years (annually for Drinking Water and DoD SOPs), and where necessary, revised to ensure continuing suitability and compliance with applicable requirements.

20.3 LABORATORY METHODS MANUAL

For each test method, the laboratory shall have available the published referenced method as well as the laboratory developed SOP.

Note: If more stringent standards or requirements are included in a mandated test method or regulation than those specified in this manual, the laboratory shall demonstrate that such requirements are met. If it is not clear which requirements are more stringent, the standard from the method or regulation is to be followed. Any exceptions or deviations from the referenced methods or regulations are noted in the specific analytical SOP.

The laboratory maintains an SOP Index for both technical and non-technical SOPs. Technical SOPs are maintained to describe a specific test method. Non-technical SOPs are maintained to describe functions and processes not related to a specific test method.

20.4 SELECTION OF METHODS *(ISO17025 5.4.1; 5.4.2; 5.4.4; 5.4.5.2; 5.4.5.3)*

Since numerous methods and analytical techniques are available, continued communication between the client and laboratory is imperative to assure the correct methods are utilized. Once client methodology requirements are established, this and other pertinent information is summarized by the Project Manager. These mechanisms ensure that the proper analytical methods are applied when the samples arrive for log-in. For non-routine analytical services (e.g., special matrices, non-routine compound lists, etc.), the method of choice is selected based on client needs and available technology. The methods selected should be capable of measuring the specific parameter of interest, in the concentration range of interest, and with the required precision and accuracy.

20.4.1 Sources of Methods *(ISO17025 5.4.2; 5.4.4; 5.4.5.1)*

Routine analytical services are performed using standard EPA-approved methodology. In some cases, modification of standard approved methods may be necessary to provide accurate analyses of particularly complex matrices. When the use of specific methods for sample analysis is mandated through project or regulatory requirements, only those methods shall be used.

When clients do not specify the method to be used or methods are not required, the methods used will be clearly validated and documented in an SOP and available to clients and/or the end user of the data.

20.4.1.1 The analytical methods used by the laboratory are those currently accepted and approved by the U. S. EPA and the state or territory from which the samples were collected. Reference methods include:

- *Method 1664, Revision A: N-Hexane Extractable Material (HEM; Oil and Grease) and Silica Gel Treated N-Hexane Extractable Material (SGT-HEM); Non-polar Material) by Extraction and Gravimetry, EPA-821-R-98-002, February 1999*
- *Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act, and Appendix A-C; 40 CFR Part 136, USEPA Office of Water. Revised as of July 1, 1995, Appendix A to Part 136 - Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater (EPA 600 Series)*
- *Methods for Chemical Analysis of Water and Wastes, EPA 600 (4-79-020), 1983.*
- *Methods for the Determination of Inorganic Substances in Environmental Samples, EPA-600/R-93/100, August 1993.*
- *Methods for the Determination of Metals in Environmental Samples, EPA/600/4-91/010, June 1991. Supplement I: EPA-600/R-94/111, May 1994.*
- *Methods for the Determination of Organic Compounds in Drinking Water, EPA-600/4-88-039, December 1988, Revised, July 1991, Supplement I, EPA-600-4-90-020, July 1990, Supplement II, EPA-600/R-92-129, August 1992. Supplement III EPA/600/R-95/131 - August 1995 (EPA 500 Series) (EPA 500 Series methods)*

- *Technical Notes on Drinking Water Methods, EPA-600/R94-173, October 1994*
- *NIOSH Manual of Analytical Methods, 4th ed., August 1994.*
- *Statement of Work for Inorganics Analysis, SOM and ISM, current versions, USEPA Contract Laboratory Program Multi-media, Multi-concentration.*
- *Standard Methods for the Examination of Water and Wastewater, 18th/19th/20th edition/ on-line edition; Eaton, A.D. Clesceri, L.S. Greenberg, A.E. Eds; American Water Works Association, Water Pollution Control Federation, American Public Health Association: Washington, D.C.*
- *Test Methods for Evaluating Solid Waste Physical/Chemical Methods (SW846), Third Edition, September 1986, Final Update I, July 1992, Final Update IIA, August 1993, Final Update II, September 1994; Final Update IIB, January 1995; Final Update III, December 1996. Final Update IV, January 2008.*
- *Annual Book of ASTM Standards, American Society for Testing & Materials (ASTM), Philadelphia, PA.*
- *Manual for the Certification of Laboratories Analyzing Drinking Water (EPA 815-R-05-004, January 2005)*
- *Code of Federal Regulations (CFR) 40, Parts 136, 141, 172, 173, 178, 179 and 261*

The laboratory reviews updated versions to all the aforementioned references for adaptation based upon capabilities, instrumentation, etc., and implements them as appropriate. As such, the laboratory strives to perform only the latest versions of each approved method as regulations allow or require.

Other reference procedures for non-routine analyses may include methods established by specific states (e.g., Underground Storage Tank methods), ASTM or equipment manufacturers. Sample type, source, and the governing regulatory agency requiring the analysis will determine the method utilized.

The laboratory shall inform the client when a method proposed by the client may be inappropriate or out of date. After the client has been informed, and they wish to proceed contrary to the laboratory's recommendation, it will be documented.

20.4.2 Demonstration of Capability *(ISO17025 5.4.3; 5.4.4)*

Before the laboratory may institute a new method and begin reporting results, the laboratory shall confirm that it can properly operate the method. In general, this demonstration does not test the performance of the method in real world samples, but in an applicable and available clean matrix sample. If the method is for the testing of analytes that are not conducive to spiking, demonstration of capability may be performed on quality control samples.

20.4.2.1 A demonstration of capability is performed whenever there is a change in instrument type (e.g., new instrumentation), method or personnel.

20.4.2.2 The initial demonstration of capability must be thoroughly documented and approved by the Department Manager/Supervisor and QA Manager prior to independently analyzing client samples. All associated documentation must be retained in accordance with the laboratories archiving procedures (refer to Section 15, Control of Records).

20.4.2.3 The laboratory must have an approved SOP, demonstrate satisfactory performance, and conduct a method detection limit study (when applicable). There may be other requirements as stated within the published method or regulations (i.e., retention time window study).

Note: In some instances, a situation may arise where a client requests that an unusual analyte be reported using a method where this analyte is not normally reported. If the analyte is being reported for regulatory purposes, the method must meet all procedures outlined within this QA Manual (SOP, MDL, and Demonstration of Capability). If the client states that the information is not for regulatory purposes, the result may be reported as long as the following criteria are met:

- The instrument is calibrated for the analyte to be reported using the criteria for the method and ICV/CCV criteria are met (unless an ICV/CCV is not required by the method or criteria are per project DQOs).
- The laboratory's nominal or default reporting limit (RL) is equal to the Limit of Quantitation (LOQ), which must be at or above the lowest non-zero standard in the calibration curve and achieve an adequate level of precision and bias. Project RLs are client specified reporting levels which may be higher than the QL. Results reported below the LOQ must be qualified as estimated values. Also see Section 20.6.1.3, Relationship of Limit of Detection (LOD) to Limit of Quantitation (LOQ).
- The client request is documented and the lab informs the client of its procedure for working with unusual compounds. The final report must be footnoted and or narrated: *Reporting Limit (RL) or Limit of Quantitation (LOQ) is based on the low standard of the calibration curve.*

20.4.3 Initial Demonstration of Capability (IDOC) Procedures

20.4.3.1 The spiking standard used must be prepared independently from those used in instrument calibration.

20.4.3.2 The analyte(s) shall be diluted in a volume of clean matrix sufficient to prepare four aliquots at the concentration specified by a method or the laboratory SOP. The laboratory will utilize the 'split and spike' procedure, i.e., a clean water or soil matrix shall be split into four portions first and then each portion is individually spiked.

20.4.3.3 At least four aliquots shall be prepared (including any applicable clean-up procedures) and analyzed according to the test method (either concurrently or over a period of several days).

20.4.3.4 Using all of the results, calculate the mean recovery in the appropriate reporting units and the standard deviations for each parameter of interest.

20.4.3.5 When it is not possible to determine the mean and standard deviations, such as for presence, absence and logarithmic values, the laboratory will assess performance against criteria described in the Method SOP.

20.4.3.6 Compare the information obtained above to the corresponding acceptance criteria for precision and accuracy in the test method (if applicable) or in laboratory generated acceptance criteria (LCS or interim criteria) if there is no mandatory criteria established. If any one of the parameters do not meet the acceptance criteria, the performance is unacceptable for that parameter.

20.4.3.7 When one or more of the tested parameters fail at least one of the acceptance criteria, the analyst must proceed according to either option listed below:

- Locate and correct the source of the problem and repeat the test for all parameters of interest beginning with 20.4.3.3 above.
- Beginning with 20.4.3.3 above, repeat the test for all parameters that failed to meet criteria. Repeated failure, however, will confirm a general problem with the measurement system. If this occurs, locate and correct the source of the problem and repeat the test for all compounds of interest beginning with 20.4.3.1 above.

Note: Results of successive LCS analyses can be used to fulfill the DOC requirement.

A certification statement (see Figure 20-1 as an example) shall be used to document the completion of each initial demonstration of capability. A copy of the certification statement is archived in the analyst's training folder.

Methods on line prior to the effective date of this Section shall be updated to the procedures outlined above as new analysts perform their demonstration of capability. A copy of the new record will replace that which was used for documentation in the past. At a minimum, the precision and accuracy of four mid-level laboratory control samples must have been compared to the laboratory's quality control acceptance limits.

20.5 LABORATORY DEVELOPED METHODS AND NON-STANDARD METHODS *(ISO17025 5.4.2; 5.4.4; 5.4.5.2; 5.4.5.3)*

Any new method developed by the laboratory must be fully defined in an SOP/Methods Manual (Section 20.2) and validated by qualified personnel with adequate resources to perform the method. Method specifications and the relation to client requirements must be clearly conveyed to the client if the method is a non-standard method (not a published or routinely accepted method). The client must also be in agreement to the use of the non-standard method. The information included in the checklist below (Figure 20-2) is needed before samples are accepted for analysis by a new method.

20.6 VALIDATION OF METHODS *(ISO17025 5.4.2; 5.4.4; 5.4.5.2; 5.4.5.3)*

Validation is the confirmation by examination and the provision of objective evidence that the particular requirements for a specific intended use are fulfilled.

All non-standard methods, laboratory designed/developed methods, standard methods used outside of their scope, and major modifications to published methods must be validated to confirm they are fit for their intended use. The validation will be as extensive as necessary to meet the needs of the given application. The results are documented with the validation procedure used and contain a statement as to the fitness for use.

20.6.1 Method Validation and Verification Activities for All New Methods

While method validation can take various courses, the following activities can be required as part of method validation. Method validation records are designated QC records and are archived accordingly.

20.6.1.1 Determination of Method Selectivity

Method selectivity is the demonstrated ability to discriminate the analyte(s) of interest from other compounds in the specific matrix or matrices from other analytes or interference. In some cases to achieve the required selectivity for an analyte, a confirmation analysis is required as part of the method.

20.6.1.2 Determination of Method Sensitivity

Sensitivity can be both estimated and demonstrated. Whether a study is required to estimate sensitivity depends on the level of method development required when applying a particular measurement system to a specific set of samples. Where estimations and/or demonstrations of sensitivity are required by regulation or client agreement, such as the procedure in 40 CFR Part 136 Appendix B, under the Clean Water Act, these shall be followed. The laboratory determinations of MDLs/DLs are described in Section 20.7.

20.6.1.3 Relationship of Limit of Detection (LOD) to the Limit of Quantitation (LOQ)

An important characteristic of expression of sensitivity is the difference in the LOD and the LOQ. The LOD is the minimum level at which the presence of an analyte can be reliably concluded. The LOQ is the minimum concentration of analyte that can be quantitatively determined with adequate precision and bias. For most instrumental measurement systems, there is a region where semi-quantitative data is generated around the LOD (both above and below the estimated MDL/DL or LOD) and below the LOQ. In this region, detection of an analyte may be confirmed but quantification of the analyte is unreliable within the accuracy and precision guidelines of the measurement system. When an analyte is detected below the LOQ, and the presence of the analyte is confirmed by meeting the qualitative identification criteria for the analyte, the analyte can be reliably reported, but the amount of the analyte can only be estimated. If data is to be reported in this region, it must be done so with a qualification that denotes the semi-quantitative nature of the result.

20.6.1.4 Determination of Interferences

A determination that the method is free from interferences in a blank matrix is performed.

20.6.1.5 Determination of Range

Where appropriate to the method, the quantitation range is determined by comparison of the response of an analyte in a curve to established or targeted criteria. Generally the upper quantitation limit is defined by the highest acceptable calibration concentration. The lower limit of quantitation or LOQ cannot be lower than the lowest non-zero calibration level, and can also be constrained by required levels of bias and precision also see Section 20.4.2. The ICP and ICPMS instrumentation utilize a low range verification check standard at the beginning of each analytical run as specified per method or project specific criteria.

20.6.1.6 Determination of Accuracy and Precision

Accuracy and precision studies are generally performed using replicate analyses, with a resulting percent recovery and measure of reproducibility (standard deviation, relative standard deviation) calculated and measured against a set of target criteria.

20.6.1.7 Documentation of Method

The method is formally documented in an SOP. If the method is a minor modification of a standard laboratory method that is already documented in an SOP, an SOP Attachment describing the specific differences in the new method is acceptable in place of a separate SOP.

20.6.1.8 Continued Demonstration of Method Performance

Continued demonstration of Method Performance is addressed in the SOP. Continued demonstration of method performance is generally accomplished by batch specific QC samples such as LCS, method blanks or PT samples.

20.7 METHOD DETECTION LIMITS (MDL/DL)/ LIMITS OF DETECTION (LOD)

(ISO17025 5.4.5.3)

Method detection limits (MDL/DL) are initially determined in accordance with 40 CFR Part 136, Appendix B or alternatively by other technically acceptable practices that have been accepted by regulators. The MDL/DL theoretically represents the concentration level for each analyte within a method at which the Analyst is 99% confident that the true value is not zero. The DL is further defined in the DoD QSM version 4.2 as representing a concentration that is 99% confident that it is distinquishable from a blank. The MDL/DL is determined for each analyte and associated surrogates initially during the method validation process and updated as required in the analytical methods, whenever there is a significant change in the procedure or equipment, or based on project specific requirements (refer to 20.7.10). The analyst prepares at least seven replicates of solution spiked at one to five times the estimated method detection limit (most often at the lowest standard in the calibration curve) into the applicable matrix with all the analytes of interest (including the surrogates for DoD QSM compliance). Each of these aliquots is extracted (including any applicable clean-up procedures) and analyzed in the same manner as the samples. Where possible, the seven replicates should be analyzed over 2-4 days to provide a more realistic MDL/DL. Refer to the laboratory SOP UP-QA-017, Method Detection Limits (MDL/DL) and the Corporate SOP No. CA-Q-S-006 for additional details and alternative approaches to the laboratory's MDL/DL process that may be used.

20.7.1 MDLs/DLs are initially performed for each individual instrument and non-microbiological method analysis. Unless there are requirements to the contrary, the laboratory will use the highest calculated MDL/DL for all instruments used for a given method as the MDL/DL for reporting purposes. This MDL/DL is not required for methods that are not readily spiked (e.g. pH, turbidity, etc.) or where the lab does not report values to the MDL/DL. Titration and gravimetric methods where there is no additional preparation involved, the MDL is based on the lowest discernable unit of measure that can be observed.

20.7.2 MDLs/DLs must be run against acceptable instrument QC, including ICV's and Tunes as per the requirement of the method that is being analyzed. This is to insure that the instrument is in proper working condition and falsely high or low MDLs/DLs are not calculated.

20.7.3 Use only clean matrix which is free of target analytes (e.g.: Laboratory reagent water, Ottawa Sand) unless a project specific MDL/DL is required in a field sample matrix.

20.7.4 The calculated MDL/DL can not be greater than the spike amount.

20.7.5 If the most recent calculated MDL/DL does not permit qualitative identification of the analyte then the laboratory may use technical judgment for establishing the MDL/DL (e.g., calculate what level would give a qualitative ID, compare with IDL (Section 20.8), spike at a level where qualitative ID is determined and assign that value as MDL/DL, minimum sensitivity requirements, Standard deviation of method blanks over time, etc.). These alternate verification procedures are documented in the laboratory SOP *UP-QA-017*, Method Detection Limits (MDLs/DLs).

20.7.6 Each of the 7 spikes must be qualitatively identifiable (e.g., appear in both columns for dual column methods, characteristic ions for GCMS mass spectra, etc). Manual integrations to force the baseline for detection are not allowed. The MDL/DL determination may be performed by the procedure in 40 CFR Part 136, Appendix B or alternatively by other technically acceptable practices (e.g., method blanks over time, single standard spikes that have been subjected to applicable sample prep processes, etc.). The procedures utilized are documented in the laboratory SOP UP-QA-017, Method Detection Limits (MDLs/DLs).

20.7.7 The initial MDL/DL is calculated as follows:

$$MDL/DL = t_{(n-1,\ 1-a\ =\ 0.99)} \times (\text{Standard Deviation of replicates})$$

where $t_{(n-1,\ 1-a\ =\ 0.99)} = 3.143$ for seven replicates.
where $t_{(n-1,\ 1-a\ =\ 0.99)} = 2.998$ for eight replicates.

20.7.8 In conjunction with the initial MDL/DL determination a MDL verification check standard (MDLV) is analyzed. The DoD QSM version 4.2 requires a quarterly verification (MDLV) on all instruments for each method represented on the DoD ELAP Scope of Accreditation. The concentration of the MDLV reflects the laboratory's Limit of Detection (LOD). DoD QSM defines the LOD as a standard that is detected at least 99% of the time at a concentration 2-3 times higher than the MDL/DL. If the MDLV is not detected, and there were no obvious instrument problems, the lab must elevate the LOD for that method/analyte.

20.7.9 Because of the inherent variability in results outside of the calibration range, TestAmerica does not recommend the reporting of results below the lowest calibration point in a curve; however, it is recognized that some projects and agencies require the reporting of results below the RL/LOQ. Any result that falls between the MDL/DL and the RL/LOQ, when reported, will be qualified as an estimated value. For DoD QSM compliance, non-detect values will be reported as less than the LOD. Positive results between the LOQ and the MDL/DL will be J-flagged as estimated.

20.7.10 Detections reported down to the MDL/DL must be qualitatively identified.

20.7.11 MDLs/DLs and RLs/LOQs are adjusted in LIMs based on moisture content and sample aliquot size.

20.8 INSTRUMENT DETECTION LIMITS (IDL)

20.8.1 The IDL is sometimes used to assess the reasonableness of the MDLs or in some cases required by the analytical method or program requirements. IDLs are most used in metals analyses but may be useful in demonstration of instrument performance in other areas.

20.8.2 IDLs are calculated to determine an instrument's sensitivity independent of any preparation method. IDLs are calculated either using 7 replicate spike analyses, like MDL but without sample preparation, or by the analysis of 10 instrument blanks and calculating 3 x the absolute value of the standard deviation.

20.8.3 If IDL is > than the MDL, it may be used as the reported MDL.

20.9 VERIFICATION OF DETECTION AND REPORTING LIMITS

20.9.1 Once an MDL/DL is established, it must be verified, on each instrument on a quarterly basis per the requirements of the DoD QSM version 4.2, by analyzing a quality control sample (MDLV), prepared as a sample, at a concentration approximately ½ the RL/LOQ which is equivalent to the LOD. This verification (MDLV) does not apply to methods that are not readily spiked (e.g. pH, turbidity, etc.) or where the lab does not report to the MDL. If the MDL/DL does not verify, then the lab will not report to the MDL/DL, or redevelop their MDL or use the level where qualitative identification is established (See 20.7). MDLs/DLs must be verified at least annually. Quarterly verification is required for methods listed on the DoD ELAP Scope of Accreditation. This verification (MDLV) is extracted and analyzed along with the annual method detection limit study and thereafter on a quarterly basis with the LOQ verification for DoD QSM compliance. Refer to the laboratory SOP *UP-QA-017*, Method Detection Limits (MDLs/DLs) for further details.

20.9.2 Limit of Quantitation (LOQ) is the lowest concentration that produces a quantitative result within specified limits of precision and bias. For DoD projects, the LOQ shall be set at or above the concentration of the lowest initial calibration standard. The laboratory's routine reporting limit is equal to the LOQ, unless project documents specifies a higher concentration to be used as the project-specific reporting limit.

When the lab establishes a Limit of Quantitation (LOQ), it must be initially verified by the analysis of a low level standard or QC sample at a concentration at or above the lowest calibration standard and quarterly thereafter for DoD QSM 4.2 compliance. The acceptance criteria of ± 30% of the mean will be applied except for those poor performers that are identified in the specific methods and/or SOPs. The QC standard will be designated in LIMs as a Method Reporting Limit check standard (MRL). There are method, program or state requirements that specify the running and reporting of the method reporting limit check standard in sequence with the analytical runs associated with their samples. This special project requirement is communicated to the laboratory via project and Job login notes.

The laboratory's nominal Reporting Limit (RL) or Limit of Quantitation (LOQ) should as general rule be between 3 and 5 times the MDL/DL. To be usable for a DoD project, the laboratory must be able to achieve specific precision and bias objectives at the LOQ. State or program specific MDL/DL criteria may be required, refer to the laboratory SOP *UP-QA-017*, Method Detection Limits (MDLs/DLs) for further details. For DoD QSM version 4.2 compliance, the Limit of Quantitation (LOQ) standard, must be analyzed and verified on a quarterly basis. The laboratory will analyze it in conjunction with the Limit of Detection (LOD) verification standard (MDLV) defined in section 20.7.8.

20.10 RETENTION TIME WINDOWS

Most organic analyses and some inorganic analyses use chromatography techniques for qualitative and quantitative determinations. For every chromatography analysis each analyte will have a specific time of elution from the column to the detector. This is known as the analyte's retention time. The variance in the expected time of elution is defined as the retention time window. As the key to analyte identification in chromatography, retention time windows must be established on every column for every analyte used for that method as specified in the reference method. These records are kept with the files associated with an instrument for later quantitation of the analytes. Complete details are available in the laboratory SOPs.

For GC, HPLC and IC methods, there must be sufficient separation between analyte peaks so as to not misidentify analytes. In the mid-level standard, the distance between the valley and peak height cannot be any less than 25% of the sum of the peak heights of the analytes. This also applies to GCMS in the case where the two compounds share the same quantitation ion.

Note: Some analytes do not separate sufficiently to be able to identify or quantitate them as separate analytes (e.g. m-xylene and p-xylene) and are quantitated and reported as a single analyte (e.g. m,p-xylenes).

Once the analyst has determined that the instrument is in optimum working condition through calibration and calibration verification procedures, he or she uses a mid-range calibration or calibration verification standard to establish the retention times for each of the individual analytes in a method. The analyst makes three injections of the same standard over a 72-hour (24 hr period for 300.0) period, tabulating the retention times for each analyte for each of the three injections. The width of retention time window is normally the average absolute retention time ± 3 Standard Deviations. A peak outside the retention time window will not be identified by the computer as a positive match of the analyte of interest.

It is possible for the statistically calculated RT window to be too tight and need to be adjusted based on analyst experience. In these instances method default retention time windows may be used (e.g., for 8000 series methods a default of 0.03 minutes may be used, and EPA CLP 0.05 minutes is used). The same concept is applied when any peak outside of that window will not be identified by the computer as a positive match.

The calibration verification standard at the beginning of a run may be used to adjust the RT for an analyte. This is essentially re-centering the window but the size of the window remains the same. The RTs are verified when all analytes are within their RT windows and are properly identified.

20.11 EVALUATION OF SELECTIVITY

The laboratory evaluates selectivity by following the checks within the applicable analytical methods, which include mass spectral tuning, second column confirmation, ICP interelement interference checks, chromatography retention time windows, sample blanks, spectrochemical, atomic absorption or fluorescence profiles, co-precipitation evaluations and specific electrode response factors.

20.12 ESTIMATION OF UNCERTAINTY OF MEASUREMENT *(ISO17025 5.1.1; 5.1.2; 5.4.6.1; 5.4.6.2; 5.4.6.3)*

20.12.1 Uncertainty is "a parameter associated with the result of a measurement, that characterizes the dispersion of the values that could reasonably be attributed to the measurand" (as defined by the International Vocabulary of Basic and General Terms in Metrology, ISO Geneva, 1993, ISBN 92-67-10175-1). Knowledge of the uncertainty of a measurement provides additional confidence in a result's validity. Its value accounts for all the factors which could possibly affect the result, such as adequacy of analyte definition, sampling, matrix effects and interferences, climatic conditions, variances in weights, volumes, and standards, analytical procedure, and random variation. Some national accreditation organizations require the use of an "expanded uncertainty": the range within which the value of the measurand is believed to lie within at least a 95% confidence level with the coverage factor k=2.

20.12.2 Uncertainty is not error. Error is a single value, the difference between the true result and the measured result. On environmental samples, the true result is never known. The measurement is the sum of the unknown true value and the unknown error. Unknown error is a combination of systematic error, or bias, and random error. Bias varies predictably, constantly, and independently from the number of measurements. Random error is unpredictable, assumed to be Gaussian in distribution, and reducible by increasing the number of measurements.

20.12.3 The minimum uncertainty associated with results generated by the laboratory can be determined by using the Laboratory Control Sample (LCS) accuracy range for a given analyte. The LCS limits are used to assess the performance of the measurement system since they take into consideration all of the laboratory variables associated with a given test over time (except for variability associated with the sub sampling and the variability due to matrix effects). The percent recovery of the LCS is compared either to the method-required LCS accuracy limits or to the statistical, historical, in-house LCS accuracy limits.

20.12.4 To calculate the uncertainty for the specific result reported, multiply the result by the decimal of the lower end of the LCS range percent value for the lower end of the uncertainty range, and multiply the result by the decimal of the upper end of the LCS range percent value for the upper end of the uncertainty range. These calculated values represent a 99%-certain range for the reported result. As an example, suppose that the result reported is 1.0 mg/l, and the LCS percent recovery range is 50 to 150%. The uncertainty range would be 0.5 to 1.5 mg/l, which could also be written as 1.0 +/- 0.5 mg/l. Refer to the laboratory SOP *UP-QA-040*, Quality Assurance – Measurement Uncertainty for additional details.

20.12.5 In the case where a well recognized test method specifies limits to the values of major sources of uncertainty of measurement (e.g. 524.2, 525, etc) and specifies the form of presentation of calculated results, no further discussion of uncertainty is required.

20.13 SAMPLE REANALYSIS GUIDELINES *(ISO17025 5.9.1)*

Because there is a certain level of uncertainty with any analytical measurement, a sample reanalysis may result in either a higher or lower value from an initial sample analysis. There are also variables that may be present (e.g., sample homogeneity, analyte precipitation over time, etc.) that may affect the results of a reanalysis. Based on the above comments, the laboratory will reanalyze samples at a client's request with the following caveats. **Client specific**

Contractual Terms & Conditions for reanalysis protocols may supercede the following items.

- Homogenous samples: If a reanalysis agrees with the original result to within the RPD limits for MS/MSD or Duplicate analyses, or within ± 1 reporting limit for samples ≤ 5x the reporting limit, the original analysis will be reported. At the client's request, both results may be reported on the same report but not on two separate reports.
- If the reanalysis does not agree (as defined above) with the original result, then the laboratory will investigate the discrepancy. Reanalysis of the sample a third time for confirmation if sufficient sample is available may be required. An NCM will be written to document the anomalies and any actions taken.
- Any potential charges related to reanalysis are discussed in the contract terms and conditions or discussed at the time of the request. The client will typically be charged for reanalysis unless it is determined that the lab was in error.
- Due to the potential for increased variability, reanalysis may not be applicable to Non-homogenous, Encore, and Sodium Bisulfate preserved samples. See the Area Supervisor/Manager or Laboratory Director if unsure.

20.14 CONTROL OF DATA *(ISO17025 5.4.7.1)*

The laboratory has policies and procedures in place to ensure the authenticity, integrity, and accuracy of the analytical data generated by the laboratory.

20.14.1 Computer and Electronic Data Related Requirements *(ISO17025 5.4.7.2)*

The three basic objectives of our computer security procedures and policies are shown below. More detail is outlined in laboratory SOP *UP-IS-014*, TestAmerica Chicago IT Procedures and Processes. The laboratory is currently running the TALs LIMS which is a custom in-house developed LIMS system that has been highly customized to meet the needs of the laboratory. It is referred to as LIMS for the remainder of this section. The LIMS utilizes Microsoft SQL Server which is an industry standard relational database platform. It is referred to as Database for the remainder of this section.

20.14.1.1 Maintain the Database Integrity: Assurance that data is reliable and accurate through data verification (review) procedures, password-protecting access, anti-virus protection, data change requirements, as well as an internal LIMS permissions procedure.

- LIMS Database Integrity is achieved through data input validation, internal user controls, and data change requirements.
- Spreadsheets and other software developed in-house must be verified with documentation through hand calculations prior to use.

20.14.1.2 Ensure Information Availability: Protection against loss of information or service through scheduled back-ups, stable file server network architecture, secure storage of media, line filter, Uninterruptible Power Supply (UPS), and maintaining older versions of software as revisions are implemented.

20.14.1.3 Maintain Confidentiality: Ensure data confidentiality through physical access controls when electronically transmitting data.

20.14.2 Data Reduction

The complexity of the data reduction depends on the analytical method and the number of discrete operations involved (e.g., extractions, dilutions, instrument readings and concentrations). The analyst calculates the final results from the raw data or uses appropriate computer programs to assist in the calculation of final reportable values. Calculations are independently verified by appropriate laboratory staff. Calculations and data reduction steps for various methods are summarized in the respective analytical SOPs or program requirements.

For manual data entry, e.g., Wet Chemistry, the data is reduced by the analyst and then verified by the Department Manager, Supervisor or alternate analyst prior to updating the data in LIMS. The spreadsheets, or any other type of applicable documents, are signed by both the analyst and alternate reviewer to confirm the accuracy of the manual entry(s).

Manual integration of peaks will be documented and reviewed and the raw data will be flagged in accordance with the TestAmerica Corporate SOP CA-Q-S-002, *Acceptable Manual Integration Practices*.

Analytical results are reduced to appropriate concentration units specified by the analytical method, taking into account factors such as dilution, sample weight or volume, etc. Blank correction will be applied only when required by the method or per manufacturer's indication; otherwise, it should not be performed. Calculations are independently verified by appropriate laboratory staff. Calculations and data reduction steps for various methods are summarized in the respective analytical SOPs or program requirements.

20.14.2.1 All raw data must be retained in the worklist folder, computer file (if appropriate), and/or runlog. All criteria pertinent to the method must be recorded. Raw data must include all pertinent information needed to reconstruct the final result from the bench notes or instrument file reported value, and accurately reflect the method used, instrument ID, analyst, and time of preparation and analysis. It may include annotated instrument hard-copies, bench notes, completed bound data book pages, or pages printed directly from the TALs LIMS system. The documentation is recorded at the time observations or calculations are made and must be signed or initialed/dated (month/day/year). It must be easily identifiable who performed which tasks if multiple people were involved.

20.14.2.2 In general, concentration results are reported in milligrams per liter (mg/l) or micrograms per liter (µg/l) for liquids and milligrams per kilogram (mg/kg) or micrograms per kilogram (µg/kg) for solids. For values greater than 10,000 mg/l, results can be reported in percent, i.e., 10,000 mg/l = 1%. Default reporting units are defined in each lab SOP and in the analytical method setup in LIMs. The reporting unit can also be designated at the project/job/method level in LIMs.

20.14.2.3 In reporting, the analyst or the instrument output records the raw data result using values of known certainty plus one uncertain digit. If final calculations are performed external to LIMS, the results should be entered in LIMS with at least three significant figures. In general, results are reported to 2 significant figures on the final report.

20.14.2.4 For those methods that do not have an instrument printout or an instrumental output compatible with the LIMS System, the raw results and dilution factors are entered directly into LIMS by the analyst, and the software calculates the final result for the analytical report. In all

cases, rounding should be limited to the final result as generated by TALs according to the method set-up.

20.14.2.5 The laboratory strives to import data directly from instruments or calculation spreadsheets to ensure that the reported data are free from transcription and calculation errors. For those analyses with an instrumental output compatible with the LIMS, the raw results and dilution factors are transferred into LIMS electronically after the initial data review has been preformed by the analyst according to review procedures identified in the individual laboratory SOPs. (For example, included in the organics data review is the evaluation of the target system's quantitation report/spectra for each sample and analyte, actions such as removing unrequested or poor spectrally-matched compounds may be necessary prior to data import into TALs). Depending on the data to be reviewed, the analyst either prints a copy or views the data on screen to check for errors in accordance with the laboratory's analytical SOPs and data review checklists. Secondary review of the data is done and recorded on the same checklist by a peer reviewer or supervisor. The raw data, including the checklist, instrument print-outs, and manual entries, and electronic files are retained for easy retrieval in accordance with the laboratory's record and retention policy outlined in Seciton 15.5.

20.14.3 Logbook / Worksheet Use Guidelines

Logbooks and worksheets are filled out 'real time' and have enough information on them to trace the events of the applicable analysis/task. (e.g. calibrations, standards, analyst, sample ID, date, time on short holding time tests, temperatures when applicable, calculations are traceable, etc.)

- Corrections are made following the procedures outlined in Section 13.
- Logbooks are controlled by the QA department. A record is maintained of all logbooks in the lab.
- Unused portions of pages must be "Z"'d out, signed and dated.
- Worksheets are created with the approval of the Department Manager, Department Supervisor and QA Manager at the facility. The QA Manager controls all worksheets following the procedures in Section 6.

20.14.4 Review / Verification Procedures

Review procedures are out lined in several SOPs [e.g. Sample Control, Project Management, QA and Method Specific SOPs] to ensure that reported data are free from calculation and transcription errors, that QC parameters have been reviewed and evaluated before data is reported. The laboratory also follows the corporate SOP on Manual Integrations to ensure the authenticity of the data [SOP No. CA-Q-S-002, Acceptable Manual Integration Practices]. The general review concepts are discussed below, more specific information can be found in the laboratory SOPs.

20.14.4.1 The data review process at TestAmerica Chicago starts at Sample Log-In, where personnel review chain-of-custody forms and input the sample information and required analyses into the TALs LIMs system. The Project Managers perform the final review of the chain-of-custody forms, the analyses logged into TALs and all other inputted information.

20.14.4.2 The next level of data review occurs with the analysts. As results are generated, analysts review their work to ensure that the results generated meet QC requirements and relevant EPA methodologies. The analysts transfer the data into the TALs LIMS in the Analyst Desktop module. Where non-compliance is observed, the analyst creates Non-Conformance Memos (NCMs) in TALs. Flags and data qualifiers can be method, project, program or QAPP specific (see Appendix 6 for list of default data qualifiers). The project manager selects a formatter in TALs that will be assigned to the project and any login associated with the project. The flags and qualifiers are then automatically applied to the data when calculated and is reviewed by the analyst as part of the data review process. The analyst documents the initial review on a data review checklist and sets the batch status in LIMs to 1st level review. The second level or peer review of the data is conducted by another individual who has been trained on the review process. This secondary review is documented on the same checklist, making any necessary corrections to the data or additions to the NCMs as necessary. The batch is then set to 2nd level review. All GC/MS Spectra and all manual integrations are reviewed. Manual integrations are also electronically reviewed utilizing auditing software to help ensure compliance to the ethics and manual integration policies.

Examples of items included in the above reviews are as follows:

- QC data are outside the specified control limits for accuracy and precision
- Reviewed sample data does not match with reported results
- Unusual detection limit changes are observed
- Samples having unusually high results
- Samples exceeding a known regulatory limit
- Raw data indicating some type of contamination or poor technique
- Inconsistent peak integration
- Transcription errors
- Results outside of calibration range

20.14.4.3 *(ISO17025 5.9.1)* When all the individual tests for a department are at 2nd level review for a job, the Supervisor or the Supervisor's designee reviews the entire job, comparing chemical relationships, reviewing for completeness and the presence of appropriate NCMs, and doing an overall evaluation of the results summary.

Examples of chemical relationships evaluated by the supervisor prior to setting data to 'Lab Complete' status include the following:

- Total Results are $\geq$ Dissolved results (e.g. metals)
- Total Solids (TS) $\geq$ TDS or TSS
- TKN $\geq$ Ammonia
- Total Phosphorus $\geq$ Orthophosphate
- COD $\geq$ BOD
- Total Cyanide $\geq$ Amenable Cyanide
- TDS $\geq$ individual anions

If the job has a deliverable designation of Level 3 or Level 4 a data package is prepared prior to setting the job to a 'Lab Complete' status.

Unacceptable analytical results may require reanalysis of the samples. Any problems are brought to the attention of the Project Manager, Department Manager/Supervisor and Quality Assurance Manager for further investigation. Corrective action is initiated whenever necessary.

20.14.4.4 Any project that requires a data package is subject to a tertiary data review for transcription errors, acceptable quality control requirements, correct application of client specific criteria and overall completeness. The report generation staff assembles the preliminary report and the Project Manager or their designee will review the results for appropriateness and completeness, approve and sign the final report. The PM will update the Final Review task and Invoice Review Task to complete. This review and approval ensures that client requirements have been met and that the final report has been properly completed. The accounting personnel will check for invoicing errors prior to its approval. When complete, the report is sent out to the client either in a hardcopy, emailed (.pdf) or facsimile format, depending on the deliverable instructions in the login. (Refer to Section 26 on Reporting Results for additional details).

20.14.4.5 A visual summary of the flow of samples and information through the laboratory, as well as data review and validation, is presented in Figure 20-3.

20.14.5 Manual Integrations

Computerized data systems provide the analyst with the ability to re-integrate raw instrument data in order to optimize the interpretation of the data. Though manual integration of data is an invaluable tool for resolving variations in instrument performance and some sample matrix problems, when used improperly, this technique would make unacceptable data appear to meet quality control acceptance limits. Improper re-integrations lead to legally indefensible data, a poor reputation, or possible laboratory decertification. Because guidelines for re-integration of data are not provided in the methods and most methods were written prior to widespread implementation of computerized data systems, the laboratory trains all analytical staff on proper manual integration techniques using TestAmerica's Corporate SOP (CA-Q-S-002) as the guideline.

20.14.5.1 The analyst must adjust baseline or the area of a peak in some situations, for example when two compounds are not adequately resolved or when a peak shoulder needs to be separated from the peak of interest. The analyst must use professional judgment and common sense to determine when manual integrating is required. Analysts are encouraged to ask for assistance from a senior analyst or manager when in doubt.

20.14.5.2 Analysts shall not increase or decrease peak areas to for the sole purpose of achieving acceptable QC recoveries that would have otherwise been unacceptable. The intentional recording or reporting of incorrect information (or the intentional omission of correct information) is against company principals and policy and is grounds for immediate termination.

20.14.5.3 Client samples, performance evaluation samples, and quality control samples are all treated equally when determining whether or not a peak area or baseline should be manually adjusted.

20.14.5.4 All manual integrations receive a second level review. Manual integrations must be indicated on an expanded scale "after" chromatograms such that the integration performed can be easily evaluated during data review. Expanded scale "before" chromatograms are also

required for all manual integrations on QC parameters (calibrations, calibration verifications, laboratory control samples, internal standards, surrogates, etc.) unless the laboratory has another documented corporate approved procedure in place that can demonstrate an active process for detection and deterrence of improper integration practices.

Figure 20-1.
Example - Demonstration of Capability Documentation

TestAmerica Chicago

Initial Demonstration of Method Capability
Certification Statement

TestAmerica Chicago
2417 Bond Street
University Park, IL 60484

Analyst Name:_____
SOP No.:_____
Method No.:_____
Description:_____
Matrix:_____
Effective Date:_____

We the undersigned certify that:

- 1. The analyst identified above, using the cited test method(s), which is in use at this laboratory for the analyses of samples under the National Environmental Laboratory Accreditation Program, have met the Demonstration of Capability.
- 2. The test method(s) was performed by the analyst identified on this certification.
- 3. A copy of the reference method and laboratory-specific SOP(s) are available for all personnel on-site.
- 4. The data associated with the demonstration capability are true, accurate, complete and self-explanatory.
- 5. All raw data (including a copy of this certification form) necessary to reconstruct and validate these analyses have been retained at the laboratory, and that the associated information is well organized and available for review by authorized assessors.

Analyst	Signature	Date
Supervisor/Manager	Signature	Date
Quality Assurance Manager	Signature	Date

Figure 20-2.

Example - New Instrument / Method / Additional Analyte Checklist

TESTAMERICA CHICAGO
INSTRUMENT VALIDATON CHECKLIST

Instrument Type: _____
Model#:_____
Serial #:_____
Lab Equip Code: _____ LIMS Equip Code: _____

Installation Date: _____
Installed By: _____

Instrument installed per specifications. Operational and functional per install guidelines.
Signature/Date of installer: _____

Outstanding items yet to be completed (If applicable):_____
_____
_____
_____

Completion Date: _____
Signature/Date Lab Representative: _____

Instrument passes all initial required lab checks and calibrations as appropriate for method of analysis: _____

Appropriate MDL's as applicable per method complete: _____
*MDL's must be forwarded to the QA Dept. for Validation to be considered complete.

Methods to be analyzed (may change over time): _____
_____

In-Service Date: _____

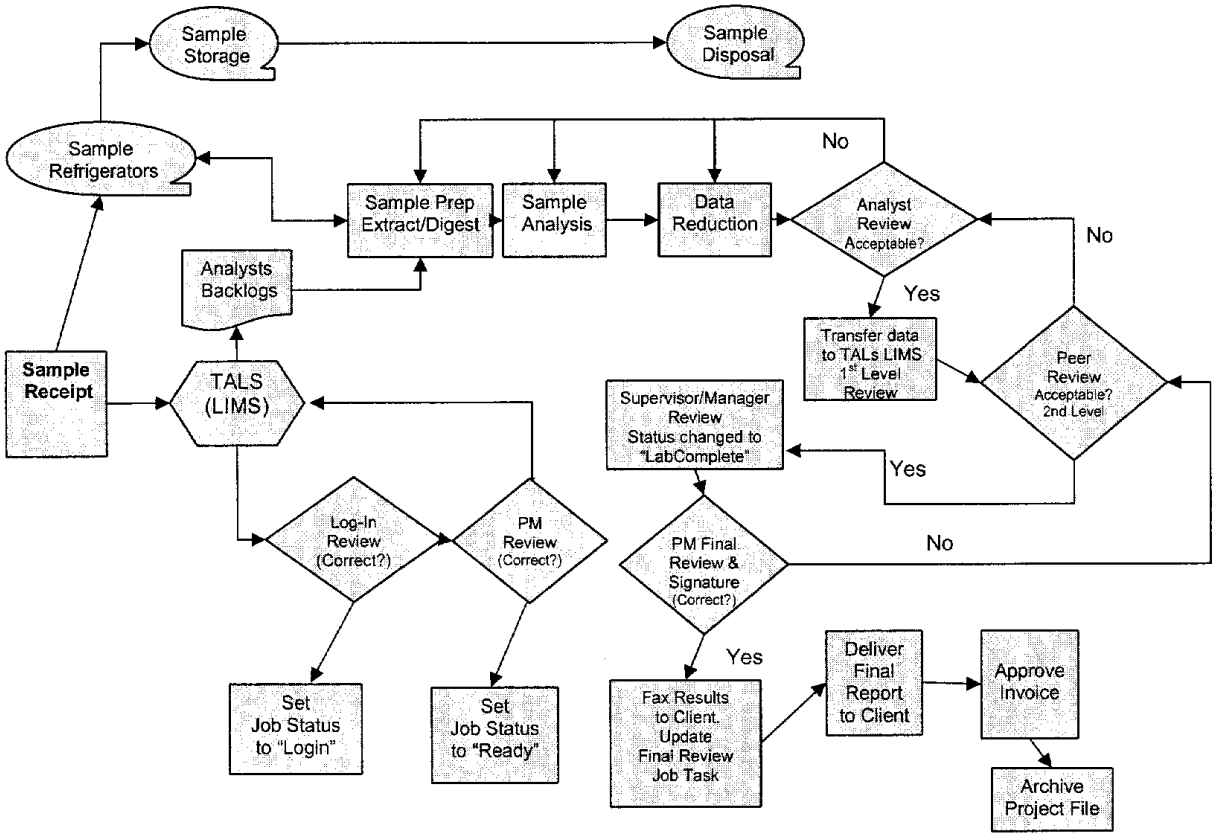
Signature/Date of Lab Representative: _____
Signature/Date of QA Representative: _____

****

QA Only
Software Verification Required (Y/N)
Verification Completed as Described: _____
_____
_____
_____
Software Verification Documentation Location:_____
Signature/Date of QA Representative: _____

Figure 20-3.

TestAmerica Chicago Work Flow



SECTION 21

EQUIPMENT (AND CALIBRATIONS) *(NELAC 5.5.5)*

(ISO17025 5.5.4; 5.5.5; 5.5.6; 5.6.1)

21.1 OVERVIEW *(ISO17025 5.5.1; 5.5.2; 5.5.3; 5.5.5; 5.5.10; 5.6.1)*

The laboratory purchases the most technically advanced analytical instrumentation for sample analyses. Instrumentation is purchased on the basis of accuracy, dependability, efficiency and sensitivity. Each laboratory is furnished with all items of sampling, preparation, analytical testing and measurement equipment necessary to correctly perform the tests for which the laboratory has capabilities. Each piece of equipment is capable of achieving the required accuracy and complies with specifications relevant to the method being performed. Before being placed into use, the equipment (including sampling equipment) is calibrated and checked to establish that it meets its intended specification. The calibration routines for analytical instruments establish the range of quantitation. Calibration procedures are specified in laboratory SOPs, the laboratory default criteria are summarized in Appendix 3 of the QAM and the DoD QSM Version 4.2 criteria are summarized in Appendix 8 of the QAM. A list of laboratory equipment and instrumentation is presented in Table 21-1.

Equipment is only operated by authorized and trained personnel. Manufacturer's instructions for equipment use are readily accessible to all appropriate laboratory personnel.

21.2 PREVENTIVE MAINTENANCE *(ISO17025 5.5.1; 5.5.3; 5.5.7; 5.5.9; 5.6.1)*

21.2.1 The laboratory follows a well-defined maintenance program to ensure proper equipment operation and to prevent the failure of laboratory equipment or instrumentation during use. This program of preventive maintenance helps to avoid delays due to instrument failure.

21.2.2 Routine preventive maintenance procedures and frequency, such as lubrication, cleaning, and replacements, should be performed according to the procedures outlined in the manufacturer's manual. Qualified personnel must also perform maintenance when there is evidence of degradation of peak resolution, a shift in the calibration curve, loss of sensitivity, or failure to continually meet one of the quality control criteria.

21.2.3 Table 21-2 lists examples of scheduled routine maintenance. It is the responsibility of each Department Managers and Supervisors to ensure that instrument maintenance logs are kept for all equipment in his/her department. Preventative maintenance procedures may be / are also outlined in analytical SOPs or instrument manuals. (Note: for some equipment, the log used to monitor performance is also the maintenance log. Multiple pieces of equipment may share the same log as long as it is clear as to which instrument is associated with an entry.)

21.2.4 Instrument maintenance logs are controlled and are used to document instrument problems, instrument repair and maintenance activities. Maintenance logs shall be kept for all major pieces of equipment. Instrument maintenance logs may also be used to specify instrument parameters.

21.2.4.1 Documentation must include all major maintenance activities such as contracted preventive maintenance and service and in-house activities such as the replacement of electrical components, lamps, tubing, valves, columns, detectors, cleaning and adjustments.

21.2.4.2 Each entry in the instrument log includes the Analyst's initials, the date, a detailed description of the problem (or maintenance needed/scheduled), a detailed explanation of the solution or maintenance performed, and a verification that the equipment is functioning properly (state what was used to determine a return to control. e.g. CCV run on *'date'* was acceptable, or instrument recalibrated on 'date' with acceptable verification, etc.) must also be documented in the instrument records.

21.2.4.3 When maintenance or repair is performed by an outside agency, service receipts detailing the service performed can be affixed into the logbooks adjacent to pages describing the maintenance performed. This stapled in page must be signed across the page entered and the logbook so that it is clear that a page is missing if only half a signature is found in the logbook.

21.2.5 If an instrument requires repair (subjected to overloading or mishandling, gives suspect results, or otherwise has shown to be defective or outside of specified limits) it shall be taken out of operation and tagged as out of service or otherwise isolated until such a time as the repairs have been made and the instrument can be demonstrated as operational by calibration and/or verification or other test to demonstrate acceptable performance. The laboratory shall examine the effect of this defect on previous analyses (refer to Sections 12 and 13).

21.2.6 In the event of equipment malfunction that cannot be resolved, service shall be obtained from the instrument vendor manufacturer, or qualified service technician, if such a service can be tendered. If on-site service is unavailable, arrangements shall be made to have the instrument shipped back to the manufacturer for repair. Back up instruments, which have been approved, for the analysis shall perform the analysis normally carried out by the malfunctioning instrument. If the back up is not available and the analysis cannot be carried out within the needed timeframe, the samples shall be subcontracted using the procedures outlined in Section 8.

21.2.7 If an instrument is sent out for service or transferred to another facility, it must be recalibrated and verified (including new initial MDL study and MDL Verification) prior to return to lab operations.

21.3 SUPPORT EQUIPMENT (ISO17025 5.5.10; 5.5.11; 5.6.2.1.2; 5.6.2.2.1; 5.6.2.2.2)

This section applies to all devices that may not be the actual test instrument, but are necessary to support laboratory operations. These include but are not limited to: balances, ovens, refrigerators, freezers, incubators, water baths, field sampling devices, temperature measuring devices, thermal/pressure sample preparation devices and volumetric dispensing devices if quantitative results are dependent on their accuracy, as in standard preparation and dispensing or dilution into a specified volume. All raw data records associated with the support equipment are retained to document instrument performance.

21.3.1 Weights and Balances

The accuracy of the balances used in the laboratory is checked every working day, before use. All balances are placed on stable counter tops.

Each balance is checked prior to initial serviceable use with at least two certified ASTM type 1 weights spanning its range of use (weights that have been calibrated to ASTM type 1 weights may also be used for daily verification). ASTM type 1 weights used only for calibration of other weights (and no other purpose) are inspected for corrosion, damage or nicks at least annually and if no damage is observed, they are calibrated at least every 5 years by an outside calibration laboratory. Any weights (including ASTM Type 1) used for daily balance checks or other purposes are recalibrated/recertified annually to NIST standards (this may be done internally if laboratory maintains "calibration only" ASTM type 1 weights).

All balances are serviced annually by a qualified service representative, who supplies the laboratory with a certificate that identifies traceability of the calibration to the NIST standards.

All of this information is recorded in logs, and the recalibration/recertification certificates are kept on file. Refer to laboratory SOP UP-QA-003, *Balance Calibration Care and Use* for additional details.

21.3.2 pH, Conductivity, and Turbidity Meters

The pH meters used in the laboratory are accurate to $\pm$ 0.1 pH units, and have a scale readability of at least 0.05 pH units. The meters automatically compensate for the temperature, and are calibrated with at least two working range buffer solutions before each use, the ATC probes are verified annually.

Conductivity meters are also calibrated before each use with a known standard to demonstrate the meters do not exceed an error of 1% or one umhos/cm, the ATC probes are verified annually.

Turbidity meters are also calibrated before each use. All of this information is documented in logs.

Consult pH and Conductivity, and Turbidity SOPs for further information.

21.3.3 Thermometers

All thermometers are calibrated on an annual basis with a NIST-traceable thermometer. IR thermometers, digital probes and thermocouples are calibrated quarterly.

The NIST thermometer is recalibrated every five years (unless thermometer has been exposed to temperature extremes or apparent separation of internal liquid) by an approved outside service and the provided certificate of traceability is kept on file. The NIST thermometer(s) have increments of 0.2 °C, and has a range applicable to all method and certification requirements. The NIST traceable thermometer is used for no other purpose than to calibrate other thermometers.

All of this information is documented in logbooks. Monitoring method-specific temperatures, including incubators, heating blocks, water baths, and ovens, is documented in method-specific logbooks. More information on this subject can be found in the laboratory SOP UP-QA-034, *Thermometer Calibration* and the method-specific SOPs.

21.3.4 Refrigerators/Freezer Units, Waterbaths, Ovens and Incubators

The temperatures of all refrigerator units and freezers used for sample and standard storage are monitored continuously via an electronic monitoring system.

Ovens, water baths and incubators are monitored on days of use.

All of this equipment has a unique identification number, and is assigned a unique thermometer for monitoring.

Sample storage refrigerator temperatures are kept between > 0 °C and ≤ 6 °C. The electronic monitoring system applies the limits of 4 ± 2°C to comply with those projects that require the tighter monitoring window.

Specific temperature settings/ranges for other refrigerators, ovens, water baths, and incubators can be found in method specific SOPs.

The temperatures that are continuously monitored are stored electronically on the Local Area Network (LAN) and the method specific temperature documentation of ovens water baths and incubators are maintained within the individual method logbooks or LIMs batch records.

21.3.5 Autopipettors, Dilutors, and Syringes

Mechanical volumetric dispensing devices including burettes (except Class A Glassware) are given unique identification numbers and the delivery volumes are verified gravimetrically, at a minimum, on a quarterly basis. Glass micro-syringes are considered the same as Class A glassware.

For those dispensers that are not used for analytical measurements, a label is / can be applied to the device stating that it is not calibrated. Any device not regularly verified can not be used for any quantitative measurements.

Micro-syringes are purchased from Hamilton Company. Each syringe is traceable to NIST. The laboratory keeps on file an "Accuracy and Precision Statement of Conformance" from Hamilton attesting established accuracy.

21.4 INSTRUMENT CALIBRATIONS *(ISO17025 5.5.8; 5.5.9; 5.5.10; 5.6.1; 5.6.2)*

Calibration of analytical instrumentation is essential to the production of quality data. Strict calibration procedures are followed for each method. These procedures are designed to determine and document the method detection limits, the working range of the analytical instrumentation and any fluctuations that may occur from day to day.

Sufficient raw data records are retained to allow an outside party to reconstruct all facets of the initial calibration. Records contain, but are not limited to, the following: calibration date, method, instrument, analyst(s) initials or signatures, analysis date, analytes, concentration, response, type of calibration (Avg RF, curve, or other calculations that may be used to reduce instrument responses to concentration.)

Sample results must be quantitated from the initial calibration and may not be quantitated from any continuing instrument calibration verification unless otherwise required by regulation, method or program.

If the initial calibration results are outside of the acceptance criteria, corrective action is performed and any affected samples are reanalyzed if possible. If the reanalysis is not possible, any data associated with an unacceptable initial calibration will be reported with appropriate data qualifiers (refer to Section 13).

Note: Instruments are calibrated initially and as needed after that and at least annually.

21.4.1 CALIBRATION STANDARDS *(ISO17025 5.6.3.1)*

Calibration standards are prepared using the procedures indicated in the Reagents and Standards section of the determinative method SOP. However, the general procedures are described below.

21.4.1.1 For each analyte and surrogate (if applicable) of interest, prepare calibration standards at the minimum number of concentrations as stated in the analytical methods. If a reference or mandated method does not specify the number of calibration standards, the minimum number is three, not including blanks or a zero standard. All of the standard solutions are prepared using Class A volumetric glassware, calibrated pipettes, and/or microsyringes and appropriate laboratory quality solvents and stock standards.

21.4.1.2 Standards for instrument calibration are obtained from a variety of sources. All standards are traceable to national or international standards of measurement or to national or international standard reference materials wherever possible. Dilution standards are prepared from stock standards purchased from commercial suppliers. The standards are entered into the reagent module of the TALs LIMs system and are maintained for each department, containing concentration, date of receipt, date of standard preparation, any dilutions made, lot number, supplier, type of solvent and a unique code number to identify the standard.

21.4.1.3 The lowest concentration calibration standard that is analyzed during an initial calibration must be at or below the stated reporting limit for the method based on the final volume of extract (or sample). The exception to these rules is ICP and ICP/MS methods or other methods where the referenced method does not specify two or more standards.

21.4.1.4 The other concentrations define the working range of the instrument/method or correspond to the expected range of concentrations found in actual samples that are also within the working range of the instrument/method. Results of samples not bracketed by initial instrument calibration standards (within calibration range to 3 significant figures) must be reported as having less certainty, e.g., defined qualifiers or flags (additional information may be included in the case narrative). The lowest calibration standard must be at or below the reporting limit. The exception to these rules is ICP and ICPMS methods or other methods where the referenced method does not specify two or more standards.

21.4.1.5 All initial calibrations are verified with a standard obtained from a second source and traceable to a national standard, when available (or vendor certified different lot if a second source is not available). For unique situations where no other source or lot is available, a standard made by a different analyst would be considered a second source. This verification occurs immediately after the calibration curve has been analyzed, and before the analysis of any samples.

21.4.2 CALIBRATION FOR ORGANIC METHODS (GC, HPLC, GC/MS)

21.4.2.1 Many of the organic analytical methods utilize an internal standard calibration (GCMS and some GC). Because of the complex nature of the multipeak chromatograms produced by the method, some instruments necessitate the use of external standard calibration (most GC and HPLC). Surrogate compounds are included in the calibration processes for all appropriate organic analyses. For more details on the calibration types listed below, refer to SOP No. CA-Q-S-005, Calibration Curves.

21.4.2.2 Once the operating parameters have been established according to the method, each instrument is calibrated for the appropriate method. The analyst prepares five or more standard solutions at various concentrations containing all of the analytes of interest, internal standards, and surrogates that are appropriate for the method. Note: There are several EPA methods that have different requirements and are exceptions (e.g. EPA 547) where a minimum of 3 calibration standards are prepared and analyzed.

21.4.2.3 The standard solutions are introduced into the instrument in the same manner as samples are; whether it be by direct injection, by headspace analysis, or by purge and trap. The calibration factor (CF) for methods that use external standards, and the response factor (RF) for methods that use internal standards are calculated for the five standards.

- External standard calibration involves comparison of instrument responses from the sample to the responses from the target compounds in the calibration standards. Sample peak areas (or peak heights) are compared to peak areas (or heights) of the standards. The ratio of the response to the amount of analyte in the calibration standard is defined as the Calibration factor (CF).

- Internal standard calibration involves the comparison of instrument responses from the target compounds in the sample to the responses of specific standards added to the sample or sample extract prior to injection. The ratio of the peak area (or height) of the target compound in the sample or sample extract to the peak area (or height) of the internal standard in the sample or sample extract is compared to a similar ratio derived for each calibration standard. The ratio is termed the response factor (RF), and may also be known as a relative response factor in other methods.

In many cases, internal standards are recommended. These recommended internal standards are often brominated, fluorinated, or stable isotopically labeled analogs of specific target compounds, or are closely related compounds whose presence in environmental samples is highly unlikely. The use of specific internal standards is available in the method SOP.

Whichever internal standards are employed, the analyst needs to demonstrate that the measurement of the internal standard is not affected by method analytes and surrogates or by matrix interferences. In general, internal standard calibration is not as useful for GC and HPLC methods with non-MS detectors because of the inability to chromatographically resolve many internal standards from the target compounds. The use of MS detectors makes internal standard calibration practical because the masses of the internal standards can be resolved from those of the target compounds even when chromatographic resolution cannot be achieved.

When preparing calibration standards for use with internal standard calibration, add the same amount of the internal standard solution to each calibration standard, such that the concentration of each internal standard is constant across all of the calibration standards, whereas the

concentrations of the target analytes will vary. The internal standard solution will contain one or more internal standards and the concentration of the individual internal standards may differ within the spiking solution (e.g., not all internal standards need to be at the same concentration in this solution). The mass of each internal standard added to each sample extract immediately prior to injection into the instrument or to each sample prior to purging must be the same as the mass of the internal standard in each calibration standard. The volume of the solution spiked into sample extracts should be such that minimal dilution of the extract occurs (e.g., 10 uL of solution added to a 1 mL final extract results in only a negligible 1% change in the final extract volume which can be ignored in the calculations).

An ideal internal standard concentration would yield a response factor of 1 for each analyte. However, this is not practical when dealing with more than a few target analytes. Therefore, as a general rule, the amount of internal standard should produce an instrument response (e.g., area counts) that is no more than 100 times that produced by the lowest concentration of the least responsive target analyte associated with the internal standard. This should result in a minimum response factor of approximately 0.01 for the least responsive target compound. Refer to SOP No. CA-Q-S-005, Calibration Curves, for specific calculations.

21.4.2.4 Policies regarding the use of calibration standard results for creating the calibration curve are as follows:

- A low calibration standard may be excluded from the calibration if the signal-to-noise ratio or spectral criteria are not suitable. The reporting level must be elevated to be the lowest calibration standard used for calibration.
- The upper calibration standard may be excluded if it saturates the detector or is obviously becoming non-linear. Any sample exceeding the upper standard used in the calibration must be diluted and re-analyzed.
- Mid-calibration standards may not be excluded unless an obvious reason is found, i.e., cracked vial, incorrectly made, etc. The failed standard should be re-run immediately and inserted into the initial calibration. If not useful, recalibration is required.
-

21.4.2.5 Percent RSD Corrective Action

Given the potentially large numbers of analytes that may be analyzed in some methods, it is likely that some analytes may exceed the acceptance limit for the RSD for a given calibration. In those instances, the following steps are recommended, but not required.

21.4.2.5.1 The first step is generally to check the instrument operating conditions. This option will apply in those instances where a linear instrument response is expected. It may involve some trade-offs to optimize performance across all target analytes. For instance, changes to the operating conditions necessary to achieve linearity for problem compounds may cause the RSD for other compounds to increase, but as long as all analytes meet the RSD limits for linearity, the calibration is acceptable.

21.4.2.5.2 If the RSD for any analyte is greater than the applicable acceptance criteria in the applicable analytical method, analytical SOP or project specific QAPP, the analyst may wish to review the results (area counts, calibration or response factors, and RSD) for those analytes to ensure that the problem is not associated with just one of the initial calibration standards. If the problem appears to be associated with a single standard, that one standard may be reanalyzed and the RSD recalculated. Replacing the standard may be necessary in some cases.

21.4.2.5.3 A third alternative is to narrow the calibration range by replacing one or more of the calibration standards with standards that cover a narrower range. If linearity can be achieved using a narrower calibration range, document the calibration linearity, and proceed with analyses. The changes to the upper end of the calibration range will affect the need to dilute samples above the range, while changes to the lower end will affect the overall sensitivity of the method. Consider the regulatory limits or action levels associated with the target analytes when adjusting the lower end of the range.

Note: When the purpose of the analysis is to demonstrate compliance with a specific regulatory limit or action level, the laboratory must ensure that the method quantitation limit is at least as low as the regulatory limit or action level.

21.4.2.6 Alternatively, the least squares regression may be used to determine linearity for organic method calibrations. A five point line must result in a correlation coefficient (r) $\geq$ 0.995 or the coefficient of determination (r^2) must be $\geq$ 0.990 or better.

21.4.2.7 Instead of a linear curve model (either Average RF or least squares regression), a second order curve (Quadratic) may be used (and preferred) as long as it contains at least six data points. As a rule of thumb, if there is a consistent trend in RFs (or CFs) in the calibration curve, either up or down, then quadratic curve fit may be indicated as the preferred calibration routine for that analyte. The coefficient of determination (COD or r^2) for the quadratic curve must be at least 0.99 for it to be considered acceptable. For more details on the calculations see Calibration Curve SOP CA-Q-S-005. Some limitations on the use of Quadratic Curve fits:

21.4.2.7.1 Care MUST be exercised to assure that the results from this equation are real, positive, and fit the range of the initial calibration.

21.4.2.7.2 They **may not** be used to mask instrument problems that can be corrected by maintenance. (Not to be used where the analyte is normally found to be linear in a properly maintained instrument).

21.4.2.7.3 They **may not** be used to compensate for detector saturation. If it is suspected that the detector is being saturated at the high end of the curve, remove the higher concentration standards from the curve and try a 1st order fit or average RF.

21.4.3 Calibration for Inorganic Analyses

EPA Method 7000 from EPA SW-846 is a general introduction to the quality control requirements for metals analysis. For inorganic methods, quality control measures set out in the individual methods and in the *Standard Methods for the Examination of Water and Wastewater* (20th Edition) may also be included. Standard Operating Procedures for the analysis and the quality control documentation measures are kept in the individual laboratory areas.

In general, inorganic instrumentation is calibrated with external standards. Some exceptions would be Inductively Coupled Plasma (ICP) and Inductively Coupled Plasma Mass Spec (ICPMS). These analyses may use an internal standard to compensate for viscosity or other matrix effects. While the calibration procedures are much the same for inorganics as they are for organics, CF's or RF's are not used. The calibration model in 21.4.2.6 is generally used for most methods, however in some instances the model from section 21.4.2.7 may be used. A correlation coefficient (r) of 0.995 or greater must be used to accept a calibration curve generated for an inorganic procedure. Correlation coefficients are determined by computer programs and

documented as part of the calibration raw data. Coefficients of calibration curves used for quantitation must be documented as part of the raw data. Curves are not allowed to be stored in calculator memories and must be written on the raw data for the purposes of data validation.

21.4.3.1 "Calibrations" for titrimetric analyses are performed by standardizing the titrants against a primary standard solution. See specific methods in *Standard Methods for the Examination of Water and Wastewater* (20th Edition) for more information.

21.4.3.2 Spreadsheets that are used for general chemistry calculations must have all cells containing calculations locked to prevent accidental changes to the calculations.

21.4.4 Calibration Verification

The calibration relationship established during the initial calibration must be verified at least daily as specified in the laboratory method SOPs in accordance with the referenced analytical methods and NELAC (2003) standard, Section 5.5.5.10. The process of calibration verification applies to both external standard and internal standard calibration techniques, as well as to linear and non-linear calibration models. Initial calibration is with a standard source secondary (second source standard) to the calibration standards, but continuing calibration verifications may use the same source standards as the calibration curve.

Note: The process of calibration verification referred to here is fundamentally different from the approach called "calibration" in some methods. As described in those methods, the calibration factors or response factors calculated during calibration are used to update the calibration factors or response factors used for sample quantitation. This approach, while employed in other EPA programs, amounts to a daily single-point calibration.

All target analytes and surrogates, including those reported as non-detects, must be included in periodic calibration verifications for purposes of retention time confirmation and to demonstrate that calibration verification criteria are being met, i.e., RPD, per NELAC (2003) Standard, Section 5.5.5.10.

All samples must be bracketed by periodic analyses of standards that meet the QC acceptance criteria (e.g., calibration and retention time). The frequency is found in the determinative methods or SOPs or project specific QAPPs.

Note: If an internal standard calibration is being used (basically GCMS) then bracketing standards are not required, only daily verifications are needed. The results from these verification standards must meet the calibration verification criteria and the retention time criteria (if applicable).

21.4.4.1 Generally, the initial calibrations must be verified at the beginning of each 12-hour analytical shift during which samples are analyzed. (Some methods may specify more or less frequent verifications). The 12-hour analytical shift begins with the injection of the calibration verification standard (or the MS tuning standard in MS methods). The shift ends after the completion of the analysis of the last sample, QC, or standard that can be injected within 12 hours of the beginning of the shift.

21.4.4.2 A continuing instrument calibration verification (CCV) must be repeated at the beginning and, for methods that have quantitation by external calibration models, at the end of each analytical batch. Some methods have more frequent CCV requirements see specific SOPs.

Most Inorganic methods require the CCV to be analyzed after ever 10 samples or injections, including matrix or batch QC samples.

21.4.4.3 The acceptance limits for calibration verifications can be found in each method SOP. As a rule of thumb: GCMS ± 20% (for CCC's), GC and HPLC ± 15%, Inorganics: ± 10 or 15%. Actual methods may have wider or tighter limits; see the method SOP for specifics, also refer to Appendix 4.

21.4.4.4 If the response (or calculated concentration) for an analyte is within the acceptance limits of the response obtained during the initial calibration, then the initial calibration is considered still valid, and the analyst may continue to use the CF, RF or % drift values from the initial calibration to quantitate sample results.

21.4.4.5 If calibration verification, ICV or CCV, acceptance criteria are not met, then the problem should be investigated and corrected prior to sample analysis. Corrective actions vary by instrument and method (as described in method SOPs), but typically include:

- Checking instrument settings and SOPs to ensure there is no operator error,
- Rerunning the calibration verification standard,
- Repreparing and reanalyzing the verification standard,
- Performing routine instrument maintenance*,
- If the CCV still fails, then the instrument should be recalibrated and associated samples reanalyzed.

*If routine maintenance such as injection port cleaning, contaminated column clipping, guard column replacement, etc. are needed to pass the CCV, the associated field samples shall be reanalyzed. The only corrective action for passing CCV without the need of reanalysis of previous samples is preparing and using fresh CCV standards.

Note: Some programs may allow reporting of samples with non-detect results when the calibration verification indicates high bias, but this should be confirmed with the client before reporting.

All target analytes and surrogates, including those reported as non-detects, must be included in periodic calibration verifications for purposes of retention time confirmation and to demonstrate that calibration verification criteria are being met.

All samples must be bracketed by periodic analyses of standards that meet the QC acceptance criteria (e.g., calibration and retention time). The frequency is found in the determinative methods or SOPs.

Note: If an internal standard calibration is being used (basically GCMS) then bracketing standards are not required, only daily verifications are needed. The results from these verification standards must meet the calibration verification criteria and the retention time criteria (if applicable).

21.4.4.6 Verification of Linear and Non-Linear Calibrations

Calibration verification for linear calibrations involves the calculation of the percent drift or the percent difference of the instrument response between the initial calibration and each subsequent

analysis of the verification standard. Use the equations below to calculate % Drift or % Difference, depending on the procedure specified in the method SOP. Verification standards are evaluated based on the % Difference from the average CF or RF of the initial calibration or based on % Drift or % Recovery if a linear or quadratic curve is used.

The Percent Difference is calculated as follows:

$$\% \text{ Difference} = \frac{(CF(v) \text{ or } RF(v)) - (\text{Avg. CF or RF})}{(\text{Avg. CF or RF})} \times 100$$

Where: CF(v) or RF(v) = CF or RF from verification standard
Avg. CF or RF = Average CF or RF from Initial Calibration.

The Percent Drift is calculated as follows:

$$\% \text{ Drift} = \frac{\text{Result - True Value}}{\text{True Value}} \times 100$$

The Percent Recovery is calculated as follows:

$$\% \text{ Recovery} = \frac{\text{Result}}{\text{True Value}} \times 100$$

Regardless of whether a linear or non-linear calibration model is used, if initial verification criterion is not met, then no sample analyses may take place until the calibration has been verified or a new initial calibration is performed that meets the specifications listed in the method SOPs. If the calibration cannot be verified after the analysis of a single verification standard, then adjust the instrument operating conditions and/or perform instrument maintenance, and analyze another aliquot of the verification standard. If the calibration cannot be verified with the second standard, then a new initial calibration is performed.

- When the acceptance criteria for the calibration verification are exceeded high, i.e., high bias, and there are associated samples that are non-detects, then those non-detects may be reported. Otherwise, the samples affected by the unacceptable calibration verification shall be reanalyzed after a new calibration curve has been established, evaluated and accepted.

- When the acceptance criteria for the calibration verification are exceeded low, i.e., low bias, those sample results may be reported if they exceed a maximum regulatory limit/decision level. Otherwise, the samples affected by the unacceptable verification shall be reanalyzed after a new calibration curve has been established, evaluated and accepted. Alternatively, a reporting limit standard may be analyzed to demonstrate that the laboratory can still support non-detects at their reporting limit.

21.5 TENTATIVELY IDENTIFIED COMPOUNDS (TICS) – GC/MS ANALYSIS

For samples containing components not associated with the calibration standards, a library search may be made for the purpose of tentative identification. The necessity to perform this type of identification will be determined by the purpose of the analyses being conducted. Data system library search routines should not use normalization routines that would misrepresent the library or unknown spectra when compared to each other.

Note: If the TIC compound is not part of the client target analyte list but is calibrated by the laboratory and is both qualitatively and/or quantitatively identifiable, it should not be reported as a TIC. If the compound is reported on the same form as true TICs, it should be qualified and/or narrated that the reported compound is qualitatively and quantitatively (if verification in control) reported compared to a known standard that is in control (where applicable).

For example, the RCRA permit or waste delisting requirements may require the reporting of non-target analytes. Only after visual comparison of sample spectra with the nearest library searches may the analyst assign a tentative identification.

21.5.1 Use the following guidelines for making tentative identifications

21.5.1.1 Major ions in the reference spectrum (ions greater than 10% of the most abundant ion) should be present in the sample spectrum.

21.5.1.2 The relative intensities of the major ions should agree within ± 20%. (Example: For an ion with an abundance of 50% in the standard spectrum, the corresponding sample ion abundance must be between 30 and 70%).

21.5.1.3 Molecular ions present in the reference spectrum should be present in the sample spectrum.

21.5.1.4 Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible background contamination or presence of coeluting compounds.

21.5.1.5 Ions present in the reference spectrum but not in the sample spectrum should be reviewed for possible subtraction from the sample spectrum because of background contamination or coeluting peaks. Data system library reduction programs can sometimes create these discrepancies.

The concentration of any non-target analytes identified in the sample (see above) should be estimated. The same formula as calibrated analytes should be used with the following modifications: The areas A_x and A_{is} should be from the total ion chromatograms, and the RF for the compound should be assumed to be 1.

The resulting concentration should be reported indicating: (1) that the value is an estimate, and (2) which internal standard was used to determine concentration. Use the nearest internal standard free of interferences.

Note: The above guidelines above are from EPA SW846 III edition, method 8260B.
For general reporting if TICs are requested, the ten (10), largest non-target analyte peaks whose area count exceeds 10% of the nearest internal standard will be termed "Tentatively Identified Compounds" (TICs). More or fewer TICs may be identified based on client requirements.

21.5.2 TIC Reporting Limits

In general Reporting limits cannot be specified because of the unknown nature of the TIC. Any reporting limit that is assigned to the TIC can only be evaluated as an estimate as the quantitation is based on the assumption that the TIC responds exactly as the IS responds which is most likely not the case. The TALs LIMs data report does not report TICs with a referenced reporting limit.

TICs that meet the above identification criteria (Section 21.5.1) at 10% area of the IS: If a RL were to be assigned, it would be 10% of the concentration of the internal standard used for quantitation. (e.g. 2.5 ug/L for 8260B, 4.0 ug/L for 8270C). In general, if the 10% area criteria are not met, the TIC RLs should be set at a level approximately 5x the level of the poorest performer in the analysis.

21.6 GC/MS TUNING

Prior to any GCMS analytical sequence, including calibration, the instrument parameters for the tune and subsequent sample analyses within that sequence must be set.

Prior to tuning/auto-tuning the mass spec, the parameters may be adjusted within the specifications set by the manufacturer or the analytical method. These generally don't need any adjustment but it may be required based on the current instrument performance. If the tune verification does not pass it may be necessary to clean the source or perform additional maintenance. Any maintenance is documented in the maintenance log.

21.6.1 The concentration of the BFB or DFTPP must be at or below the concentrations that are referenced in the analytical methods. Part of the purpose of the tune is to demonstrate sensitivity and analyzing solutions at higher concentrations does not support this purpose. Tune failures may be due to saturation and a lower BFB/DFTPP concentration may be warranted.

21.6.2 Tune evaluations usually utilize the "Autofind" function and are set up to look at the apex +/- 1 scan and average the three scans. Background correction is required prior to the start of the peak but no more than 20 scans before. Background correction cannot include any part of the target peak.

21.6.3 Other Options or if Auto Tune Fails:

21.6.3.1 Sometimes the instrument does not always correctly identify the apex on some peaks when the peak is not perfectly shaped. In this case, manually identify and average the apex peak +/- 1 scan and background correct as in 21.6.2 above. This is consistent with EPA 8260 and 8270.

21.6.3.2 Or the scan across the peak at one half peak height may be averaged and background corrected. This is consistent with Standard Methods 6200, EPA 624 and EPA 625.

21.6.3.3 Adjustments such as adjustments to the repeller and ion focus lenses, adjusting the EM Voltage, etc. may be made prior to tune verification as long as all of the subsequent injections in the 12 hour tune cycle are analyzed under the same MS tune settings and it is documented in the run sequence log and/or maintenance log that an adjustment was made. Excessive adjusting (more than 2 tries) without clear documentation is not allowed. Necessary maintenance is performed and documented in instrument log.

21.6.3.4 A single scan at the Apex (only) may also be used for the evaluation of the tune or as specified in the reference method. For SW 846 and EPA 600 series methods, background correction is still required.

21.6.3.5 Cleaning the source or other maintenance may be performed and then follow steps for tune evaluation above. Note: If significant maintenance was performed, see methods 8000B or 8000C then the instrument may require recalibration prior to proceeding.

21.6.4 Tune evaluation printouts must include the chromatogram and spectra as well as the Tune evaluation information. In addition, the verifications must be sent directly to the printer or pdf file (no screen prints for DFTPP or BFB tunes). This ability should be built into the instrument software.

21.6.5 All MS tune settings must remain constant between running the tune check and all other samples. It is recommended that a separate tune method not be used, however a separate method may be used as long as the MS conditions between the methods are the same as the sample analysis method and tracked so any changes that are made to the analysis method are also made to the tune method.

Table 21-1.

Example - Laboratory Equipment and Instrumentation *(ISO17025 5.5.4; 5.5.5)*

Equipment/ Instrument	Manufacturer	Model Number	Serial Number	Year Put into Service	Condition When Received
GC Extractable INST0304	HP 6890 GC Plus with Dual FID	6890 (G1530A)	US00001850	2007	NEW
GC Extractable INST09	HP 5890A GC with FID	5890A	S/N 2750A18141	1988	NEW
GC Extractable INST1516	Agilent 6890N GC System with dual ECD	G6890N (G1530N)	S/N CN10411048	2004	NEW
GC Extractable INST1920	Agilent 7890A GC System with dual FID	7890A (G3440A)	S/N CN10501115	2011	NEW
GC Extractable INST2324	Agilent 6890N GC System with Dual ECD	G6890N (G1530N)	S/N CN10421024	2004	NEW
GC Extractable INST2526	Agilent 7890A GC System with dual FID	7890A (G3440A)	S/N CN10371134	2010	NEW
GC Extractable INST3132	Agilent 6890N GC System with dual ECD	6890N (G1530N)	S/N CN10411047	2004	NEW
GC Extractable INST3738	HP 6890 Series GC with Dual ECD	6890 (G1530A)	S/N US00004455	1996	NEW
GC Extractable INST4142	HP 6890 Series GC System: G1530A with Dual ECD	6890 (G1530A)	S/N US00006539	1997	NEW
GC Extractable INST4748	Agilent 6890A Series GC Plus System: G1530A with Dual ECD	6890A (G1530A)	S/N US00037876	2007	NEW
GC Volatile INST1112	HP5890A GC with FID	5890A	S/N 2750A17322	1992	NEW
GC Volatile INST1314	HP5890A GC with FID	5890A	S/N 2750A17321	1988	NEW
GC Volatile INST1718	HP5890E Series II Plus GC with FID	5890E	S/N 3336A53465	2010	USED
GC Volatile INST2122	Agilent 7890 GC with FID	7890	S/N CN10291030	2010	NEW
GC Volatile INST3334	HP 5890A GC with FID	5890A	S/N 2541A06735	2010	USED
HPLC INST4546	Agilent 1100 HPLC - Detector: Fluorescence Agilent 1100 HPLC - Detector: UV Variable Wavelength	G1321A FLD G1314A VWD	S/N DE23905110 S/N JP24020956	2003	NEW
HPLC INST35	Agilent 1100 HPLC - Detector: Variable wavelength	G1314A VWD	S/N JP11614612	2001	NEW
HPLC INST40	Agilent 1100 HPLC - Detector: Variable wavelength	G1314A VWD	S/N JP11414170	2001	NEW

Equipment/ Instrument	Manufacturer	Model Number	Serial Number	Year Put into Service	Condition When Received
HPLC INST43	Agilent 1100 HPLC - Detector: Variable wavelength	G1314A VWD	S/N JP92112755	2001	NEW
HPLC INST44	Agilent 1100 HPLC - Detector: Variable wavelength	G1314A VWD	S/N JP92112931	2001	NEW
GPC GPC2	J2 Accuprep 170 GPC Controller/Autosampler D-Star Fixed WvL Det Solvent Pump/ UPS	J2M 170 DFW-20	S/N 4237A1292 S/N None	2002	NEW
GPC GPC3	J2 Accuprep MPS GPC Injector/Autosampler /Detector APC-UPC	J2M 3300 J2 330	S/N 05C-1143-4.0 S/N DS00005388	2005	NEW
GPC GPC6	J2 Accuprep MPS GPC Injector/Autosampler /Detector	P/N 54022	S/N PUM-S13H-000	2008	NEW
GC/MS Semivolatile CMS01	Agilent 6890N GC System Agilent 5973 MS Detector	6890N (G1530N) G2578A	S/N US10250131 S/N US21854134	2004	NEW
GC/MS Semivolatile CMS11	Agilent 6890N GC System Agilent 5973 MS Detector	6890N (G1530N) G2578A	S/N CN10308018 S/N US30955129	2003	NEW
GC/MS Semivolatile CMS12	Agilent 6890N GC System Agilent 5973 MS Detector	6890N (G1530N) G2578A	S/N CN10308019 S/N US21854871	2003	NEW
GC/MS Semivolatile CMS20	Agilent 6890N GC System Agilent 5975 MS Detector	6890N (G1530N) G3171A	S/N CN10615045 S/N US861622903	2006	NEW
GC/MS Semivolatile CMS21	Agilent 6890A GC System Agilent 5973 MS Detector	6890A (G1530A) G2577A	S/N US00035156 S/N US10442182	2008	USED
GC/MS Semivolatile CMS23	Agilent 7890A GC System Agilent 5975C MS Detector	7890A (G3440A) 5975C (G3172A)	S/N CN10271149 S/N US10283612	2010	NEW
GC/MS Volatile MS02	Agilent 6890N GC System Agilent 5973 MS Detector	6890N (G1530N) G2579A	S/N CN10340024 S/N US33220076	2003	NEW
GC/MS Volatile MS03 (Screener)	HP 5890 Series II GC HP 5972 Series MS Detector	5890 Series II 5972	S/N 3310A47330 S/N 3609A03585	1998	NEW

Equipment/ Instrument	Manufacturer	Model Number	Serial Number	Year Put into Service	Condition When Received
GC/MS Volatile MS06	Agilent 6890 Series GC Plus G1530A Agilent 5973 Network MS Detector G2579A	6890 (G1530A) 5973 (G2579A)	S/N US10250132 S/N US21854172	2003	NEW
GC/MS Volatile MS09 (Screener)	HP Series 5890 Series II Plus GC HP 5972 Series MS Detector	5890 Series II 5972	S/N 3336A60300 S/N 3435A01881	1998	NEW
GC/MS Volatile MS16	Agilent 6890 Series GC Plus G1530A Agilent 5973 Network MS Detector G2579A	6890 (G1530A) 5973 (G2579A)	S/N US00041196 S/N US10360253	2001	NEW
GC/MS Volatile MS18	Agilent 6890N Series GC Agilent 5975 Series MS Detector	6890N Series (G1530N) 5975 (G3172A)	S/N CN10528010 S/N US51530111	2005	NEW
GC/MS Volatile MS19	Agilent 6890N Series GC Agilent 5975 Series MS Detector	6890N Series (G1530N) 5975 (G3172A)	S/N CN10527059 S/N US52430414	2005	NEW
GC/MS Volatile MS22	Agilent 6890N Series GC Agilent 5973 Series MS Detector	6890N Series (G1530N) 5973 (G2571A)	S/N US10202110 S/N US10442062	2008	USED
Autoanalyzer AQ2 SEAL	AQ2 Analyzer	AQ2	S/N 090321	2004	NEW
Autoanalyzer PC Titrate	Burivar - I/2 Buret Module	PC-1104-00	S/N MS-0E3-585	2004	NEW
Ion Chromatography IC-4	Dionex DX-120 Ion Chromatograph	DX-120	S/N 99070500	1999	NEW
TOC TOC3	Tekmar Dohrmann Phoenix 8000 TOC	8000	S/N 97231001	1997	NEW
TOC TOC4	Tekmar Dohrmann Phoenix 8000 TOC w/Boat	8000	S/N 98239017	1999	NEW
TOC TOC5	Teledyne-Tekmar TOC Analyzer	FUSION	S/N US10216006	2010	NEW
TOX TOX2	Thermo Electron	ECS1200	SN 2005.0179	2005	NEW
Dissolved Oxygen Meter HACH-DO1	Sension6 Dissolved Oxygen Meter	Sension6	S/N 990400000150	1999	NEW
Dissolved Oxygen Meter HACH-DO2	Sension6 Portable Dissolved Oxygen Meter	Sension6	S/N None	2002	NEW
Flashpoint Tester FP1	Flashpoint Tester - Cleveland Open Cup		S/N 10AY-2	1990	USED
Flashpoint Tester FP4	Flashpoint Tester – Herzog Pensky Marten – Closed Cup	HFP339	S/N 073390090	2007	NEW

Equipment/ Instrument	Manufacturer	Model Number	Serial Number	Year Put into Service	Condition When Received
pH Meter pH2	pH Meter: Thermo Orion	410	S/N 074127	2003	NEW
pH Meter pH4	pH Meter: Beckman	Φ250	S/N 4188	2007	NEW
pH Meter pH5	pH Meter: Hanna pH/ORP Meter	HI98183	S/N 0824706	2010	NEW
Oil & Grease	Horizon Technology: Oil & Grease Machine – Extractor	SPE-3000XL Plus-SS	S/N 04-2008	2005	NEW
Oil & Grease	Horizon Technology: Oil & Grease Controller	SPE-3000	S/N 04-1279	2005	NEW
Oil & Grease	Speed Vap II – Evaporator	SPEED-VAP 9000	SPEED-VAP 9000	2002	NEW
Spectrophotometer SPEC3	Spectrophotometer: Genesys 10vis	10 vis	S/N 2D7D054001	2001	NEW
Spectrophotometer SPEC5	UV mini 1240V Shimadzu	1240V	S/N A10934634610	2009	NEW
Conductivity Meter	Specific Conductivity Meter: VWR EC Meter	1056	S/N 0104022	2001	NEW
Turbidimeter TURB1	HACH Turbidimeter	2100A	S/N 773A35	1996	NEW
Mercury CV Analyzer HG5	Teledyne Leeman Hydra AA Auto Mercury Analyzer	HYDRA AA	S/N 7014	2008	USED
Mercury CV Analyzer HG6	Teledyne Leeman Hydra II AA Auto Mercury Analyzer	HYDRA II	S/N 0023	2010	NEW
ICP ICP5	TJA ICAP 61E Trace	13559500	S/N 10792	2001	NEW
ICP ICP6	Thermo Fisher ICAP 6500 DUO	6500 DUO	S/N 20083806	2008	NEW
ICP-MS ICPMS2	ThermoElectron Corp ICP-MS X-Series II ID100 Autodiluter ESI FAST Autosampler SC4-DX	X-Series II ID100 SC4-DX	S/N 01189C S/N 00293 S/N X4DX-HS-TSP-16-091013	2007	NEW
TCLP-4 Extraction Apparatus	TCLP Millipore Rotary (4 place extractor)	115V	S/N 455R44033	1991	NEW
TCLP-48 Extraction Apparatus	TCLP Rotation System: Associated Designer & Manufacturer	3740-48BRE	S/N 2244	2010	NEW

Table 21-2.

Example: Schedule of Routine Maintenance

Instrument	Procedure	Frequency
Mercury Analyzer	Check tubing for wear	Daily
	Fill rinse tank with 10% HCl	Daily
	Insert clean drying tube filled with Magnesium Perchlorate	Daily
	Fill reductant bottle with 10% Stannous Chloride	Daily
ICP	Check pump tubing	Daily
	Check fluid level in waste container	Daily
	Check filters	Monthly (ICP5)
	Clean or replace filters	As required (ICP5)
	Check torch	Daily
	Check sample spray chamber for debris	Monthly
	Check nebulizer	Monthly
	Check entrance slit for debris	Yearly by Service Rep.
	Check/Refill recirculator	Monthly
ICP MS	Check pump tubing	Daily
	Change pump tubing	As required
	Check torch	Weekly
	Check / clean nebulizer	As required
	Clean cones	As required
	Check air filters	As required
	Check multiplier voltages & do cross calibration	Weekly
	Check/Change vacuum pump oil	Monthly
	Check chiller water level/Refill	Monthly
UV-Vis Spectrophotometer	Clean ambient flow cell	As required
	Precision check/alignment of flow cell	As required
	Wavelength verification check	Annually
Auto Analyzers	Clean sampler	As required
	Check all tubing	As required
	Clean inside of colorimeter	As required
	Clean pump well and pump rollers	As required
	Clean wash fluid receptacle	As required
	Oil rollers/chains/side rails	As required
	Clean optics and cells	As required
HP / Agilient GC/MS	Pump oil-level check	Monthly
	Pump oil changing	Annually
	Analyzer bake-out	As required
	Analyzer cleaning	As required
	Resolution adjustment	As required
	Change multiplier	As required
	Change filaments	As required

Instrument	Procedure	Frequency
Gas Chromatograph	Compare standard response to previous day or since last initial calibration	Daily
	Check carrier gas flow rate in column	Daily via use of known compound retention
	Check temp. of detector, inlet, column oven	Daily
	Septum replacement	As required
	Check system for gas leaks (with SNOOP-MS VOA)	W/cylinder change as required
	Check for loose/frayed wires and insulation	As Required
	Bake injector/column	As Required
	Change/remove sections of guard column	As Required
	Replace connectors/liners	As Required
	Change/replace column(s)	As Required
Electron Capture Detector (ECD)	Detector wipe test (Ni-63)	Semi-annually
	Detector cleaning	As required
Flame Ionization Detector (FID)	Detector cleaning	As required
HPLC	Change guard columns	As required
	Change lamps	As required
	Change pump seals	Semi-annually or as required
	Replace tubing	As required
	Change frit	As required
	Filter all samples	Daily
	Change autosampler rotor/stator	As required
Autosamplers (GC/MS Volatiles GC Volatiles)	Check lines	As required
	Change filters	As required
	Replace Needles	As required
	Check Gaskets	As required
	Re-calibrate	As required
	Replace Sensors	As required
Autosamplers (GC/MS Semivolatiles)	Replace Syringe	As required
Concentrators (GC/MS Volatiles GC Volatiles)	Change Trap	As required
	Replace Lines	As required
Balances	Class "S" traceable weight check	Daily, when used
	Clean pan and check if level	Daily
	Field service	At least Annually
Conductivity Meter	KCl calibration	Daily, applicable standards
	Conductivity cell cleaning	As required
Turbidimeter	Check light bulb	Daily, when used
Deionized/Distilled Water	Conductivity Point Sources	Water Quality SOP *UP-QA-035*
	Daily conductivity check	Daily
	Check deionizer light	Daily
	Monitor for VOA's	As required
	System cleaning	As required
	Replace cartridge & large mixed bed resins	As required

Instrument	Procedure	Frequency
Drying Ovens	Temperature monitoring Temperature adjustments	Daily As required
Refrigerators/ Freezers	Temperature monitoring Temperature adjustment Defrosting/cleaning	Continuous As required As required
Vacuum Pumps/ Air Compressor	Lubricate	As required
pH/Specific Ion Meter	Calibration/check slope Clean electrode	Daily As required
BOD Incubator	Temperature monitoring Coil and incubator cleaning	Continuous As required
Centrifuge	None	Serviced as required
Water baths	Temperature monitoring Water replaced	Daily As needed

SECTION 22

MEASUREMENT TRACEABILITY *(NELAC 5.5.6)*

22.1 OVERVIEW *(ISO17025 5.6.2.1.2; 5.6.2.2.2; 5.6.3.1)*

Traceability of measurements shall be assured using a system of documentation, calibration, and analysis of reference standards. Laboratory equipment that are peripheral to analysis and whose calibration is not necessarily documented in a test method analysis or by analysis of a reference standard shall be subject to ongoing certifications of accuracy. At a minimum, these must include procedures for checking specifications of ancillary equipment: balances, thermometers, temperature, Deionized (DI) and Reverse Osmosis (RO) water systems, automatic pipettes and other volumetric measuring devices. (Refer to Section 21.3) With the exception of Class A Glassware (including glass microliter syringes that have a certificate of accuracy), quarterly accuracy checks are performed for all mechanical volumetric devices. Microsyringes are verified at least semi-annually or disposed of after 6 months of use. Wherever possible, subsidiary or peripheral equipment is checked against standard equipment or standards that are traceable to national or international standards. Class A Glassware should be routinely inspected for chips, acid etching or deformity. If the Class A glassware is suspect, the accuracy of the glassware will be assessed prior to use. The following definitions are provided by the American Association for Laboratory Accreditation (A2LA):

"Traceability is the property of a measurement result whereby it can be related to stated references, usually national or international standards, through an unbroken chain of comparisons, each step in the chain having stated uncertainties." There are six essential elements:

- An unbroken chain of comparison
- A calculated measurement uncertainty for each step in the chain to allow for an overall uncertainty calculation
- Documentation of each step in each calibration report
- All steps in the chain are performed by individuals with evidence of technical competence and accredited by a recognized accreditation body
- Reference to International Standard (SI) units
- Recalibration at appropriate intervals to preserve traceability

Calibration is defined as "determining and documenting the deviation of the indication of a measuring instrument (or the stated value of a material measure) from the conventional 'true' value of the measurand."

Uncertainty is defined as "a parameter associated with the result of a measurement that characterizes the dispersion of the value that could reasonably be attributed to the measurand." Measurement of Uncertainty is discussed is Section 20.12 of this QA Manual.

Note: Total measurement error is a function of both precision and bias.

22.2 NIST-TRACEABLE WEIGHTS AND THERMOMETERS *(ISO17025 5.6.3.1; 5.6.3.2)*

Reference standards of measurement shall be used for calibration only and for no other purpose, unless it can be shown that their performance as reference standards would not be invalidated.

For NIST-traceable weights and thermometers, the laboratory requires that all calibrations be conducted by a calibration laboratory accredited by A2LA, NVLAP (National Voluntary Laboratory Accreditation Program), APLAC (Asia-Pacific Laboratory Accreditation Cooperation), or EA (European Cooperation for Accreditation). A certificate and scope of accreditation is kept on file at the laboratory. (Refer to Section 21.3 for calibration of weights and thermometers).

An external certified service engineer services laboratory balances on an annual basis. This service is documented on each balance with a signed and dated certification sticker. Balance calibrations are checked each day of use. All in-service thermometers are calibrated annually against a traceable reference thermometer. Temperature readings of ovens, refrigerators, and incubators are checked on each day of use or continuously monitored.

22.3 REFERENCE STANDARDS / MATERIALS *(ISO17025 5.6.3.1; 5.6.3.2; 5.6.3.3; 5.6.3.4; 5.9.1)*

Reference standards/materials, where commercially available, are traceable to certified reference materials. Commercially prepared standard materials are purchased from vendors accredited by A2LA or NVLAP with an accompanying Certificate of Analysis that documents the standard purity. If a standard cannot be purchased from a vendor that supplies a Certificate of Analysis, the purity of the standard is documented by analysis. (Refer to Section 9 for additional information on purchasing). The receipt of all reference standards must be documented. Reference standards are labeled with a unique Standard Name in TALs LIMs and expiration date. All documentation received with the reference standard is retained as a QC record and references the LIMs assigned Standard Name.

All reference, primary and working standards/materials, whether commercially purchased or laboratory prepared, must be checked regularly to ensure that the variability of the standard or material from the 'true' value does not exceed method requirements. The accuracy of calibration standards is checked by comparison with a standard from a second source. In cases where a second standard manufacturer is not available, a vendor certified different lot is acceptable for use as a second source. For unique situations, where no other source or lot is available, a standard made by a different analyst would be considered a second source. The appropriate Quality Control (QC) criteria for specific standards are defined in laboratory SOPs. In most cases, the analysis of an Initial Calibration Verification (ICV) or LCS (where there is no sample preparation) is used as the second source confirmation. These checks are generally performed as an integral part of the analysis method (e.g. calibration checks, laboratory control samples).

All standards and materials must be stored and handled according to method or manufacturer's requirements in order to prevent contamination or deterioration. Refer to Table 9-1 in Section 9 for general storage requirements and Table 22-1 for additional storage information as well as the Corporate Environmental Health & Safety Manual or individual laboratory SOPs. For safety requirements, please refer to method SOPs and the laboratory Environmental Health and Safety Manual.

22.4 DOCUMENTATION AND LABELING OF STANDARDS, REAGENTS, AND REFERENCE MATERIALS

Reagents must be at a minimum the purity required in the test method. The date of reagent receipt and the expiration date are documented. The lots for most of the common solvents and acids are tested for acceptability prior to company wide purchase. (Refer to TestAmerica's Corporate SOP (CA-Q-S-001), Solvent and Acid Lot Testing and Approval.)

All manufacturer or vendor supplied Certificate of Analysis or Purity must be retained, stored appropriately, and readily available for use and inspection. These records are maintained in the various laboratory sections. Records must be kept of the date of receipt and date of expiration of standards, reagents and reference materials. In addition, records of preparation of laboratory standards, reagents, and reference materials must be retained, stored appropriately, and be readily available for use and inspection. For detailed information on documentation and labeling, please refer to the method specific SOPs.

Commercial materials purchased for preparation of calibration solutions, spike solutions, etc.., are usually accompanied with an assay certificate or the purity is noted on the label. If the assay purity is 96% or better, the weight provided by the vendor may be used without correction. If the assay purity is less than 96% a correction will be made to concentrations applied to solutions prepared from the stock commercial material.

22.4.1 All standards, reagents, and reference materials must be labeled in an unambiguous manner. Standards are logged into the laboratory's LIMS system in the reagent module, and are assigned a unique identification number. The following information is typically recorded in the electronic database within the LIMS and in some departments a Standards Log book is used in conjunction with the LIMs record.

- Standard Name
- Description of Standard
- Department
- Preparer's name
- Final volume and number of vials prepared
- Solvent type and lot number
- Preparation Date
- Expiration Date
- Standard source type (stock or daughter)
- Standard type (spike, surrogate, other)
- Parent standard ID (if applicable)
- Parent Standard Analyte Concentration (if applicable)
- Parent Standard Amount used (if applicable)
- Component Analytes
- Final concentration of each analyte
- Applicable methods
- Applicable QC types

Records are maintained electronically for standard and reference material preparation. These records show the traceability to purchased stocks or neat compounds. These records also include method of preparation, date of preparation, expiration date and preparer's name or initials. Preparation procedures are provided in the Method SOPs.

22.4.2 All standards, reagents, and reference materials must be clearly labeled with a minimum of the following information:

- Expiration Date (include prep date for reagents)
- Standard ID – TALs LIMs Name
- Special Health/Safety warnings if applicable

22.4.3 In addition, the following information may be helpful:

- Date of receipt for commercially purchased items or date of preparation for laboratory prepared items
- Date opened (for multi-use containers, if applicable)
- Description of standard (if different from manufacturer's label or if standard was prepared in the laboratory)
- Concentration (if applicable)
- Initials of analyst preparing standard or opening container

All containers of prepared reagents must include a preparation date, expiration date and an ID number to trace back to preparation.

Procedures for preparation of reagents can be found in the Method SOPs.

Standard ID numbers must be traceable through associated logbooks, worksheets and raw data.

All reagents and standards must be stored in accordance to the following priority: 1) with the manufacturer's recommendations; 2) with requirements in the specific analytical methods; as specified in the laboratory SOP and 3) according to Table 22-1.

Table 22-1.

Standard Sources and Preparation *(ISO17025 5.9.1)*

Instrument	Source	How Received	Stock Storage	Preparation	Intermediate & Working Standard Storage	Frequency
ICP	Environmental Express; Inorganic Ventures; Ultra	Custom Prepared Solutions	Room Temperature (Standards with Ag protect from light)	Working standards from stock	Room Temperature	Daily
ICPMS	Environmental Express; Inorganic Ventures; Ultra	Custom Prepared Solutions	Room Temperature (Standards with Ag protect from light)	Working standards from stock	Room Temperature	Daily
CVAA	Inorganic Ventures; Ultra	1 ppm solutions	Room Temperature	Working standards from stock	Room Temperature	Daily
GC	Restek; Supelco; Cerilliant; Ultra ; Absolute ; Aldrich	Solutions and Neat	Refrigerate	Working standards from stock	Refrigerate	As needed or every 6 months
GC (Herbicide)	Chemserv Ultra; Supelco; Accustandard	Solutions and Neat	Room Temperature or Refrigerate	Working standards from stock	Refrigerate	As needed or every 6 months
HPLC	Ultra; Restek; Supelco; Cerilliant Absolute ; Aldrich	Solutions and Neat	Room Temperature or Refrigerate	Working standards from stock	Refrigerate	As needed or every 6 months
TOX	VWR; Aldrich; Sigma	Solid	Desiccate	Working standards from stock	Refrigerate	Monthly
TOC	VWR	Solid	Refrigerate	Working standards from stock	Refrigerate	Daily
Volatile Organics	Accustandard; Absolute; Ultra; Chemservice; Restek; Supelco; CPI International; o2si Smart Solutions	Ampoule/ Solutions and Neat	Freezer (-10°C) Refrigerate Per manufacturer recommendation	Working standards from stock	Freezer Refrigerate Per manufacturer recommendation	Bi-weekly; Gas, weekly (Per SOP)
Semi-Volatile Organics (GC/MS)	Accustandard; Restek; Supleco; Cambridge; Cerilliant	Ampoule/ Solutions and Neat	Freezer (-10°C) or Room temp.	Working standards from stock	Freezer	As needed or every 6 months
Ion Chromatography	APG; RICCA	Solutions	Refrigerate	Working standards from stock	N/A	Daily
AQ2 Seal	APG RICCA VWR	Solutions, Salts	Refrigerate	Working standards from stock	Refrigerate	Daily
PC Titrate	APG RICCA VWR	1000 ppm Solutions; Salts	Room Temperature or Refrigerate	Working standards from stock	Room Temperature or Refrigerate	Daily

SECTION 23.0

SAMPLING *(NELAC 5.5.7)*

23.1 OVERVIEW *(ISO17025 5.7.1; 5.7.3)*

TestAmerica Chicago provides sampling services. Sampling procedures for the bulleted items below are described in the SOP: UP-FS-001, *Field Services: Groundwater Sampling-Bailing Method.*

- Groundwater Sampling
- Wastewater Sampling
- Potable Sampling
- Field Parameter Analysis
- Cleaning and Decontamination of Field Equipment

23.2 SAMPLING CONTAINERS

The laboratory offers clean sampling containers for use by clients. These containers are obtained from reputable container manufacturers and meet EPA specifications as required. Any certificates of cleanliness that are provided by the supplier are maintained at the laboratory.

23.2.1 Preservatives

Upon request, preservatives are provided to the client in pre-cleaned sampling containers. The containers are purchased pre-preserved from the container supplier where the grades of the preservatives are at a minimum:

- Hydrochloric Acid – Reagent ACS (Certified VOA Free) or equivalent
- Methanol – Purge and Trap grade
- Nitric Acid – Instra-Analyzed or equivalent
- Sodium Bisulfate – ACS Grade or equivalent
- Sodium Hydroxide – Instra-Analyzed or equivalent
- Sulfuric Acid – Instra-Analyzed or equivalent
- Sodium Thiosulfate – ACS Grade or equivalent
- Sodium Hydroxide/Zinc Acetate – Instra-Analyzed or equivalent

23.2.2 Preparing Container Orders

When new containers arrive at the laboratory, the date of receipt is recorded on the packing list received with them for retained documentation. Periodically, containers are evaluated for cleanliness based upon their intended parameter sample analysis. Upon request, the containers are then sent to clients for use in collecting samples. The shipping date, type and number of containers are maintained on file by the lab. Shipping personnel insure that container stock is rotated so that "first in" is "first out." When a client requests containers, a client services representative creates a container request in LIMS; it is then stored permanently in LIMS with a unique container order

number. Copies of the container request are printed for the shipping department. One copy goes to the client with the containers; one copy is filed in the shipping department.

The laboratory also provides EnCore, TerraCore or other soil sampling devices when requested.

If containers are provided directly to the client from the manufacturer or from other sources, the laboratory will not be responsible for any of the above records.

23.3 FIELD QUALITY CONTROL (QC) *(ISO17025 5.7.1)*

Common field quality control samples are defined in the following paragraphs. The frequency of field quality control samples should be specified in the site specific Quality Assurance Project Plan (QAPP) or by the client. TestAmerica provides trip blanks for VOC analysis with the sample containers for all volatile organic analyses. Blanks generated in the field will be analyzed along with the field samples (exception soil samples where the blank is aqueous).

23.3.1 Equipment Blank / Rinsate Blank - The equipment blank, sometimes referred to as a rinsate blank, is a sample of the water used to decontaminate sampling equipment. The source water should be as free of target analytes as possible. An aliquot of this water is poured over or through the sample collection device after decontamination, collected in a sample container, preserved with appropriate reagents, and returned to the laboratory. This serves as a check on sampling device cleanliness, and will also be affected by the site and sample handling conditions evaluated by the other types of blanks. The sampling time for the equipment blank should begin when the equipment is rinsed and the water is collected.

23.3.2 Field Blank - The field blank is water that is as free of target analytes as possible and from the same source as the equipment blank. The water is poured into a sampling container at the sampling site, preserved with the appropriate reagents, and returned to the laboratory. This serves as a check on reagent and environmental contamination. The sampling time for the field blank should be when the blank is prepared in the field.

23.3.3 Trip Blank - The trip blank pertains to volatile analysis only. This serves as a check on sample contamination originating from sample transport, sample container contamination, shipping and storage, or from certain site conditions. Trip blanks are often referred to as travel blanks. They are prepared using pre-cleaned sample containers. They are filled with organic-free water (the source of the organic free water is the same source of water used to prepare volatile standards, method blanks, LCS and sample dilutions), sealed and taken into the field with the empty containers which will be used for sampling. The recommended frequency is one trip blank per cooler (in duplicate or triplicate), per volatiles method. Unless otherwise specified, the sampling time for the trip blank is the time of receipt at the laboratory (When the "Trip" ends).

23.3.4 Field Duplicates - Field duplicates are replicate samples collected from the same sampling point or location during a field collection event. This control sample is used to demonstrate the ability of both the sampling and analytical process to generate data of acceptable precision.

23.4 DEFINITION OF HOLDING TIME

The date and time of sampling documented on the chain-of-custody (COC) form establishes the day and time zero. As a general rule, when the maximum allowable holding time is expressed in "days" (e.g 14 days, 28 days), the holding time is based on calendar day measured. Holding times expressed in "hours" (e.g. 6 hours, 24 hours, etc.) are measured from date and time zero. The first day of holding time ends twenty-four hours after sampling. Holding times for analysis include any necessary reanalysis. However there are some special programs and projects that require the holding time to be calculated based on the date and specific time of analysis compared to the time of sampling. This method of calculating holding time can be accommodated by the laboratory through the Project setup in the TALs LIMs system.

23.4.1 Semi-Volatile - Holding times for sample preparation for semi-volatile organics are measured from the sampling date (and time where applicable) until the day (and time where applicable) solvent contacts the sample. If a sample is to be extracted on the day of expiration, the actual time of extraction must be recorded on the sample preparation worksheet and in the LIMs preparation batch. Holding times for analysis are measured from the date (and time where applicable) of initiation of extraction to the time of injection into the gas chromatograph.

23.4.2 Volatiles - Holding times for volatile organics are measured from the date (and time where applicable) of sampling to the date and time of injection into the gas chromatograph. The time of initiation of purging is considered the injection time, but data systems record the start of the chromatographic run rather than the start of purging. Hence, if a sample is so near expiration that the start-of-purging time rather than the chromatographic run time is needed to document the integrity of the sample; the analyst must observe and record the start-of-purging time in the instrument log. Extractions, e.g. for high level soils, must be completed in time to allow for analysis to be initiated within the maximum allowable holding time.

23.4.3 Inorganics - For inorganic and metals analysis, the preparation/digestion/distillation and analytical procedure must be completed within the maximum holding time as measured from the sampling date (and time where applicable).

23.5 SAMPLING CONTAINERS, PRESERVATION REQUIREMENTS, HOLDING TIMES

The preservation and holding time criteria specified in the following tables are derived from the source documents for the methods. If method required holding times (this information is in the laboratory SOPs) or preservation requirements are not met, the reports will be qualified using a flag, footnote or case narrative. As soon as possible or "ASAP" is an EPA designation for tests for which rapid analysis is advised, but for which neither EPA nor the laboratory have a basis for a holding time.

23.6 SAMPLE ALIQUOTS / SUBSAMPLING *(ISO17025 5.7.1)*

Taking a representative sub-sample from a container is necessary to ensure that the analytical results are representative of the sample collected in the field. The size of the sample container, the quantity of sample fitted within the container, and the homogeneity of the sample need consideration when sub-sampling for sample preparation. It is the laboratory's responsibility to take a representative sub-sample or aliquot of the sample provided for analysis. In that regard the following guidelines apply to analysts:

Analysts should handle each sample as if it is potentially dangerous. At a minimum, safety glasses, gloves, and lab coats must be worn when preparing aliquots for analysis. Refer to the laboratory SOP UP-QA-039, *Sample Homogenization and Subsampling Procedures* for further details.

23.6.1 For water samples, before taking each aliquot for analysis, invert the sample container end-over-end three times and immediately pour off the aliquot. Especially when suspended solids are present, adequate mixing of the sample is extremely important.

23.6.2 For solid samples, when volatile organics are not requested, if the solid can be mixed, stir before removing the aliquot. Mix more than is needed for the analysis to be performed (e.g. if 30 g are needed, mix 50-100 g, if 1 g is needed, mix 20 g, etc...).

- If the solid cannot be easily mixed: After thoroughly mixing the sample within the sample container or, for non-organic methods, the sample can be transferred to a wip bag (or other suitable plastic bag) for manual mixing, a sub-sample from various quadrants and depths of the sample are taken to acquire the required sample weight.
- For soil samples, avoid debris in the sub-sample aliquot as much as possible (e.g. gravel, sticks, roots and grass); note this information in the sample preparation record.
- If the solid is extremely heterogeneous, and the client has given no instructions, utilize the following technique: separate the like materials into groups on a clean surface and take portions of masses from each group, proportional to their contribution to the original sample, to make a composite. Record in detail exactly how the composite was created. For very unusual samples, consult with the Project Manager, QA department or Department Supervisor/Manager.

23.6.3 For solid samples, when volatile organics analysis is requested, the sample should be manipulated as little as possible. In most cases, the sample will arrive already preserved or in an EnCore™ sampler of the correct mass (requiring quick preservation of the entire amount). If the client requests volatiles on a solid sample which has been collected in a jar and is in a common container from which aliquots for other test methods must be taken, login should deliver the container to the volatiles department for preparing a proper aliquot prior to any other aliquots being taken out.

23.6.4 For multiphasic samples, the client should instruct the laboratory as to the intent of the testing and how to handle the sample. If the entire sample is to be accounted for, and the phases do not mix easily with inversion/stirring, such that a representative aliquot can be taken, the analyst should record the percent by volume of each phase. The analysis must be conducted on each phase separately; the final results are combined mathematically, weighting the individual phase results by volume. One exception to this procedure is the situation addressed in the TCLP and SPLP methods for wastes containing free liquids. However, if the leachate and final filtrate are not miscible, it is necessary to combine mathematically the concentrations of the two (or more) solutions by volume.

SECTION 24

HANDLING OF SAMPLES (NELAC 5.5.8)

(ISO17025 5.8.1)

Sample management procedures at the laboratory ensure that sample integrity and custody are maintained and documented from sampling/receipt through disposal.

24.1 CHAIN OF CUSTODY (COC) *(ISO17025 5.7.2; 5.9.1)*

The COC form is the written documented history of any sample and can be initiated when bottles are sent to the field, or at the time of sampling. This form is completed by the sampling personnel and accompanies the samples to the laboratory where it is received and stored under the laboratory's custody. The purpose of the COC form is to provide a legal written record of the handling of samples from the time of collection until they are received at the laboratory. It also serves as the primary written request for analyses from the client to the laboratory. The COC form acts as a purchase order for analytical services when no other contractual agreement is in effect. An example of a COC form may be found in Figure 24-1.

24.1.1 Field Documentation

The information the sampler needs to provide at the time of sampling on the container label is:

- Sample identification
- Date and time
- Preservative

During the sampling process, the COC form is completed and must be legible (see Figure 24-1). This form includes information such as:

- Client name, address, phone number and fax number (if available)
- Project name and/or number
- The sample identification
- Date, time and location of sampling
- Sample collectors name
- The matrix description
- Preservatives used
- Analysis requested
- Requested turnaround time (TAT)
- Any special instructions
- Purchase Order number or billing information (e.g. quote number) if available
- The date and time that each person received or relinquished the sample(s), including their signed name.

The samples are stored in a cooler with ice, as applicable, and remain solely in the possession of the client's field technician until the samples are delivered to the laboratory. The sample collector must assure that each container is in his/her physical possession or in his/her view at all times, or stored in such a place and manner to preclude tampering. The field technician

relinquishes the samples in writing on the COC form to the sample control personnel at the laboratory or to a TestAmerica courier. Samples are only considered to be received by lab when personnel at the laboratory have physical contact with the samples.

Note: Independent couriers are not required to sign the COC form. The COC is usually kept in the sealed sample cooler. The sample receipt log is stored in log-in and it maintains a list of all receipts by date.

24.1.2 Legal / Evidentiary Chain-of-Custody *(ISO17025 5.8.4)*

If samples are identified for legal/evidentiary purposes on the COC or prearranged between the client and the project manager, login will complete the custody seal (Figure 24-2), retain the shipping record with the COC, and initiate an internal COC (Figure 24-3) for laboratory use by analysts and a sample disposal record (Figure 24-4).

24.2 SAMPLE RECEIPT *(ISO17025 5.8.2)*

Samples are received at the laboratory by designated sample receiving personnel and a unique laboratory project identification number is assigned. Each sample container shall be assigned a unique sample identification number that is cross-referenced to the client identification number such that traceability of test samples is unambiguous and documented. Each sample container is affixed with a durable sample identification label. Sample acceptance, receipt, tracking and storage procedures are summarized in the following sections.

Refer to laboratory SOP *UP-SR-001*, Sample Receipt: Handling and Processing Procedures for complete details.

24.2.1 Laboratory Receipt *(ISO17025 5.8.3)*

When samples arrive at the laboratory, sample receiving personnel inspect the coolers and samples. The integrity of each sample must be determined by comparing sample labels or tags with the COC and by visual checks of the container for possible damage. Any non-conformance, irregularity, or compromised sample receipt must be documented on a Non-Conformance Memo (NCM) in TALs LIMs and brought to the immediate attention of the project manager and the client. The COC, shipping documents, documentation of any non-conformance, irregularity, or compromised sample receipt, record of client contact, and resulting instructions become part of the project record.

24.2.1.1 Inspection of samples include a check for:

- Complete documentation to include sample identification, location, date and time of collection, collector's name, preservation type, sample type and any additional comments concerning the samples.
- Complete sample labels to include unique identification in indelible ink.
- Use of appropriate sample containers (see Section 23)
- Adherence to holding times as specified in the test method and/or summarized in Section 23.
- Adequate sample volume for required analyses (see Section 23).
- Damage or signs of contamination to sample container. Volatile vials are also inspected for headspace.

Refer to Figure 24-5 (Sample Acceptance Policy) and Figure 24-6 (Login Sample Receipt Checklist) for additional details.

24.2.1.2 Check and record the temperature of the samples or temperature blanks within each cooler that require thermal preservation using the IR gun located in the sample Login department.

- Samples shall be deemed acceptable if arrival temperature is just above freezing and less than or equal to 6.0° C (routinely 4°C +/-2). Samples that are hand-delivered immediately after collection may not be at the required temperatures; however, if there is evidence that the chilling process has begun, such as the arrival on ice, the samples shall be considered acceptable. This will be documented on the COC (Figure 24-1) and on the sample receipt checklist (Figure 24-6) in TALs LIMs.

- If the samples were shipped in ice and solid ice is still present and in direct contact with samples, report the samples as "received on ice." Direct contact means samples must be surrounded by ice cubes or crushed ice. Ice present in a plastic bottle or other container does not constitute direct contact. Samples shipped with only "blue ice" will be reported as "received on blue ice".

24.2.1.3 Verify sample preservation as specified in the test method. Check for correct pH as specified in the test method. The results are documented on the sample receipt checklist in TALs LIMs. In the case of volatiles it is recorded by the analyst after analysis on the sample tracking sheet. Certain analytes are negatively affected by the presence of residual chlorine. BOD/cBOD and TOX are checked for chlorine content at the time of analysis. Cyanide, ammonia, and phenolics should be treated immediately upon collection, which is accomplished by providing treatment ampules with the sample bottles for those samples that might be suspected of containing chlorine, such as wastewaters or drinking waters. The chlorine treatment should be confirmed at log-in.

24.2.1.4 After inspecting the samples, the sample receiving personnel sign and date the COC form, make any necessary notes of the samples' conditions and store them in appropriate refrigerators or storage locations.

24.2.1.5 If samples are received without a COC, TestAmerica will provide a generic COC form to be completed by the client when the samples are brought to the laboratory. The client is always provided with a copy of the completed COC form for their records.

24.2.1.6 If analyses with short holding times are requested, the dates and times are inspected to ensure that holding times have not already expired. The appropriate laboratory staff is notified when short hold analysis is required.

24.2.1.7 Samples received after normal working hours are left in their coolers and placed in a cold storage location typically Walk-In Cooler #8. The person receiving the samples must record the date and time received, the presence or absence of ice and custody seals, the temperature of samples, presence and type of packing material, and initials.

24.2.1.8 *(ISO17025 5.8.3)* Any deviations from these checks described in Section 24.2.1 that question the suitability of the sample for analysis, or incomplete documentation as to the tests required will be resolved by consultation with the client. If the sample acceptance policy criteria (Section 24.3) are not met, the laboratory shall either:

- Retain all correspondence and/or records of communications with the client regarding the disposition of rejected samples The project manager may be able to make decisions on samples with prior knowledge from the client, but documentation of acceptable scenarios must be provided and acknowledge by the client, and records of these decisions must be documented, or

- Fully document any decision to proceed with sample analysis that does not meet sample acceptance criteria.

Note: North Carolina requires that they be notified when samples are processed that do not meet sample acceptance criteria.

Once Sample acceptance is verified, the samples are logged into the LIMs according to the laboratory SOP UP-SR-001, Sample Receipt: Handling and Processing Procedures.

24.2.2 Sample Log-in *(ISO17025 5.8.2)*

All samples that are received by the laboratory are logged into the LIMS to allow the laboratory to track and evaluate sample progress in accordance with the laboratory SOP *UP-SR-001*, Sample Receipt: Handling and Processing Procedures. Each group of samples that are logged in together (typically one project from a given client/sampling event) is assigned a unique job number. Within each job, each sampling point (or sample) receives a unique number. Sample numbers are generated sequentially over time, and are not re-assigned. A sample may be composed of more than one bottle since different preservatives may be required to perform all analyses requested. Even if multiple containers are received for a single sample, each container is uniquely identified with a container ID. The LIMS generates sample labels that are attached to each bottle for a given sample.

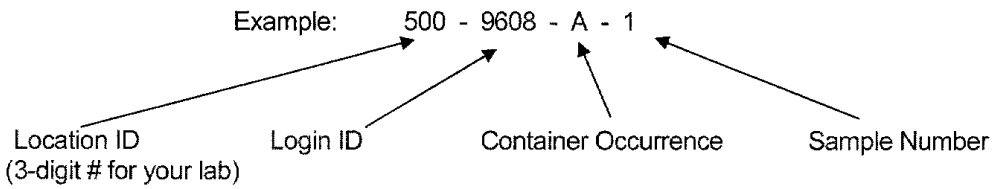
Each job/set of samples is logged into LIMS with a minimum of the following information:

- Client Name, Project Name, Address, Phone, Fax, Report to information, invoice to information (most of this information is "default information" that is stored in the LIMS).
- Date and time sampled;
- Date and time received;
- Job and/or project description, sample description;
- Sample matrix, special sample remarks;
- Reporting requirements (i.e., QC level, report format, invoicing format);
- Turn-around-time requirements;
- Parameters (methods and reporting limits or MDLs are default information for a given parameter)

24.2.2.1 Unique Sample Identification

All samples that are processed through the laboratory receive a unique sample identification to ensure that there can be no confusion regarding the identity of such samples at anytime. This system includes identification for all samples, subsamples and subsequent extracts and/or digestates.

The laboratory assigns a unique identification (e.g., Sample ID) code to each sample container received at the laboratory. This Primary ID is made up of the following information (consisting of 4 components):



The above example states that TestAmerica Chicago Laboratory (Location 500). Login ID is 9608 (unique to a particular client/job occurrence). The container code indicates it is the first container ("A") of Sample #1.

If the primary container goes through a prep step that creates a "new" container, then the new container is considered secondary and gets another ID. An example of this being a client sample in a 1-Liter amber bottle is sent through a Liquid/Liquid Extraction and an extraction vial is created from this step. The vial would be a SECONDARY container. The secondary ID has 5 components.

Example: 500 - 9608 - A - 1 - **A** ←——**Secondary Container Occurrence**

Example: 500-9608-A-1-A, would indicate the PRIMARY container listed above that went through a step that created the 1st occurrence of a Secondary container.

With this system, a client sample can literally be tracked throughout the laboratory in every step from receipt to disposal.

24.3 SAMPLE ACCEPTANCE POLICY

The laboratory has a written sample acceptance policy (Figure 24-5) that clearly outlines the circumstances under which samples shall be accepted or rejected. These include:

- a COC filled out completely;
- samples must be properly labeled;
- proper sample containers with adequate volume for the analysis (Sampling Guide) and necessary QC;
- samples must be preserved according to the requirements of the requested analytical method (Sampling Guide);
- sample holding times must be adhered to (Sampling Guide);
- all samples submitted for water/solid Volatile Organic analyses must have a Trip Blank submitted at the same time;
- the project manager will be notified if any sample is received in damaged condition.

Data from samples which do not meet these criteria are flagged and the nature of the variation from policy is defined. A copy of the sample acceptance policy is provided to each client prior to shipment of samples.

24.4 SAMPLE STORAGE *(ISO17025 5.8.4)*

In order to avoid deterioration, contamination or damage to a sample during storage and handling, from the time of receipt until all analyses are complete, samples are stored in refrigerators suitable for the sample matrix. Metals digestates are stored unrefrigerated in a sample storage closet in the metals instrument lab. In addition, samples to be analyzed for volatile organic parameters are stored in separate refrigerators designated for volatile organic parameters only. Samples are never to be stored with reagents, standards or materials that may create contamination.

To ensure the integrity of the samples during storage, refrigerator blanks are maintained in the volatile sample refrigerators and analyzed at a minimum every two weeks. Refer to laboratory SOP *UP-QA-022, Refrigerated Storage Monitoring –Volatile Samples* for additional details.

Analysts and technicians retrieve the sample container allocated to their analysis from the designated refrigerator and place them on carts, analyze the sample, and return the remaining sample or empty container to the refrigerator from which it originally came. All unused portions of samples, including empty sample containers, are returned to the secure sample control area. All samples are kept in the refrigerators for four to six weeks after analysis, which meets or exceeds most sample holding times. In general, samples that are not used for validation purposes are retained for 30 days and samples that are being used for validation purposes are retained for 60 days after the final report has been submitted to the client. This holding period allows samples to be checked if a discrepancy or question is raised by the client. Special arrangements may be made to store samples for longer periods of time.

Access to the laboratory is controlled such that sample storage need not be locked at all times unless a project specifically demands it. Samples are accessible to laboratory personnel only. Visitors to the laboratory are prohibited from entering the refrigerator and laboratory areas unless accompanied by an employee of TestAmerica.

24.5 HAZARDOUS SAMPLES AND FOREIGN SOILS

To minimize exposure to personnel and to avoid potential accidents, known or found to be hazardous samples may be stored in an isolated area, in a fume hood or with secondary containment. The sample itself is clearly marked with a sample label reading "HAZARDOUS" or "FOREIGN SOIL". The date, log number, lab sample number, and the result or brief description of the hazard are all written on the bottle. Analysts will notify a Sample Custodian or the Health and Safety Manager of any sample determined to be hazardous after completion of analysis. All hazardous samples are either returned to the client or disposed of appropriately in accordance with current regulations through a hazardous waste disposal firm that lab-packs all hazardous samples and removes them from the laboratory. Foreign soil samples are autoclaved by the laboratory or sent out for incineration by a USDA-approved waste disposal facility.

24.6 SAMPLE SHIPPING *(ISO17025 5.8.2)*

In the event that the laboratory needs to ship samples, the samples are placed in a cooler with enough ice to ensure the samples remain just above freezing and at or below 6.0°C during transit. The samples are carefully surrounded by packing material to avoid breakage (yet maintain appropriate temperature). A trip blank, that which was originally received with the samples, is enclosed for those samples requiring water/solid volatile organic analyses (see Note).. The chain-of-custody form is signed by the sample Login personnel and attached to the shipping paperwork. Samples are generally shipped overnight express or hand-delivered by a TestAmerica courier to maintain sample integrity. All personnel involved with shipping and receiving samples must be trained to maintain the proper chain-of-custody documentation and to keep the samples intact and on ice. The Environmental, Health and Safety Manual contains additional shipping requirements.

Note: If a client does not request trip blank analysis on the COC or other paperwork, the laboratory will not analyze the trip blanks that were supplied. However, in the interest of good client service, the laboratory will advise the client at the time of sample receipt that it was noted that they did not request analysis of the trip blank; and that the laboratory is providing the notification to verify that they are not inadvertently omitting a key part of regulatory compliance testing.

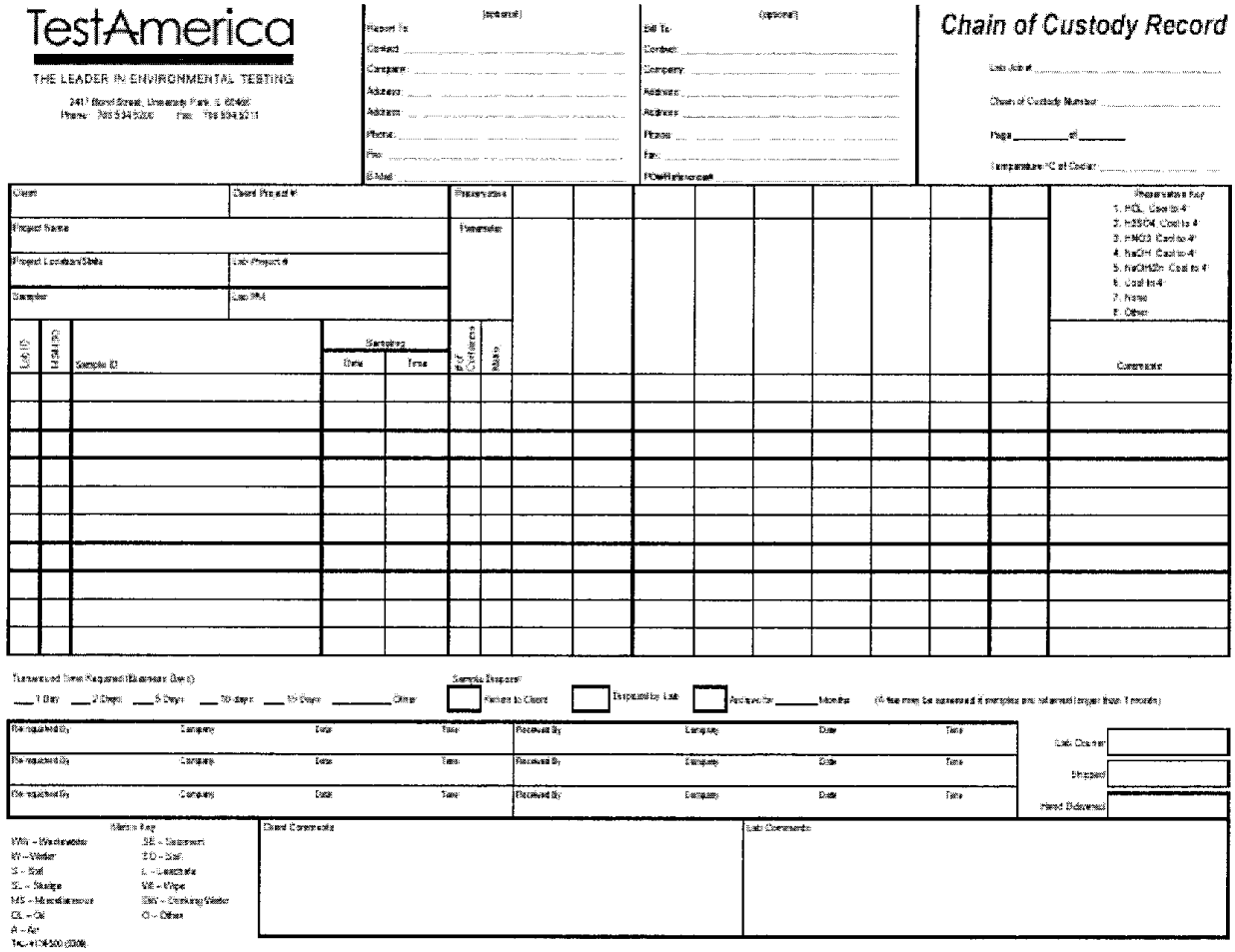
24.7 SAMPLE DISPOSAL

Samples should be retained for a minimum of 30 days after the project report is sent, however, provisions may be made for earlier disposal of samples once the holding time is exceeded. Some samples are required to be held for longer periods based on regulatory or client requirements (e.g., 60 days after project report is sent). The laboratory must follow the longer sample retention requirements where required by regulation or client agreement. Several possibilities for sample disposal exist: the sample may be consumed completely during analysis, the sample may be returned to the customer or location of sampling for disposal, or the sample may be disposed of in accordance with the laboratory's waste disposal procedures (SOP *UP—WM-001*, Laboratory Waste Disposal Procedure). All procedures in the laboratory Environmental, Health and Safety Manual are followed during disposal. Samples are normally maintained in the laboratory no longer than three months from receipt unless otherwise requested. Unused portions of samples found or suspected to be hazardous according to state or federal guidelines may be returned to the client upon completion of the analytical work.

If a sample is part of a known litigation, the affected legal authority, sample data user, and/or submitter of the sample must participate in the decision about the sample's disposal. All documentation and correspondence concerning the disposal decision process must be kept on file. Pertinent information includes the date of disposal, nature of disposal (such as sample depletion, hazardous waste facility disposal, or return to client), names of individuals who conducted the arrangements and physically completed the task. The laboratory will remove or deface sample labels prior to disposal unless this is accomplished through the disposal method (e.g., samples are incinerated). A Waste Disposal Record (Figure 24-4) should be completed.

Figure 24-1.

Example: Chain of Custody (COC)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Chain of Custody Record

Figure 24-2.

Example: Custody Seal

Figure 24-3.

Example: Internal Chain of Custody (COC)

TestAmerica Chicago
Internal Sample Custody Transfer Record

Job#:_____ **Client:**_____

Sample No.	Analysis	Relinquished by:	Received by:	Date	Time	Comments

Figure 24-4.

Example: Sample Disposal Records

Sample Disposal via Aeration and/or Neutralization **Page No.:**_____

Drum No.	Aeration Start Date/Time	Aeration Stop Date/Time	Neutralized (pH)	Disposed By	Disposal Date
	/	/			
	/	/			
	/	/			
	/	/			

Figure 24-5.

Example: Sample Acceptance Policy

All incoming work will be evaluated against the criteria listed below. Where applicable, data from any samples that do not meet the criteria listed below will be documented on an NCM and on the laboratory report defining the nature and substance of the variation. In addition the client will be notified either by telephone, fax or e-mail ASAP after the receipt of the samples. For further details, refer to the following TestAmerica Chicago SOP: *UP-SR-001- Sample Receipt: Handling and Processing Procedures*

TestAmerica Chicago Sample Acceptance Policy

The following describes TestAmerica Chicago's Sample Acceptance Policy. Upon receipt of samples at the facility, the laboratory will assess all samples based upon the following criteria. The purpose of such criteria is to maintain the integrity of the samples and ensure that proper sampling and preservation procedures have been followed. Samples found to be in 'non-compliance' with this policy will be formally addressed and conditions documented according to internal operating procedures. Subsequent analysis of such samples may or may not proceed and will be determined by discussion with the appropriate parties involved.

Samples are considered "compromised" if the following conditions are observed upon sample receipt:

- Cooler and/or samples are received outside of temperature specification.
- Samples are received broken or leaking.
- Samples are received beyond holding time.
- Samples are received without appropriate preservation.
- Samples are received in inappropriate containers.
- COC does not match samples received.
- COC is not properly completed or not received.
- Breakage of any Custody Seal.
- Apparent tampering with cooler and/or samples.
- Headspace in volatiles samples.
- Seepage of extraneous water or materials into samples.
- Inadequate sample volume.
- Illegible, impermanent, or non-unique sample labeling.

This policy will be made available to all TestAmerica Chicago clients where applicable.

Figure 24-6.

Example: Cooler Receipt Form *(ISO17025 5.8.3)*

LOGIN SAMPLE RECEIPT CHECK LIST

Client:_____ **Job Number:**_____

Login Number:_____

Question	Answer	Comment
Radioactivity either was not measured or, if measured, is at or below background	True	
The Cooler's custody seal, if present, is intact	True	
The cooler or samples do not appear to have been compromised or tampered with	True	
Samples were received on ice.	True	3.3, 3.1, 2.7, 2.5
Cooler Temperature is acceptable	True	
Cooler Temperature is recorded	True	
COC is present	True	
COC is filled out in ink and legible	True	
COC is filled out with pertinent information	True	
Is the Field Sampler's name present on COC?	True	
There are no discrepancies between the sample IDs on the containers and the COC	True	
Samples are received within Holding Time	True	
Sample containers have legible labels	True	
Samples are not broken or leaking	True	
Sample collection dates/times are provided	True	
Appropriate sample containers are used	True	
Sample bottles are completely filled	True	
Sample Preservation Verified	True	
There is sufficient vol. for all requested analyses including any requested MS/MSDs	True	
VOA sample vials do not have headspace or bubble is < 6mm (1/4") in diameter	True	
If necessary, staff have been informed of any short hold time or quick TAT needs	True	
Multiphasis samples are not present	True	
Samples do not require splitting or compositing	True	

SECTION 25.0

ASSURING THE QUALITY OF TEST RESULTS
(NELAC 5.5.9)

25.1 OVERVIEW *(ISO17025 5.9.2)*

In order to assure our clients of the validity of their data, the laboratory continuously evaluates the quality of the analytical process. The analytical process is controlled not only by instrument calibration as discussed in Section 21, but also by routine process quality control measurements (e.g. Blanks, Laboratory Control Samples (LCS), Matrix Spikes (MS), duplicates (DUP), surrogates, Internal Standards (IS)). These quality control checks are performed as required by the method or regulations to assess precision and accuracy. In addition to the routine process quality control samples, Proficiency Testing (PT) Samples (concentrations unknown to laboratory) are analyzed to help ensure laboratory performance.

25.2 CONTROLS *(ISO17025 5.9.2)*

Sample preparation or pre-treatment is commonly required before analysis. Typical preparation steps include homogenization, grinding, solvent extraction, sonication, acid digestion, distillation, reflux, evaporation, drying and ashing. During these pre-treatment steps, samples are arranged into discreet manageable groups referred to as preparation (prep) batches. Prep batches provide a means to control variability in sample treatment. Control samples are added to each prep batch to monitor method performance and are processed through the entire analytical procedure with investigative/field samples.

25.3 NEGATIVE CONTROLS *(ISO17025 5.9.2)*

25.3.1 **Method Blanks** are used to assess preparation and analysis for possible contamination during the preparation and processing steps.

25.3.1.1 The method blank is prepared from a clean matrix similar to that of the associated samples that is free from target analytes (e.g., Reagent water, Ottawa sand, glass beads, etc.) and is processed along with and under the same conditions as the associated samples.

25.3.1.2 The method blank goes through all of the steps of the process (including as necessary: filtration, clean-ups, etc.).

25.3.1.3 The specific frequency of use for method blanks during the analytical sequence is defined in the specific standard operating procedure for each analysis. Generally it is 1 for each batch of samples; not to exceed 20 environmental samples.

25.3.1.4 Evaluation criteria and corrective action for method blanks is defined in the specific standard operating procedure for each analysis. Corrective action is taken if the concentration of a target analyte in the blank is greater than the reporting limit for the project being performed. Note that some programs have more stringent criteria (DoD requires method blanks to be < ½ the reporting limit.)

- The source of contamination is investigated
- Measures are taken to minimize or eliminate the source of the contamination
- Affected samples are reprocessed or the results are qualified on the final report.

25.3.2 **Calibration Blanks** are prepared and analyzed along with calibration standards where applicable. They are prepared using the same reagents that are used to prepare the standards. In some analyses the calibration blank may be included in the calibration curve.

25.3.3 **Instrument Blanks** are blank reagents or reagent water that may be processed during an analytical sequence in order to assess contamination in the analytical system. In general, instrument blanks are used to differentiate between contamination caused by the analytical system and that caused by the sample handling or sample prep process. Instrument blanks may also be inserted throughout the analytical sequence to minimize the effect of carryover from samples with high analyte content.

25.3.4 **Trip Blanks** are required to be submitted by the client with each shipment of samples requiring aqueous and solid volatiles analyses. Additionally, trip blanks may be prepared and analyzed for volatile analysis of air samples, when required by the client. A trip blank may be purchased (certified clean) or is prepared by the laboratory by filling a clean container with pure deionized water that has been purged to remove any volatile compounds. Appropriate preservatives are also added to the container. The trip blank is sent with the bottle order and is intended to reflect the environment that the containers are subjected to throughout shipping and handling and help identify possible sources if contamination is found. The field sampler returns the trip blank in the cooler with the field samples. Trip Blanks are also sometimes referred to as Travel Blanks.

25.3.5 **Field Blanks** are sometimes used for specific projects by the field samplers. A field blank prepared in the field by filling a clean container with pure reagent water and appropriate preservative, if any, for the specific sampling activity being undertaken. (EPA OSWER)

25.3.6 **Equipment Blanks** are also sometimes created in the field for specific projects. An equipment blank is a sample of analyte-free media which has been used to rinse common sampling equipment to check effectiveness of decontamination procedures. (NELAC)

25.3.7 **Holding Blanks**, also referred to as refrigerator or freezer blanks, are used to monitor the sample storage units for volatile organic compounds during the storage of VOA samples in the laboratory (refer to section 24.4).

25.3.8 Field blanks, equipment blank and trip blanks, when received, are analyzed in the same manner as other field samples. When known, blanks should not be selected for matrix QC, as it does not provide information on the behavior of the target compounds in the field samples. Usually, the client sample ID will provide information to identify the field blanks with labels such as "FB", "EB", or "TB".

Evaluation criteria and corrective action for these controls are defined in the specific standard operating procedure for each analysis.

25.4 POSITIVE CONTROLS *(ISO17025 5.9.2)*

Control samples (e.g., QC indicators) are analyzed with each batch of samples to evaluate data based upon (1) Method Performance (Laboratory Control Sample (LCS) or Blank Spike (BS)), which entails both the preparation and measurement steps; and (2) Matrix Effects (Matrix Spike (MS) or Sample Duplicate (MD, DUP), which evaluates field sampling accuracy, precision,

representativeness, interferences, and the effect of the matrix on the method performed. Each regulatory program and each method within those programs specify the control samples that are prepared and/or analyzed with a specific batch

Note that frequency of control samples vary with specific regulatory, methodology and project specific criteria. Complete details on method control samples are as listed in each analytical SOP also refer to Appendix 3 for a listing of the laboratory's default criteria and Appendix 8 for the DoD QSM Version 4.2 criteria that is followed.

25.4.1 Method Performance Control - Laboratory Control Sample (LCS)

25.4.1.1 The LCS measures the accuracy of the method in a blank matrix and assesses method performance independent of potential field sample matrix affects in a laboratory batch.

25.4.1.2 The LCS is prepared from a clean matrix similar to that of the associated samples that is free from target analytes (for example: Reagent water, Ottawa sand, glass beads, etc.) and is processed along with and under the same conditions as the associated samples. The LCS is spiked with verified known amounts of analytes or is made of a material containing known and verified amounts of analytes, taken through all preparation and analysis steps along with the field samples. Where there is no preparation taken for an analysis (such as in aqueous volatiles), or when all samples and standards undergo the same preparation and analysis process (such as Phosphorus), a calibration verification standard is reported as the LCS. In some instances where there is no practical clean solid matrix available, aqueous LCS's may be processed for solid matrices; final results may be calculated as mg/kg or ug/kg, assuming 100% solids and a weight equivalent to the aliquot used for the corresponding field samples, to facilitate comparison with the field samples.

25.4.1.3 Certified pre-made reference material purchased from a NIST/A2LA accredited vendor may also be used for the LCS when the material represents the sample matrix or the analyte is not easily spiked (e.g. solid matrix LCS for metals, TDS, etc.).

25.4.1.4 As stated in the opening of this section, the LCS goes through all of the steps of the process (including as necessary: filtration, clean-ups, etc.).

25.4.1.5 The specific frequency of use for LCS during the analytical sequence is defined in the specific standard operating procedure for each analysis, it is also defined in Appendix 3 (Default criteria) and in Appendix 8 (DoD QSM Version 4.2 criteria). The frequency is generally 1 for each batch of samples; not to exceed 20 environmental samples.

25.4.1.6 If the mandated or requested test method, or project requirements, do not specify the spiking components, the laboratory shall spike all reportable components to be reported in the Laboratory Control Sample (and Matrix Spike) where applicable (e.g. no spike of pH). However, in cases where the components interfere with accurate assessment (such as simultaneously spiking chlordane, toxaphene and PCBs in Method 608), the test method has an extremely long list of components or components are incompatible, at a minimum, a representative number of the listed components (see below) shall be used to control the test method. The selected components of each spiking mix shall represent all chemistries, elution patterns and masses, permit specified analytes and other client requested components. However, the laboratory shall ensure that all reported components are used in the spike mixture within a two-year time period.

25.4.1.6.1 For methods that have 1-10 target analytes, spike all components.

25.4.1.6.2 For methods that include 11-20 target analytes, spike at least 10 or 80%, whichever is greater.

25.4.1.6.3 For methods with more than 20 target analytes, spike at least 16 components.

25.4.1.6.4 Exception: Due to analyte incompatibility in pesticides, separate Toxaphene and Chlordane standards are only spiked at client request based on specific project needs. If required to meet project objectives, separate LCSs can be prepared and analyzed for Toxaphene, Chlordane, or an alternate Aroclor and project specified limits will be applied. The project / job notes in LIMs will be used to identify which projects require the additional LCS spikes.

25.4.1.6.5 Exception: Due to analyte incompatibility between the various PCB aroclors, aroclors 1016 and 1260 are used for spiking as they cover the range of all of the aroclors. Specific aroclors may be used by request on a project specific basis.

25.4.1.7 **Accuracy Calculation**: Percent Recovery (%R) Calculation (applies to LCS, CCV, Surrogates, and Matrix Spikes).

$$\%R = \frac{AV}{TV} \times 100$$

Where: AV = Analyzed Value
TV = True Value

25.5 SAMPLE MATRIX CONTROLS *(ISO17025 5.9.2)*

25.5.1 Matrix Spikes (MS)

25.5.1.1 The Matrix spike is used to assess the effect sample matrix of the spiked sample has on the precision and accuracy of the results generated by the method used.

25.5.1.2 An MS is essentially a sample fortified with a known amount of the test analyte(s). At a minimum, with each matrix-specific batch of samples processed, an MS is carried through the complete analytical procedure. Unless specified by the client, samples used for spiking are randomly selected and rotated between different client projects.

25.5.1.3 If the mandated or requested test method does not specify the spiking components, the laboratory shall spike all reportable components to be reported in the Laboratory Control Sample and Matrix Spike. However, in cases where the components interfere with accurate assessment (such as simultaneously spiking chlordane, toxaphene and PCBs in Method 608), the test method has an extremely long list of components or components are incompatible, a representative number of the listed components (see LCS analytes discussion in Section 25.4.1.6 above) may be used to control the test method. The selected components of each spiking mix shall represent all chemistries, elution patterns and masses, permit-specified analytes and other client requested components. However, the laboratory shall ensure that all reported components are used in the spike mixture within a two-year time period.

25.5.1.4 The percent recovery calculation for matrix spikes is essentially the same as the calculation shown in 25.4.1.7 except that:

AV = Sp – Sa

Where: Sp = Spike result
Sa = Sample result

25.5.2 Surrogate Spikes

25.5.2.1 Surrogate Spikes are similar to matrix spikes except the analytes are compounds with properties that mimic the analyte of interest and are unlikely to be found in environment samples for organic analysis.

25.5.2.2 Surrogate compounds are added to all samples, standards, and blanks, for all organic chromatography methods except when the matrix precludes its use or when a surrogate is not available. The recovery of the surrogates is compared to the acceptance limits for the specific method (also refer to Section 25.5). Poor surrogate recovery may indicate a problem with sample composition and shall be reported, with data qualifiers, to the client whose sample produced poor recovery.

25.5.3 Duplicates

25.5.3.1 For a measure of analytical precision, with each matrix-specific batch of samples processed, a matrix duplicate (MD or DUP) sample, matrix spike duplicate (MSD), or LCS duplicate (LCSD) is carried through the complete analytical procedure. Duplicate samples are usually analyzed with methods that do not require matrix spike analysis. LCSD's are normally not performed except when regulatory agencies or client specifications require them. The recoveries for the spiked duplicate samples must meet the same laboratory established recovery limits as the accuracy QC samples. If an LCSD is analyzed both the LCS and LCSD must meet the same recovery criteria and be included in the final report. The precision measurement is reported as "Relative Percent Difference" (RPD). Poor precision between duplicates (except LCS/LCSD) may indicate non-homogeneous matrix or sampling.

25.5.3.2 **Precision Calculation** (Relative Percent Difference - RPD)

$$RPD = \frac{|S - D|}{\frac{(S + D)}{2}} \times 100$$

Where: S=Sample Concentration
D=Duplicate Concentration

25.5.4 Internal Standards

25.5.4.1 In most organic analyses, internal standards are spiked into all environmental and quality control samples (including the initial calibration standards). An internal standard is also used with some metals analyses (ICP and ICPMS). It is added to sample extracts after the extraction (post-prep). The acceptance criteria in most organic methods are 50% to 200% of the responses in the mid-point of the corresponding calibration curve. Consult the method-specific SOPs for details on the internal standard compounds, calculations and acceptance criteria.

25.5.4.2 When the internal standard recoveries fall outside these limits, if there are not obvious chromatographic interferences, reanalyze the sample to confirm a possible matrix effect. If the recoveries confirm or there was obvious interference, results are reported from the original analysis and a qualifier is added. If the reanalysis meets internal standard recovery criteria, the second run is reported (or both are reported if requested by the client).

25.6 ACCEPTANCE CRITERIA (CONTROL LIMITS)

25.6.1 As mandated by the test method or regulation, each individual analyte in the LCS, MS, or Surrogate Spike are evaluated against the control limits as published in the test method. Where there are no established acceptance criteria, the laboratory calculates in-house control limits with the use of control charts or, in some cases, utilizes client project specific or regulatory mandated control limits. When this occurs, the regulatory or project limits will supersede the laboratory's in-house limits.

Control charts are monitored to detect trends such as shifts in mean recovery, changes in standard deviation, and to assist in troubleshooting apparent trends. Control charts are also used as a visual aid to help ensure that an appropriate set of data is being selected when setting control limits. More rigorous statistical tests can also be used to determine if different portions of the selected data belong to the same population and may be pooled. For example, the t-test can be used to test means and the F-test can be used to test variances.

Note: For methods, analytes and matrices with very limited data (e.g., unusual matrices not analyzed often), interim limits are established using available data or by analogy to similar methods or matrices.

25.6.2 Once control limits have been established, they are verified, reviewed, and updated if necessary on an annual basis unless the method requires more frequent updating or significant changes to the method or preparation procedure warrant re-evaluation of the limits (e.g. EPA SW846 8000 series methods). Control limits are established per method (as opposed to per instrument) regardless of the number of instruments utilized. Refer to Section 5.6.1 for additional details on control charts.

25.6.2.1 The lab should consider the effects of the spiking concentration on the control limits. The acceptance criteria for recovery and precision are often a function of the spike concentration used. Therefore, caution must be used when pooling data to generate control limits.

25.6.2.2 Not only should the results all be from a similar matrix, but the spiking levels should also be approximately the same (within a factor of 2). Similarly, the matrix spike and surrogate results should all be generated using the same set of extraction, cleanup and analysis techniques. For example, results from solid samples extracted by ultrasonic extraction are not mixed with those extracted by Soxhlet.

25.6.2.3 The laboratory should try and avoid discarding data that do not meet a preconceived notion of acceptable performance. This results in a censored data set, which, when used to develop acceptance criteria, will lead to unrealistically narrow criteria. For a 99% confidence interval, 1 out of every 100 observations likely will still fall outside the limits. For methods with long analyte lists this may mean occasional failures every batch or two. While professional

judgment is important in evaluating data to be used to develop acceptance criteria, specific results are not discarded simply because they do not meet one's expectations. However, data points shall be discarded if they were the result of human or mechanical error. Matrix spike results where the native analyte concentration is > 4 times the spike level are calculated and reported in the TestAmerica Chicago LIMs system with qualification. Further evaluation and narration of the MS/MSD results based on program specific criteria may be required.

25.6.3 Laboratory generated % Recovery acceptance (control) limits are generally established by taking $\pm$ 3 Standard Deviations (99% confidence level) from the average recovery of a minimum of 20-30 data points (more points are preferred).

25.6.3.1 Regardless of the calculated limit, the limit should be no tighter than the Calibration Verification (ICV/CCV), unless the analytical method specifies a tighter limit.

25.6.3.2 In-house limits cannot be any wider than those mandated in a regulated analytical method. Client or contract required control limits are evaluated against the laboratory's statistically derived control limits to determine if the data quality objectives (DQOs) can be achieved. If the laboratory control limits are not consistent with DQOs, then alternatives must be considered, such as method improvements or use of an alternate analytical method.

25.6.3.3 The lowest acceptable recovery limit will be 10% (the analyte must be detectable and identifiable). Exception: The lowest acceptable recovery limit for Benzidine will be 5% and the analyte must be detectable and identifiable.

25.6.3.4 The maximum acceptable recovery limit will be 150%.

25.6.3.5 The maximum acceptable RPD limit will be 20% for waters and 30% for soils. The minimum RPD limit is 0%.

25.6.3.6 If the calculated control limit changes by $\leq$ 5% from previous, the control chart is visually inspected and, using professional judgment, they may be left unchanged if there is no affect on laboratory ability to meet the existing limits. Additional statistical tests (e.g., t and F tests) may be applied to the mean and variance to determine if the new data belong to the same population and may be pooled to establish new control limits.

25.6.4 The lab must be able to generate a current listing of their control limits and track when the updates are performed. In addition, the laboratory must be able to recreate historical control limits.

25.6.4.1 The QA department generates a Control Limit Summary table for each method and applicable matrix that contains the precision and accuracy acceptability limits for analyses performed at TestAmerica Chicago. This summary includes the date range from which the limits were generated and an approval date. The effective date for the control limits are governed by the date that the limits are entered into the LIMs system. The tables and LIMs are updated each time new limits are generated. The current summary tables and any historical limits are located in the W:/QC/LIMITS/STATS directory. Unless otherwise noted, limits within these tables are laboratory generated. The control limits that are entered into the Laboratory Information Management System (LIMS) are used by the methods in the evaluation of the data. Each applicable method and matrix is associated with a Method Limit Group in LIMs which houses all applicable limits used by the system in the evaluation of the data. The Quality Assurance department maintains an archive of all limits used within the laboratory.

25.6.5 A LCS that is within the acceptance criteria establishes that the analytical system is in control and is used to validate the process. Samples that are analyzed with an LCS with recoveries outside of the acceptance limits may be determined as out of control and should be reanalyzed if possible. If reanalysis is not possible, then the results for all affected analytes for samples within the same batch must be qualified when reported. The internal corrective action process (see Section 13) is also initiated if an LCS exceeds the acceptance limits. Sample results may be qualified and reported without reanalysis if:

25.6.5.1 The analyte results are below the reporting limit and the LCS is above the upper control limit.

25.6.5.2 If the analytical results are above the relevant regulatory limit and the LCS is below the lower control limit.

25.6.5.3 Or, for NELAC and Departement of Defense (DoD) work, there are an allowable number of Marginal Exceedances (ME):

- <11 analytes – 0 marginal exceedances are allowed.
- 11 – 30 Analytes – 1 marginal exceedance is allowed
- 31-50 Analytes – 2 marginal exceedances are allowed
- 51-70 Analytes – 3 marginal exceedances are allowed
- 71-90 Analytes – 4 marginal exceedances are allowed
- > 90 Analytes – 5 marginal exceedances are allowed

25.6.5.3.1 Marginal exceedances are recovery exceedances between 3 SD and 4 SD from the mean recovery limit (NELAC).

25.6.5.3.2 Marginal exceedances must be random. If the same analyte exceeds the LCS control limit repeatedly, it is an indication of a systematic problem. The source of the error must be located and corrective action taken. The laboratory has a system to monitor marginal exceedances to ensure that they are random.

25.6.5.3.3 Though marginal excedences may be allowed, the data must still be qualified to indicate it is outside of the normal limits.

25.6.5.3.4 The DoD QSM 4.2, Appendix G requires that the "when a laboratory's in-house limits are outside the DoD control limits (upper and/or lower), they must report their in-house limits in the laboratory report even if the LCS associated with the batch fell within the DoD limits." The laboratory will evaluate the DoD QSM 4.2 control limits against the laboratories internally derived statistical limits. Those internal limits that do not meet the DoD QSM 4.2 control limits will be used in the laboratory report. Those analytes which utilize the statistical limits as opposed to the DoD QSM 4.2 designated control limits will be clearly outlined in the case narrative of the report.

25.6.6 If the MS/MSDs do not meet acceptance limits, the MS/MSD and the associated spiked sample is reported with a qualifier for those analytes that do not meet limits. If obvious preparation errors are suspected, or if requested by the client, unacceptable MS/MSDs are reprocessed and reanalyzed to prove matrix interference. A more detailed discussion of acceptance criteria and corrective action can be found in Appendix 3 (Default criteria), Appendix 8 (DoD QSM 4.2 criteria), within the method specific SOPs and in Section 13.

25.6.7 If a surrogate standard falls outside the acceptance limits, if there is not obvious chromatographic matrix interference, reanalyze the sample to confirm a possible matrix effect. If the recoveries confirm or there was obvious chromatographic interference, results are reported from the original analysis and a qualifier is added. If the reanalysis meets surrogate recovery criteria, the second run is reported (or both are reported if requested by the client). Under certain circumstances, where all of the samples are from the same location and share similar chromatography, the reanalysis may be performed on a single sample rather than all of the samples and if the surrogate meets the recovery criteria in the reanalysis, all of the affected samples would require reanalysis. A more detailed discussion of acceptance criteria and corrective action can be found in Appendix 3 (Default criteria), Appendix 8 (DoD QSM 4.2 criteria) and within the method specific SOPs.

25.7 METHOD DETECTION LIMITS (MDLs/DLs)

MDLs/DLs, calculated as described in Section 20.7, are updated annually, or as required by the method or program. Methods that reported for compliance with the DoD QSM version 4.2 are verified on a quarterly basis.

25.8 ADDITIONAL PROCEDURES TO ASSURE QUALITY CONTROL

25.8.1 The laboratory has written and approved method SOPs to assure the accuracy of the test method including calibration (see Section 21), use of certified reference materials (see Section 22) and use of PT samples (see Section 16).

25.8.2 A discussion regarding MDLs, Limit of Detection (LOD) and Limit of Quantitation (LOQ) can be found in Section 20.

25.8.3 Use of formulae to reduce data is discussed in the method standard operating procedures and in Section 21.

25.8.4 Selection of appropriate reagents and standards is included in Section 9 and 22.

25.8.5 A discussion on selectivity of the test is included in Section 5.

25.8.6 Constant and consistent test conditions are discussed in Section 19.

25.8.7 The laboratories sample acceptance policy is included in Section 24.

25.8.8 A listing of the type of test result correlations that are looked at during report review (e.g. Total Chromium should be greater or equal to Hexavalent Chromium) is included in Section 20.14.4.3.

SECTION 26.0

REPORTING RESULTS *(NELAC 5.5.10)*

26.1 OVERVIEW *(ISO17025 5.10.1; 5.10.2; 5.10.8)*

The results of each test are reported accurately, clearly, unambiguously, and objectively in accordance with State and Federal regulations as well as client requirements. Analytical results are issued in a format that is intended to satisfy customer and laboratory accreditation requirements as well as provide the end user with the information needed to properly evaluate the results. Where there is a conflict between the client requests and laboratory ethics or regulatory requirements the laboratory's ethical and legal requirements are paramount, and an adequate solution will be devised working with the client at the time of project set up. (Refer to section 7 for additional details.)

A variety of report formats are available to meet specific needs.

In cases where a client asks for simplified reports, there must be a written request from the client. There still must be enough information that would show any analyses that were out of conformance (QC out of limits) and there should be a reference to a full report that is made available to the client.

Review of reported data is included in Section 20.

26.2 TEST REPORTS *(ISO17025 5.10.1; 5.10.2; 5.10.3.1; 5.10.3.2; 5.10.5; 5.10.6; 5.10.7; 5.10.8)*

Analytical results are reported in a format that is satisfactory to the client and meets all requirements of applicable accrediting authorities and agencies. A variety of report formats are available to meet specific needs. The report cover page is printed with the corporate letterhead logo and the laboratory name. It is reviewed and signed by the appropriate project manager or designee. At a minimum, the standard laboratory report shall contain the following information:

26.2.1 The cover page of the report is printed with the company letterhead logo, which includes the laboratory name, address, telephone number, the Project Manager's name and email address.

26.2.2 A report title (e.g. Sample Results or Analytical Data) with a "Results" column header.

26.2.3 A unique identification of the report (e.g. Job Number: 500-6101-1) and on each page an identification in order to ensure the page is recognized as part of the report and a clear identification of the end.

Note: Page numbers of report are represented as Page # of ##. Where the first number is the page number and the second is the total number of pages.

26.2.4 A copy of the chain of custody (COC).

- Any COCs involved with Subcontracting are included.
- In all cases, the applicable COC is scanned and placed in the shipping and receiving documents folder of the data deliverable for its respective job number and is paginated as an integral part of the report.
- Any additional addenda to the report must be treated in a similar fashion so it is a recognizable part of the report and cannot accidentally get separated from the report (eg. Sampling information).

26.2.5 The name and address of client and a project name/number, if applicable.

26.2.6 Client project manager or other contact

26.2.7 Description and unambiguous identification of the tested sample(s) including the client identification code.

26.2.8 Date of receipt of sample, date and time of collection, and date(s) of test preparation and performance, and time of preparation or analysis if the required holding time for either activity is less than or equal to 72 hours.

26.2.9 Date reported or date of revision, if applicable.

26.2.10 Method of analysis including method code (EPA, Standard Methods, etc).

26.2.11 Reporting limit.

26.2.12 Method detection limits (if requested)

26.2.13 Definition of Data qualifiers and reporting acronyms (e.g. ND).

26.2.14 Sample results.

26.2.15 QC data consisting of method blank, surrogate, LCS, and MS/MSD recoveries and control limits.

26.2.16 Condition of samples at receipt including temperature. This may be accomplished in a narrative or by attaching sample login sheets (Refer to Sec. 26.2.4 – Item 3 regarding additional addenda). A sample receipt checklist is completed by login personnel, temperatures are documented on the COC and on the checklist and are included as part of the data deliverable to the client.

26.2.17 A statement to the effect that the results relate only to the items tested and the sample as received by the laboratory.

26.2.18 A statement that the report shall not be reproduced except in full, without prior express written approval by the laboratory coordinator.

26.2.19 A signature and title of the person(s) accepting responsibility for the content of the report and date of issue. Signatories are appointed by the Lab Director.

26.2.20 When NELAC accreditation is required, the lab shall certify that the test results meet all requirements of NELAC or provide reasons and/or justification if they do not. The cover page of each report contains the following text: These test results meet all the requirements of NELAC for accredited parameters. The Lab Certification ID# is 100201. All questions regarding this test report should be directed to the TestAmerica Project Manager whose signature appears on this report. All pages of this report are integral parts of the analytical data. Therefore, this report should be reproduced only in its entirety.

26.2.21 Where applicable, a narrative to the report that explains the issue(s) and corrective action(s) taken in the event that a specific accreditation or certification requirement was not met.

26.2.22 When Soil samples are analyzed, a specific identification as to whether soils are reported on a "wet weight" or "dry weight" basis.

26.2.23 Appropriate laboratory certification number for the state of origin of the sample, if applicable.

26.2.24 If only part of the report is provided to the client (client requests some results before all of it is complete), it must be clearly indicated on the report (e.g., *'Preliminary Report'* is printed across each page of the report), and that a complete report will follow once all of the work has been completed.

26.2.25 Any non-TestAmerica subcontracted analysis results are provided as a separate report on the official letterhead of the subcontractor and may be appended to the TestAmerica Chicago report in its entirety for submission to the client. All TestAmerica subcontracting is clearly identified on the report as to which laboratory performed a specific analysis.

Note: Refer to the Corporate SOP on Electronic Reporting and Signature Policy (No. CA-I-P-002) for details on internally applying electronic signatures of approval.

26.3 REPORTING LEVEL OR REPORT TYPE *(ISO17025 5.10.1; 5.10.7; 5.10.8)*

The laboratory offers four levels of quality control reporting. Each level, in addition to its own specific requirements, contains all the information provided in the preceding level. The packages provide the following information in addition to the information described above:

- Level I is a report with the features described in Section 26.2 above with the exception that no QC results are reported.
- Level II is a Level I report plus batch QC summary information, including results for the method blank, percent recovery for laboratory control samples and matrix spike samples, the RPD values for all MSD, sample duplicate analyses and surrogate recoveries.
- Level III contains all the information supplied in Level II, supplemented with the presentation of data on CLP-like forms, including relevant tune (GC/MS), internal standard (GC/MS) and calibration information. No raw data is provided.
- Level IV is the same as Level III with the addition of all raw supporting data.

In addition to the various levels of QC packaging, the laboratory also provides reports in CD deliverable form. Initial reports may be provided to clients by facsimile, as a .pdf file sent via email or CD, as a hardcopy report or by any combination of the three. Procedures used to ensure client confidentiality are outlined in Section 26.6.

26.3.1 Electronic Data Deliverables (EDDs)

EDDs are routinely offered as part of TestAmerica's services. TestAmerica Chicago offers a variety of EDD formats including Environmental Resources Program Information System (ERPIMS), Environmental Restoration Information System (ERIS), Automated Data Review (ADR), Staged Electronic Data Deliverable (SEDD), EQUIS ™, GISKEY ™ and other State deliverables besides general formats in Dbase ™, Excel ™ and text.

EDD specifications are submitted to an EDD Specialist by the PM or AE for review. The specification undergoes review for contract and LIMs compatibility. Once the facility has committed to providing data in a specific electronic format, the coding of the format may need to be performed. This coding is documented and validated by the Corporate IT TALs LIMs staff. The validation of the code is retained by the Corporate IT staff coding the EDD. Changes to the format must be directed by the program or client and approved by the EDD Specialist and Corporate IT TALs LIMs staff.

EDDs shall be subject to a review to ensure their accuracy and completeness. If EDD generation is automated, review may be reduced to periodic screening if the laboratory can demonstrate that it can routinely generate that EDD without errors. Any revisions to the EDD format must be reviewed until it is demonstrated that it can routinely be generated without errors. If the EDD can be reproduced accurately and if all subsequent EDDs can be produced error-free, each EDD does not necessarily require a review. If an EDD checker program is provided to the laboratory then the EDDs will be screened using this tool.

26.4 SUPPLEMENTAL INFORMATION FOR TEST *(ISO17025 5.10.1; 5.10.3.1; 5.10.5)*

The lab identifies any unacceptable QC analyses or any other unusual circumstances or observations such as environmental conditions and any non-standard conditions that may have affected the quality of a result. This is typically in the form of a footnote or a qualifier and/or a narrative explaining the discrepancy in the front of the report. Refer to Appendix 6 for a list of the laboratory's standard footnotes and qualifiers.

26.4.1 Numeric results with values outside of the calibration range, either high or low are qualified as 'estimated'.

26.4.2 Where quality system requirements are not met, a statement of compliance/non-compliance with requirements and/or specifications is required, including identification of test results derived from any sample that did not meet NELAC sample acceptance requirements such as improper container, holding time, or temperature.

26.4.3 Where applicable, a statement on the estimated uncertainty of measurements; information on uncertainty is needed when a client's instructions so require.

26.4.4 Opinions and Interpretations - The test report contains objective information, and generally does not contain subjective information such as opinions and interpretations. If such information is required by the client, the Laboratory Director will determine if a response can be prepared. If so, the Laboratory Director will designate the appropriate member of the management team to prepare a response. The response will be fully documented, and reviewed by the Laboratory Director, before release to the client. There may be additional fees charged to the client at this time, as this is a non-routine function of the laboratory.

Note: Review of data deliverable packages for submittal to regulatory authorities requires responses to non-conforming data concerning potential impact on data quality. This necessitates a limited scope of interpretation, and this work is performed by the Department Manager/Supervisor, PM or QA Department depending on the type of non-conformance. This is the only form of "interpretation" of data that is routinely performed by the laboratory.

When opinions or interpretations are included in the report, the laboratory provides an explanation as to the basis upon which the opinions and interpretations have been made. Opinions and interpretations are clearly noted as such and where applicable, a comment should be added suggesting that the client verify the opinion or interpretation with their regulator.

26.5 ENVIRONMENTAL TESTING OBTAINED FROM SUBCONTRACTORS *(ISO17025 5.10.1; 5.10.6)*

If the laboratory is not able to provide the client the requested analysis, the samples would be subcontracted following the procedures outlined in the Corporate SOP on Subcontracting (SOP No. CA-L-S-002) also refer to in Section 8.

Data reported from analyses performed by a subcontractor laboratory are clearly identified as such on the analytical report provided to the client. Results from a subcontract laboratory outside of the TestAmerica network are reported to the client on the subcontract laboratory's original report stationary in its entirety.

26.6 CLIENT CONFIDENTIALITY *(ISO17025 4.1.5; 5.10.7)*

In situations involving the transmission of environmental test results by telephone, facsimile or other electronic means, client confidentiality must be maintained.

TestAmerica will not intentionally divulge to any person (other than the Client or any other person designated by the Client in writing) any information regarding the services provided by TestAmerica or any information disclosed to TestAmerica by the Client. Furthermore, information known to be potentially endangering to national security or an entity's proprietary rights will not be released.

Note: This shall not apply to the extent that the information is required to be disclosed by TestAmerica under the compulsion of legal process. TestAmerica will, to the extent feasible, provide reasonable notice to the client before disclosing the information.

Note: Authorized representatives of an accrediting authority are permitted to make copies of any analyses or records relevant to the accreditation process, and copies may be removed from the laboratory for purposes of assessment.

26.6.1 Report deliverable formats are discussed with each new client. If a client requests that reports be faxed or e-mailed, the reports are faxed with a cover sheet or e-mailed with the following note that includes a confidentiality statement similar to the following:

Confidentiality Notice: This e-mail communication, including any attachments, may contain privileged or confidential information for specific individuals and is protected by law. If you are not the intended recipient(s), you are hereby notified that any dissemination, distribution or copying of this communication is strictly prohibited and you should delete this message and its attachments from your computer without retaining any copies. If you have received this communication in error, please reply to the sender immediately. We appreciate your cooperation.
Please consider the environment before printing this e-mail.

26.7 FORMAT OF REPORTS *(ISO17025 5.10.8)*

The formats of reports are designed to accommodate each type of environmental test carried out and to minimize the possibility of misunderstanding or misuse.

26.8 AMENDMENTS TO TEST REPORTS *(ISO17025 5.10.1; 5.10.9)*

Corrections, additions, or deletions to reports are only made when justification arises through supplemental documentation. Justification is documented using the laboratory's corrective action system (refer to Section 13).

The revised report is retained on the document server in the TALs LIMs system, as is the original report. The revised report can be accessed through PM Desktop under the Job Number in the Deliverable folder. The revised deliverable is identified as Rev(1) next to the report which has been revised.

When the report is re-issued, a notation of "Revision: 1" is printed on the cover page with the date of the submittal. The statement regarding the nature of the revision is placed at the top of the Job Narrative page with a brief explanation of reason for the re-issue. For example: The following report required a revision: 500-6101-1. Details are as follows: The client has added MTBE to the VOC compound list.

26.9 POLICIES ON CLIENT REQUESTS FOR AMENDMENTS *(ISO17025 5.10.1; 5.10.9)*

26.9.1 Policy on Data Omissions or Reporting Limit Increases *(ISO17025 5.9.1; 5.10.5; 5.10.9)*

Fundamentally, our policy is simply to not omit previously reported results (including data qualifiers) or to not raise reporting limits and report sample results as ND. This policy has few exceptions. Exceptions are:

- Laboratory error.
- Sample identification is indeterminate (confusion between COC and sample labels).
- An incorrect analysis (not analyte) was requested (e.g., COC lists 8315 but client wanted 8310). A written request for the change is required.
- Incorrect limits reported based on regulatory requirements.
- The requested change has absolutely *no possible* impact on the interpretation of the analytical results and there is *no possibility* of the change being interpreted as misrepresentation by anyone inside or outside of our company.

26.9.2 Multiple Reports

TestAmerica does not issue multiple reports for the same workorder where there is different information on each report (this does not refer to copies of the same report) unless required to meet regulatory needs and approved by QA.

Appendix 1. *(ISO17025 4.1.5)*

TESTAMERICA
ETHICS POLICY No. CA-L-P-001

Refer to CA-L-P-001 for complete policy.

TestAmerica
EMPLOYEE ETHICS STATEMENT

I understand that TestAmerica Laboratories, Inc. and its affiliates ("TestAmerica"), are committed to ensuring the highest standard of ethical and professional conduct in all business activities. The Company and its employees will comply with all applicable laws, regulations and policies. We will ensure the highest standards of quality and integrity of the data and services provided to our clients. I have read the Ethics Policy of the Company.

With regard to the duties I perform, the data I report in connection with my employment at the Company, and all business activities, I agree that:

- *I shall not make false statements to, or seek to otherwise deceive, members of Management or their representatives, agents, or clients/customers in any aspect of my job, including timekeeping, accounting, and compliance withal safety, environmental and employment regulations.*
- *I will not, through acts of commission, omission, erasure, or destruction, improperly report measurement standards, quality control data, test results or conclusions; nor will I intentionally alter or omit dates, dollar values or other business related information in order to achieve desired financial results.*
- *I will not share the pricing or cost data of Vendors or Suppliers with anyone outside of the TestAmerica family of companies.*
- *I shall not accept gifts of a value that would adversely influence judgment.*
- *I shall avoid conflicts of interest and report any potential conflicts to the management (e.g., employment or consulting with competitors, clients, or vendors);*
- *I shall not participate in unfair competition practices (e.g., slandering competitors, collusion with other labs to restrict others from bidding on projects);*
- *I shall not take any action, personally, or on behalf of the Company, which violates any applicable law, regulation, or internal policy, or which causes the Company to incur financial risk or loss or causes the Company to report incorrect financial information.*
- *I will not intentionally report data values that are inconsistent with the actual values observed or measured.*
- *I will not intentionally report the dates, times, sample or QC identifications, or method citations of data analyses that are not the actual dates, times, sample or QC identifications, or method citations.*
- *I will not intentionally misrepresent another individual's work as my own or represent my own work as someone else's.*
- *I will not intentionally misrepresent any data where data does not meet Method or QC requirements. If it is to be reported, I will report it with all appropriate notes and/or qualifiers; I shall not modify data (either sample or QC data) unless the modification can be technically justified through a measurable analytical process, such as one deemed acceptable to the laboratory's Standard Operating Procedures, Quality Assurance Manual or Technical Director. All such modifications must be clearly and thoroughly documented in the appropriate laboratory notebooks/worksheets and/or raw data and include my initials or signature and date.*
- *I shall not compare or disclose results for any Performance Testing (PT) sample, or other similar QA or QC requirements, with any employee of any other laboratory, including any other TestAmerica*

facility, prior to the required submission date of the results to the person, organization, or entity supplying the PT sample.

- *I understand the critical importance of accurately reporting data, measurements, and results, whether initially requested by a client, or retained by TestAmerica and submitted to a client at a later date, or retained by TestAmerica for subsequent internal use;*
- *I shall not misrepresent certifications and status of certifications to clients or regulators;*
- *I shall not intentionally discharge wastes illegally down the drain or onto the ground.*
- *I shall immediately inform my supervisor or other member of management regarding any intentional or unintentional reporting of my own inauthentic data. Such report shall be given both orally and in writing to the supervisor or other member of management contacted and to the local Facility Director and Quality Assurance Officer/Manager (where applicable). The Facility Director or Quality Assurance Officer/Manager (where applicable) will initial and date the information and return a copy to me. I shall not condone any accidental or intentional reporting of inauthentic data by other employees and will immediately report its occurrence. If I have actual knowledge of such acts committed by any other employees, and I do not report such information to designated members of Management, it shall be considered as serious as if I personally committed the offense. Accordingly, in that event, I understand that I may be subject to immediate termination of employment.*
- *I understand that if any supervisor, manager, or representative of TestAmerica management instructs, requests, or directs me to perform any of the aforementioned improper laboratory practices, or illegal or unethical business activities, or if I am in doubt or uncertain as to whether or not such laboratory practices or business activities are proper, I will not comply. In fact, I must report such event to all appropriate members of Management including, but not limited to, the Facility Director, all supervisors and managers with direct line reporting relationship between me and the Facility Director, and the local Quality Assurance representative (where applicable), excluding such individuals who participated in such perceived improper instruction, request, or directive. In addition, I may contact Corporate Quality Assurance / Ethics Compliance Officer(s) for assistance.*
- *I understand that any attempt by management or an employee to circumvent these policies will be subject to disciplinary action.*

As a TestAmerica employee, I understand that I have the responsibility to conduct myself with integrity in accordance with the ethical standards described in the Ethics Policy. I will also report any information relating to possible kickbacks or violations of the Procurement Integrity Act, or other questionable conduct in the course of sales or purchasing activities. I will not knowingly participate in any such activity and will report any actual or suspected violation of this policy to management.

I understand that all of my dealings as an employee must be in compliance with applicable Federal and State laws, including regulations, environmental regulations, accounting rules, and employment laws, such as the Drug Free Workplace Act and anti-discrimination and harassment legislation.

I understand that if my job includes supervisory responsibilities, I shall not instruct, request, or direct any subordinate to perform any laboratory practice which is unethical or improper. Also, I shall not discourage, intimidate, or inhibit an employee who may choose to appropriately appeal my supervisory instruction, request, or directive which the employee perceives to be improper, nor retaliate against those who do.

The Ethics Policy has been explained to me by my supervisor or at a training session, and I have had the opportunity to ask questions if I did not understand any part of it. I understand that any violation of this policy subjects me to disciplinary action, which can include termination. In addition, I understand that any violation of this policy which relates to work under a government contract or subcontract could also subject me to the potential for prosecution under federal law.

Employee Printed Name _____

EMPLOYEE SIGNATURE _____ Date _____

Form No. CW-L-WI-002

CONFIDENTIALITY AND PROPRIETARY INFORMATION AGREEMENT

TestAmerica and their predecessors, in their businesses, have developed and use commercially valuable technical and non-technical information and to guard the legitimate interests of TestAmerica and its clients, it is necessary to protect certain information as confidential and proprietary.

I, _____(printed name), understand and acknowledge that during the term of my employment by TestAmerica, I will be privy to and entrusted with certain confidential information and trade secrets of TestAmerica and its clients.

Confidential information and trade secrets include, but are not limited to: customer and client lists; price lists; marketing and sales strategies and procedures; operational and equipment techniques; standard operating procedures; business plans and systems; quality control procedures and systems; special projects and technological research, including projects, research and reports for any government entity or client; client's plans and processes; client's manner of operation; the trade secrets of clients; client's data; vendor or supplier pricing; employee lists and personal information, and any other records, data, files, drawings, inventions, discoveries, applications, or processes which are not in the public domain.

I agree as follows:

1. I will not in any way, during the term of my employment, or at any time thereafter, except as authorized in writing by the Legal Department of TestAmerica or the client where client data is involved, disclose to others, use for my own benefit, remove from TestAmerica's premises (except to the extent off-site work is approved by my supervisor), copy or make notes of any confidential information and/or trade secrets of TestAmerica or its clients, excepting only that information which may be public knowledge through no act of my own. Technical and business information of any previous employer or other third party which I may disclose to TestAmerica shall be limited to that which was acquired legitimately and disclosed to me without restriction as to secrecy.

2. I agree that all inventions (whether or not patentable) conceived or made by me during the period of my employment by TestAmerica shall belong to TestAmerica, provided such inventions grow out of my work for TestAmerica and are related to the business of TestAmerica. I agree to disclose and assign such inventions to TestAmerica. In California, this provision shall not apply to any invention which qualifies fully under Section 2870 of the California Labor Code.

3. On termination of my employment from TestAmerica, I will deliver to TestAmerica all documents, records, notes, data, memoranda, files, manuals, equipment and things of any nature which relate in any way to confidential information and/or trade secrets of TestAmerica or its clients and which are in my possession or under my control.

4. I agree that during the period of my employment and for one (1) year from and after the termination (for any reason) of my employment with TestAmerica, I shall not directly or indirectly (without first obtaining the written permission of TestAmerica), recruit for employment, or induce to terminate his or her employment with TestAmerica, any person who is an active employee of TestAmerica on the last day of my employment with TestAmerica.

5. I acknowledge that if I were to breach any provision of this Confidentiality Agreement, money damages will be inadequate, and I hereby agree that TestAmerica shall be entitled, where appropriate, to specific performance and/or injunctive relief (i.e. to require me to comply with this Agreement). In the event that any provision of this Agreement is held to be unenforceable because of the scope, duration or area of its applicability, the court making such determination shall have the power to modify any or all such terms, and those terms shall then be applicable in such modified form and the other provisions of this Agreement shall remain in force.

6. I further acknowledge that the willingness of TestAmerica to hire me or to continue my employment constitutes full and adequate consideration for the agreements, and obligations to which I have agreed as set forth in this document.

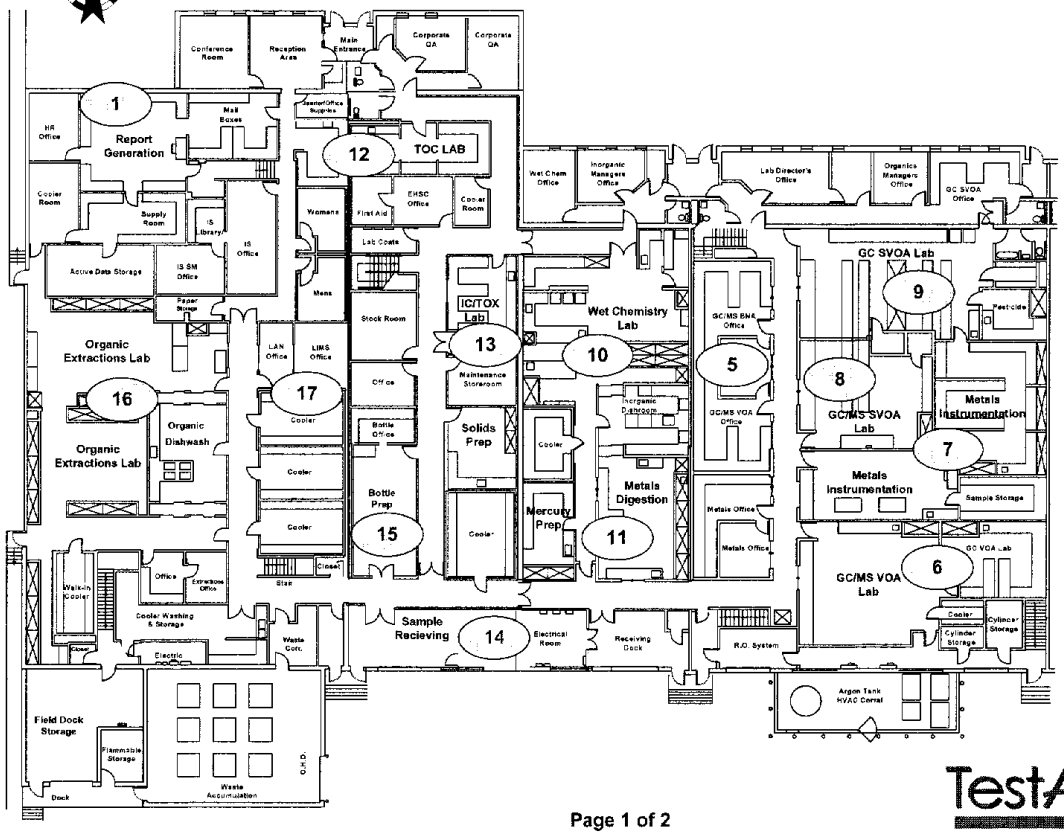
I have executed this Agreement, intending to be legally bound.

_____	_____	_____
Printed Name	*Signature*	*Date*

Form No. CW-L-WI-006

Appendix 2. **Laboratory Floor Plan**

Floor Plan of TestAmerica Chicago



Key Areas*

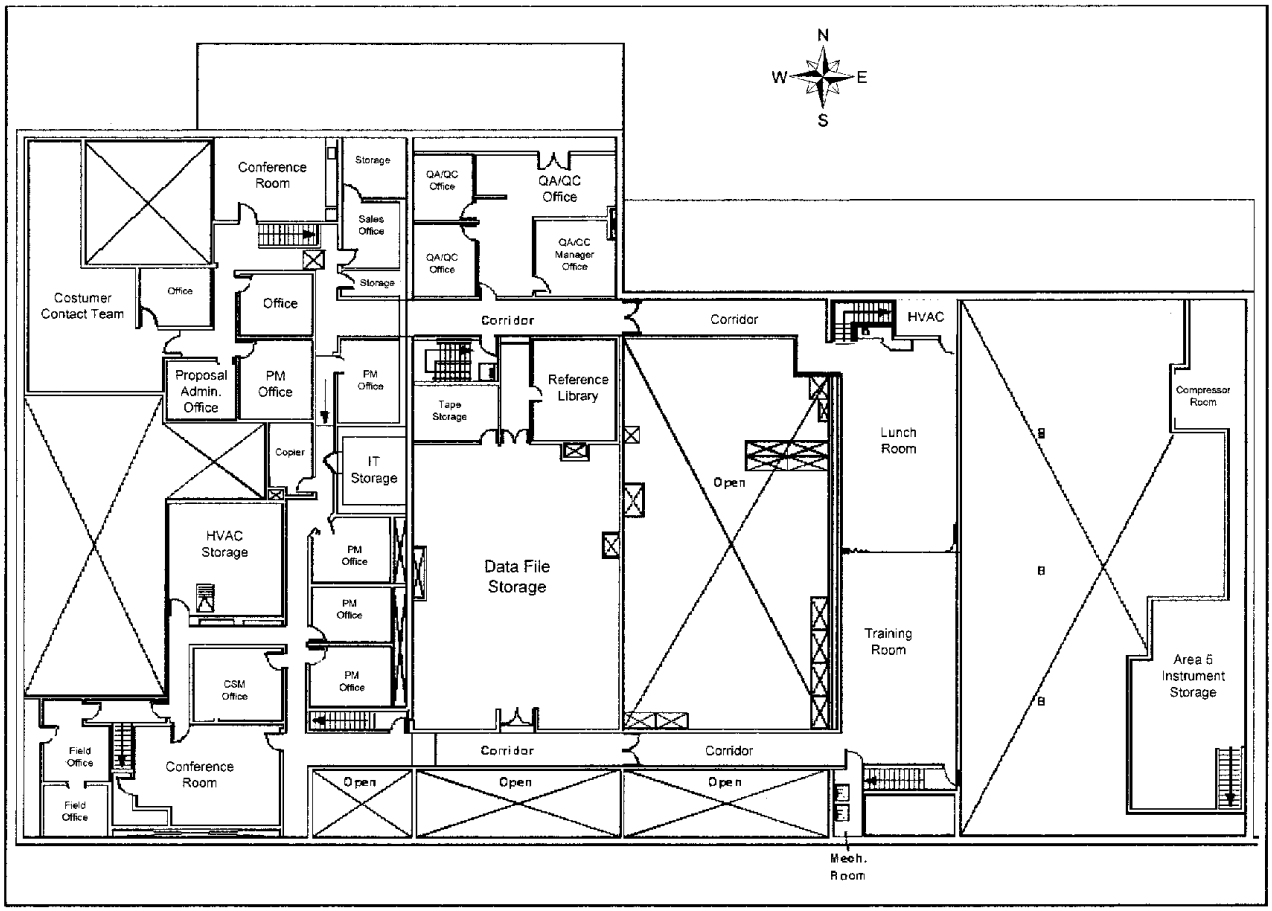
1. Report Generation
2. QA/QC
3. Training Center
4. Project Management
5. GC/MS Office
6. GC/MS GC VOA
7. Metals Instrumentation
8. GC/MS SVOA Lab
9. GC SVOA
10. Wet Chemistry
11. Metals Prep.
12. TOC Lab
13. TOX / IC Lab
14. Sample Recieving
15. Bottle Prep
16. Organic Extractions
17. LIMS/LAN

Page 1 of 2

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

Upper Floor Plan of TestAmerica Chicago



Appendix 3: Summary of Calibration and QC Procedures for GC Organics

Method	QC Check	Frequency	Acceptance Criteria[3]	Corrective Action[4]
SW8081A SW8081B SW8082 SW8082A SW8151A	Minimum five-point initial calibration for all target analytes[2]	Initial calibration prior to sample analysis. Perform instrument re-calibration once per year minimum.	Linear regression correlation coefficient $r^2 \geq 0.99$, $r \geq 0.995$. RSD of CF ≤ 20%	Correct problem then repeat initial calibration
	Initial calibration verification (ICV) must be from a 2nd source	Once immediately following initial calibration	All target analytes within 15% of expected value	Correct problem then repeat initial calibration
	Continuing calibration verification (CCV)	Before sample analysis, every 12 hours, and at the end of the analysis sequence	All analytes within 15% of expected value and within the RT Window. Average of all analytes 15%.	Repeat CCV once, if it again fails criteria, identify and correct problem, repeat initial CCV (re-calibrate if necessary) and re-analyze all samples since last successful CCV.
	Breakdown check (Endrin and DDT)[1]	Before sample analysis	Degradation ≤15% for either Endrin or DDT.	Inlet/column maintenance; repeat breakdown check and re-analyze all samples since last successful breakdown check.
	Method blank	One per analytical prep batch, not to exceed 20 samples in a batch.	No analytes detected RL values between the RL and MDL will be evaluated and flagged with a 'J'	Correct problem then re-prep and analyze method blank and all samples processed with the contaminated blank
	LCS for all analytes	One per prep batch, not to exceed 20 samples in a batch.	Statistical Control Limits	Re-prep and analyze the LCS and all samples in the affected analytical batch
	Surrogate(s)	Every sample, spike, standard, and method blank	Statistical Control Limits	Check system, re-inject, re-extract
	MS/MSD	One per batch per matrix, if insufficient sample for MS/MSD, then a LCS/LCSD will be analyzed.	Statistical Control Limits	None (LCS is used to determine if data is acceptable).
	Second-column confirmation	100% for all positive results	Same as for initial or primary column analysis	Same as for initial or primary column analysis. If the relative % difference of results between the 2 columns is greater than 40%, a comment should be placed in LIMS.
	Retention time window calculated for each analyte (see section 9 for how to calculate RTWs).	System set-up, with each new column or major instrument maintenance. Update the mid-RTW at the start of the run or daily.	Each analyte of the LCS, MS/MSD and CCV must be within the calculated RTW.	Correct the problem and re-process or re-analyze samples. If questions, see the supervisor or technical director.
	MDL verification	Minimum - quarterly	Detectible	Re-evaluate MDL standard used and MDL

1 --8081A only
2 – Method 8082, a five-point calibration is only analyzed for Aroclors 1016 and 1260.
3 - This is a summary of the acceptance criteria, refer to the method SOP for specific or more information.
4 - All abnormalities must be noted on the data, the benchsheet and in LIMS.

Appendix 3: Summary of Calibration and QC Procedures for GC Organics

Method	QC Check	Frequency	Acceptance Criteria[3]	Corrective Action[4]
EPA608	Minimum three-point (preferably five) initial calibration for all target analytes	Initial calibration prior to sample analysis. Perform instrument re-calibration once per year minimum.	**RSD of CF ≤ 10%** Linear regression - correlation coefficient $r^2 > 0.990$, $r \geq 0.995$.	Correct problem then repeat initial calibration
	Initial calibration verification (ICV) must be from a 2nd source.	Immediately following initial calibration	All analytes within 15% of expected value	Correct problem then repeat initial calibration
	Continuing calibration verification (CCV)	Before sample analysis, after every 10 samples, and at the end of the analysis sequence	All analytes within **15% of expected value** and within the RTW.	Repeat CCV once, if it again fails criteria, identify and correct problem, repeat initial CCV (re-calibrate if necessary) and re-analyze all samples since last successful CCV.
	Breakdown check (Endrin and DDT)[1]	Before sample analysis	Degradation ≤15% for either Endrin or DDT.	Inlet/column maintenance; repeat breakdown check and re-analyze all samples since last successful breakdown check.
	Method blank	One per analytical prep batch, **not to exceed 10 samples in a batch**.	No analytes detected ≥ RL values between the RL and MDL will be evaluated and flagged with a 'J'	Correct problem then re-prep and analyze method blank and all samples processed with the contaminated blank
	LCS (QC check standard)	One per prep batch, **not to exceed 10 samples in a batch.**	Method Control Limits	Re-prep[7] and analyze the LCS and all samples in the affected analytical batch
	Surrogate(s)	Every sample, spiked sample, standard, and method blank	Method Control Limits	Check system, re-inject, re-extract
	MS	One per batch per matrix, 10%, if insufficient sample for MS, then an additional LCS will be analyzed.	Method Control Limits	All target compounds should be reported, and any compounds that are outside criteria must be within criteria in the LCS.
	Second-column confirmation	100% for all positive results	Same as for initial or primary column analysis	Same as for initial or primary column analysis. If the relative % difference of results between the 2 columns is greater than 40%, a comment should be placed in LIMS.
	Retention time window calculated for each analyte (see section 9 for how to calculate RTWs).	System set-up, with each new column or major instrument maintenance. Update the mid-RTW at the start of the run or as needed.	Each analyte of the LCS, MS/MSD and CCV must be within the calculated RTW.	Correct the problem and re-process or re-analyze samples. If questions, see the supervisor or technical director.
	MDL verification	Minimum yearly	Detectible	Re-evaluate MDL standard used and MDL; see Technical Director.

3 - This is a summary of the acceptance criteria, refer to the method SOP for specific or more information.
4 - All abnormalities must be noted on the data, the benchsheet and in LIMS.

Appendix 3: Summary of Calibration and QC Procedures for GC/MS Organics

Method	QC Check	Frequency	Acceptance Criteria[2]	Corrective Action[3]
SW8260B SW8270C SW8270D	Check of mass spectral ion intensities[1], i.e., Tune	Prior to initial calibration or Continuing calibration verification, every 12 hours	Refer to criteria listed in the method SOP for Tune criteria, including DDT, Benzidine and Pentachlorophenol requirements for 8270.	Retune the instrument and verify (instrument maintenance may be needed).
SW8260B	Minimum five-point initial calibration for all target analytes	Initial calibration prior to sample analysis. Perform instrument re-calibration as necessary and after major instrument maintenance.	SPCCs average RF ≥ 0.30 or 0.1 depending on the compound and %RSD for RFs for CCCs ≤ 30% and all other target analytes %RSD grand mean <15%	Correct problem then repeat initial calibration
SW8270C			SPCCs average RF ≥ 0.050 and %RSD for RFs for CCCs ≤ 30% and all other target analytes grand mean <15%.	Correct problem then repeat initial calibration
			option (if %RSD is > 15%)–linear regression $r^2 \geq 0.990$, $r \geq 0.995$.	Refer to individual SOP for corrective action procedure.
SW8270D			All analytes < 20% RSD, up to 10% of analytes can be > 20% RSD Most analytes must meet minimum RF's (see Table 4 in 8270D SOP)	Correct problem then repeat initial calibration
SW8260B SW8270C SW8270D	Initial calibration verification (ICV) must be from a 2nd source.	Immediately following five-point initial calibration and before samples are analyzed.	All analytes within 25% of expected value (8260B; 8270C) All analytes within 30% of expected value (8270D)	Correct problem then repeat ICV, if criteria fails it may be necessary to repeat initial calibration or report outlier with PM/QA approval and qualification (NCM required).
	Relative Retention time window	Each sample	Relative retention time (RRT) of the analyte within 0.06 RRT units of the RRT of the internal standard	Correct problem then reprocess or re-analyze all samples analyzed since the last retention time check
SW8260B	Continuing calibration verification (CCV)	Daily, before sample analysis and every 12 hours of analysis time	SPCCs average RF ≥ 0.30 or 0.1 depending on the compound; and	Correct problem then repeat CCV, if criteria is met, repeat associated samples. If criteria is not met, it may be necessary to repeat initial calibration and re-analyze all samples since last successful CCV.
SW8270C			SPCCs average RF ≥ 0.050; and	
SW8260B SW8270C			CCCs: ≤20% difference (when using RFs) or drift (when using least squares regression).	
SW8270D			All analytes < 20% RSD, up to 10% of analytes can be > 20% RSD Most analytes must meet minimum RF's (see Table 4 in 8270D SOP)	

Method	QC Check	Frequency	Acceptance Criteria[2]	Corrective Action[3]
SW8260B SW8270C SW8270D	Method blank	One per analytical prep batch	No analytes detected ≥ RL values between the RL and MDL will be evaluated and flagged with a 'J'	Correct problem then re-prep[5] and analyze method blank and all samples processed with the contaminated blank
SW8260B SW8270C	Internal Standards	Every sample/standard and blank	Retention time ±30 seconds from retention time of the mid-point std. in the CCV/ICAL (sample/standard). EICP area within -50% to +100% of ICAL mid-point std for the CCV and samples.	Refer to corrective action section of method SOP.
SW8270D			Retention time ±30 seconds from retention time of the mid-point std. in the CCV/ICAL (sample/standard). EICP area within -50% to +100% of ICAL mid-point standard Area based on CCV	Refer to corrective action section of method SOP.
	LCS for all target analytes	Volatiles: one per tune per 12 hour sequence. Semivolatiles: One per prep batch, not to exceed the 20 samples in a batch.	Statistical Control Limits	Refer to corrective action section of method SOP.
	MS/MSD	One set per batch of 20 samples per matrix, if insufficient sample for MS/MSD, then a LCS/LCSD will be analyzed.	Statistical Control Limits	None (the LCS is used to evaluate and determine if the batch is acceptable).
	Surrogate(s)	Every sample, spike, standard, and blank	Statistical Control Limits	Refer to corrective action section of method SOP.
SW8260B	pH check	All 8260 water samples All low level 5035 soils are prepped in sodium sulfate (refer to the method SOP for exceptions).	pH ≤2.	If the pH is > 2, then comment the data, in the PIPE database, and LIMS.
SW8260B	Residual chlorine check (samples suspected of coming from a chlorinated waste stream)	Each sample.	Residual chlorine should be negative.	If the residual chlorine is positive, then comment the data, in the PIPE database, and LIMS.
	MDL verification	Minimum - quarterly	Detectible	Re-evaluate MDL standard used and MDL;

1 – SW8260B requires BFB; SW8270C and SW8270D requires DFTPP
2 - This is a summary of the acceptance criteria, refer to the method SOP for specific or more information.
3 - All abnormalities must be noted on the data, the bench sheet and in LIMS.

Appendix 3: Summary of Calibration and QC Procedures for GC/MS Organics

Method	QC Check	Frequency	Acceptance Criteria[1]	Corrective Action[2]
EPA624 EPA625	Check of mass spectral ion intensities (i.e. Tune) 624-BFB; 625-DFTPP	Prior to initial calibration or Continuing calibration verification every 24 hours.	Refer to criteria listed in the method SOP for Tune requirements including DDT, Benzidine and Pentachlorophenol criteria for 625.	Retune instrument and verify instrument maintenance may be needed.
	625 Minimum of Three - point initial calibration for all target analytes. 624: Minimum of Four – point initial calibration for all target analytes.	Initial calibration prior to sample analysis. Perform instrument re-calibration once per year minimum.	%RSD < 35%	If the calibration is not considered linear by either %RSD or linear regression, then correct problem then repeat initial calibration
	Relative Retention time window	Each sample	Retention time (RT) of the analyte within 30 seconds of the RT (± 0.25 min. RTW is used) of the target.	Correct problem then reprocess or re-analyze all samples analyzed since the last retention time check
EPA624 EPA625	Continuing calibration verification (CCV)	Daily, before sample analysis	All calibration analytes within "Range of Q" of expected value. (Method Control Limits)	Correct problem then repeat initial calibration and re-analyze all samples since last successful CCV.
EPA624 EPA625	Method blank	624: One per tune 625:One per prep batch (not to exceed 20 samples per batch).	No analytes detected ≥ RL values between the RL and MDL will be evaluated & flagged with a 'J'	Correct problem then re-prep[5] and analyze method blank and all samples processed with the contaminated blank
	LCS for all analytes in method specified spike list.	624: One per tune 625: One per prep batch (not to exceed 20 samples per batch)	Method Control Limits (Range of P)	Refer to the corrective action section of the Method SOP
	MS for all analytes in method specified spike list.	One per batch of 20 per matrix, if insufficient sample for MS, then a duplicate LCS will be analyzed.	Method Control Limits (Range of P)	None (the LCS is used to evaluate and determine if the batch is acceptable).
	Surrogate(s)	Every sample, spiked sample, standard, and method blank	Statistical Control Limits	Refer to the corrective action section of the Method SOP
EPA624 EPA625	Internal Standards	Every sample/standard	Retention time ±30 seconds from retention time of the mid-point std. in the CCV/ICAL (sample/standard). EICP area within -50% to +100% of ICAL mid-point std for the CCV and –50% to +100% of the prior CCV for the samples.	Refer to the corrective action section of the Method SOP
EPA624	pH check	All 624 samples	pH should be ≤ 2.	If the pH is > 2, then comment the data, in the PIPE database, and LIMS.
EPA624	Residual chlorine check (NC samples only)	All samples	Residual chlorine should be negative.	If the residual chlorine is positive, then comment the data, in the PIPE database, and LIMS.
	MDL verification	Minimum yearly	Detectible	Re-evaluate MDL standard used and MDL

1 - This is summary of the acceptance criteria, refer to the method SOP for specific or more information.
2 - All abnormalities must be noted on the data, the benchsheet and in LIMS.

Appendix 3: Summary of Calibration and QC Procedures for Method SW8310

Method	QC Check	Frequency	Acceptance Criteria[1]	Corrective Action[2]
SW8310 SW8310A (HPLC)	Minimum five-point initial calibration for all target analytes	Initial calibration prior to sample analysis. Perform instrument re-calibration once per year minimum.	CF RSD for each analyte ≤20% or mean RSD for all analytes ≤20% linear – $r^2 \geq 0.990$, $r \geq 0.995$.	Correct problem then repeat initial calibration
	Initial calibration verification (ICV) must be from a 2nd source.	Immediately following initial calibration	All analytes within 15% of expected value	Correct problem then repeat initial calibration
	Retention time verification	Update at start of run or daily	All standards within window	Correct problem then re-analyze all samples analyzed since the last retention time check
	Continuing calibration verification (CCV)	Before sample analysis, after every 10 samples, and at the end of the analysis sequence	All analytes within 15% of expected value	Repeat CCV, if criteria fails, correct problem then repeat initial CCV and re-analyze all samples since last successful CCV.
	Method blank	One per prep batch (not to exceed more than 20 samples per batch).	No analytes detected ≥ RL values between the RL and MDL will be evaluated and flagged with a 'J'	Correct problem then re-prep and analyze method blank and all samples processed with the contaminated blank
	LCS for all analytes	One per prep batch (not to exceed more than 20 samples per batch).	Statistical Control Limits	Correct problem then re-prep and analyze the LCS and all samples in the affected analytical batch
	Surrogate	Every sample, spike, standard, and method blank	Statistical Control Limits	Check system, re-inject, re-extract
	MS/MSD	One per batch per matrix, if insufficient sample for MS/MSD, then a LCS/LCSD will be analyzed.	Statistical Control Limits	None (LCS is used to determine if the batch is acceptable).
	Confirmation	100% for all positive results (use response of both detectors)	Same as for initial or primary analysis. Comment LIMS if >40% difference in compound response between detectors.	Same as for initial or primary analysis.
	MDL verification	Minimum quarterly	Detectible	Re-evaluate MDL standard used and MDL

1 - This is a summary of the acceptance criteria, refer to the method SOP for specific information or more information.

2 - All abnormalities must be noted on the data, the benchsheet and in LIMS.

Appendix 3: Summary of Calibration and QC Procedures for Method EPA610 (HPLC)

Method	QC Check	Frequency	Acceptance Criteria[1]	Corrective Action[2]
EPA610 (HPLC)	Minimum five-point initial calibration for all target analytes	Initial calibration prior to sample analysis. Perform instrument re-calibration once per year minimum.	RSD of CF of each analyte **<10%,** $r^2 \geq$ 0.990, r ≥ 0.995, or linear regression.	Correct problem then repeat initial calibration
	Initial calibration verification (ICV) must be from a 2nd source.	Immediately following initial calibration	All analytes within 15% of expected value	Correct problem then repeat initial calibration
	Retention time verification	Update at start of run or daily	All standards within window	Correct problem then reprocess or re-analyze all samples analyzed since the last retention time check
	Continuing calibration verification (CCV)	Before sample analysis and at the end of the analysis sequence	All analytes within 15% of expected value	Repeat CCV, if criteria fails, correct problem then repeat initial CCV and re-analyze all samples since last successful CCV.
	Method blank	One per prep batch **(not to exceed more than 10 samples per batch).**	No analytes detected ≥ RL values between the RL and MDL will be evaluated and flagged with a 'J'	Correct problem then re-prep and analyze method blank and all samples processed with the contaminated blank
	LCS for all analytes	One per prep batch **(not to exceed more than 10 samples per batch).**	Statistical Control Limits	Correct problem then re-prep and analyze the LCS and all samples in the affected analytical batch
	Surrogate	Every sample, spiked sample, standard, and method blank	Statistical Control Limits	Check system, re-inject, re-extract
	MS	One per batch per matrix, if insufficient sample for MS, then an additional LCS will be analyzed.	Statistical Control Limits	All target compounds should be reported, and any compound that is outside criteria must be within criteria in the LCS.
	Confirmation	100% for all positive results (use response of both detectors)	Same as for initial or primary analysis. Comment LIMS if >40% difference in compound response between detectors.	Same as for initial or primary analysis
	MDL verification	Minimum yearly	Detectible	Re-evaluate MDL standard used and MDL

1 - This is a summary of the acceptance criteria, refer to the method SOP for specific information or more information.
2 - All abnormalities must be noted on the data, the benchsheet and in LIMS.

Appendix 3: Summary of Calibration and QC Procedures for Method SW8330

Method	QC Check	Frequency	Acceptance Criteria[1]	Corrective Action[2]
SW8330 SW8330A SW8332 (HPLC)	Five-point initial calibration for all target analytes	Initial calibration prior to sample analysis. Perform instrument re-calibration once per year minimum.	RSD of CF of each analyte ≤20% or mean RSD for all analytes ≤20% linear – $r^2 \geq 0.990$, $r \geq 0.995$	Correct problem then repeat initial calibration
	Initial calibration verification (ICV) must be from a 2nd source.	Immediately following initial calibration	All analytes within 15% of expected value	Correct problem then repeat initial calibration
	Retention time verification	Update at start of run or daily	All standards within RT window	Correct problem then reprocess or re-analyze all samples analyzed since the last retention time check
	Continuing calibration verification (CCV)	Before sample analysis, after every 10 samples, and at the end of the analysis sequence	All analytes within 15% of expected value	Repeat CCV, if criteria fails, correct problem then repeat initial CCV and re-analyze all samples since last successful CCV.
	Method blank	One per prep batch not to exceed more than 20 samples per batch.	No analytes detected ≥ RL values between the RL and MDL will be evaluated and flagged with a 'J'	Correct problem then re-prep and analyze method blank and all samples processed with the contaminated blank
	LCS for all analytes	One per prep batch (not to exceed more than 20 samples per batch).	Statistical Control Limits	Correct problem then re-prep and analyze the LCS and all samples in the affected analytical batch
	Surrogate	Every sample, spike, standard, and blank	Statistical Control Limits	Check system, re-inject, re-extract
	MS/MSD	One per batch per matrix, if insufficient sample for MS/MSD, then a LCS/LCSD will be analyzed.	Statistical Control Limits	None (LCS is used to determine if the batch is acceptable).
	Confirmation	100% for all positive results; 2nd column (phenyl Hexyl column) confirmation	Same as for initial or primary analysis. Comment LIMS if >40% difference in compound response between detectors.	Same as for initial or primary analysis
	MDL verification	Minimum quarterly	Detectible	Re-evaluate MDL standard used and MDL

1 - This is a summary of the acceptance criteria, refer to the method SOP for specific or more information.
2 - All abnormalities must be noted on the data, the benchsheet and in LIMS.

Appendix 3: Summary of Calibration and QC Procedures for GC Organics

Method	QC Check	Frequency	Acceptance Criteria[1]	Corrective Action[2]
SW8015B[3] SW8015C	Five-point initial calibration for all target analytes	Initial calibration prior to sample analysis. Perform instrument re-calibration once per year minimum.	RSD of CF ≤ 20% Linear – least squares regression $r^2 \geq 0.990$, $r \geq 0.995$	Correct problem then repeat initial calibration
	Initial calibration verification (ICV), must be from a 2nd source.	Immediately following five-point initial calibration	All analytes within 15% of expected value	Correct problem then repeat initial calibration
	LCS for all analytes	One per prep batch, not to exceed 20 samples in a batch.	Statistical Control Limits	Re-prep and analyze the LCS and all samples in the affected analytical batch
	Continuing calibration verification (CCV)	Before sample analysis, after every 10 samples, and at the end of the analysis sequence	All analytes within 15% of expected value and within the RTW.	Repeat CCV, if criteria fails, correct problem then repeat initial CCV (re-calibrate if necessary) and re-analyze all samples since last successful CCV.
	Method blank	One per analytical prep batch, not to exceed 20 samples in a batch.	No analytes detected ≥ RL values between the RL and MDL will be evaluated and flagged with a 'J'	Correct problem then re-prep and analyze method blank and all samples processed with the contaminated blank
	Surrogate	Every sample, spiked sample, standard, and method blank	Statistical Control Limits	Check system, re-analyze, re-prep
	MS/MSD	One per batch per matrix, if insufficient sample for MS/MSD, then a LCS/LCSD will be analyzed.	Statistical Control Limits	None (LCS is used to determine if data is acceptable).
	GC/MS confirmation.	At the clients request or analyst judgment.		
	Retention time window calculated for each analyte (see section 9 for how to calculate RTWs).	System set-up, with each new column or major instrument maintenance. Update the mid-RTW as the start of the run or daily.	Each analyte of the LCS, MS/MSD and CCV must be within the calculated RTW.	Correct the problem and re-process or re-analyze samples. For questions, see the supervisor or technical director.
SW8015B SW8015C – GRO	pH Check	All water samples after analysis.	pH should be less than 2.	If pH is > 2, then place a comment on the benchsheet and in LIMS.
	MDL verification	Minimum quarterly	Detectible	Re-evaluate MDL standard used and MDL

1 - This is a summary of the acceptance criteria, refer to the method SOP for specific or more information.
2 - All abnormalities must be noted on the data, the benchsheet and in LIMS.
3 - For GRO and DRO, see state specific SOP/Method for acceptance criteria. If there is not a specific method for that state, then follow the acceptance criteria in this table.

Appendix 3: Summary of Calibration and QC Procedures for Method SW6010B / SW6010C

Method	QC Check	Frequency	Acceptance Criteria	Corrective Action
SW6010B SW6010C	Initial calibration (three standards for each element- and a blank)	Daily initial calibration prior to sample analysis.	>/= 0.995 (6010B) >/= 0.998 (6010C)	Re-calibrate
	Curve high-standards read-back	Daily after curve	+/- 5% of known concentration	Re-calibrate, re-run samples for affected elements
	Second-source calibration verification (ICV)	Daily after initial calibration	All analytes within 10% of expected value	Correct problem then repeat initial calibration; may report non-detects if biased high.
	Second-source calibration verification – Low Level (ICVL) 6010C only	Daily after mid-range ICV (Conc = RL Level)	All analytes within 30% of expected value	Correct problem then repeat initial calibration; may report non-detects if biased high.
	Initial Calibration blank (ICB)	After ICV	</= RL	Correct problem then re-analyze for affected elements; may report results >10X contamination level or non-detects if biased high
	CRI (2X RL)	At the beginning of an analytical run	50-150%	NA
	Interference check solution (ICSA)	At the beginning of an analytical run	+/- 20% of expected value of spiked elements. Non-spiked elements: < 2x RL (6010B); < RL (6010C)	Correct problem; re-analyze ICSA; re-analyze all affected samples; may report non-detects if biased high.
	Interference check solution (ICSB)	At the beginning of an analytical run	+/- 20% of expected value of spiked elements. Non-spiked elements: < 2x RL (6010B); < RL (6010C)	Correct problem; re-analyze ICSB; re-analyze all affected samples; may report non-detects if biased high.
	Method Blank	One per prep batch of </= 20 samples	+/- RL	Correct problem, re-prep and re-analyze the method blank and all samples for affected elements; may report results >10X contamination level or non-detects if biased high
	LCS	One per prep batch of </= 20 samples	+/- 20% of known concentration	Correct problem, then re-prep and analyze the LCS and all samples for affected elements; may report non-detects if biased high.
	Method duplicate	One per prep batch per matrix upon request	</= 20 RPD	Repeat
	MS/MSD	One per prep batch per matrix	+/- 25% of known concentration	None
	Dilution test	Each new sample matrix	1:5 dilution must agree within 10% of the original determination	Perform post digestion spike addition upon request
	Post digestion spike addition	When dilution test fails for some programs	Recovery within 25% of expected results	None
	Continuing Calibration Verification (CCV)	After every 10 readings and at the end of the analytical sequence	All analytes within 10% of expected value	Correct problem and re-analyze affected elements of bracketed samples; may report non-detects if biased high.

Method 6010 cont.	QC Check	Frequency	Acceptance Criteria	Corrective Action
	Second-source calibration verification – Low Level (CCVL) 6010C only	After mid-range CCV (Conc = RL Level)	All analytes within 30% of expected value	Correct problem and re-analyze affected elements of bracketed samples; may report non-detects if biased high.
	Continuing Calibration blank (CCB)	After each CCV	+/- RL	Correct problem and re-analyze affected elements of bracketed samples; may report results >10X contamination level or non-detects if biased high
	MDL verification	Minimum – Quarterly	Detectible	Re-evaluate MDL standard used and MDL; see Technical Director.

Appendix 3: Summary of Calibration and QC Procedures for Method SW6020 / SW6020A

Method	QC Check	Frequency	Acceptance Criteria	Corrective Action
SW6020 SW6020A	Initial calibration (three standards for each element- and a blank)	Daily initial calibration prior to sample analysis.	>/= 0.995	Re-calibrate
	Curve high-standards read-back	Daily after curve	+/- 5% of known concentration	Re-calibrate, re-run samples for affected elements
	Second-source calibration verification (ICV)	Daily after initial calibration	All analytes within 10% of expected value	Correct problem then repeat initial calibration; may report non-detects if biased high.
	Second-source calibration verification – Low Level (ICVL) 6020A only	Daily after mid-range ICV (Conc = RL Level)	All analytes within 30% of expected value	Correct problem then repeat initial calibration; may report non-detects if biased high.
	Initial Calibration blank (ICB)	After ICV	</= RL	Correct problem then re-analyze for affected elements; may report results >10X contamination level or non-detects if biased high
	CRI (2X RL)	At the beginning of an analytical run and every 12 hours	50-150%	NA
	Interference check solution (ICSA)	At the beginning of an analytical run and every 12 hours	+/- 20% of expected value of spiked elements. Non-spiked elements: < 2X RL (6020); < RL (6020A)	Correct problem; re-analyze ICSA; re-analyze all affected samples; may report non-detects if biased high.
	Interference check solution (ICSB)	At the beginning of an analytical run and every 12 hours	+/- 20% of expected value of spiked elements. Non-spiked elements: < 2X RL (6020); < RL (6020A)	Correct problem; re-analyze ICSB; re-analyze all affected samples; may report non-detects if biased high.
	Method Blank	One per prep batch of </= 20 samples	+/- RL	Correct problem, re-prep and re-analyze the method blank and all samples for affected elements; may report results >10X contamination level or non-detects if biased high
	LCS	One per prep batch of </= 20 samples	+/- 20% of known concentration	Correct problem, then re-prep and analyze the LCS and all samples for affected elements; may report non-detects if biased high.
	Method duplicate	One per prep batch per matrix upon request	</= 20 RPD	Repeat
	MS/MSD	One per prep batch per matrix	+/- 25% of known concentration	None
	Dilution test	Each new sample matrix	1:5 dilution must agree within 10% of the original determination	Perform post digestion spike addition upon request
	Post digestion spike addition	When dilution test fails for some programs	Recovery within 25% of expected results	None
	Continuing Calibration Verification (CCV)	After every 10 readings and at the end of the analytical sequence	All analytes within 10% of expected value	Correct problem and re-analyze affected elements of bracketed samples; may report non-detects if biased high.

Method 6020 cont.	QC Check	Frequency	Acceptance Criteria	Corrective Action
	Second-source calibration verification – Low Level (CCVL) 6020A only	After mid-range CCV (Conc = RL Level)	All analytes within 30% of expected value	Correct problem and re-analyze affected elements of bracketed samples; may report non-detects if biased high.
	Continuing Calibration blank (CCB)	After each CCV	+/- RL	Correct problem and re-analyze affected elements of bracketed samples; may report results >10X contamination level or non-detects if biased high
	MDL verification	Minimum quarterly	Detectible	Re-evaluate MDL standard used and MDL

Appendix 3: Summary of Calibration and QC Procedures for Method SW7196A

Method	QC Check	Frequency	Acceptance Criteria	Corrective Action
SW7196A	Initial calibration (6 or 7 standards and a blank)	Initial calibration prior to sample analysis.	r ≥ 0.995	Correct problem then repeat initial calibration
	Second-source calibration verification (ICV)	Immediately following initial calibration	All analytes within 10% of expected value	Correct problem then repeat initial calibration; may report non-detects if biased high.
	Initial Calibration Blank (ICB)	After ICV	+/- RL	Correct problem then proceed; may report results >10X contamination level or non-detects if biased high
	Method blank	One per prep batch	No analytes detected ≥ ½ RL or MDL, whichever is greater[1]	Correct problem then re-prep and analyze method blank and all samples processed with the contaminated blank. may report results >10X contamination level or non-detects if biased high
	LCS - soluble	One per batch	+/- 20% of known concentration	Re-prep, re-analyze all affected samples. ; may report non-detects if biased high.
	LCS – insoluble (soil sets only)	One per batch	+/- 20% of known concentration	Re-prep, re-analyze all affected samples. ; may report non-detects if biased high.
	Matrix duplicate	One per batch upon request	</= 20 RPD	Repeat once
	MS/MSD - soluble	One pair per batch	+/- 25% of known concentration	Persistent interference indicates the need to evaluate matrix further for reducing agents.
	MS/MSD – insoluble (soil sets only)	One pair per batch	+/- 25% of known concentration	Persistent interference indicates the need to evaluate matrix further for reducing agents.
	Post Digestion Spike (soil sets only)	One per batch	+/- 15% of known concentration	If check indicates interference, dilute and re-analyze sample. Persistent interference indicates the need to evaluate matrix further for reducing agents.
	Continuing calibration verification (CCV)	Beginning and after every 10 samples and at the end of the analysis sequence	All analytes within 10% of expected value	Correct problem and re-analyze all samples since last successful calibration; may report non-detects if biased high.
	Continuing Calibration Blank (CCB)	After every CCV	+/- RL	Correct problem and re-analyze all samples since last successful calibration. May report results >10X contamination level or non-detects if biased high

Appendix 3: Summary of Calibration and QC Procedures for Method SW7470A/ SW7471A/ SW7471B

Method	QC Check	Frequency	Acceptance Criteria	Corrective Action
SW7470A SW7471A SW7471B	Calibration curve – minimum 5 standards and blank	Quarterly, monthly, or daily – see individual SOP	r ≥ 0.995	Recalibrate
	Initial Calibration Verification – mid-level, second-source required (ICV)	Immediately following initial calibration and daily at the beginning of each analytical sequence.	±10% of known concentration	Correct problem then repeat initial calibration; may report non-detects if biased high.
	Initial Calibration Blank (ICB)	After ICV	</= RL	Correct problem then proceed; may report results >10X contamination level or non-detects if biased high
	Method blank	One per batch of 20 or fewer samples	</= RL	Correct problem then re-prep and analyze method blank and all samples processed with the contaminated blank. may report results >10X contamination level or non-detects if biased high
	CRA (at RL level)	One per batch of 20 or fewer samples	No established limits	None
	LCS	One per batch of 20 or fewer samples	+/- 20% of known concentration	Correct problem, re-prep and repeat LCS, MB and all samples in the batch; may report non-detects if the LCS is biased high.
	Matrix duplicate	One per matrix per batch of 20 or fewer samples as requested	</= 20 RPD	Repeat once
	MS/MSD	One pair per matrix per batch of 20 or fewer samples	+/- 25% of known concentration	None
	Continuing calibration verification (CCV)	Beginning, every 10 samples, and at end of sequence	±10% of known concentration	Correct problem then repeat CCV, CCB and all samples since last successful CCB; may report non-detects if biased high.
	Continuing Calibration Blank (CCB)	After every CCV	</= RL	Correct problem then repeat CCV,CCB and all samples since last successful CCB; may report results >10X contamination level or non-detects if biased high
	MDL verification	Minimum quarterly	Detectible	Re-evaluate MDL standard used and MDL; see Technical Director.

Appendix 3: Summary of Calibration and QC Procedures for Cyanide

Method	QC Check	Frequency	Acceptance Criteria	Corrective Action
SW9010B SW9010C SW9014 EPA 335.2 SM4500CNC	Initial calibration (six standards and a calibration blank)	Initial daily calibration prior to sample analysis.	$r \geq 0.995$	Correct problem then repeat initial calibration
	Distilled standards (one high and one low)	Once per calibration	+/- 10% of known concentration (high) +/- 25% of known concentration (low)	Correct problem then repeat distilled standards and related samples
	Second-source calibration verification (ICV)	Immediately following initial daily calibration	+/- 10% of known concentration	Correct problem. Repeat, then repeat initial calibration if necessary
	Initial Calibration Blank (ICB)	Following ICV	</= RL	Correct problem. Repeat, then repeat initial calibration, ICV and ICB if necessary; may report non-detects if CCV is biased high
	Method blank	One per prep batch of 20 or fewer samples	</= RL	Correct problem then re-prep and analyze method blank and all samples processed with the contaminated blank; may report non-detects and results >10X the contamination level.
	LCS	One per prep batch of 20 or fewer samples	+/- 20% of known concentration	Re-prep, re-analyze affected samples; may report non-detects if biased high
	Matrix duplicate	One per matrix per prep batch of 20 or fewer samples upon request	</= 20 RPD	None
	MS/MSD	One per matrix per prep batch	+/- 25% of known concentration	None unless both spike recoveries very low, then PDS
	Post Digestion Spike (PDS)	When both MS and MSD recoveries are very low	+/- 15% of known concentration	None
	Continuing calibration verification (CCV)	After every 10 readings and at the end of the analysis sequence	+/- 10% of known concentration	Correct problem then re-read the CCV, CCB and all samples since last successful CCV/CCB; may report non-detects if CCV is biased high
	Continuing Calibration Blank (CCB)	After every CCV	</= RL	Correct problem then re-read the CCV, CCB and all samples since last successful CCV/CCB; may report non-detects and results >10X the contamination level.

Appendix 3: Summary of Calibration and QC Procedures for Mercury

Method	QC Check	Frequency	Acceptance Criteria	Corrective Action
EPA245.1 EPA245.5	Calibration curve – minimum 5 standards and blank	Quarterly, monthly, or daily – see individual SOP	r ≥ 0.995	Recalibrate
	Initial Calibration Verification – mid-level, second-source required (ICV)	Immediately following initial calibration and daily at the beginning of each analytical sequence.	±5% of known concentration	Correct problem then repeat initial calibration; may report non-detects if biased high.
	Initial Calibration Blank (ICB)	After ICV	</= RL	Correct problem then proceed; may report results >10X contamination level or non-detects if biased high
	CRA	One per calibration	No established limits	None
	Method blank	One per batch of 20 or fewer samples	</= RL	Correct problem then re-prep and analyze method blank and all samples processed with the contaminated blank. may report results >10X contamination level or non-detects if biased high
	LCS	One per batch of 20 or fewer samples	+/- 15% of known concentration	Correct problem, re-prep and repeat LCS, MB and all samples in the batch; may report non-detects if the LCS is biased high.
	Matrix duplicate	One per matrix per batch of 20 or fewer samples as requested	</= 20 RPD	Repeat once
	MS/MSD	One pair per matrix per batch of 20 or fewer samples	+/- 30% of known concentration	None
	Continuing calibration verification (CCV)	Beginning, every 10 samples, and at end of sequence	±10% of known concentration	Correct problem then repeat CCV, CCB and all samples since last successful CCB; may report non-detects if biased high.
	Continuing Calibration Blank (CCB)	After every CCV	</= RL	Correct problem then repeat CCV,CCB and all samples since last successful CCB; may report results >10X contamination level or non-detects if biased high
	MDL verification	Minimum yearly	Detectible	Re-evaluate MDL standard used and MDL

Appendix 3: Summary of Calibration and QC Procedures for ICP Method 200.7

Method	QC Check	Frequency	Acceptance Criteria	Corrective Action
EPA200.7	Initial calibration (three standards for each element- and a blank)	Daily initial calibration prior to sample analysis.	>/= 0.995	Re-calibrate
	Curve high-standards read-back	Daily after curve	+/- 5% of known concentration	Re-calibrate, re-run samples for affected elements
	Second-source calibration verification (ICV)	Daily after initial calibration	All analytes within 5% of expected value	Correct problem then repeat initial calibration; may report non-detects if biased high.
	Initial Calibration blank (ICB)	After ICV	</= RL	Correct problem then re-analyze for affected elements; may report results >10X contamination level or non-detects if biased high
	CRI (2X RL)	At the beginning of an analytical run	50-150%	NA
	Interference check solution (ICSA)	At the beginning of an analytical run	+/- 20% of expected value of spiked elements or < 2X RL	Correct problem; re-analyze ICSA; re-analyze all affected samples; may report non-detects if biased high.
	Interference check solution (ICSB)	At the beginning of an analytical run	+/- 20% of expected value of spiked elements or < 2X RL	Correct problem; re-analyze ICSB; re-analyze all affected samples; may report non-detects if biased high.
	Method Blank	One per prep batch of </= 20 samples	+/- RL	Correct problem, re-prep and re-analyze the method blank and all samples for affected elements; may report results >10X contamination level or non-detects if biased high
	LCS	One per prep batch of </= 20 samples	+/- 15% of known concentration	Correct problem, then re-prep and analyze the LCS and all samples for affected elements; may report non-detects if biased high.
	Method duplicate	One per 10 or fewer samples per matrix	</= 20 RPD	Repeat
	MS	One per10 or fewer samples per matrix	+/- 30% of known concentration	None
	Dilution test	Each new sample matrix	1:5 dilution must agree within 10% of the original determination	Perform post digestion spike addition
	Continuing Calibration Verification (CCV)	After every 10 readings and at the end of the analytical sequence	All analytes within 10% of expected value	Correct problem and re-analyze affected elements of bracketed samples; may report non-detects if biased high.
	Continuing Calibration blank (CCB)	After each CCV	+/- RL	Correct problem and re-analyze affected elements of bracketed samples; may report results >10X contamination level or non-detects if biased high
	MDL verification	Minimum yearly	Detectible	Re-evaluate MDL standard used and MDL; see Technical Director.

Appendix 3: Summary of Calibration and QC Procedures for ICP/MS Method 200.8

Method	QC Check	Frequency	Acceptance Criteria	Corrective Action
EPA200.8	Initial calibration (three standards for each element- and a blank)	Daily initial calibration prior to sample analysis.	>/= 0.995	Re-calibrate
	Curve high-standards read-back	Daily after curve	+/- 5% of known concentration	Re-calibrate, re-run samples for affected elements
	Second-source calibration verification (ICV)	Daily after initial calibration	All analytes within 10% of expected value	Correct problem then repeat initial calibration; may report non-detects if biased high.
	Initial Calibration blank (ICB)	After ICV	</= RL	Correct problem then re-analyze for affected elements; may report results >10X contamination level or non-detects if biased high
	CRI (2X RL)	At the beginning of an analytical run and every 12 hours	50-150%	NA
	Interference check solution (ICSA)	At the beginning of an analytical run and every 12 hours	+/- 20% of expected value of spiked elements or < 2X RL	Correct problem; re-analyze ICSA; re-analyze all affected samples; may report non-detects if biased high.
	Interference check solution (ICSB)	At the beginning of an analytical run and every 12 hours	+/- 20% of expected value of spiked elements or < 2X RL	Correct problem; re-analyze ICSB; re-analyze all affected samples; may report non-detects if biased high.
	Method Blank	One per prep batch of </= 20 samples	+/- RL	Correct problem, re-prep and re-analyze the method blank and all samples for affected elements; may report results >10X contamination level or non-detects if biased high
	LCS	One per prep batch of </= 20 samples	+/- 15% of known concentration	Correct problem, then re-prep and analyze the LCS and all samples for affected elements; may report non-detects if biased high.
	Method duplicate	One per 10 or fewer samples per matrix	</= 20 RPD	Repeat
	MS	One per10 or fewer samples per matrix	+/- 30% of known concentration	None
	Dilution test	Each new sample matrix	1:5 dilution must agree within 10% of the original determination	Perform post digestion spike addition
	Continuing Calibration Verification (CCV)	After every 10 readings and at the end of the analytical sequence	All analytes within 10% of expected value	Correct problem and re-analyze affected elements of bracketed samples; may report non-detects if biased high.
	Continuing Calibration blank (CCB)	After each CCV	+/- RL	Correct problem and re-analyze affected elements of bracketed samples; may report results >10X contamination level or non-detects if biased high
	MDL verification	Minimum yearly	Detectible	Re-evaluate MDL standard used and MDL.

Appendix 3: Summary of Calibration and QC Procedures for Gravimetric Analyses

Method	QC Check	Frequency	Acceptance Criteria	Corrective Action
EPA160.1 SM2540 C (TDS) EPA160.2 SM2540D (TSS) EPA160.3 SM2540B (TS) EPA160.4 (TVS) ASTM D482-87 (Ash) ASTM D2974 (FOM) SM2540 G (Moisture) ASTM D5050 (Density)	LCS	Applicable to some gravimetric testing - one per batch of 20 or fewer samples	+/- 20% of known concentration	Repeat LCS, MB and all samples in batch; May report non-detects if LCS is biased high.
	Method blank	One per batch of 20 or fewer samples	</= RL	Repeat MB, LCS and all samples in batch; May report non-detects and samples > 10X the contamination level.
	Duplicate	10% of samples	</=20 RPD	None
	MS/MSD	Applicable to some gravimetric testing - one pair per set of 20 or fewer samples	+/- 25% of known concentration	None
	MDL verification	Minimum yearly	Detectible	Re-evaluate MDL standard used and MDL.

Appendix 3: Summary of Calibration and QC Procedures for Titrimetric Analyses

Method	QC Check	Frequency	Acceptance Criteria	Corrective Action
EPA310.1 SM2320B Alkalinity forms EPA376.1 SM4500 SF Sulfide Hach 8000 COD EPA330.4 SM4500ClF Cl_2 Res.	LCS	One per batch of 20 or fewer samples	± 20% of known concentration	Correct problem and repeat LCS, MB and all samples in the batch; May report non-detects if the LCS is biased high.
	Method blank	One per batch of 20 or fewer samples	No analyte detected > report limit	Correct problem and repeat MB, LCS and all samples in the batch; May report non-detects and samples >10X the contamination level
	Duplicate	One per batch of 20 or fewer samples	±20%	None
	MS/MSD	Available for some titrimetric tests at one pair per batch of 20 or fewer samples.	+/- 25% of known concentration	None
	Standardization	Per specific SOP	NA	NA

Appendix 3: Summary of Calibration and QC Procedures for Spectrophotometric Analyses

Method	QC Check	Frequency	Acceptance Criteria	Corrective Action
EPA350.2 SM4500NH3C: NH_3. EPA325.2 SM4500ClE: 9251: Cl^-. SM3500CrB Cr+6. EPA375.4: SW9038: SO_4^{-2}. EPA415.1 SM5310C 9060: TOC. EPA 354.1 $SM4500NO_2B$ Nitrite EPA 353.2 $SM4500NO_3F$ Nitrate 9066 EPA420.4 Phenols EPA365.2 SM4500P Phosphorus 353.2 $SM4500NH_3C$ TKN	Calibration curve – minimum 5 standards and blank	Quarterly, monthly, or daily – see individual SOP	r ≥ 0.995	Recalibrate
	Initial Calibration Verification – mid-level, second-source required (ICV)	Immediately following initial calibration and daily at the beginning of each analytical sequence.	±10% of known concentration	Correct problem then repeat initial calibration; may report non-detects if biased high.
	Initial Calibration Blank (ICB)	After ICV	</= RL	Correct problem then proceed; may report results >10X contamination level or non-detects if biased high
	Method blank	One per batch of 20 or fewer samples	</= RL	Correct problem then re-prep and analyze method blank and all samples processed with the contaminated blank. may report results >10X contamination level or non-detects if biased high
	LCS	One per batch of 20 or fewer samples	+/- 20% of known concentration	Correct problem, re-prep and repeat LCS, MB and all samples in the batch; may report non-detects if the LCS is biased high.
	Matrix duplicate	One per matrix per batch of 20 or fewer samples as requested	</= 20 RPD	Repeat once
	MS/MSD	One pair per matrix per batch of 20 or fewer samples	+/- 25% of known concentration	None
	Continuing calibration verification (CCV)	Beginning, every 10 samples, and at end of sequence	±10% of known concentration	Correct problem then repeat CCV, CCB and all samples since last successful CCB; may report non-detects if biased high.
	Continuing Calibration Blank (CCB)	After every CCV	</= RL	Correct problem then repeat CCV,CCB and all samples since last successful CCB; may report results >10X contamination level or non-detects if biased high
	MDL verification	Minimum yearly	Detectible	Re-evaluate MDL standard used and MDL.

1 - Report all targets identified in the method blank above the MDL.

Appendix 3: Summary of Calibration and QC Procedures for Electrometric Analyses

Method	QC Check	Frequency	Acceptance Criteria	Corrective Action
EPA405.1: BOD[1], CBOD[1]. EPA120.1: SW9050: Cond.[1] EPA360.1: DO[1]. EPA340.2: F⁻. SW9214: F⁻. EPA150.1: SW9040, 9045:pH[1]. SM5320B: SW9020: TOX.	Calibration Curve – minimum of 5 standards and blank	Initial Calibration before each batch (where applicable)	r ≥ 0.995.	Recalibrate
	Initial Calibration Verification (second source) (ICV)	Immediately after initial calibration and daily at beginning of analytical sequence	±10% of known concentration	Correct problem then repeat initial calibration; may report non-detects if biased high.
	Initial Calibration Blank	With ICV	</= RL	Correct problem then proceed; may report results >10X contamination level or non-detects if biased high
	Method blank	One per batch of 20 or fewer samples	</= RL	Correct problem then re-prep and analyze method blank and all samples processed with the contaminated blank; may report results >10X contamination level or non-detects if biased high.
	LCS	One per batch of 20 or fewer samples	±20% of known concentration	Correct problem, re-prep and repeat LCS, MB and all samples in the batch; may report non-detects if the LCS is biased high.
	MS/MSD	Each batch	± 20%	None
	Duplicate	When spike not available	±20%	None
	Continuing Calibration verification (CCV)	Beginning, every 10 samples, and end of batch (every eight burns for TOX)	±10% of known concentration	Correct problem then repeat CCV, CCB and all samples since last successful CCB; may report non-detects if biased high.
	Continuing Calibration blank	With each CCV	</= RL (used to correct CCV and following samples in TOX analysis)	Correct problem then repeat CCV,CCB and all samples since last successful CCB; may report results >10X contamination level or non-detects if biased high
	Calibration checks	Alternate pH buffers every 10 readings. Read-back DO calibration bottle. Conductivity standards must bracket samples in each range.	pH buffers +/- 0.2 SU DO bottle +/- 0.5 mg/L Cond. stds. After samples, <10% drift	Re-calibrate and re-read samples since last in-control check.
	MDL verification	Minimum yearly	Detectible	Re-evaluate MDL standard used and MDL

[1]Calibration curve and related QC items do not apply.

Appendix 3: Summary of Calibration and QC Procedures for Ion Chromatographic Analyses

Method	QC Check	Frequency	Acceptance Criteria	Corrective Action
EPA300 & SW9056 SW9045A: Bromide Chloride Fluoride Nitrate Nitrite Sulfate.	Calibration Curves	Initial calibration. Perform instrument re-calibration once per month minimum.	R ≥ 0.995.	Recalibrate
	Initial Calibration verification (ICV), second source	Immediately following initial calibration and daily before the analytical sequence	±10% of known concentration	Correct problem then repeat initial calibration; may report non-detects if biased high.
	Initial Calibration Blank	After ICV	</= RL	Correct problem then proceed; may report results >10X contamination level or non-detects if biased high
	Method blank	One per batch of 20 or fewer samples	</= RL	Correct problem then re-prep and analyze method blank and all samples processed with the contaminated blank; may report results >10X contamination level or non-detects if biased high.
	LCS	One per batch of 20 or fewer samples	±10% of known concentration for 300.0, 20% for 9056	Correct problem, re-prep and repeat LCS, MB and all samples in the batch; may report non-detects if the LCS is biased high.
	MS/MSD	One pair per batch of 20 or fewer samples	±25% of known concentration for 9056, 20% for 300.0	None, control batch on LCS
	Duplicate	One per batch of 20 or fewer samples upon request	</= 20 RPD	None
	Continuing calibration verification (CCV)	After every 10 readings and at the end of the analytical sequence	± 10% of known concentration	Correct problem then repeat CCV, CCB and all samples since last successful CCB; may report non-detects if biased high.
	Continuing Calibration blank (CCB)	After every CCV	</= RL	Correct problem then repeat CCV,CCB and all samples since last successful CCB; may report results >10X contamination level or non-detects if biased high
	Retention time window	Based on RTs of ICV anions	+/- 5% drift	Evaluate data
	MDL verification	Minimum yearly	Detectible	Re-evaluate MDL standard used and MDL.

Appendix 3: Summary of Calibration and QC Procedures for Oil & Grease Analyses

Method	QC Check	Frequency	Acceptance Criteria	Corrective Action
9071B 1664A HEM SGT-HEM	Method blank	One per batch of 20 or fewer samples	</= RL	Correct problem then re-prep and analyze method blank and all samples processed with the contaminated blank; may report results >10X contamination level or non-detects if biased high.
	LCS	One per batch of 20 or fewer samples	78-114% for HEM, 64-132% for SGT-HEM	Correct problem, re-prep and repeat LCS, MB and all samples in the batch; may report non-detects if the LCS is biased high.
	MS/MSD	One pair per batch of 20 or fewer samples as available	78-114% for HEM, 64-132% for SGT-HEM	None, use LCS

Appendix 3: Summary of Calibration and QC Procedures for Physical Analyses

Method	QC Check	Frequency	Acceptance Criteria	Corrective Action
SW1010 / SW1010A D-93: Flash Point. SW9095A: Paint Filter. EPA160.5, SM 2540 F: Settleable Solids.				
	LCS	One per batch of 20 or fewer samples – applicable for flash point only	Statistical limits	Re-analyze
	Duplicate	One for each batch of 20 or fewer samples	Report results	None

Appendix 4. Glossary/Acronyms

Glossary:

Acceptance Criteria:
Specified limits placed on characteristics of an item, process, or service defined in requirement documents. (ASQC)

Accreditation:

The process by which an agency or organization evaluates and recognizes a laboratory as meeting certain predetermined qualifications or standards, thereby accrediting the laboratory. In the context of the National Environmental Laboratory Accreditation Program (NELAP), this process is a voluntary one. (NELAC)

Accrediting Authority:
The Territorial, State, or Federal Agency having responsibility and accountability for environmental laboratory accreditation and which grants accreditation (NELAC) [1.5.2.3]

Accuracy:
The degree of agreement between an observed value and an accepted reference value. Accuracy includes a combination of random error (precision) and systematic error (bias) components which are due to sampling and analytical operations; a data quality indicator. (QAMS)

Analyst:
The designated individual who performs the "hands-on" analytical methods and associated techniques and who is the one responsible for applying required laboratory practices and other pertinent quality controls to meet the required level of quality. (NELAC)

Batch:
Environmental samples which are prepared and/or analyzed together with the same process and personnel, using the same lot(s) of reagents. A preparation batch is composed of one to 20 environmental samples of the same matrix, meeting the above mentioned criteria and with a maximum time between the start of processing of the first and last sample in the batch to be 24 hours. An analytical batch is composed of prepared environmental samples (extracts, digestates or concentrates) and /or those samples not requiring preparation, which are analyzed together as a group using the same calibration curve or factor. An analytical batch can include samples originating from various environmental matrices and can exceed 20 samples. (NELAC Quality Systems Committee)

Blank:
A sample that has not been exposed to the analyzed sample stream in order to monitor contamination during sampling, transport, storage or analysis. The blank is subjected to the usual analytical and measurement process to establish a zero baseline or background value and is sometimes used to adjust or correct routine analytical results. (ASQC)

Blind Sample:
A sample for analysis with a composition known to the submitter. The analyst/laboratory may know the identity of the sample but not its composition. It is used to test the analyst's or laboratory's proficiency in the execution of the measurement process.

Calibration:
To determine, by measurement or comparison with a standard, the correct value of each scale reading on a meter, instrument, or other device. The levels of the applied calibration standard should bracket the range of planned or expected sample measurements. (NELAC)

Calibration Curve:
The graphical relationship between the known values, such as concentrations, of a series of calibration standards and their instrument response. (NELAC)

Calibration Method:
A defined technical procedure for performing a calibration. (NELAC)

Calibration Standard:
A substance or reference material used to calibrate an instrument (QAMS)

Certified Reference Material (CRM):
A reference material one or more of whose property values are certified by a technically valid procedure, accompanied by or traceable to a certificate or other documentation which is issued by a certifying body. (ISO Guide 30–2.2)

Chain of Custody:
An unbroken trail of accountability that ensures the physical security of samples and includes the signatures of all who handle the samples. (NELAC) [5.12.4]

Clean Air Act:
The enabling legislation in 42 U>S>C> 7401 et seq., Public Law 91-604, 84 Stat. 1676 Pub. L. 95-95, 91 Stat., 685 and Pub. L. 95-190, 91 Stat., 1399, as amended, empowering EPA to promulgate air quality standards, monitor and enforce them. (NELAC)

Comprehensive Environmental Response, Compensation and Liability Act (CERCLA/SUPERFUND):
The enabling legislation in 42 U.S.C. 9601-9675 et seq., as amended by the Superfund Amendments and Reauthorization Act of 1986 (SARA), 42 U.S.C. 9601 et seq., to eliminate the health and environmental threats posed by hazardous waste sites. (NELAC)

Compromised Samples:
Those samples which are improperly sampled, insufficiently documented (chain of custody and other sample records and/or labels), improperly preserved, collected in improper containers, or exceeding holding times when delivered to a laboratory. Under normal conditions, compromised samples are not analyzed. If emergency situation require analysis, the results must be appropriately qualified. (NELAC)

Confidential Business Information (CBI):
Information that an organization designates as having the potential of providing a competitor with inappropriate insight into its management, operation or products. NELAC and its representatives agree to safeguarding identified CBI and to maintain all information identified as such in full confidentiality.

Confirmation:
Verification of the identity of a component through the use of an approach with a different scientific principle from the original method. These may include, but are not limited to:

Second column confirmation
Alternate wavelength
Derivatization
Mass spectral interpretation
Alternative detectors or
Additional Cleanup procedures
(NELAC)

Conformance:
An affirmative indication or judgement that a product or service has met the requirements of the relevant specifications, contract, or regulation; also the state of meeting the requirements. (ANSI/ASQC E4-1994)

Correction:
Actions necessary to correct or repair analysis specific non-conformances. The acceptance criteria for method specific QC and protocols as well as the associated corrective actions. The analyst will most frequently be the one to identify the need for this action as a result of calibration checks and QC sample analysis. No significant action is taken to change behavior, process or procedure.

Corrective Action:
The action taken to eliminate the causes of an existing nonconformity, defect or other undesirable situation in order to prevent recurrence. (ISO 8402)

Data Audit:
A qualitative and quantitative evaluation of the documentation and procedures associated with environmental measurements to verify that the resulting data re of acceptable quality (i.e., that they meet specified acceptance criteria). (NELAC)

Data Reduction:
The process of transforming raw data by arithmetic or statistical calculations, standard curves, concentration factors, etc., and collation into a more useable form. (EPA-QAD)

Deficiency:
An unauthorized deviation from acceptable procedures or practices, or a defect in an item. (ASQC)

Demonstration of Capability (DOC):
Procedure to establish the ability to generate acceptable accuracy and precision.

Detection Limit Check Standard (DLCK):
A non-processed standard spiked at the method reporting limit or lowest calibration standard. Used in conjunction with the MRL Check standard in LCG analysis.

Detection Limit:
The lowest concentration or amount of the target analyte that can be identified, measured, and reported with confidence that the analyte concentration is not a false positive value. See Method Detection Limit. (NELAC)

Document Control:
The act of ensuring that documents (and revisions thereto) are proposed, reviewed for accuracy, approved for release by authorized personnel, distributed properly, and controlled to ensure use of the correct version at the location where the prescribed activity if performed. (ASQC)

Duplicate Analyses:
The analyses or measurements of the variable of interest performed identically on two subsamples of the same sample. The results from duplicate analyses are used to evaluate analytical or measurement precision but not the precision of sampling, preservation or storage internal to the laboratory. (EPA-QAD)

Environmental Detection Limit (EDL):
The smallest level at which a radionuclide in an environmental medium can be unambiguously distinguished for a given confidence interval using a particular combination of sampling and measurement procedures, sample size, analytical detection limit, and processing procedure. The EDL shall be specified for the 0.95 or greater confidence interval. The EDL shall be established initially and verified annually for each test method and sample matrix. (NELAC Radioanalysis Subcommittee)

Equipment Blank:
Sample of analyte-free media which has been used to rinse common sampling equipment to check effectiveness of decontamination procedures. (NELAC)

External Standard Calibration:
Calibrations for methods that do not utilize internal standards to compensate for changes in instrument conditions.

Extraction Blank (LB, LB2, LB3):
A blank that has been taken through the extraction procedure such as TCLP/SPLP, 5035 AVS/SEM.

Federal Water Pollution Control Act (Clean Water Act, CWA):
The enabling legislation under 33 U.S.C. 1251 et seq., Public Law 92-50086 Stat 816, that empowers EPA to set discharge limitations, write discharge permits, monitor, and bring enforcement action for non-compliance. (NELAC)

Field Blank:
Blank prepared in the field by filing a clean container with pure de-ionized water and appropriate preservative, if any, for the specific sampling activity being undertaken (EPA OSWER)

Field Duplicate:
Duplicate field-collected sample.

Field of Testing:
NELAC's approach to accrediting laboratories by program, method and analyte. Laboratories requesting accreditation for a program-method-analyte combination or for an up-dated/improved method are required to submit to only that portion of the accreditation process not previously addressed (see NELAC, section 1.9ff). (NELAC)

Good Laboratory Practices (GLP):
Formal regulations for performing basic laboratory operations outlined in 40 CFR Part 160 and 40 CFR Part 729 and required for activities performed under FIFRA and TSCA.

Holding Times (Maximum Allowable Holding Times):
The maximum times that samples may be held prior to analyses and still be considered valid or not compromised. (40 CFR Part 136)

Instrument Blank:
A clean sample (e.g., distilled water) processed through the instrumental steps of the measurement process; used to determine instrument contamination. (EPA-QAD)

Instrument Detection Limit (IDL):
The minimum amount of a substance that can be measured with a specified degree of confidence that the amount is greater than zero using a specific instrument. The IDL is associated with the instrumental portion of a specific method only, and sample preparation steps are not considered in its derivation. The IDL is a statistical estimation at a specified confidence interval of the concentration at which the relative uncertainty is ± 100%. The IDL represents a range where qualitative detection occurs on a specific instrument. Quantitative results are not produced in this range.

Internal Chain of Custody:
An unbroken trail of accountability that ensures the physical security of samples, data and records. Internal COC refers to additional documentation procedures implemented within the laboratory that includes special sample storage requirements, and documentation of all signatures and/or initials, dates, and times of personnel handling specific samples or sample aliquots.

Internal Standard:
A known amount of standard added to a test portion of a sample and carried through the entire measurement process as a reference for evaluating and controlling the precision and bias of the applied analytical test method. (NELAC)

Internal Standard Calibration:
Calibrations for methods that utilize internal standards to compensate for changes in instrument conditions.

Laboratory Control Sample (however named, such as laboratory fortified blank, spiked blank, or QC check sample):
A sample matrix, free from the analytes of interest, spiked with verified known amounts of analytes or a material containing known and verified amounts of analytes, taken through all preparation and analysis steps. Where there is no preparation taken for an analysis (such as in aqueous volatiles), or when all samples and standards undergo the same preparation and analysis process (such as Phosphorus), there is no LCS. It is generally used to establish intra-laboratory or analyst specific precision and bias or to assess the performance of all or a portion of the measurement system.

An LCS shall be prepared at a minimum of 1 per batch of 20 or less samples per matrix type per sample extraction or preparation method except for analytes for which spiking solutions are not available such as total suspended solids, total dissolved solids, total volatile solids, total solids, pH, color, odor, temperature, dissolved oxygen or turbidity. The results of these samples shall be used to determine batch acceptance.

Note: NELAC standards allow a matrix spike to be used in place of this control as long as the acceptance criteria are as stringent as for the LCS. (NELAC)

Laboratory Duplicate:
Aliquots of a sample taken from the same container under laboratory conditions and processed and analyzed independently. (NELAC)

Least Squares Regression (1st Order Curve):
The least squares regression is a mathematical calculation of a straight line over two axes. The y axis represents the instrument response (or Response ratio) of a standard or sample and the x axis represents the concentration. The regression calculation will generate a correlation coefficient (r) that is a measure of the "goodness of fit" of the regression line to the data. A value of 1.00 indicates a perfect fit. In order to be used for quantitative purposes, r must be greater than or equal to 0.99 for organics and 0.995 for inorganics.

Limit of Detection (LOD):
An estimate of the minimum amount of a substance that an analytical process can reliably detect. An LOD is analyte- and matrix-specific and may be laboratory dependent. (Analytical Chemistry, 55, p.2217, December 1983, modified) See also Method Detection Limit.

Limit or Quantitation (LOQ):
Limit of Quantitation (LOQ) is the lowest concentration that produces a quantitative result within specified limits of precision and bias. For DoD projects, the LOQ shall be set at or above the concentration of the lowest initial calibration standard. The laboratory's routine reporting limit is equal to the LOQ, unless project documents specifies a higher concentration to be used as the project-specific reporting limit.

Matrix:
The component or substrate that contains the analyte of interest. For purposes of batch and QC requirement determinations, the following matrix distinctions shall be used:

Aqueous: Any aqueous sample excluded from the definition of Drinking Water matrix or Saline/Estuarine source. Includes surface water, groundwater, effluents, and TCLP or other extracts.

Drinking Water: any aqueous sample that has been designated as a potable or potential potable water source.

Saline/Estuarine: any aqueous sample from an ocean or estuary, or other salt water source such as the Great Salt Lake.

Non-aqueous Liquid: any organic liquid with <15% settleable solids.

Biological Tissue: any sample of a biological origin such as fish tissue, shellfish, or plant material. Such samples shall be grouped according to origin.

Solids: includes soils, sediments, sludges, and other matrices with ≥15% settleable solids.

Chemical Waste: a product or by-product of an industrial process that results in a matrix not previously defined.

Air: whole gas or vapor samples including those contained in flexible or rigid wall containers and the extracted concentrated analytes of interest from a gas or vapor that are collected with a sorbant tube, impinger solution, filter, or other device. (NELAC)

Matrix Spike (spiked sample or fortified sample):

Prepared by adding a known mass of target analyte to a specified amount of matrix sample for which an independent estimate of target analyte concentration is available. Matrix spikes are used, for example, to determine the effect of the matrix on a method's recovery efficiency.

Matrix spikes shall be performed at a frequency of one in 20 samples per matrix type per sample extraction or preparation method except for analytes for which spiking solutions are not available such as, total suspended solids, total dissolved solids, total volatile solids, total solids, pH, color, odor, temperature, dissolved oxygen or turbidity. The selected sample(s) shall be rotated among client samples so that various matrix problems may be noted and/or addressed. Poor performance in a matrix spike may indicate a problem with the sample composition and shall be reported to the client whose sample was used for the spike. (QAMS)

Matrix Spike Duplicate (spiked sample or fortified sample duplicate):
A second replicate matrix spike is prepared in the laboratory and analyzed to obtain a measure of the precision of the recovery for each analyte.

Matrix spike duplicates or laboratory duplicates shall be analyzed at a minimum of 1 in 20 samples per matrix type per sample extraction or preparation method. The laboratory shall document their procedure to select the use of an appropriate type of duplicate. The selected sample(s) shall be rotated among client samples so that various matrix problems may be noted and/or addressed. Poor performance in the duplicates may indicate a problem with the sample composition and shall be reported to the client whose sample was used for the duplicate. (QAMS)

Method Blank:
A sample of a matrix similar to the batch of associated samples (when available) that is free from the analytes of interest and is processed simultaneously with and under the same conditions as samples through all steps of the analytical procedures, and in which no target analytes or interferences are present at concentrations that impact the analytical results for sample analyses. (NELAC)

Method Detection Limit:
The minimum concentration of a substance (an analyte) that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte. (40 CFR Part 136, Appendix B) The DoD QSM version 4.1 further defines the DL as representing a concentration that is 99% confident that it is distinguishable from a blank.

Method Detection Limit Check Standard (MDLCK):
A standard that is processed with the MDL Study that is spiked at ½ the spike level used for the MDL/DL study or ½ the method reporting limit or ½ the lowest calibration standard (RL/LOQ). This check standard is also required by the DOD QSM and is referred to as the MDLV which is to be analyzed on a quarterly basis for methods listed on the DoD ELAP Accreditation.

Method Reporting Limit Check Standard (MRL):
A standard that is not processed with the samples. It is analyzed in the analytical sequence at a spike concentration approximately 2x the low standard or reporting limit. This standard check is used in conjunction with the USACE LCG analysis and for DoD QSM sample analysis.

Negative Control:
Measures taken to ensure that a test, its components, or the environment do not cause undesired effects, or produce incorrect test results. (NELAC)

Non-conformance:
An indication, judgment, or state of not having met the requirements of the relevant specifications, contract, or regulation.

Non-conformance Memo (NCM):
The term for the mechanism used in the TALs LIMs to document a non-conformance to a job, project, program or method analysis in the system.

Performance Audit:
The routine comparison of independently obtained qualitative and quantitative measurement system data with routinely obtained data in order to evaluate the proficiency of an analyst or laboratory. (NELAC)

Performance Based Measurement System (PBMS):
A set of processes wherein the data quality needs, mandates or limitations of a program or project are specified and serve as criteria for selecting appropriate test methods to meet those needs in a cost-effective manner. (NELAC)

Positive Control:
Measures taken to ensure that a test and/or its components are working properly and producing correct or expected results from positive test subjects. (NELAC)

Precision:
The degree to which a set of observations or measurements of the same property, obtained under similar conditions, conform to themselves; a data quality indicator. Precision is usually expressed as standard deviation, variance or range, in either absolute or relative terms. (NELAC)

Preservation:
Refrigeration and/or reagents added at the time of sample collection (or later) to maintain the chemical and/or biological integrity of the sample. (NELAC)

Proficiency Testing:
A means of evaluating a laboratory's performance under controlled conditions relative to a given set of criteria through analysis of unknown samples provided by an external source. (NELAC) [2.1]

Proficiency Testing Program:
The aggregate of providing rigorously controlled and standardized environmental samples to a laboratory for analysis, reporting of results, statistical evaluation of the results and the collective demographics and results summary of all participating laboratories. (NELAC)

Proficiency Test Sample (PT):
A sample, the composition of which is unknown to the analyst and is provided to test whether the analyst/laboratory can produce analytical results within specified acceptance criteria. (QAMS)

Proprietary:
Belonging to a private person or company.

Quality Assurance:
An integrated system of activities involving planning, quality control, quality assessment, reporting and quality improvement to ensure that a product or service meets defined standards of quality with a stated level of confidence. (QAMS)

Quality Assurance [Project] Plan (QAPP):
A formal document describing the detailed quality control procedures by which the quality requirements defined for the data and decisions pertaining to a specific project are to be achieved. (EAP-QAD)

Quality Control:
The overall system of technical activities which purpose is to measure and control the quality of a product or service so that it meets the needs of users. (QAMS)

Quality Control Sample:
An uncontaminated sample matrix spiked with known amounts of analytes from a source independent from the calibration standards. It is generally used to establish intra-laboratory or analyst specific precision and bias or to assess the performance of all or a portion of the measurement system. (EPA-QAD)

Quality Manual:
A document stating the management policies, objectives, principles, organizational structure and authority, responsibilities, accountability, and implementation of an agency, organization, or laboratory, to ensure the quality of its product and the utility of its product to its users. (NELAC)

Quality System:
A structured and documented management system describing the policies, objectives, principles, organizational authority, responsibilities, accountability, and implementation plan of an organization for ensuring quality in its work processes, products (items), and services. The quality system provides the framework for planning, implementing, and assessing work performed by the organization and for carrying out required QA and QC (ANSI/ASQC-E-41994)

Quantitation Limits:
The minimum levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be quantified with specified degree of confidence. (NELAC)

Range:
The difference between the minimum and the maximum of a set of values. (EPA-QAD)

Raw Data:
Any original information from a measurement activity or study recorded in laboratory notebooks, worksheets, records, memoranda, notes, or exact copies thereof and that are necessary for reconstruction and evaluation of the report of the activity or study. Raw data may include photography, microfilm or microfiche copies, computer printouts, magnetic/optical media, including dictated observations, and recorded data from automated instruments. Reports specifying inclusion of "raw data" do not need all of the above included, but sufficient information to re-create the reported data.

Reagent Blank (method reagent blank):
A sample consisting of reagent(s), without the target analyte or sample matrix, introduced into the analytical procedure at the appropriate point and carried through all subsequent steps to determine the contribution of the reagents and of the involved analytical steps. (QAMS)

Record Retention:
The systematic collection, indexing and storing of documented information under secure conditions.

Reference Material:
A material or substance one or more properties of which are sufficiently well established to be used for the calibration of an apparatus, the assessment of a measurement method, or for assigning values to materials. (ISO Guide 30-2.1)

Reference Standard:
A standard, generally of the highest metrological quality available at a given location, from which measurements made at that location are derived. (VIM-6.0-8)

Replicate Analyses:
The measurements of the variable of interest performed identically on two or more sub-samples of the same sample within a short time interval. (NELAC)

Reporting Limit (RL):
The level to which data is reported for a specific test method and/or sample. The reporting limit is either the laboratory nominal Limit of Quantitation (LOQ) or the level of sensitivity required by the client based on a sensitivity requirement that meets project objectives. The reporting limit (RL) cannot be lower than the quantitation limit (QL).

Resource Conservation and Recovery Act (RCRA):
The enabling legislation under 42 USC 321 et seq. (1976), that gives EPA the authority to control hazardous waste from the "cradle-to-grave", including its generation, transportation, treatment, storage, and disposal. (NELAC)

Safe Drinking Water Act (SDWA):
The enabling legislation, 42 USC 300f et seq. (1974), (Public Law 93-523), that requires the EPA to protect the quality of drinking water in the U.S. by setting maximum allowable contaminant levels, monitoring, and enforcing violations. (NELAC)

Sample Duplicate:
Two samples taken from and representative of the same population and carried through all steps of the sampling and analytical procedures in an identical manner. Duplicate samples are used to assess variance of the total method including sampling and analysis. (EPA-QAD)

Sampling and Analysis Plan (SAP):
A formal document describing the detailed sampling and analysis procedures for a specific project.

Second Order Polynomial Curve (Quadratic):
The 2nd order curves are a mathematical calculation of a slightly curved line over two axis. The y axis represents the instrument response (or Response ratio) of a standard or sample and the x axis represents the concentration. The 2nd order regression will generate a coefficient of determination (COD or r^2) that is a measure of the "goodness of fit" of the quadratic curvature the data. A value of 1.00 indicates a perfect fit. In order to be used for quantitative purposes, r^2 must be greater than or equal to 0.99.

Selectivity:
(Analytical chemistry) the capability of a test method or instrument to respond to a target substance of constituent in the presence of non-target substances. (EPA-QAD)

Sensitivity:
The capability of a method or instrument to discriminate between measurement responses representing different levels (e.g., concentrations) of a variable of interest. (NELAC)

Spike:
A known mass of target analyte added to a blank, sample or sub-sample; used to determine recovery efficiency or for other quality control purposes.

If the mandated or requested test method does not specify the spiking components, the laboratory shall spike all reportable components to be reported in the Laboratory Control Sample and Matrix Spike. However, in cases where the components interfere with accurate assessment (such as simultaneously spiking chlordane, toxaphene and PCBs in Method 608), the test method has an extremely long list of components or components are incompatible, a representative number (at a minimum 10%) of the listed components may be used to control the test method. The selected components of each spiking mix shall represent all chemistries, elution patterns and masses permit specified analytes and other client requested components. However, the laboratory shall ensure that all reported components are used in the spike mixture within a two-year time period.. (NELAC)

Standard:
The document describing the elements of laboratory accreditation that has been developed and established within the consensus principles of NELAC and meets the approval requirements of NELAC procedures and policies. (ASQC)

Standard Operating Procedures (SOPs):
A written document which details the method of an operation, analysis, or action whose techniques and procedures are thoroughly prescribed and which is accepted as the method for performing certain routine or repetitive tasks. (QAMS)

Standardized Reference Material (SRM):
A certified reference material produced by the U.S. National Institute of Standards and Technology or other equivalent organization and characterized for absolute content, independent of analytical method. (EPA-QAD)

Storage Blank:
A blank matrix stored with field samples of a similar matrix (volatiles only) that measures storage contribution to any source of contamination.

Surrogate:
A substance with properties that mimic the analyte of interest. It is unlikely to be found in environment samples and is added to them for quality control purposes.

Surrogate compounds must be added to all samples, standards, and blanks, for all organic chromatography methods except when the matrix precludes its use or when a surrogate is not available. Poor surrogate recovery may indicate a problem with sample composition and shall be reported to the client whose sample produced poor recovery. (QAMS)

Systems Audit (also Technical Systems Audit):
A thorough, systematic, qualitative on-site assessment of the facilities, equipment, personnel, training, procedures, record keeping, data validation, data management, and reporting aspects of a total measurement system. (EPA-QAD)

Toxic Substances Control Act (TSCA):
The enabling legislation in 15 USC 2601 et seq., (1976) that provides for testing, regulating, and screening all chemicals produced or imported into the United States for possible toxic effects prior to commercial manufacture. (NELAC)

Traceability:
The property of a result of a measurement whereby it can be related to appropriate standards, generally international or national standards, through an unbroken chain of comparisons. (VIM-6.12)

Trip Blank:
A blank matrix placed in a sealed container at the laboratory that is shipped, held unopened in the field, and returned to the laboratory in the shipping container with the field samples.

Uncertainty:
A parameter associated with the result of a measurement that characterizes the dispersion of the value that could reasonably be attributed to the measured value.

Acronyms:

CAR – Corrective Action Report
CCV – Continuing Calibration Verification
CF – Calibration Factor
CFR – Code of Federal Regulations
COC – Chain of Custody
CRS – Change Request Form
DL – Detection Limit
DLCK – Detection Limit Check Standard
DOC – Demonstration of Capability
DQO – Data Quality Objectives
DU – Duplicate
EHS – Environment, Health and Safety
EPA – Environmental Protection Agency
GC - Gas Chromatography
GC/MS - Gas Chromatography/Mass Spectrometry
HPLC - High Performance Liquid Chromatography
ICP - Inductively Coupled Plasma Atomic Emission Spectroscopy
ICV – Initial Calibration Verification
IDL – Instrument Detection Limit
IH – Industrial Hygiene
IS – Internal Standard
LCS – Laboratory Control Sample
LCSD – Laboratory Control Sample Duplicate
LOD – Limit of Detection
LOQ – Limit of Quantitation
LIMS – Laboratory Information Management System
MDL – Method Detection Limit
MDLCK – MDL Check Standard
MDLV – MDL Verification Check Standard
MRL – Method Reporting Limit Check Standard
MS – Matrix Spike
MSD – Matrix Spike Duplicate
MSDS - Material Safety Data Sheet
NCM – Non-Conformance Memo
NELAC - National Environmental Laboratory Accreditation Conference
NELAP - National Environmental Laboratory Accreditation Program
PT – Performance Testing
QAM – Quality Assurance Manual
QA/QC – Quality Assurance / Quality Control
QAPP – Quality Assurance Project Plan
RF – Response Factor
RPD – Relative Percent Difference
RSD – Relative Standard Deviation
SD – Serial Dilution
SOP- Standard Operating Procedure
TAT – Turn-Around-Time
VOA – Volatiles
VOC – Volatile Organic Compound

Appendix 5.

Laboratory Certifications, Accreditations, Validations

TestAmerica Chicago maintains certifications, accreditations, certifications, and validations with numerous state and national entities. Programs vary but may include on-site audits, reciprocal agreements with another entity, performance testing evaluations, review of the QA Manual, Standard Operating Procedures, Method Detection Limits, training records, etc. At the time of this QA Manual revision, the laboratory has accreditation/certification/licensing with the following organizations:

Organization	Lab ID Number
DoD-ELAP	ADE-1429
ISO/IEC 17025:2005	AT-1428
California	01132CA
Georgia	939
Hawaii	None
Illinois (NELAC)	100201
Indiana	C-IL-02
Iowa	82
Kansas	E-10161
Kentucky	90023 + UST 0066
Louisianna	02046
Massachusetts	M-IL035
Mississippi	None
North Carolina	291
Oklahoma	8908
South Carolina	77001003
Texas	T104704252-10-TX
USDA	P330-09-00027
Wisconsin	999580010
Wyoming	IL01 (8TMS-Q)

The certificates and parameter lists (which may differ) for each organization may be found on the corporate web site, the laboratory's public server, the final report review table, and in the following offices: QA, marketing, and project management.

Appendix 6. Example: Data Qualifiers – Standard Flagging Suite

Flag	Flag Text
*	Recovery or RPD exceeds control limits
*	ISTD response or retention time outside acceptable limits
*	ICPMS Relative Intensity is outside the method limits
<	Not detected at or above the reporting limit
>	The analyte exceeded the indicated concentration
^	Instrument related QC exceeds the control limits. (ICV, CCV, MRL,CRA, LRS, ICSA, ICSB, ICB, CCB)
4	MS, MSD: The analyte present in the original sample is 4 times greater than the matrix spike concentration; therefore, control limits are not applicable.
+	MSA correlation coefficient is less than 0.995.
A	The tentatively identified compound is a suspected aldol-condensation product.
B	Compound was found in the blank and sample.
b	Result Detected in the USB
C	Pesticide identification was confirmed by GC/MS.
D	Surrogate or matrix spike recoveries were not obtained because the extract was diluted for analysis; also compounds analyzed at a dilution may be flagged with a D.
D	Sample results are obtained from a dilution; the surrogate or matrix spike recoveries reported are calculated from diluted samples.
DW	Result has been dry weight corrected
E	Result exceeded calibration range.
F	MS or MSD Recovery or RPD exceeds the control limits
g	Result fails applicable drinking water standards
h	Alternate peak selection upon analytical review.
H	Sample was prepped or analyzed beyond the specified holding time
HF	Field parameter with a holding time of 15 minutes
I	Indicates the presence of an interference, recovery is not calculated.
J	Indicates an Estimated Value for TICs
J	Result is less than the RL but greater than or equal to the MDL and the concentration is an approximate value.
L	A negative instrument reading lower than the absolute value of the reporting limit
N	This flag indicates the presumptive evidence of a compound.
ND	Compound not detected.
P	The %RPD between the primary and confirmation column/detector is >40%. The higher value has been reported.
p	The %RPD between the primary and confirmation column/detector is >40%. The lower value has been reported.
Q	Result was qualitatively confirmed, but not quantitated.
R	The instrument was not calibrated for this compound. A non-detect indicates that the characteristic ions were not present and the compound was not qualitatively identified. No controls were present to determine either sample preparation efficiency or the instrument sensitivity for the compound. As a result, the limit of detection is not known and the reported concentrations are estimates.
S	Result was determined by the Method of Standard Additions
s	SCB Recovery Exceeds Limits
T	Result is a tentatively identified compound (TIC) and an estimated value.
U	Indicates the analyte was analyzed for but not detected.
V	Serial Dilution exceeds the control limits
W	PS: Post-digestion spike was outside control limits
X	Surrogate is outside control limits
Y	The chromatographic response resembles a typical fuel pattern.
Z	The chromatographic response does not resemble a typical fuel pattern.

Appendix 7 Method Capability Listing *(ISO17025 4.2.5)*

Unit	Parameter	Method No.	NELAP	Matrix
GCE	Pesticides/PCBs	EPA 608	X	W
GCE	Organochlorine Pesticides	SW 8081A / 8081B	X	W/S
GCE	PCBs	SW 8082 / 8082A	X	W/S
GCE	Chlorinated Herbicides by GC	SW 8151A	X	W/S
GCV	Petroleum Hydrocarbons (DRO)	SW 8015B / 8015C	X	W/S
GCV	Petroleum Hydrocarbons (GRO)	SW 8015B / 8015C	X	W/S
GCV	Glycol_Direct Inject	SW 8015B	X	W/S
HPLC	PAHs by HPLC	E 610	X	W
HPLC	PAHs by HPLC	SW 8310	X	W/S
HPLC	Explosives	SW 8330 / 8330A	X	W/S
M	Hardness (Ca, Mg)	E 200.7	X	W
M	Hardness (Ca, Mg)	SM 2340B	X	W
M	ICP Metals	E 200.7	X	W
M	ICP Metals- Soil Fractionation Analysis	M1213		S
M	ICP Metals- SEM Simultaneously Extracted Metals	SEM		S
M	ICP-MS Metals	E 200.8	X	W
M	CVAA Mercury	SW 7470A	X	W
M	CVAA Mercury	SW 7471A / 7471B	X	S
M	CVAA Mercury	E 245.1	X	W
M	ICP Metals	SW 6010B / 6010C	X	W/S
M	ICP-MS Metals	SW 6020/6020A	X	W/S
M	Metals-ICP	ILM04.0		W/S
M	Metals-Mercury	ILM04.0		W/S
MSB	GC/MS Semi-Volatiles	E 625	X	W
MSB	GC/MS Semi-Volatiles	SW 8270C / 8270D	X	W/S
MSB	GC/MS Semi-Volatiles	SW 8270C (SIM)		W
MSV	VOAs by GC/MS	E 624	X	W
MSV	GC/MS Volatiles	SW 8260B	X	W/S
P	GC/MS Soil VOAs in EnCore Samples	SW 5035 / 5035A		S
P	TCLP	SW 1311	X	S
P	SPLP	SW 1312	X	S
P	Extractable Organics; Accel. Liq.-Liq. Waters	SW 3520C (Limited Application)		W
P	Extractable Organics; Separatory Funnel	SW 3510C		W
P	Extractable Organics; Accel. Soxhlet	SW 3541A		S
P	Extractable Organics; Sonication	SW 3550B / 3550C		S

Unit	Parameter	Method No.	NELAP	Matrix
P	Acid Cleanup	SW 3665A		W/S
P	Alumina Cleanup	SW 3610B		W/S
P	Florisil Clean-up	SW 3620B		W/S
P	Gel Permeation Column Clean-up	SW 3640B		S
P	Sulfur Clean-up	SW 3660B		W/S
P	Waste Dilution	SW 3580A		S
P	Metals Digestions; Surface/Ground Water for ICP/ICPMS	SW 3005A		W
P	Metals Digestions; Waters/Extracts for ICP	SW 3010A		W
P	Metals Digestions; Soils/Wastes for ICP/ICPMS	SW 3050B		S
W	Alkalinity	SM 2320B	X	W/S
W	Ammonia - Nessl.	SM 4500NH3B&C,18thEd	X	W/S
W	BOD - 5 Day	SM 5210B	X	W
W	Bromide, IC	EPA 300.0	X	W
W	Bromide, IC	SW-846 9056 /9056A	X	W/S
W	Carbonaceous BOD	SM 5210B	X	W
W	Chloride (AQ2 Seal)	SM 4500Cl E	X	W/S
W	Chloride (AQ2 Seal)	SW-846 9251	X	W/S
W	Chloride, IC	EPA 300.0	X	W
W	Chloride, IC	SW-846 9056 /9056A	X	W/S
W	Chlorine, Residual	SM 4500 Cl F	X	W
W	COD - High Level	SM 5220C	X	W/S
W	COD - Low Level	SM 5220C	X	W/S
W	Chromium, Hexavalent	SM 3500-CrB	X	W/S
W	Chromium, Hexavalent	SW-846 3060A/7196A	X	W/S
W	Cyanide, Amenable	SM 4500CN G & E	X	W/S
W	Cyanide	SW-846 9010B/9010C/ 9014	X	W/S
W	Cyanide	SM 4500CN C, E	X	W/S
W	Cyanide	ILM04.0		W/S
W	Ferrous Iron	SM 3500 Fe B		W/S
W	Flashpoint (Ignitability)	SW-846 1010 /1010A	X	W/S
W	Fluoride / Fluorine	SM 4500F C	X	W/S
W	Fluoride, IC	EPA 300.0	X	W
W	Fluoride, IC	SW-846 9056 /9056A	X	W/S
W	Langlier Index (Corrosivity)	SM 2330A+B	X	W/S
W	Nitrate-NO2 (AQ2 Seal)	EPA 353.2	X	W/S
W	Nitrate-NO2 (AQ2 Seal)	SM 4500NO3F	X	W/S

Unit	Parameter	Method No.	NELAP	Matrix
W	Nitrate, IC	EPA 300.0	X	W
W	Nitrate, IC	SW-846 9056 /9056A	X	W/S
W	Nitrite	SM 4500NO2B	X	W/S
W	Nitrite, IC	EPA 300.0	X	W
W	Nitrite, IC	SW-846 9056 /9056A	X	W/S
W	Oil & Grease (HEM; SGT-HEM)	E 1664	X	W
W	Oil & Grease (HEM; SGT-HEM)	SW-846 9071B	X	S
W	Oxygen, Dissolved	SM 4500 O G	X	W
W	pH - Low/High	SM 4500H+B	X	W
W	pH - Low/High	SW-846 9045C / 9045D SW-846 9040B /9040C	X	W/S
W	Paint Filter	SW-846 9095A / 9095B	X	W
W	Phenol (AQ2 Seal)	EPA 420.4	X	W/S
W	Phenol (AQ2 Seal)	SW-846 9066	X	W/S
W	Phosphate, Ortho	SM 4500 PE	X	W/S
W	Phosphate, Ortho , IC	EPA 300.0	X	W
W	Phosphate, Ortho , IC	SW-846 9056 /9056A	X	W/S
W	Phosphorus	SM 4500 P E	X	W/S
W	Specific Conductance	EPA 120.1	X	W
W	Specific Conductance	SM 2510B	X	W
W	Specific Conductance	SW-846 9050A	X	W/S
W	Specific Gravity	ASTM D2710F		W/S
W	Sulfate - Turbidimetric	SW-846 9038M SM4500SO4E	X	W/S
W	Sulfate, IC	EPA 300.0	X	W
W	Sulfate, IC	SW-846 9056 /9056A	X	W/S
W	Sulfide	SM 4500SF	X	W/S
W	Sulfide	SW-846 9030B/9034	X	W/S
W	AVS- Acid Volatile Sulfide	AVS		S
W	Solids, Settleable	SM2540F		W
W	Sulfide, Reactive	SW 7.3.4.2	X	W/S
W	TKN - Nesslerization	SM 4500NorgC, 18th Ed. (SM4500NH3C, 18th Ed.)	X	W/S
W	TOC (TIC/DIC) [Organic Carbon; Inorganic & Dissolved]	SM 5310C	X	W
W	TOC (TIC/DIC) [Organic Carbon; Inorganic & Dissolved]	SW-846 9060 /9060A	X	W
W	TOC (TIC/DIC) [Organic Carbon; Inorganic & Dissolved]	Lloyd Kahn		S
W	TOX (Total Organic Halogens)	SM 5320B		W
W	TOX (Total Organic Halogens)	SW-846 9020B	X	W
W	TS - Water (Total Solids-Residue)	SM 2540B	X	W

Unit	Parameter	Method No.	NELAP	Matrix
W	TS - Solid (Total Solids-Residue)	SM 2540G	X	S
W	TSS (Total Suspended Solids)	SM 2540D	X	W
W	TDS (Total Dissolved Solids)	SM 2540C	X	W
W	TVS (Total Volatile Solids)	160.4 / SM2540E	X	W
W	TVDS (Total Volatile Dissolved Solids)	160.4 / SM2450E	X	W
W	TVSS (Total Volatile Suspended Solids)	160.4 / SM2540E	X	W

Matrix: W (Water) S (Soil/Solid) O (Other)
Note: NELAP accreditation may be matrix and program specific. Refer to TestAmerica Chicago's IL NELAP Accreditation Number: 100201

DoD QSM Version 4.2: Appendix F - Quality Control Requirements Summary
Table F-1 Summary of QC Check Definitions, Purpose, and Evaluation – Inorganics (Metals)

QC Check	Definition	Purpose	Evaluation
Calibration Blank	Reagent water containing no analytes of interest.	To determine the zero point of the calibration curve for all initial and continuing calibrations.	This is a required QC procedure. Continuing calibration blank responses above the LOD require corrective action
Continuing calibration verification (CCV)	This verification of the ICAL that is required during the course of analysis at periodic intervals. Continuing calibration verification applies to both external standard and internal standard calibration techniques, as well as to linear and non-linear calibration models	To verify that instrument response is reliable, and has not changed significantly from the current ICAL curve.	If the values for the analytes are outside the acceptance criteria, the ICAL may not be stable. Results associated with out-of-control CCV results require reanalysis or flagging
Demonstrate Acceptable Analytical Capability	QC samples are analyzed in series to verify ability to produce data of acceptable precision and bias.	To verify the ability to produce data of acceptable precision and bias for a specific instrument type, matrix, method, and analyst.	The average recovery of the spikes and standard deviation of the replicates must be within designated acceptance criteria. Analysis of field samples may not be conducted until this check is successful.
Dilution test (Metals only)	Analysis of a positive sample, which has been diluted to a concentration 1/5th of the original, to confirm that there is no interference in the original sample analysis. (Modified COE)	To assess matrix interference	Agreement within 10% between the concentration for the undiluted sample and 5x the concentration for the diluted sample indicates the absence of interferences, and such samples may be analyzed without using the MSA. Results outside acceptance limits indicate a possible matrix effect. For ICP a post-digestion spike must be run.
Duplicate Sample (replicate)	Two identical portions of material collected for chemical analysis, and identified by unique alphanumeric codes. The duplicate may be portioned from the same sample, or may be two identical samples taken from the same site. The two portions are prepared and analyzed identically. (modified QSM)	To provide information on the heterogeneity of the sample matrix or to determine the precision of the intralaboratory analytical process for a specific sample matrix	A duplicate sample will provide information on the heterogeneity of the sample matrix. The greater the heterogeneity of the matrix, the greater the relative percent difference between the sample and the sample duplicate. If the sample matrix is homogeneous (such as with drinking water) and the relative percent difference is high, this could indicate a problem in the analytical system.
Initial calibration for all analytes (ICAL)	Analysis of analytical standards at different concentrations that are used to determine and calibrate the quantitation range of the response of the analytical detector or method	To establish a calibration curve for the quantification of the analytes of interest	Statistical procedures are used to determine the relationship between the signal response and the known concentration of analytes of interest. The ICAL must be successful before any samples or other QC check samples can be analyzed.
Instrument Detection Limit (IDL) Study (6010 and 6020 only)	The process to determine the minimum concentration of a substance (analyte) that an instrument can differentiate from noise. The procedure for calculating varies by method.	To provide evaluation of instrument sensitivity	IDLs must be established before samples can be analyzed.

F-1 (cont.)			
QC Check	**Definition**	**Purpose**	**Evaluation**
Interference check solutions (ICP and ICP/MS only)	A pair of solutions containing interfering elements that are used to verify the correction factors of analytes of concern.	To verify the established correction factors by analyzing the interference check solution at the beginning of the analytical sequence	No samples can be run if this check does not pass acceptance criteria.
Internal Standards	A substance that is introduced in known amount into each calibration standard and field and QC sample of the analyte.	The ratio of the analyte signal to the internal standard signal is then used to determine the analyte concentration.	Any sample associated with out-of-control results must be reanalyzed.
Laboratory control sample (LCS) containing all analytes to be reported	A sample matrix, free from the analytes of interest, spiked with known amounts of analytes or a material containing known and verified amounts of analytes.	Used to evaluate the performance of the total analytical system, including all preparation and analysis steps. Assesses the ability of the laboratory/analyst to successfully recover the target analytes from a control (clean) matrix. Control limits for LCS recovery, typically expressed as percent recovery, are used for the development of statistical control limits and serve as acceptance criteria for determining whether an analytical run is in control (batch acceptance).	This is a required QC Check. The inability to achieve acceptable recoveries in the LCS indicate problems with the precision and bias of the measurement system. Failure to achieve acceptable recoveries in a "clean" matrix is an indicator of possible problems achieving acceptable recoveries in field samples.
Linear dynamic range or high-level check standards (ICP and ICP.MS only)	High-level check standard periodically analyzed to verify the linearity of the calibration curve at the upper end.	To verify quantitative accuracy of data up to the high=level standard.	The QC check establishes the upper linear range of the calibration.
Low-Level calibration check standard (ICP only)	A reference standard that contains a quantity of analyte equal to or less than the reporting limit.	To confirm the accuracy of measurements at or near the RL.	This QC check must be within acceptance criteria before any samples are analyzed.
Matrix Spike (MS)	A sample prepared by adding a know mass of target analyte to a specified amount of matrix sample for which an independent estimate of target analyte concentration is available.	To assess the performance of the method as applied to a particular matrix. Matrix spikes are used, for example, to determine the effect of the matrix on a method's recovery efficiency. The recovery of target analytes from the matrix spike sample is used to determine the bias of the method in the specific sample matrix.	The lack of acceptable recoveries in the matrix spike often points to problems with the sample matrix. One test of this is a comparison to the LCS recoveries. If the corresponding LCS recoveries are within acceptable limits, a matrix effect is likely. The lab should not correct for recovery; only report the results of the analyses and the associated MS results and indicate that the results from these analyses have increased uncertainty
Matrix Spike Duplicate (MSD)	A second replicate matrix spike prepared in the laboratory and analyzed to obtain a measure of the precision of recovery for each analyte	To assess the performance of the method as applied to a particular matrix and provide information on the homogeneity of the matrix. Also used to determine the precision of the intralaboratory analytical process for a specific sample matrix.	When compared to the MS, the MSD will provide information on the heterogeneity of the sample matrix. The greater the heterogeneity of the matrix, the greater the RPD between the matrix spike and the matrix spike duplicate. If the sample matrix is homogeneous, such as with drinking water, and the RPD is high, this could indicate a problem in the analytical system.

F-1 (cont.)			
QC Check	**Definition**	**Purpose**	**Evaluation**
MB	A sample of a matrix similar to the batch of associated samples (when available) that is free from the analytes of interest and is processed simultaneously with an under the same conditions as samples through all steps of the analytical procedures, and in which no target analytes or interferences are present at concentrations that impact the analytical results for sample analyses.	To assess background interferences or contamination in the analytical system that might lead to high bias or false positive data. Results of method blanks provide an estimate of the within-batch variability of the blank response and an indication of bias introduced by the preparation and analytical procedure.	This is one of the QC samples used to measure lab accuracy/bias. The sample could indicate whether contamination is occurring during sample prep and analysis. If analytes are detected > ½ RL, reanalyze or qualify (B-flag) all results for the specific analytes(s) in all samples in the associated prep batch as appropriate. For common lab contaminants, no analytes detected > RL. See Section D.1.1.1 and Box D-1
MSA (ICP only)	A set of procedures adding one or more increments of a standard solution to sample aliquots of the same size in order to overcome inherent matrix effects. The procedures encompass the extrapolation back to obtain the sample concentration. (This process is also called spiking the sample.)	To compensate for a sample constituent that enhances or depresses the analyte signal, thus producing a different slope from that of the calibration standards. It will not correct for additive interferences that cause a baseline shift.	This is the method used when matrix interferences are present and do not allow determination of accurate sample results
Post digestion spike addition (ICP and ICP/MS only)	An analyte spike added to a portion of prepared sample to verify absence or presence of matrix effects	To confirm the presence of a matrix interference. Assess matrix effects based on: 1. the occurrence of new and unusual matrices included within the batch, or 2. contingency analysis based on SD or MS failures	To verify the absence of an interference, the spike recovery must be between 75%-125% Results outside the acceptance limits require MSA for all samples within the batch
Second source calibration verification (ICV)	A standard obtained or prepared from a source independent of the source of standards for the ICAL. Its concentration should be at or near the middle of the calibration range. It is done after the ICAL.	To verify the accuracy of the ICAL.	The concentration of the 2nd source calibration verification, determined from the analysis, is compared with the known value of the standard to determine the accuracy of the ICAL. This independent verification of the ICAL must be acceptable before sample analysis can begin.

Notes:
1. Project-specific requirements identified by the client supersede any requirements listed. The requirements are meant to be default, to be used when project-specific direction based on DQOs is not available.
2. If there is a contradiction between the method and the DoD tables, the requirements specified in the tables shall be followed.

DoD QSM Version 4.2: Appendix F - Quality Control Requirements Summary
Table F-7: Inorganic Analysis by ICP and CVAA - Methods 6010 and 7000 Series

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Demonstrate acceptable analytical capability	Prior to using any test method and at any time there is a significant change in instrument type, personnel, test method, or sample matrix.	QC acceptance criteria published by DoD, if available; otherwise method- specified criteria.	Recalculate results; locate and fix problem, then rerun demonstration for those analytes that did not meet criteria (see Section C.1.f).	NA	This is a demonstration of analytical ability to generate acceptable precision and bias per the procedure in Appendix C. No analysis shall be allowed by analyst until successful demonstration of capability is complete.
LOD Determination and verification (See Box D-13)					
LOQ Establishment and verification (See Box D-14)					
Instrument detection limit (IDL) study (ICP only)	At initial set-up and after significant change in instrument type, personnel, test method, or sample matrix.	IDLs shall be $\leq$ LOD	NA	NA	Samples may not be analyzed without a valid IDL
Linear dynamic range (LRS) or high-level check standard (ICP only)	Every 6 months	Within $\pm$ 10% of expected value	NA	NA	
Initial Calibration (ICAL) for all analytes ICP: min 1 high std and a Calib.Blank CVAA: min 5 stds and a Calib. Blank	Daily ICAL prior to sample analysis	If more than one calibration standard is used $r \geq 0.995$.	Correct problem then repeat ICAL.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until ICAL has passed.
2nd Source calibration verification (ICV)	Once after each ICAL, prior to beginning a sample run.	Value of 2nd source for all analyte(s) within $\pm$ 10% of true value.	Correct problem and verify 2nd source standard. Rerun ICV. If that fails, correct problem and repeat ICAL.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until calibration has been verified.

F-7 (cont.)					
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action**	**Flagging Criteria**	**Comments**
Continuing Calibration verification (CCV)	After every 10 field samples and at the end of analysis sequence.	ICP: within ± 10% of true value. CVAA: within ± 20% of true value.	Correct problem, rerun CCV. If that fails, then repeat ICAL. Reanalyze all samples since the last successful CCV.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to all results for the specific analyte(s) in all samples since the last acceptable calibration verification.	Problem must be corrected. Results may not be reported without a valid CCV. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Low-level calibration check standard (ICP only)	Daily, after one-point ICAL.	Within ± 20% of true value	Correct problem, then reanalyze.	Flagging criteria are not appropriate.	No samples may be analyzed without a valid low-level calibration check standard. Low-level calibration check standard should be less than or equal to the reporting limit.
Method Blank (MB)	One per prep batch	No analytes detected > ½ RL and greater than 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater). Blank results must not otherwise affect sample results. For common lab contaminants, no analytes detected >RL (see Box D-1)	Correct problem, then see criteria in box D-1; If required, reprep and reanalyze MB and all samples processed with the contaminated blank.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply B-flag to all results for the specific analyte(s) in all samples in the associated prep batch.	Problem must be corrected. Results may not be reported without a valid method blank. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Calibration Blank (ICB / CCB)	Before beginning a sample run, after every 10 samples, and at the end of the analysis sequence	No analytes detected > LOD.	Correct problem. Reprep and reanalyze calibration blank. All samples following the last acceptable calibration blank must be reanalyzed.	Apply B-flag to all results for the specific analyte(s) in all samples associated with the blank.	
Interference check solutions (ICS) (ICP only)	At the beginning of an analytical run.	ICS-A: Absolute value of concentration for all non-spiked analytes < LOD (unless they are verified trace impurity from one of the spiked analytes) ICS-AB: ± 20% of true value.	Terminate analysis; locate and correct problem; reanalyze ICS, reanalyze all samples.	If corrective action fails, apply Q-flag to all results for specific analyte(s) in all samples associated with the ICS.	

F-7 (cont.)					
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action**	**Flagging Criteria**	**Comments**
LCS containing all analytes to be reported	One per prep batch	QC acceptance criteria specified by DoD, if available; see Box D-3 and Appendix G.	Correct problem., then reprep and reanalyze the LCS and all samples in the associated prep batch for failed analytes, if sufficient sample material is available (see full explanation in Appendix G).	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to specific analyte(s) in the associated prep batch.	Problem must be corrected. Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Matrix Spike (MS)	One per prep batch per matrix (see Box D-7)	For matrix evaluation, use QC acceptance criteria specified by DoD for LCS	Examine the project-specific DQOs. If the matrix spike fails outside of DoD criteria, additional quality control tests are required to evaluate matrix effects.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met.	For matrix evaluation only. If MS results are outside the LCS limits, the data shall be evaluated to determine the source of difference and to determine if there is a matrix effect or analytical error.
Matrix Spike Duplicate (MSD) or Sample Duplicate	One per prep batch per matrix (see Box D-7)	MSD: For matrix evaluation use QC acceptance criteria specified by DoD for LCS. MSD or sample duplicate: RPD $\leq$ 20% (between MS and MSD or sample and sample duplicate)	Examine the project-specific DQOs. Contact client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met.	The data shall be evaluated to determine the source of difference.
Dilution Test (ICP only)	One per prep batch .	Five-fold dilution must agree within $\pm$ 10% of the original measurement.	ICP: Perform post-digestion spike (PDS) addition.	Flagging criteria are not appropriate.	Only applicable for samples with concentrations > 50x LOQ.
Post-digestion spike (PDS) addition (ICP only)	When dilution test fails or analyte concentration in all samples < 50 x LOD	Recovery within 75-125% (see Table B-1)	Run all associated samples in the prep batch by MSA or see flagging criteria.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met.	Spike addition should produce a concentration of 10 – 100 x LOQ.
Method of Standard Addition (MSA)	When matrix interference is confirmed.	NA	NA	NA	Document use of MSA in the case narrative.
Results reported between DL and LOQ	NA	NA	NA	Apply J-flag to all results between LOD and LOQ	

Notes:
1. Project-specific requirements identified by the client supersede any requirements listed. The requirements are meant to be default, to be used when project-specific direction based on DQOs is not available.
2. If there is a contradiction between the method and the DoD tables, the requirements specified in the tables shall be followed.

DoD QSM Version 4.2: Appendix F - Quality Control Requirements Summary
Table F-1 Summary of QC Check Definitions, Purpose, and Evaluation – Inorganics (WC)

QC Check	Definition	Purpose	Evaluation
Calibration Blank	Reagent water containing no analytes of interest.	To determine the zero point of the calibration curve for all initial and continuing calibrations.	This is a required QC procedure. Continuing calibration blank responses above the LOD require corrective action
Continuing calibration verification (CCV)	This verification of the ICAL that is required during the course of analysis at periodic intervals. Continuing calibration verification applies to both external standard and internal standard calibration techniques, as well as to linear and non-linear calibration models	To verify that instrument response is reliable, and has not changed significantly from the current ICAL curve.	If the values for the analytes are outside the acceptance criteria, the ICAL may not be stable. Results associated with out-of-control CCV results require reanalysis or flagging
Demonstrate Acceptable Analytical Capability	QC samples are analyzed in series to verify ability to produce data of acceptable precision and bias.	To verify the ability to produce data of acceptable precision and bias for a specific instrument type, matrix, method, and analyst.	The average recovery of the spikes and standard deviation of the replicates must be within designated acceptance criteria. Analysis of field samples may not be conducted until this check is successful.
Duplicate Sample (replicate)	Two identical portions of material collected for chemical analysis, and identified by unique alphanumeric codes. The duplicate may be portioned from the same sample, or may be two identical samples taken from the same site. The two portions are prepared and analyzed identically. (modified QSM)	To provide information on the heterogeneity of the sample matrix or to determine the precision of the intralaboratory analytical process for a specific sample matrix	A duplicate sample will provide information on the heterogeneity of the sample matrix. The greater the heterogeneity of the matrix, the greater the relative percent difference between the sample and the sample duplicate. If the sample matrix is homogeneous (such as with drinking water) and the relative pecent difference is high, this could indicate a problem in the analytical system.
Initial calibration for all analytes (ICAL)	Analysis of analytical standards at different concentrations that are used to determine and calibrate the quantitation range of the response of the analytical detector or method	To establish a calibration curve for the quantification of the analytes of interest	Statistical procedures are used to determine the relationship between the signal response and the known concentration of analytes of interest. The ICAL must be successful before any samples or other QC check samples can be analyzed.
Laboratory control sample (LCS) containing all analytes to be reported	A sample matrix, free from the analytes of interest, spiked with known amounts of analytes or a material containing known and verified amounts of analytes.	Used to evaluate the performance of the total analytical system, including all preparation and analysis steps. Assesses the ability of the laboratory/analyst to successfully recover the target analytes from a control (clean) matrix. Control limits for LCS recovery, typically expressed as percent recovery, are used for the development of statistical control limits and serve as acceptance criteria for determining whether an analytical run is in control (batch acceptance).	This is a required QC Check. The inability to achieve acceptable recoveries in the LCS indicate problems with the precision and bias of the measurement system. Failure to achieve acceptable recoveries in a "clean" matrix is an indicator of possible problems achieving acceptable recoveries in field samples.

F-1 (cont.)			
QC Check	**Definition**	**Purpose**	**Evaluation**
MS	A sample prepared by adding a know mass of target analyte to a specified amount of matrix sample for which an independent estimate of target analyte concentration is available.	To assess the performance of the method as applied to a particular matrix. Matrix spikes are used, for example, to determine the effect of the matrix on a method's recovery efficiency. The recovery of target analytes from the matrix spike sample is used to determine the bias of the method in the specific sample matrix.	The lack of acceptable recoveries in the MS often points to problems with the sample matrix. One test of this is a comparison to the LCS recoveries. If the corresponding LCS recoveries are within acceptable limits, a matrix effect is likely. The lab should not correct for recovery; only report the results of the analyses and the associated MS results and indicate that the results from these analyses have increased uncertainty
MSD	A second replicate matrix spike prepared in the laboratory and analyzed to obtain a measure of the precision of recovery for each analyte	To assess the performance of the method as applied to a particular matrix and provide information on the homogeneity of the matrix. Also used to determine the precision of the intralaboratory analytical process for a specific sample matrix.	When compared to the MS, the MSD will provide information on the heterogeneity of the sample matrix. The greater the heterogeneity of the matrix, the greater the RPD between the matrix spike and the matrix spike duplicate. If the sample matrix is homogeneous, such as with drinking water, and the RPD is high, this could indicate a problem in the analytical system.
Matrix Verification sample (CR+6 only)	A pH-adjusted filtrate that has been spiked with CR+6 to ensure that the sample matrix does not have a reducing condition or other interferents that could affect color development. (Modified Method)	To ensure that the sample matrix does not have a reducing condition or other interferents that affect color development.	To verify the absence of an interference, the spike recovery must be between 85% and 115%. If the result of verification indicates a suppressive interference, the sample should be diluted and reanalyzed. If the interference persists after sample dilution, an alternative method (Method 7195, Coprecipitation, or Method 7197, Chelation/Extraction) should be used.
MB	A sample of a matrix similar to the batch of associated samples (when available) that is free from the analytes of interest and is processed simultaneously with an under the same conditions as samples through all steps of the analytical procedures, and in which no target analytes or interferences are present at concentrations that impact the analytical results for sample analyses.	To assess background interferences or contamination in the analytical system that might lead to high bias or false positive data. Results of method blanks provide an estimate of the within-batch variability of the blank response and an indication of bias introduced by the preparation and analytical procedure.	This is one of the QC samples used to measure lab accuracy/bias. The sample could indicate whether contamination is occurring during sample prep and analysis. If analytes are detected > ½ RL, reanalyze or qualify (B-flag) all results for the specific analytes(s) in all samples in the associated prep batch as appropriate. For common lab contaminants, no analytes detected > the RL. See Section D.1.1.1 and Box D-1
RT window position establishment for each analyte (and surrogate) (all chromatographic methods only)	Determination of the placement of the RT window (i.e. start/stop time) of each analyte or group of analytes as it elutes through the chromatographic column so that analyte identification can be made during sample analysis. This is done during the ICAL.	To idendify analytes of interest	Incorrect window position may result in false negatives, require additional manual integrations, or cause unnecessary reanalysis of samples when surrogates or spiked compounds are erroneously not identified.

F-1 (cont.)			
QC Check	**Definition**	**Purpose**	**Evaluation**
RT window width calculated for each analyte (and surrogate) (non-MS chromatographic methods only)	Determination of the length of time between sample injection and the appearance of a peak at the detector. The total length of time (window) is established for each analyte or group of analytes and is set for complete elution of analyte peaks. It is based upon a series of analyses and statistical calculations that establish the measured band on the chromatogram that can be associated with a specific analyte or group of analytes.	To ensure that the chromatographic system is operating reliably and that the system conditions have been optimized for the target anaytes and surrogates in the standards and sample matrix to be analyzed. It is done to minimize the occurrence of both false positive and false negative results.	Used to evaluate continued system performance. Tight RT windows may result in false negatives or may cuase unnecessary reanalysis of samples when surrogates or spiked compounds are erroneously not identified. Overly wide RT windows may result in false positive results that cannot be confirmed upon further analysis.
Second source calibration verification (ICV)	A standard obtained or prepared from a source independent of the source of standards for the ICAL. Its concentration should be at or near the middle of the calibration range. It is done after the ICAL.	To verify the accuracy of the ICAL.	The concentration of the 2nd source calibration verification, determined from the analysis, is compared with the known value of the standard to determine the accuracy of the ICAL. This independent verification of the ICAL must be acceptable before sample analysis can begin.

Notes:
1. Project-specific requirements identified by the client supersede any requirements listed. The requirements are meant to be default, to be used when project-specific direction based on DQOs is not available.
2. If there is a contradiction between the method and the DoD tables, the requirements specified in the tables shall be followed.

DoD QSM Version 4.2: Appendix F - Quality Control Requirements Summary
Table F-9: Inorganic Analysis by Colorimetric Hexavalent Chromium: Method 7196

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Demonstrate acceptable analytical capability	Prior to using any test method and at any time there is a significant change in instrument type, personnel, test method, or sample matrix.	QC acceptance criteria published in method; otherwise QC acceptance criteria established in-house by laboratory.	Recalculate results; locate and fix problem, then rerun demonstration for the analyte that did not meet criteria (see Section C.1.f).	NA	This is a demonstration of analytical ability to generate acceptable precision and bias per the procedure in Appendix C. No analysis shall be allowed by analyst until successful demonstration of capability is complete.
LOD Determination and verification (See Box D-13)					
LOQ Establishment and verification (See Box D-14)					
Reference Blank (reagent water)	Before beginning standards or sample analysis.	NA	NA	NA	Use for blank subtraction of standards, field and QC samples. For Turbid field samples, a turbidity blank must be used instead of the reference blank (using a sample aliquot prepped in accordance with method 7196A (Section 7.1)).
Initial Calibration (ICAL) (minimum 3 standards and a Calib. Blank)	Daily ICAL prior to sample analysis	$r \geq 0.995$.	Correct problem and repeat ICAL.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until ICAL has passed.
2nd Source calibration verification (ICV)	Before beginning a sample run.	Value of 2nd source within ± 10% of true value.	Correct problem and verify 2nd source standard. Rerun ICV. If that fails, correct problem and repeat ICAL.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until calibration has been verified.
Continuing Calibration verification (CCV)	After every 15 field samples and at the end of analysis sequence.	Value of CCV within ± 10% of true value.	Correct problem then repeat CCV and reanalyze all samples since the last successful CCV.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to all results for the specific analyte(s) in all samples since the last acceptable calibration verification.	Problem must be corrected. No samples may be run until calibration has been verified. Flagging is only appropriate in cases where the samples cannot be reanalyzed.

F-9 (cont.)					
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action**	**Flagging Criteria**	**Comments**
Method Blank (MB)	One per prep batch	No analytes detected > ½ RL and greater than 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater). Blank results must not otherwise affect sample results (see Box D-1).	Correct problem, then see criteria in box D-1; If required, reprep and reanalyze MB and all samples processed with the contaminated blank.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply B-flag to all results for the specific analyte(s) in all samples in the associated prep batch.	Problem must be corrected. Results may not be reported without a valid method blank. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
LCS containing all analytes to be reported	One per prep batch	QC acceptance criteria specified by DoD; see Box D-3 and Appendix G.	Correct problem, then reprep and reanalyze the LCS and all samples in the associated batch for the failed analyte in all samples in the associated prep batch, if sufficient sample material is available (see full explanation in Appendix G).	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to specific analyte(s) in all samples in the associated prep batch.	Problem must be corrected. Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Sample matrix Verification (also known as Matrix Spike - MS)	One for every sample matrix analyzed	Spike recovery within 85 – 115%	If check indicates interference, dilute and reanalyze sample; persistent interference indicates the need to use alternative method or analytical conditions, or to use MSA.	Flagging criteria are not appropriate.	Verification check ensures lack of reducing condition or interference from matrix. Additional corrective actions are identified in mtheo 7196A (Sections 7.4 and 7.5)
Matrix Spike Duplicate (MSD) or Sample Duplicate	Aqueous matrix: One per every 10 project samples per matrix. Solid matrix: One per prep batch per matrix.	Aqueous matrix: RPD ≤ 20% (between MS and MSD or sample and sample duplicate). Solid matrix: RPD ≤ 30%..	Examine the project-specific DQOs. Contact client as to additional measures to be taken.	Flagging criteria are not appropriate.	Refer to sample matrix verification sample for MS data evaluation.
Pre-digestion MS (Solid matrix samples only, Method 3060)	One soluble and insoluble pre-digestion MS analyzed per prep batch prior to analysis	MS recoveries within 75-125%	Correct problem and rehomogenize, redigest, and reanalyze samples. If that fails, evaluate against LCS results	If corrective action fails, apply J-flag to the analyte in all samples in the associated prep batch.	

F-9 (cont.)					
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action**	**Flagging Criteria**	**Comments**
Post-digestion matrix spike (PDS)	One per prep batch	Recovery within 85-115%	Correct problem and rehomogenize, redigest, and reanalyze samples. Persistent interference indicates the need to use an alternative method or analytical conditions, or to use MSA.	NA	
Results reported between DL and LOQ	NA	NA	NA	Apply J-flag to all results between DL and LOQ.	

Notes:

1. Project-specific requirements identified by the client supersede any requirements listed. The requirements are meant to be default, to be used when project-specific direction based on DQOs is not available.
2. If there is a contradiction between the method and the DoD tables, the requirements specified in the tables shall be followed.

DoD QSM Version 4.2: Appendix F - Quality Control Requirements Summary
Table F-10: Inorganic Analysis by Cyanide: Method 9010 and 9014

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Demonstrate acceptable analytical capability	Prior to using any test method and at any time there is a significant change in instrument type, personnel, test method, or sample matrix.	QC acceptance criteria published by DoD, if available; otherwise method- specified criteria.	Recalculate results; locate and fix problem, then rerun demonstration for those analytes that did not meet criteria (see Section C.1.f).	NA	This is a demonstration of analytical ability to generate acceptable precision and bias per the procedure in Appendix C. No analysis shall be allowed by analyst until successful demonstration of capability is complete.
LOD Determination and verification (See Box D-13)					
LOQ Establishment and verification (See Box D-14)					
Initial Calibration (ICAL) 6 stds and a Calib. Blank	Daily ICAL prior to sample analysis	$r \geq 0.995$.	Correct problem then repeat ICAL.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until ICAL has passed. All calibration standards must be distilled if samples are expected to contain sulfides.
Distilled Standards (one high and one low)	Once per multipoint calibration	Within $\pm$ 15% of true value	Correct problem, then repeat distilled standards	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until distilled standards have passed.
2nd Source calibration verification (ICV)	Once after each ICAL, prior to beginning a sample run.	Within $\pm$ 15% of true value.	Correct problem, then repeat distilled standards.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until calibration has been verified.
Method Blank (MB)	One per prep batch	No analytes detected > ½ RL and greater than 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater). Blank results must not otherwise affect sample results. For common lab contaminants, no analytes detected >RL (see Box D-1)	Correct problem, then see criteria in box D-1; If required, reprep and reanalyze MB and all samples processed with the contaminated blank.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply B-flag to all results for the specific analyte(s) in all samples in the associated prep batch.	Problem must be corrected. Results may not be reported without a valid method blank. Flagging is only appropriate in cases where the samples cannot be reanalyzed.

F-10 (Cont.)					
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action**	**Flagging Criteria**	**Comments**
LCS containing all analytes to be reported	One per prep batch	QC acceptance criteria specified by DoD, if available; see Box D-3 and Appendix G.	Correct problem, then reprep and reanalyze the LCS and all samples in the associated prep batch for failed analytes, if sufficient sample material is available (see full explanation in Appendix G).	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to specific analyte(s) in all samples in the associated prep batch.	Problem must be corrected. Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Matrix Spike (MS)	One per prep batch per matrix (see Box D-7)	For matrix evaluation, use QC acceptance criteria specified by DoD for LCS	Examine the project-specific DQOs. If the matrix spike fails outside of DoD criteria, MSA shall be used for the analysis..	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met.	If MS results are outside the LCS limits, the data shall be evaluated to determine the source of difference and to determine if there is a matrix effect or analytical error.
Matrix Spike Duplicate (MSD) or Sample Duplicate	One per prep batch per matrix (see Box D-7)	MSD: For matrix evaluation use QC acceptance criteria specified by DoD for LCS. MSD or sample duplicate: RPD ≤ 20% (between MS and MSD or sample and sample duplicate)	Correct problem and reanalyze sample and duplicate.	Apply J-flag if sample cannot be rerun or reanalysis does not correct problem..	The data shall be evaluated to determine the source of difference.
Results reported between DL and LOQ	NA	NA	NA	Apply J-flag to all results between DL and LOQ.	

Notes:
1. Project-specific requirements identified by the client supersede any requirements listed. The requirements are meant to be default, to be used when project-specific direction based on DQOs is not available.
2. If there is a contradiction between the method and the DoD tables, the requirements specified in the tables shall be followed.

DoD QSM Version 4.2 Appendix F - Quality Control Requirements Summary
Table F-11: Inorganic Analysis by Common Anions: Method 9056

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Demonstrate acceptable analytical capability	Prior to using any test method and at any time there is a significant change in instrument type, personnel, test method, or sample matrix.	QC acceptance criteria published by DoD, if available; otherwise method- specified criteria.	Recalculate results; locate and fix problem, then rerun demonstration for those analytes that did not meet criteria (see Section C.1.f).	NA	This is a demonstration of analytical ability to generate acceptable precision and bias per the procedure in Appendix C. No analysis shall be allowed by analyst until successful demonstration of capability is complete.
LOD Determination and verification (See Box D-13)					
LOQ Establishment and verification (See Box D-14)					
RT window width calculated for each analyte	After method set-up and after major maintenance (e.g., column change).	RT width $\pm$ 3 times standard deviation for each analyte RT over a 24-hour period.	NA	NA	
Initial Calibration (ICAL) for all analytes min 3 standards and one calibration blank	ICAL prior to sample analysis	$r \geq 0.995$.	Correct problem, then repeat ICAL.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until ICAL has passed.
2nd Source calibration verification (ICV)	Once after each ICAL, prior to beginning a sample run.	All analytes within $\pm$ 10% of true value and RTs within appropriate windows.	Correct problem and verify 2nd source standard. Rerun ICV. If that fails, correct problem and repeat ICAL.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until calibration has been verified.
RT window position establishment for each analyte	Once per ICAL	Position shall be set using the midpoint standard of the ICAL curve when ICAL is performed. On days when ICAL is not performed, the initial CCV is used.	NA	NA	
Midrange continuing calibration verification (CCV)	After every 10 field samples and at the end of the analysis sequence.	All project analytes within established RT windows. Within $\pm$ 10% of true value.	Correct problem, then rerun calibration verification. If that fails, then repeat ICAL. Reanalyze all samples since the last successful calibration verification.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to all results for the specific analyte(s) in all samples since the last acceptable CCV.	Problem must be corrected. Results may not be reported without a valid CCV. Flagging is only appropriate in cases where the samples cannot be reanalyzed. RT windows are updated per the method.

F-11 (cont.)					
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action**	**Flagging Criteria**	**Comments**
Method Blank (MB)	One per prep batch	No analytes detected > ½ RL and greater than 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater). Blank results must not otherwise affect sample results (see Box D-1).	Correct problem, then see criteria in box D-1; If required, reprep and reanalyze MB and all samples processed with the contaminated blank.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply B-flag to all results for the specific analyte(s) in all samples in the associated prep batch.	Problem must be corrected. Results may not be reported without a valid method blank. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
LCS containing all analytes to be reported	One per prep batch	Laboratory in-house limits not to exceed ± 20%. Control limits may be not greater than ± 3 times the standard deviation of the mean LCS recovery. See Box D-3.	Correct problem, then reprep and reanalyze the LCS and all samples in the associated prep batch for failed analytes, if sufficient sample material is available (see full explanation in Appendix G).	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to specific analyte(s) in all samples in the associated prep batch.	Problem must be corrected. Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Matrix Spike (MS)	One per prep batch per matrix (see Box D-7)	For matrix evaluation, use laboratory in-house LCS limits (not to exceed ± 20%).	Examine the project-specific DQOs. Contact client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met.	For matrix evaluation only. If MS results are outside the LCS limits, the data shall be evaluated to determine the source of difference and to determine if there is a matrix effect or analytical error.
Matrix Spike Duplicate (MSD)	One per prep batch per matrix (see Box D-7)	For matrix evaluation use laboratory in-house LCS limits (not to exceed ± 20%). RPD ≤ 15% (between MS and MSD).	Examine the project-specific DQOs. Contact client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met.	The data shall be evaluated to determine the source of difference.
Sample duplicate (replicate)	One per every 10 samples.	% D ≤ 10% (between sample and sample duplicate).	Correct problem and reanalyze sample and duplicate.	Apply J-flag if sample cannot be rerun or reanalysis does not correct problem.	The data shall be evaluated to determine the source of difference.
Results reported between DL and LOQ	NA	NA	NA	Apply J-flag to all results between DL and LOQ	

Notes:
1. Project-specific requirements identified by the client supersede any requirements listed. The requirements are meant to be default, to be used when project-specific direction based on DQOs is not available.
2. If there is a contradiction between the method and the DoD tables, the requirements specified in the tables shall be followed.

DoD QSM Version 4.2: Appendix F - Quality Control Requirements Summary
Table F-1 Summary of QC Check Definitions, Purpose, and Evaluation – Organics (GC/HPLC)

QC Check	Definition	Purpose	Evaluation
Breakdown Check 8081: Endrin, DDT 8270: DDT	Analysis of s standard solution containing Endrin and DDT. Area counts of these compounds and their breakdown products are evaluated to assess instrument conditions.	To verify the inertness of the injection port because DDT and Endrin are easily degraded in the injection port.	If degredation of either DDT or Endrin exceeds method–specified criteria, corrective action must be taken before proceeding with calibration
Confirmation of positive results (organics only)	Use of alternative analytical techniques (another method, dissimilar column, or different detector such as MS detector) to validate the presence of target analytes identified	To verify the identification of an analyte	All positive results must be confirmed.
Continuing calibration verification (CCV)	This verification of the ICAL that is required during the course of analysis at periodic intervals. Continuing calibration verification applies to both external standard and internal standard calibration techniques, as well as to linear and non-linear calibration models	To verify that instrument response is reliable, and has not changed significantly from the current ICAL curve.	If the values for the analytes are outside the acceptance criteria, the ICAL may not be stable. Results associated with out-of-control CCV results require reanalysis or flagging
Demonstrate Acceptable Analytical Capability	QC samples are analyzed in series to verify ability to produce data of acceptable precision and bias.	To verify the ability to produce data of acceptable precision and bias for a specific instrument type, matrix, method, and analyst.	The average recovery of the spikes and standard deviation of the replicates must be within designated acceptance criteria. Analysis of field samples may not be conducted until this check is successful.
Duplicate Sample (replicate)	Two identical portions of material collected for chemical analysis, and identified by unique alphanumeric codes. The duplicate may be portioned from the same sample, or may be two identical samples taken from the same site. The two portions are prepared and analyzed identically. (modified QSM)	To provide information on the heterogeneity of the sample matrix or to determine the precision of the intralaboratory analytical process for a specific sample matrix	A duplicate sample will provide information on the heterogeneity of the sample matrix. The greater the heterogeneity of the matrix, the greater the relative percent difference between the sample and the sample duplicate. If the sample matrix is homogeneous (such as with drinking water) and the relative pecent difference is high, this could indicate a problem in the analytical system.
Initial calibration for all analytes (ICAL)	Analysis of analytical standards at different concentrations that are used to determine and calibrate the quantitation range of the response of the analytical detector or method	To establish a calibration curve for the quantification of the analytes of interest	Statistical procedures are used to determine the relationship between the signal response and the known concentration of analytes of interest. The ICAL must be successful before any samples or other QC check samples can be analyzed.
Internal Standards	A substance that is introduced in known amount into each calibration standard and field and QC sample of the analyte.	The ratio of the analyte signal to the internal standard signal is then used to determine the analyte concentration.	Any sample associated with out-of-control results must be reanalyzed.

Table F-1 Cont.			
QC Check	**Definition**	**Purpose**	**Evaluation**
Laboratory control sample (LCS) containing all analytes to be reported	A sample matrix, free from the analytes of interest, spiked with known amounts of analytes or a material containing known and verified amounts of analytes.	Used to evaluate the performance of the total analytical system, including all preparation and analysis steps. Assesses the ability of the laboratory/analyst to successfully recover the target analytes from a control (clean) matrix. Control limits for LCS recovery, typically expressed as percent recovery, are used for the development of statistical control limits and serve as acceptance criteria for determining whether an analytical run is in control (batch acceptance).	This is a required QC Check. The inability to achieve acceptable recoveries in the LCS indicate problems with the precision and bias of the measurement system. Failure to achieve acceptable recoveries in a "clean" matrix is an indicator of possible problems achieving acceptable recoveries in field samples.
MS	A sample prepared by adding a know mass of target analyte to a specified amount of matrix sample for which an independent estimate of target analyte concentration is available.	To assess the performance of the method as applied to a particular matrix. Matrix spikes are used, for example, to determine the effect of the matrix on a method's recovery efficiency. The recovery of target analytes from the matrix spike sample is used to determine the bias of the method in the specific sample matrix.	The lack of acceptable recoveries in the MS often points to problems with the sample matrix. One test of this is a comparison to the LCS recoveries. If the corresponding LCS recoveries are within acceptable limits, a matrix effect is likely. The lab should not correct for recovery; only report the results of the analyses and the associated MS results and indicate that the results from these analyses have increased uncertainty
MSD	A second replicate matrix spike prepared in the laboratory and analyzed to obtain a measure of the precision of recovery for each analyte	To assess the performance of the method as applied to a particular matrix and provide information on the homogeneity of the matrix. Also used to determine the precision of the intralaboratory analytical process for a specific sample matrix.	When compared to the MS, the MSD will provide information on the heterogeneity of the sample matrix. The greater the heterogeneity of the matrix, the greater the RPD between the matrix spike and the matrix spike duplicate. If the sample matrix is homogeneous, such as with drinking water, and the RPD is high, this could indicate a problem in the analytical system.
MB	A sample of a matrix similar to the batch of associated samples (when available) that is free from the analytes of interest and is processed simultaneously with an under the same conditions as samples through all steps of the analytical procedures, and in which no target analytes or interferences are present at concentrations that impact the analytical results for sample analyses.	To assess background interferences or contamination in the analytical system that might lead to high bias or false positive data. Results of method blanks provide an estimate of the within-batch variability of the blank response and an indication of bias introduced by the preparation and analytical procedure.	This is one of the QC samples used to measure lab accuracy/bias. The sample could indicate whether contamination is occurring during sample prep and analysis. If analytes are detected > ½ RL, reanalyze or qualify (B-flag) all results for the specified analytes(s) in all samples in the associated prep batch as appropriate. For common lab contaminants, no analytes detected > RL. See Section D.1.1.1 and Box D-1
RT window position establishment for each analyte (and surrogate) (all chromatographic methods only)	Determination of the placement of the RT window (i.e. start/stop time) of each analyte or group of analytes as it elutes through the chromatographic column so that analyte identification can be made during sample analysis. This is done during the ICAL.	To idendify analytes of interest	Incorrect window position may result in false negatives, require additional manual integrations, or cause unnecessary reanalysis of samples when surrogates or spiked compounds are erroneously not identified.

Table F-1 Cont.			
QC Check	**Definition**	**Purpose**	**Evaluation**
RT window width calculated for each analyte (and surrogate) (non-MS chromatographic methods only)	Determination of the length of time between sample injection and the appearance of a peak at the detector. The total length of time (window) is established for each analyte or group of analytes and is set for complete elution of analyte peaks. It is based upon a series of analyses and statistical calculations that establish the measured band on the chromatogram that can be associated with a specific analyte or group of analytes.	To ensure that the chromatographic system is operating reliably and that the system conditions have been optimized for the target anaytes and surrogates in the standards and sample matrix to be analyzed. It is done to minimize the occurrence of both false positive and false negative results.	Used to evaluate continued system performance. Tight RT windows may result in false negatives or may cuase unnecessary reanalysis of samples when surrogates or spiked compounds are erroneously not identified. Overly wide RT windows may result in false positive results that cannot be confirmed upon further analysis.
Second source calibration verification (ICV)	A standard obtained or prepared from a source independent of the source of standards for the ICAL. Its concentration should be at or near the middle of the calibration range. It is done after the ICAL.	To verify the accuracy of the ICAL.	The concentration of the 2nd source calibration verification, determined from the analysis, is compared with the known value of the standard to determine the accuracy of the ICAL. This independent verification of the ICAL must be acceptable before sample analysis can begin.
Surrogate spike (organic analysis only)	A substance with properties that mimic the analyte of interest. It is unlikely to be found in environmental samples and is added to them for quality control purposes.	To assess the ability of the method to successfully recover specific non-target analytes from an actual matrix. Because surrogates are generally added to each sample in a batch, they can be used to monitor recovery on a sample-specific, rather than batch-specific basis.	Whereas the MS is normally done on a batch-specific basis, the surrogate spike is done on a sample-specific basis. Taken with the information derived from other spikes (LCS; MS), the bias in the analytical system can be determined.

Notes:
1. Project-specific requirements identified by the client supersede any requirements listed. The requirements are meant to be default, to be used when project-specific direction based on DQOs is not available.
2. If there is a contradiction between the method and the DoD tables, the requirements specified in the tables shall be followed.

DoD QSM Version 4.2: Appendix F - Quality Control Requirements Summary
Table F-2: Organic Analysis by GC/HPLC - Methods 8015; 8081; 8082; 8151; 8310; 8330

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Demonstrate acceptable analytical capability	Prior to using any test method and at any time there is a significant change in instrument type, personnel, test method, or sample matrix.	QC acceptance criteria published by DoD, if available; otherwise method-specified criteria.	Recalculate results; locate and fix problem, then rerun demonstration for those analytes that did not meet criteria (see Section C.1.f).	NA	This is a demonstration of analytical ability to generate acceptable precision and bias per the procedure in Appendix C. No analysis shall be allowed by analyst until successful demonstration of capability is complete.
LOD Determination and verification (See Box D-13)					
LOQ Establishment and verification (See Box D-14)					
RT window width calculated for each analyte and surrogate	At method set-up and after major maintenance (e.g. column change).	RT width is $\pm$ 3 times standard deviation for each analyte RT from a 72-hour study.	NA	NA	
Breakdown check (Endrin/DDT Method 8081 only)	At the beginning of each 12-hour period, prior to analysis of samples	Degradation $\leq$ 15% for both DDT and Endrin	Correct problem then repeat breakdown check.	Flagging criteria are not appropriate.	No samples shall be run until degradation $\leq$ 15% .for both DDT and Endrin.
Minimum five-point initial calibration (ICAL) for all analytes	ICAL prior to sample analysis.	One of the options below. **Option 1:** RSD for each analyte $\leq$ 20% **Option 2:** linear least squares regression: $r \geq 0.995$ **Option 3:** non-linear regression: coefficient of determination (COD) $r^2 \geq 0.99$ (6 points shall be used for 2nd order, 7 points shall be used for 3rd order)	Correct problem then repeat initial calibration	NA	Problem must be corrected. No samples may be run until ICAL has passed. Caliration may not be forced through the origin. Quantitation for multicomponent analytes such as chlordane, toxaphene, and Aroclors must be performed using a 5-point calibration. Results may not be quantitated using a single point.
RT window position establishement for each analyte and surrogate	Once per ICAL and at the beginning of the analytical shift.	Position shall be set using midpoint standard of the ICAL curve when ICAL is performed. On days when ICAL is not performed, the initial CCV is used.	NA	NA	

Table F-2 Cont.					
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action**	**Flagging Criteria**	**Comments**
2nd Source calibration verification (ICV)	Immediately following ICAL	All project analytes within established RT windows. GC Methods: All project analytes within ± 20% of expected value from the ICAL; HPLC Methods: All project analytes within ± 15% of expected value from the ICAL.	Correct problem, rerun, ICV. If that fails, repeat ICAL.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until calibration has been verified.
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action**	**Flagging Criteria**	**Comments**
Continuing calibration verification (CCV)	Prior to sample analysis, after every 10 field samples, and at the end of the analysis sequence.	All project analytes within established RT windows. GC Methods: All project analytes within ± 20% of expected value from the ICAL; HPLC Methods: All project analytes within ± 15% of expected value from the ICAL.	Correct problem, then rerun calibration verification. If that fails, then repeat ICAL. Reanalyze all samples since last successful calibration verification.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to all results for the specific analyte(s) in all samples since the last acceptable calibration verification.	Problem must be corrected. Results may not be reported without a valid CCV. Flagging is only appropriate in cases where the samples cannot be reanalyzed. RT windows are updated per the method.
Method Blank (MB)	One per prep batch	No analytes detected > ½ RL and > 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater). Blank result must not otherwise affect sample results (see Box D-1).	Correct problem, then see criteria in box D-1; if required, reprep and reanalyze MB and all samples processed with the contaminated blank.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply B-flag to all results for the specific analyte(s) in all samples in the associated prep batch.	Problem must be corrected. Results may not be reported without a valid MB. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
LCS containing all analytes to be reported, including surrogates	One per prep batch	QC acceptance criteria specified by DoD, if available. Otherwise, use in-house control limits. In-house control limits may not be greater than ± 3 times the standard deviation of the mean LCS recovery. See Box D-3 and Appendix G.	Correct problem, then reprep and reanalyze the LCS and all samples in the associated prep batch for failed analytes, if sufficient sample material is available (see full explanation in Appendix G).	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to specific analyte(s) in all samples in the associated prep batch.	Problem must be corrected. Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Matrix Spike (MS)	One per prep batch per matrix (see Box D-7).	For matrix evaluation, use LCS acceptance criteria specified by DoD, if available. Otherwise, use in-house LCS control limits.	Examine the project-specific DQOs. Contact the client as to additional measures to be taken.	For specific analytes(s) in the parent sample, apply J-flag if acceptable criteria are not met.	For matrix evaluation only. If MS results are outside the LCS limits, the data shall be evaluated to determine the source of difference and to determine if there is a matrix effect or analytical error.

Table F-2 Cont.					
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action**	**Flagging Criteria**	**Comments**
Matrix spike duplicate MSD or Sample Duplicate	One per prep batch per matrix (see Box D-7).	MSD: For matrix evaluation, use LCS acceptance criteria specified by DoD, if available. Otherwise, use in-house LCS control limits. MSD or sample duplicate: RPD ≤ 30% (between MS and MSD or sample and sample duplicate).	Examine the project-specific DQOs. Contact client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met.	The data shall be evaluated to determine the source of difference.
Surrogate spike	All field and QC samples	QC acceptance criteria specified by DoD, if available. Otherwise use in-house control limits.	For QC and field samples, correct problem then reprep and reanalyze all failed samples for failed surrogates in the associated prep batch, if sufficient sample material is available. If obvious chromatographic interferences with surrogate is present, reanalysis may not be necessary.	Apply Q-flag to all associated analytes if acceptance criteria are not met.	Alternative surrogates are recommended when there is obvious chromatographic interferences.
Confirmation of positive results (second column or second detector)	All positive results must be confirmed (with the exception of Method 8015).	Calibration and QC criteria same as for initial or primary column analysis. Results between primary and second column RPD ≤ 40%.	NA	Apply J-flag if RPD > 40%. Discuss in the case narrative.	Using project-specific reporting requirements if available; otherwise, use method reporting requirements; otherwise, report the result from the primary column (see Box D-16).
Results reported between DL and LOQ	NA	NA	NA	Apply J-flag to all results between DL and LOQ.	

Notes:
1. Project-specific requirements identified by the client supersede any requirements listed. The requirements are meant to be default, to be used when project-specific direction based on DQOs is not available.
2. If there is a contradiction between the method and the DoD tables, the requirements specified in the tables shall be followed.

DoD QSM Version 4.2: Appendix F - Quality Control Requirements Summary
Table F-1 Summary of QC Check Definitions, Purpose, and Evaluation – Organics (GC/MS)

QC Check	Definition	Purpose	Evaluation
Breakdown Check 8081: Endrin, DDT 8270: DDT	Analysis of s standard solution containing Endrin and DDT. Area counts of these compounds and their breakdown products are evaluated to assess instrument conditions.	To verify the inertness of the injection port because DDT and Endrin are easily degraded in the injection port.	If degredation of `either DDT or Endrin exceeds method–specified criteria, corrective action must be taken before proceeding with calibration
Confirmation of positive results (organics only)	Use of alternative analytical techniques (another method, dissimilar column, or different detector such as MS detector) to validate the presence of target analytes identified	To verify the identification of an analyte	All positive results must be confirmed.
Continuing calibration verification (CCV)	This verification of the ICAL that is required during the course of analysis at periodic intervals. Continuing calibration verification applies to both external standard and internal standard calibration techniques, as well as to linear and non-linear calibration models	To verify that instrument response is reliable, and has not changed significantly from the current ICAL curve.	If the values for the analytes are outside the acceptance criteria, the ICAL may not be stable. Results associated with out-of-control CCV results require reanalysis or flagging
Demonstrate Acceptable Analytical Capability	QC samples are analyzed in series to verify ability to produce data of acceptable precision and bias.	To verify the ability to produce data of acceptable precision and bias for a specific instrument type, matrix, method, and analyst.	The average recovery of the spikes and standard deviation of the replicates must be within designated acceptance criteria. Analysis of field samples may not be conducted until this check is successful.
Duplicate Sample (replicate)	Two identical portions of material collected for chemical analysis, and identified by unique alphanumeric codes. The duplicate may be portioned from the same sample, or may be two identical samples taken from the same site. The two portions are prepared and analyzed identically. (modified QSM)	To provide information on the heterogeneity of the sample matrix or to determine the precision of the intralaboratory analytical process for a specific sample matrix	A duplicate sample will provide information on the heterogeneity of the sample matrix. The greater the heterogeneity of the matrix, the greater the relative percent difference between the sample and the sample duplicate. If the sample matrix is homogeneous (such as with drinking water) and the relative percent difference is high, this could indicate a problem in the analytical system.
Initial calibration for all analytes (ICAL)	Analysis of analytical standards at different concentrations that are used to determine and calibrate the quantitation range of the response of the analytical detector or method	To establish a calibration curve for the quantification of the analytes of interest	Statistical procedures are used to determine the relationship between the signal response and the known concentration of analytes of interest. The ICAL must be successful before any samples or other QC check samples can be analyzed.
Internal Standards	A substance that is introduced in known amount into each calibration standard and field and QC sample of the analyte.	The ratio of the analyte signal to the internal standard signal is then used to determine the analyte concentration.	Any sample associated with out-of-control results must be reanalyzed.

Table F-1 Cont.			
QC Check	**Definition**	**Purpose**	**Evaluation**
Laboratory control sample (LCS) containing all analytes to be reported	A sample matrix, free from the analytes of interest, spiked with known amounts of analytes or a material containing known and verified amounts of analytes.	Used to evaluate the performance of the total analytical system, including all preparation and analysis steps. Assesses the ability of the laboratory/analyst to successfully recover the target analytes from a control (clean) matrix. Control limits for LCS recovery, typically expressed as percent recovery, are used for the development of statistical control limits and serve as acceptance criteria for determining whether an analytical run is in control (batch acceptance).	This is a required QC Check. The inability to achieve acceptable recoveries in the LCS indicate problems with the precision and bias of the measurement system. Failure to achieve acceptable recoveries in a "clean" matrix is an indicator of possible problems achieving acceptable recoveries in field samples.
MS	A sample prepared by adding a know mass of target analyte to a specified amount of matrix sample for which an independent estimate of target analyte concentration is available.	To assess the performance of the method as applied to a particular matrix. Matrix spikes are used, for example, to determine the effect of the matrix on a method's recovery efficiency. The recovery of target analytes from the matrix spike sample is used to determine the bias of the method in the specific sample matrix.	The lack of acceptable recoveries in the matrix spike often points to problems with the sample matrix. One test of this is a comparison to the LCS recoveries. If the corresponding LCS recoveries are within acceptable limits, a matrix effect is likely. The lab should not correct for recovery; only report the results of the analyses and the associated MS results and indicate that the results from these analyses have increased uncertainty
MSD	A second replicate matrix spike prepared in the laboratory and analyzed to obtain a measure of the precision of recovery for each analyte	To assess the performance of the method as applied to a particular matrix and provide information on the homogeneity of the matrix. Also used to determine the precision of the intralaboratory analytical process for a specific sample matrix.	When compared to the MS, the MSD will provide information on the heterogeneity of the sample matrix. The greater the heterogeneity of the matrix, the greater the RPD between the matrix spike and the matrix spike duplicate. If the sample matrix is homogeneous, such as with drinking water, and the RPD is high, this could indicate a problem in the analytical system.
MB	A sample of a matrix similar to the batch of associated samples (when available) that is free from the analytes of interest and is processed simultaneously with an under the same conditions as samples through all steps of the analytical procedures, and in which no target analytes or interferences are present at concentrations that impact the analytical results for sample analyses.	To assess background interferences or contamination in the analytical system that might lead to high bias or false positive data. Results of method blanks provide an estimate of the within-batch variability of the blank response and an indication of bias introduced by the preparation and analytical procedure.	This is one of the QC samples used to measure lab accuracy/bias. The sample could indicate whether contamination is occurring during sample prep and analysis. If analytes are detected > ½ RL, reanalyze or qualify (B-flag) all results for the specific analytes(s) in all samples in the associated prep batch as appropriate. For common lab contaminants, no analytes detected > RL. See Section D.1.1.1 and Box D-1
RT window position establishment for each analyte (and surrogate) (all chromatographic methods only)	Determination of the placement of the RT window (i.e. start/stop time) of each analyte or group of analytes as it elutes through the chromatographic column so that analyte identification can be made during sample analysis. This is done during the ICAL.	To idendify analytes of interest	Incorrect window position may result in false negatives, require additional manual integrations, or cause unnecessary reanalysis of samples when surrogates or spiked compounds are erroneously not identified.

Table F-1 Cont.			
QC Check	**Definition**	**Purpose**	**Evaluation**
Second source calibration verification (ICV)	A standard obtained or prepared from a source independent of the source of standards for the ICAL. Its concentration should be at or near the middle of the calibration range. It is done after the ICAL.	To verify the accuracy of the ICAL.	The concentration of the 2nd source calibration verification, determined from the analysis, is compared with the known value of the standard to determine the accuracy of the ICAL. This independent verification of the ICAL must be acceptable before sample analysis can begin.
Surrogate spike (organic analysis only)	A substance with properties that mimic the analyte of interest. It is unlikely to be found in environmental samples and is added to them for quality control purposes.	To assess the ability of the method to successfully recover specific non-target analytes from an actual matrix. Because surrogates are generally added to each sample in a batch, they can be used to monitor recovery on a sample-specific, rather than batch-specific basis.	Whereas the MS is normally done on a batch-specific basis, the surrogate spike is done on a sample-specific basis. Taken with the information derived from other spikes (LCS; MS), the bias in the analytical system can be determined.
Tuning (MS methods only)	The analysis of a standard compound to verify that the mass spectrometer meets standard mass spectra abundance criteria prior to sample analysis. (COE)	To verify the proper working of the mass spectrometer	Proper tuning of the mass spectrometer must be verified prior to sample analysis

Notes:
1. Project-specific requirements identified by the client supersede any requirements listed. The requirements are meant to be default, to be used when project-specific direction based on DQOs is not available.
2. If there is a contradiction between the method and the DoD tables, the requirements specified in the tables shall be followed.

DoD QSM Version 4.2: Appendix F - Quality Control Requirements Summary
Table F-4: Organic Analysis by GC/MS - Methods 8260 and 8270

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Demonstrate acceptable analytical capability	Prior to using any test method and at any time there is a significant change in instrument type, personnel, test method, or sample matrix.	QC acceptance criteria published by DoD, if available; otherwise method- specific criteria.	Recalculate results; locate and fix problem, then rerun demonstration for those analytes that did not meet criteria (see Section C.1.f).	NA	This is a demonstration of analytical ability to generate acceptable precision and bias per the procedure in Appendix C. No analysis shall be allowed by analyst until successful demonstration of capability is complete.
LOD Determination and verification (See Box D-13)					
LOQ Establishment and verification (See Box D-14)					
Tuning	Prior to ICAL and at the beginning of each 12- hour period.	Refer to method specific ion criteria	Retune instrument and verify. Rerun affected samples	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be accepted without a valid tune.
Breakdown check DDT (8270 only)	At the beginning of each 12-hour period, prior to analysis of samples	Degradation ≤ 20% for DDT Benzidine & PCP should be present at their normal responses and should not exceed a tailing factor of 2.	Correct problem then repeat breakdown check.	Flagging criteria are not appropriate.	No samples shall be run until degradation ≤ 20%.
Minimum five-point initial calibration (ICAL) for all analytes	ICAL prior to sample analysis.	1. Average response factor (RF) for SPCCs: VOCs ≥ 0.30 for Chlorobenzene and 1,1,2,2-tetrachloroethane, ≥ 0.1 for chloromethane, bromoform, and 1,1-dichloroethane SVOCs ≥ 0.050	Correct problem then repeat initial calibration	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until ICAL has passed. Calibration may not be forced through the origin.

Table F-4 Cont.					
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action**	**Flagging Criteria**	**Comments**
Minimum five-point initial calibration (ICAL) Cont.		2. RSD for RFs for CCCs: VOCs and SVOCs ≤ 30% and one option below. **Option 1:** RSD for each analyte ≤ 15% **Option 2:** linear least squares regression: $r \geq 0.995$ **Option 3:** non-linear regression-coefficient of determination (COD) $r^2 \geq 0.99$ (6 points shall be used for 2nd order, 7 points shall be used for 3rd order)			
2nd Source calibration verification (ICV)	Once after each ICAL	All project analytes within ± 20% of true value	Correct problem and verify 2nd source standard. Rerun, 2nd source verification. If that fails, correct problem and repeat ICAL.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until calibration has been verified.
RT window position establishement for each analyte and surrogate	Once per ICAL	Position shall be set using midpoint standard of the ICAL curve when ICAL is performed. On days when ICAL is not performed, the initial CCV is used.	NA	NA	
Evaluation of relative retention times (RRT)	With each sample	RRT of each target analyte within ± 0.06 RRT units.	Correct problem, then rerun ICAL.	Flagging criteria are not appropriate.	Labs may update the RTs based on the CCV to account for minor performance fluctuations or after routine system maintenance (such as column clipping). With each sample, the RRT shall be compared with the most recently updated RRT. If the RRT has changed by more than ± 0.06 RRT units since the last update, this indicates a significant change in system performance and the lab must take appropriate corrective actions as required by the method and rerun the ICAL to reestablish the RTs.

Table F-4 Cont.					
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action**	**Flagging Criteria**	**Comments**
Continuing calibration verification (CCV)	Daily before sample analysis and every 12 hours of analysis time.	1. Average RF for SPCCs: VOCs ≥ 0.30 for Chlorobenzene and 1,1,2,2-tetrachloroethane, ≥ 0.1 for chloromethane, bromoform, and 1,1-dichloroethane SVOCs ≥ 0.050 2. %Difference/Drift for all target compounds and surrogates: VOCs and SVOCs ≤ 20% D (Note: D = difference when using RFs or drift when using least squares regression or non-linear calibration).	DoD project level approval must be obtained for each of the failed analytes or corrective action must be taken. Correct problem, then rerun calibration verification. If that failes, then repeat ICAL. Reanalyze all samples since last acceptable CCV.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to all results for the specific analyte(s) in all samples since the last acceptable CCV.	Problem must be corrected. Results may not be reported without a valid CCV. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Internal Standards verification	Every field sample, standard, and QC sample.	RT ± 30 seconds from RT of the midpoint standard in the ICAL EICP area within -50% to +100% of ICAL midpoint standard.	Inspect mass spectrometer and GC for malfunctions. Reanalysis of samples analyzed while system was malfunctioning is mandatory.	If corrective action fails in field samples, apply Q-flag to analytes associated with the non-compliant IS. Flagging criteria are not appropriate for failed standards.	Sample results are not acceptable without a valid IS verification.
Method Blank (MB)	One per prep batch	No analytes detected > ½ RL and > 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater). Blank result must not otherwise affect sample results. For common lab contaminants, no analytes detected > RL (see Box D-1).	Correct problem, then see criteria in box D-1; if required, reprep and reanalyze MB and all samples processed with the contaminated blank.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply B-flag to all results for the specific analyte(s) in all samples in the associated prep batch.	Problem must be corrected. Results may not be reported without a valid MB. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
LCS containing all analytes to be reported, including surrogates	One per prep batch	QC acceptance criteria specified by DoD, if available. Otherwise, use in-house control limits. In-house control limits may not be greater than ± 3 times the standard deviation of the mean LCS recovery. See Box D-3 and Appendix G.	Correct problem, then reprep and reanalyze the LCS and all samples in the associated prep batch for failed analytes, if sufficient sample material is available (see full explanation in Appendix G).	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to specific analyte(s) in all samples in the associated prep batch.	Problem must be corrected. Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed.

Table F-4 Cont.					
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action**	**Flagging Criteria**	**Comments**
Matrix Spike (MS)	One per prep batch per matrix (see Box D-7).	For matrix evaluation, use LCS acceptance criteria specified by DoD, if available. Otherwise, use in-house LCS control limits.	Examine the project-specific DQOs. Contact the client as to additional measures to be taken.	For specific analytes(s) in the parent sample, apply J-flag if acceptable criteria are not met.	For matrix evaluation only. If MS results are outside the LCS limits, the data shall be evaluated to determine the source of difference and to determine if there is a matrix effect or analytical error.
Matrix spike duplicate MSD or Sample Duplicate	One per prep batch per matrix (see Box D-7).	MSD: For matrix evaluation, use LCS acceptance criteria specified by DoD, if available. Otherwise, use in-house LCS control limits. MSD or sample duplicate: RPD $\leq$ 30% (between MS and MSD or sample and sample duplicate).	Examine the project-specific DQOs. Contact client as to additional action measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met.	The data shall be evaluated to determine the source of difference.
Surrogate spike	All field and QC samples	QC acceptance criteria specified by DoD, if available. Otherwise use in-house control limits.	For QC and field samples, correct problem then reprep and reanalyze all failed samples for the failed surrogates in the associated prep batch, if sufficient sample material is available. If obvious chromatographic interferences with surrogate is present, reanalysis may not be necessary.	Apply Q-flag to all associated analytes if acceptance criteria are not met.	Alternative surrogates are recommended when there is obvious chromatographic interferences.
Results reported between DL and LOQ	NA	NA	NA	Apply J-flag to all results between DL and LOQ.	

Notes:
1. Project-specific requirements identified by the client supersede any requirements listed. The requirements are meant to be default, to be used when project-specific direction based on DQOs is not available.
2. If there is a contradiction between the method and the DoD tables, the requirements specified in the tables shall be followed.

TITLE: Metals Analysis Mercury by EPA Methods 245.1/245.5; SW-846 7470A/ 7471A/ 7471B; and U.S. EPA CLP Doc No. ILM04.0

Approvals (Signature/Date):

Debbie Johnson 10-4-10
Debbie Johnson Date
Supervisor, Metals

Diane L. Harper 10-4-10
Diane L. Harper Date
Inorganics Manager

Terese A. Preston 10/5/10
Terese A. Preston Date
Quality Assurance Manager

10-4-10
John D. Nagel Date
Env. Health & Safety Coor.

Michael J. Healy 10/5/10
Michael J. Healy Date
Laboratory Director

Facility Distribution No: _____ **Distributed To:** Uncontrolled

1.0 SCOPE / APPLICATION

This Standard Operating Procedure (SOP) outlines the digestion and analytical procedure for the determination of the mercury concentration in aqueous and non-aqueous media. This SOP was written using EPA 600/4-79-020 Methods 245.1 and 245.5; SW-846, 3rd Edition, Methods 7470A/7471A, and Update IV, 7471B; and U.S. EPA CLP Document No. ILM04.0 as references.

On occasion, clients request slight modifications to this SOP. These modifications are addressed on a case-by-case basis with the range of accuracy (i.e., MDLs, linearity check or PT sample) verified prior to implementation. Any modifications would be written into a Quality Assurance Plan (QAP), authorized via laboratory signature approval, and mentioned in the data package's case narrative.

Specific requirements pertaining to the DoD QSM Version 4.1 are located in Attachment 3. These requirements are additionally applicable to all NFESC projects. Any deviations from these procedures and/or variances from must be addressed appropriately in accordance with standard operating protocol and pre-approved on a project by project basis.

1.1 Method Sensitivity

1.1.1 Method Detection Limits

The method detection limit (MDL), referred to as the detection limit (DL) in NELAC and DOD QSM documents, is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to Appendix B of 40 CFR 136, "Guidelines Establishing Test Procedures for the Analysis of Pollutants" with additional details are provided in the TestAmerica Corporate SOP, *CA-QS-006, Detection Limits* and the TestAmerica Chicago SOP, *UP-QA-017, Method Detection Limit Studies.* MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; the MDL will be verified on a quarterly basis to meet the requirements of the DoD QSM version 4.1.

1.1.2 Demonstration of Capability

Before the laboratory may institute a new method and begin reporting results, the laboratory shall confirm that it can properly operate the method. A demonstration of capability is performed whenever there is a change in instrument type, method or personnel. An Initial Demonstration of Capability (IDOC) must be thoroughly documented and approved by the Department Manager/Supervisor and QA Manager prior to independently analyzing client samples. All associated documentation must be retained in the QA Department and in the Analyst Training files. For additional details on the demonstration of capability procedures followed, refer to the laboratory SOP, *UP-QA-QAM, Quality Assurance Manual, Sections 20.4.2 and 20.4.3.*

1.1.3 Instrument Detection Limits

Instrument Detection Limits (IDLs) are performed quarterly for each element by the metals laboratory for each instrument as specified in CLP. These limits are used to gauge instrument sensitivity and when routinely evaluated, instrument performance without the introduction of method variance can be determined. For DoD QSM, the IDL must be ≤ the Limit of Detection (LOD), which = ½ RL. Refer to IDL SOP, *UP-QA-010* for additional details.

1.1.4 Reporting Limits

Reporting Limits are defined as the lowest concentration of an analyte determined by a given method in a given matrix that the laboratory feels can be reported with acceptable quantitative error or client requirements, values specified by the EPA methods or other project and client requirements. The laboratory maintains reporting limits that are higher than the MDL. Wherever possible, reporting is limited to values approximately 3-5x the respective MDL to ensure confidence in the value reported. Client specific requests for reporting to the IDL or MDL are special circumstances not to be confused with the previous statement.

Matrix	Reporting Limit [1,3]	CRDL [2]
Water	0.2 ug/L	0.2 ug/L
Soil	0.017 mg/kg	0.1 mg/kg

[1] Reporting Limit is used for EPA Method 245.1 and SW-846 7470A/7471A. Reporting Limits may vary depending on sample volume/size, dilution factors, and changes in the MDL.
[2] CRDL (Contract Required Detection Limit) is used for U.S. EPA CLP ILM04.0.
[3] The DoD QSM, version 4.1 and NELAC use the term Limit of Quantitation (LOQ) for the RL.

1.1.5 Definitions

Refer to Section 3.0 of the Laboratory's Quality Assurance Manual (UP-QA-QAM).

1.2 Summary of Method

This flameless cold vapor AA procedure is a physical method based on the absorption of radiation at 253.7 nm by mercury vapor. The mercury is reduced to the elemental state and swept from solution and passed through a cell of a double beam AA. Absorbance is a function of mercury concentration.

2.0 INTERFERENCES

- Chloride may be converted to free chlorine during the oxidation step, and since free chlorine absorbs at 253.7 nm, it must be removed before the Hg vapors are swept into the cell. Samples high in chloride, such as brines or certain effluents, may require excess hydroxylamine reagent, up to 25 ml for water bath preparations (6.25 mL for hot block preparations) and gentle agitation to prevent chlorine interference. Sulfide and certain volatile organic materials may also interfere with the oxidation step.
- Certain volatile organics may interfere by absorbing at the Hg wavelength, but TestAmerica Chicago does not routinely pre-treat samples for organics.

3.0 SAFETY

Employees must abide by the policies and procedures in the Corporate Safety Manual, Radiation Safety Manual, Lab Specific Addendum to the CSM, and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coat and closed-toe, nonabsorbent shoes are a minimum.

3.1 Specific Safety Concerns or Requirements

- Samples that contain high concentrations of carbonates or organic material or samples that are at elevated pH can react violently when acids are added.
- To avoid mercury inhalation, the vapors are passed through a charcoal filter and vented.

3.2 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **NOTE: This list does not include all materials used in the method.** The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table. A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Mercury (1,000 ppm in Reagent)	Oxidizer Corrosive Poison	0.1 Mg/M3 Ceiling (Mercury Compounds)	Extremely toxic. Causes irritation to the respiratory tract. Causes irritation. Symptoms include redness and pain. May cause burns. May cause sensitization. Can be absorbed through the skin with symptoms to parallel ingestion. May affect the central nervous system. Causes irritation and burns to eyes. Symptoms include redness, pain, and blurred vision; may cause serious and permanent eye damage.
Sulfuric Acid	Corrosive Oxidizer Dehydrator Poison	1 Mg/M3-TWA	Inhalation produces damaging effects on the mucous membranes and upper respiratory tract. Symptoms may include irritation of the nose and throat, and labored breathing. Symptoms of redness, pain, and severe burn can occur. Contact can cause blurred vision, redness, pain and severe tissue burns. Can cause blindness.
Nitric Acid	Corrosive Oxidizer Poison	2 ppm-TWA 4 ppm-STEL	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Hydrochloric Acid	Corrosive Poison	5 ppm-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Potassium Permanganate	Oxidizer	5 Mg/M3 for Mn Cmpds.	Causes irritation to the respiratory tract. Symptoms may include coughing, shortness of breath. Dry crystals and concentrated solutions are caustic causing redness, pain, severe burns, brown stains in the contact area and possible hardening of outer skin layer. Diluted solutions are only mildly irritating to the skin. Eye contact with crystals (dusts) and concentrated solutions causes severe irritation, redness, and blurred vision and can cause severe damage, possibly permanent.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Potassium Persulfate	Oxidizer	None	Causes irritation to the respiratory tract. Symptoms may include coughing, shortness of breath. Causes irritation to skin and eyes. Symptoms include redness, itching, and pain. May cause dermatitis, burns, and moderate skin necrosis.
1 – Always add acid to water to prevent violent reactions. 2 – Exposure limit refers to the OSHA regulatory exposure limit.			

4.0 EQUIPMENT AND SUPPLIES

All laboratory glassware used in this test must be acid rinsed, followed by deionized water rinsing.

- 1 Leeman Lab HydraAA Automated Mercury Analyzer, with built in autosampler, running with WinHg Version 1.7 software
- 1 Teledyne Leeman Hydra II_{AA} automated Mercury Analyzer, with built in autosampler, running with Envoy software
- Various printers.
- Class A volumetric glassware
- Eppendorf pipettes

5.0 REAGENTS AND STANDARDS

5.1 Reagents

All reagents and chemicals used in this test must be reagent grade or better.

5.1.1 Miscellaneous Reagents

- Hydrochloric Acid [HCl], Concentrated
- Nitric Acid [HNO_3], Concentrated
- Sulfuric Acid [H_2SO_4], Concentrated
- Anhydrone desiccant for in-line moisture trap
- Deionized (DI) Water, Type II

5.1.2 Sodium Chloride-Hydroxylamine Hydrochloride Solution

Dissolve 240 g of sodium chloride and 240 g of hydroxylamine hydrochloride in sufficient DI water to make 2-liters of solution.

- Life of Reagent: 1 Year
- Storage Requirements: None

5.1.3 Stannous Chloride Solution

Dissolve 100 g of stannous chloride in 10% hydrochloric acid to make 1-liter of solution.

- Life of Reagent: 1 Month
- Storage Requirements: None

5.1.4 Potassium Permanganate, 5%

Dissolve 175 g of potassium permanganate into 3.5-liters of DI water.

- Life of Reagent: 1 Year
- Storage Requirements: None

5.1.5 Potassium Persulfate, 5%

Dissolve 175 g of potassium persulfate into 3,500 mLs of DI water.

- Life of Reagent: 1 Year
- Storage Requirements: None

5.2 Standards

All standards are prepared in Class A volumetric flasks.

5.2.1 Standard Stock Solution I; 1,000 ppm

A 1,000 ppm concentrated mercury standard is purchased from an outside supplier.

- Life of Standard: 1 Year
- Storage Requirements: None

5.2.2 Working Standard Solution I; 100 ppb

To a 1.0 L volumetric flask filled with ~800 mLs DI water, transfer 100 uLs of Stock Solution I to the flask using a 100 uL Eppendorf pipette. Add 2.5 mLs conc. nitric acid as a preservative. Dilute to volume with DI Water. Invert and mix to insure complete mixture.

*For use in spiking Matrix Spikes, CRAs & the Standard Curve.

- Life of Standard: 24 Hours
- Storage Requirements: None

5.2.2.1 Working Standard Solution IA; 25 ppb

To a 100 mL volumetric flask filled with ~80 mLs DI water, transfer 25 mLs of Working Standard Solution I (Item 5.2.2) to the flask using a graduated cylinder. Dilute to volume with DI Water. Invert and mix to insure complete mixture.

*For use in spiking Matrix Spikes, CRAs & the Standard Curve.

- Life of Standard: 24 Hours
- Storage Requirements: None

5.2.3 Standard Stock Solution II; 1,000 ppm

Purchased from an outside supplier as a 1,000 ppm solution and is from an alternate source than that of Standard Stock Solution I (Rgt. 5.2.1).

- Life of Standard: 1 Year
- Storage Requirements: None

5.2.4 Working Standard Solution II; 200 ppb

To a 1.0 L volumetric flask filled with ~800 mLs DI water, add 2.5 mLs concentrated nitric acid (as a preservative) and 200 uLs of Standard Stock Solution II to the flask (using a 200 uL Eppendorf pipette). Dilute to volume with DI water and invert several times to mix.

*For use in spiking the ICV/CCV and LCS.

- Life of Standard: 24 Hours
- Storage Requirements: None

5.2.4.1 Working Standard Solution IIA; 50 ppb

To a 100 mL volumetric flask filled with ~80 mLs DI water, add 25 mLs of Working Standard Solution II (Item 5.2.4) to the flask using a graduated cylinder. Dilute to volume with DI Water. Invert and mix to insure complete mixture.

*For use in spiking the ICV/CCV and LCS.

- Life of Standard: 24 Hours
- Storage Requirements: None

5.2.5 Working Standards for Mercury in Water

Standard (ug/L)	mLs of Working Solution IA 5.2.2.1	Final Volume (mLs) Hot Block
Blank	0.0	25
0.2	0.2	25
0.5	0.5	25
1.0	1.0	25
3.0	3.0	25
5.0	5.0	25
CRA (0.2 ug/L)	0.2	25
Matrix Spike (1.0 ug/L)	1.0	25

Standard (ug/L)	mLs of Working Solution IIA 5.2.4.1	Final Volume (mLs) Hot Block
Init. Cal. Verif. (ICV) (2.0 ug/L)	1.0	25
Cont. Cal. Verif. (CCV) (1.0 ug/L)	0.5	25
Lab Control Sample (LCS) (2.0 ug/L)	1.0	25

CLP Standard (ug/L)	mLs of Working Solution IIA 5.2.4.1	Final Volume (mLs) Hot Block
Init. Cal. Verif (ICV) (2.0 ug/L)	1.0	25
Cont. Cal. Verif. (CCV) (1.0 ug/L)	0.5	25

NOTE: ILM04.0 and NELAC require the ICV and CCV to be at different levels.

5.2.6 Working Standards for Mercury in Soil

Standard (ug/L)	mLs of Working Soln. I 5.2.2	Final Volume (mLs) Hot Block
Blank	0.00	50
0.2	0.10	50
0.5	0.25	50
1.0	0.50	50
3.0	1.50	50
5.0	2.50	50
CRA (0.2 ug/L)	0.10	50
Matrix Spike (1.0 ug/L)	0.50	50

Standard (ug/L)	mLs of Working Soln. II 5.2.4	Final Volume (mLs) Hot Block
Init. Cal. Verif. (ICV) (2.0 ug/L)	0.50	50
Cont. Cal. Verif. (CCV) (1.0 ug/L)	0.25	50
Lab Control Sample (LCS) (2.0 ug/L)	0.50	50

CLP Standard (ug/L)	mLs of Working Soln. II 5.2.4	Final Volume (mLs) Hot Block
Init. Cal. Verif (ICV) (2.0 ug/L)	0.50	50
Cont. Cal. Verif. (CCV) (1.0 ug/L)	0.25	50

NOTE: ILM04.0 and NELAC require the ICV and CCV to be at different levels.

6.0 CALIBRATION (NON-DAILY)

All calibration procedures are performed on a daily basis. Refer to Section 7.4 for details.

7.0 PROCEDURE

7.1 Quality Control Checks

The following Quality Control samples are performed with each batch of samples. Refer to Section 8.0 for additional details.

QC Sample	Frequency [1]	Control Limits
Method Blank (MB)	1 in 20 samples	• < Reporting Limit (EPA 245.1; 245.5 / SW-846) • < ½ RL, or < LOD (DOD QSM)[3] • < CRDL (CLP)
LCS	1 in 20 samples	• 80-120% Recovery (EPA 245.5 / SW-846 / CLP) • 85-115% Recovery (EPA 245.1)
Matrix Duplicate (DU)[2]	1 in 20 samples	• 20 RPD unless the sample conc. is <5x RL, then ± RL. (EPA 245.1; 245.5 / SW-846) • 20 RPD unless the sample conc. is <5x CRDL, then ± CRDL. (CLP)
Matrix Spike (MS) MS Duplicate (MSD)[2]	1 in 20 samples	• 75 – 125% Recovery unless the sample concentration > spike level by 4x (EPA 245.5 / SW-846 / CLP) • 70 – 130% Recovery (EPA 245.1) • 80 - 120% (DoD QSM) • > 50% Recovery; if <50% Recovery, Method of Standard Additions (MSA) is required (TCLP)

[1] Drinking waters by EPA 245.1; and CLP analyses are analyzed at a frequency of 1 in 10 samples.
[2] The sample selection for MS/MSD or MS/MD, where appropriate, is rotated among client samples so that various matrix problems may be noted and/or addressed. MD's are performed only when requested by the client/project/contract. The MS/MSD are the routinely performed matrix QC indicators.
[3] For DoD QSM v.4.1, ½ the RL = Limit of Detection (LOD)

7.2 Sample Preservation and Storage

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client request. Listed below are the holding times and preservations for the referenced programs.

Program	Preservation [1]	Holding Time [2]
SDWA	pH < 2, Cool 4 ± 2°C	28 days VTS [3]
CWA	pH < 2, Cool 4 ± 2°C	28 days VTS
RCRA	pH < 2, Cool 4 ± 2°C	28 days VTS
CLP	pH < 2, Cool 4 ± 2°C	26 days VTSR [4]

[1] Waters are preserved with nitric acid at pH <2; Soils are preserved at Cool 4 ± 2°C.
[2] Holding times include digestion and analysis.
[3] VTS: Verified Time of Sampling.
[4] VTSR: Verified Time of Sample Receipt.

7.3 Sample Preparation

7.3.1 Mercury Water Digestion Procedure - EPA Method 245.1 / CLP ILM04.0

Item	Hot Block
Sample Volume	25 mLs
Reaction Vessel	Sample Vials, 50 mLs
Sulfuric Acid (conc.)	1.25 mLs
Nitric Acid (conc.)	0.625 mLs
Potassium Permanganate, 5% Sol. (W/V)	3.75 mLs
Potassium Persulfate, 5% Sol. (W/V)	2 mLs
Preparation	2 hrs. @ 90 - 95°C, Cool
Hydroxylamine Addition	1.5 mLs
Total Volume	34.125 mLs

NOTE: The sample should remain purple for 15 minutes after adding the potassium permanganate. If the sample does not maintain the purple color, a second addition of potassium permanganate is added to all samples of the batch to maintain the purple color.

After the hydroxylamine addition, place the sample digestates on the instrument for analysis, where the reducing agent, stannous chloride, is added by the analyzer from a reservoir to form elemental Hg.

7.3.2 Mercury Water Digestion Procedure - SW-846 Method 7470A

Item	Hot Block
Sample Volume	25 mLs
Reaction Vessel	Sample Vials, 50 mLs
Sulfuric Acid (conc.)	1.25 mLs
Nitric Acid (conc.)	0.625 mLs
Potassium Permanganate, 5% Sol. (W/V)	3.75 mLs
Potassium Persulfate, 5% Sol. (W/V)	2 mLs
Preparation	2 hrs. @ 90 - 95°C, Cool
Hydroxylamine Addition	1.5 mLs
Total Volume	34.125 mLs

NOTE: The sample should remain purple for 15 minutes after adding the potassium permanganate. If the sample does not maintain the purple color, a second addition of potassium permanganate is added to all samples of the batch to maintain the purple color.

After the hydroxylamine addition, place the sample digestates on the instrument for analysis, where the reducing agent, stannous chloride, is added by the analyzer from a reservoir to form elemental Hg.

7.3.3 Mercury Soil Digestion Procedure - SW-846 Method 7471A/7471B

NOTE: A prep batch must be started prior to weighing the solids aliquots. The balance is interfaced with TALS and the sample weights are transferred directly into the ADII batch, eliminating potential transcription errors. The sample IDs can also be scanned directly into the batch from the barcodes on the containers. Three aliquots of soils (~0.2 g each) are combined and digested as one sample, although this requirement has been withdrawn in 7471B. Soil samples are homogenized prior to the removal of the three aliquots according to SOP UP-QA-039, *Quality Assurance – Sample Homogenization and Subsampling Procedures.*

Item	Hot Block
Sample Weight	~ 0.6 – 0.7 grams
Reaction Vessel	Digestion Vessel
DI Water, Type II	2.5 mLs
Aqua Regia [3:1 HCl (conc.) to HNO_3 conc.)]	2.5 mLs
Preparation	2 min. @ 90-95°C, Cool
DI Water, Type II	25 mLs
Potassium Permanganate, 5% Sol. (W/V)	7.5 mLs
Preparation	30 min. @90-95°C, Cool
Hydroxylamine Addition	3 mLs
Total Volume	50 mLs

NOTE: The sample should remain purple for 15 minutes after adding the potassium permanganate. If the sample does not maintain the purple color, a second addition of potassium permanganate is added to all samples of the batch to maintain the purple color.

After the hydroxylamine addition, place the sample digestates on the instrument for analysis, where the reducing agent, stannous chloride, is added by the analyzer from a reservoir to form elemental Hg.

7.3.4 Mercury Soil Digestion Procedure - EPA Method 245.5 / CLP ILM04.0

NOTE: A prep batch must be started prior to weighing the solids aliquots. The balance is interfaced with TALS and the sample weights are transferred directly into the ADII batch, eliminating potential transcription errors. The sample IDs can also be scanned directly into the batch from the barcodes on the containers.

Item	Hot Block
Sample weight	0.2 - 0.3 grams
Reaction Vessel	Digestion Vessel
Sulfuric Acid (conc.)	5 mLs
Nitric Acid (conc.)	2.5 mLs
Preparation	2 min. @ 90 -95°C, Cool
DI Water, Type II	50 mLs
Potassium Permanganate, 5% Sol. (W/V)	15 mLs
Potassium Persulfate, 5% Sol. (W/V)	8 mLs
Preparation	30 min. @ 90 - 95°, Cool
Hydroxylamine Addition	6 mLs
Total Volume	Dilute to 100 mLs

NOTE: The sample should remain purple for 15 minutes after adding the potassium permanganate. If the sample does not maintain the purple color, a second addition of potassium permanganate is added to all samples of the batch to maintain the purple color.

After the hydroxylamine addition, place the sample digestates on the instrument for analysis, where the reducing agent, stannous chloride, is added by the analyzer from a reservoir to form elemental Hg.

7.4 Calibration / Standardization

Before the instrument is used as a measurement device, the instrument response to known reference materials must be determined, in this case by establishing the working linear curve. All sample measurements must be made within this linear range of the instrument. Periodic known standards and blanks are read throughout the run at the given frequency to verify that the calibration is holding to specified criteria.

Standard	Frequency	Control Limit
Calibration Curve	Initially	Corr. Coeff. $\geq$ 0.995
ICV	After the Calibration Curve	• 90 –110% Recovery (EPA 245.5/SW-846/CLP/DoD) • 95 – 105% Recovery (EPA 245.1)
ICB	After the ICV	• < Reporting Limit (EPA 245.1; 245.5 / SW-846) • < ½ RL, or LOD (DOD QSM) • < CRDL (CLP)
CRA	After ICB	• No established limits.
CCV	Every 10 readings; end of each run	• 90 – 110% Recovery (EPA 245.1; 245.5 / CLP) • 80 – 120% Recovery (SW-846, and DoD QSM)
CCB	Every 10 readings; End of each run	• < Reporting Limit (EPA 245.1; 245.5 / SW-846) • < ½ RL, or LOD (DOD QSM) • < CRDL (CLP)

7.5 Preventive Maintenance

The instruments require some routine daily maintenance as well as some scheduled and non-scheduled periodic maintenance. All maintenance will be recorded in the instruments maintenance logbook. The following schedule lists some of the various maintenance procedures and when they should be performed. Additionally, the scheduled maintenance procedures given in the software should be checked and followed.

Equipment	Schedule
Drying Tube	Replace as needed.
Pump Tubing	Weekly, or as needed.
Lamp	Replace as needed (avg. 4 mos. - 1 yr.).
Optical Cell	Clean as needed (typically monthly).
Liquid Gas Separator	Replace every 1-3 yrs., as needed.
Internal Tubing	Should not require replacement under normal circumstances.

7.6 Sample Analysis

7.6.1 Preparing the System

The following procedures must be performed each morning before warming up the system:

- Release the clamps and check the pump tubing for wear.
- Check the reductant volume and rinse volume. Refresh, if needed.
- If the lamp has been off then turn on the lamp power and allow the lamp to warm up for at least 45 minutes for the HydrAA and 20 minutes for the Hydra II_{AA}.
- Start up the system.

7.6.2 Sample analysis

Refer to the work instructions found in Appendices 1 and 2 for details specific to the daily operation of each instrument.

7.7 Documentation

7.7.1 Raw Data and TALS Documentation

The analysis of samples and standards is documented in TALS ADII, supported by the instrument print-out. The digestion batch notes must include the following information, when applicable:

Filter Lot #
HCl Lot #
HNO_3 Lot #
H_2SO_4 Lot #
H_2O_2 Lot #
Potassium Persulfate Lot #
Potassium Permanganate Lot #
Stannous Chloride Lot #
NaCl Lot #
Hood ID
Hot Block ID
Hot Block temperature
Thermometer ID #
Digestion Tube Lot #
Re-pipet Check
Balance ID #

The instrument print-out must be labeled with the instrument identification, analyst signature, date of analysis, and batch ID. All errors and unused data must be clearly identified with appropriate cross-outs, initials, and dates.

7.7.2 Traceability of Standards

Custom made and single element stock standard solutions which are traceable to NIST or EPA are purchased. On receipt, each standard is recorded in the TALs reagent module and is issued a unique source ID #. The manufacturer, lot #, date received, expiration date, date of verification, concentration, volume received, and the initials of the recording analyst are entered into the system.

The intermediate standards are prepared daily in according to the instructions in section 5.2. The volumetric is labeled with the standard concentration, the preparatory instructional section of the SOP and states "prepared daily from stock". The date and time of preparation must also be recorded on the flask. The bar-coded stock label is affixed to each volumetric containing an intermediate prepared from that stock. Both stock IDs must be included in the analytical or preparatory batches in the TALS Analysts' Desktop module.

7.7.3 Data Review

Analytical data goes through a 200% review cycle. As results are generated, analysts review their work to ensure that the results generated meet QC requirements and relevant EPA methodologies. The analyst transfers the data into TALs in the Analyst Desktop module. All data that include real world samples, even entire runs that are not reported, are transferred to TALS. Where non-compliance is observed, the analyst creates Non-Conformance Memos (NCMs) in TALs. Flags and data qualifiers can be method, project, program or QAPP specific. The analyst documents the initial review on a data review checklist (Attachment 2) and sets the batch status in LIMs to 1st level review. The second level or peer review of the data is conducted by another individual who has been trained on the review process. This secondary review is documented on the same checklist, making any necessary corrections to the data or additions to the NCMs as necessary. The batch is then set to 2nd level review. The raw data, including the checklist, instrument print-outs, and manual entries, and electronic files are retained for easy retrieval in accordance with the laboratory's record and retention policy outlined in the SOP, *UP-QA-QAM, Section 15.*

Examples of items included in the above reviews are as follows:

- QC data are outside the specified control limits for accuracy and precision
- Unusual detection limit changes are observed
- Samples having unusually high results
- Samples exceeding a known regulatory limit
- Raw data indicating some type of contamination or poor technique
- Transcription errors
- Results outside of calibration range

8.0 QUALITY CONTROL

8.1 QC Summary

The laboratory generates annual statistically generated control limits and these can be used when requested by the client, contract or QAPP. These limits are based on the successive analysis of LCSs.

8.1.1 Calibration curve must be composed of a minimum of a blank and 5-standards. A least square fit linear calibration curve must have a minimum correlation coefficient of 0.995, which must be reported with the raw data.

8.1.2 ICV and ICB will be performed at the beginning of an analytical sequence. The ICV must not vary more than - a) 10% for EPA 245.5, SW-846 including for DoD & CLP methods or b) 5% for EPA 245.1 method from its true value and must be prepared from a different source than the calibration curve standards.

Calibration verification will be performed with a CCV and CCB every 10 samples and at the end of the analysis. The CCV must not vary more than a) 20% for SW-846 methods including DoD or b) 10% for EPA 245.1; 245.5 & CLP methods from its true value and must be prepared from a different source than the calibration curve standards. The CCB must be < Reporting Limit (EPA / SW-846); < ½ RL, or LOD (DOD QSM) and < CRDL (CLP).

8.1.3 Dilute samples if they are more concentrated than the highest standard or if they fall on the plateau of a calibration curve (dilute with a digested blank containing all reagents, or repeat the analysis using a smaller sample volume).

8.1.4 A minimum of one MB must be analyzed per sample batch to determine if contamination has occurred

8.1.5 An LCS will be included with each batch of 10 (drinking waters and EPA 245.1) or 20 (EPA 245.5, SW-846 or CLP) samples. The analyzed result must not vary more than 20% from the true value. For EPA Method 245.1, the LCS acceptance limits are 85-115%.

8.1.6 Matrix spike and duplicate samples are analyzed with each batch of 10 (drinking waters and EPA 245.1) or 20 (EPA 245.5, SW-846 or CLP) samples.

8.2 Corrective Actions

When an out-of-control situation occurs, the analysts must use his/her best analytical judgment and available resources to determine the corrective action to be taken. The out-of-control situation may be caused by more than one variable. The analyst should seek the assistance of his/her immediate supervisor, section manager, QA personnel, or other experienced staff if he/she is uncertain of the cause of the out-of-control situation. The test must not be resumed until the source of the problem and an in-control status is attained. All samples associated with the out-of-control situation should be reanalyzed. Out-of-control data must never be released without approval of the supervisor, section manager, project manager, QA personnel or the laboratory manager.

Listed below are steps that must be taken when an out-of-control situation occurs:

- demonstrate that all the problems creating the out-of-control situation were addressed
- document the problem and the action which was taken to correct the problem in a Non-Conformance Memo (NCM) in TALs LIMs.
- document in the NCM that an in-control situation has been achieved and receive approval of the supervisor, section manager, QA personnel, or the laboratory manager prior to the release of any analytical data associated with the problem.

QC Indicator	Suggested Corrective Actions
Calibration Curve	• reanalyze the standard curve; • prepare a new stock and/or working standards; • check the reagents/solutions and prepare fresh if necessary.
Initial Calibration Verification (ICV)	• repeat ICV to verify proper preparation; • prepare new ICV from original stock; • recalibrate with a new standard curve; • prepare new stock and/or working standards; • check reagents/solutions and prepare fresh if necessary.
Initial Calibration Blank (ICB)	• prepare new ICB to verify proper preparation; • verify that the instrument base-line is stable and perform necessary maintenance, cleaning, etc.. to achieve stability; • determine the source of contamination by the process of elimination, carryover from a previous analysis or reagent contamination and correct the problem; • check reagents/solutions and prepare fresh if necessary; • correct for any contamination and reanalyze ICB and any associated samples.
Laboratory Control Sample (LCS)	If LCS is low: • reanalyze LCS to verify that it is out-of-control; • determine the source of error within the preparation procedure, repeat the sample set, write a NCM If the LCS is high: • reanalyze LCS to verify that it is out-of-control; • determine the source of error within the preparation procedure, repeat the sample set; • determine if the high result is due to contamination; • check for contamination of reagents, LCS stock solution, or preparation area; • correct for contamination, reanalyze.

QC Indicator	Suggested Corrective Actions
Method Blank (MB)	• reanalyze the MB to verify that it is beyond the reporting limit; • determine the source of contamination; • determine if the high result is due to contamination; • check for contamination of reagents or preparation area; • correct for contamination, reanalyze set; • in the extreme case where all samples in the set are at least 10X > the MB, reanalysis will not be required. However, a NCM and approval will be necessary.
Matrix Duplicate (DU)	• the sample must be reprocessed and reanalyzed; • if the reanalysis results in data that is still out of the control limit, then the sample will be flagged; • regardless of the outcome of the reanalysis, an NCM will be written and approved by the Supervisor or Section Manager.
Matrix Spike (MS)	• the sample must be reprocessed and reanalyzed; • if the reanalysis results in data that is still out of the control limit, then the sample will be flagged; • regardless of the outcome of the reanalysis, an NCM will be written and approved by the Supervisor or Section Manager.
Continuing Calibration Verification (CCV)	• repeat CCV to verify proper preparation; • prepare new CCV from original stock; • check for instrument base-line drift or a change in one or more of the reagents; • check reagents/solutions and prepare fresh if necessary; • recalibrate with a new standard curve and repeat all samples since the previous in control CCV; • never dispose of any samples until you are sure that all QC, especially the CCV, are within the control limits.
Continuing Calibration Blank (CCB)	• prepare new CCB to verify proper preparation; • verify that the instrument base-line is stable and/or perform necessary maintenance, cleaning, etc.. to achieve stability; • determine the source of contamination by the process of elimination, carryover from a previous analysis or reagent contamination and correct the problem, • check reagents/solutions and prepare fresh if necessary; • correct for any contamination and reanalyze CCB and any associated samples; • never dispose of any samples until you are sure that all QC, especially the CCB are within the control limits.
Summary	• If any of the ICV, ICB, CCV or CCB results are out-of-control for any element, the instrument is restandardized and the samples associated with the out-of-control elements are reanalyzed. • If the MB or LCS are out-of-control for any element, the samples are redigested. An exception is if the sample concentrations are ≥ 10X the MB contamination, the results are reported as is. • If any of the DU or MS results are out-of-control, a reanalysis is performed if there is sufficient sample. If there is insufficient sample, or the reanalysis is still out-of-control, the client is notified of the poor results via an NCM. • NCMs are available for out-of-control MB, LCS, MS and DU problems. These are initiated by the analyst performing the analysis. The NCM is then reviewed by the supervisor or section manager. The NCM is stored electronically within the TALS and used to prepare the case narrative (if applicable).

9.0 DATA ANALYSIS AND CALCULATIONS

Perform a linear regression or quadratic fit analysis of the calibration standard results. Compare sample results to the curve to determine the mercury concentration.

9.1 Water ug/L Hg = ug/L x Dilution Factor

9.2 Soil $\text{mg/kg Hg} = \frac{\text{(ug/L) x L x Dilution Factor}}{\text{wt(g)}}$

(Where L = Final digestate volume)

NOTE: All dry weight corrections are made in ADII at the time the batch is calculated.

9.3 Accuracy $\%R = \frac{(A_T - A_O)}{A_F} \times 100$

Where:
A_T = Total amount recovered in fortified sample
A_O = Amount recovered in unfortified sample
A_F = Amount added to sample

9.4 Precision $RPD = \frac{|C_1 - C_2|}{(C_1+C_2)/2} \times 100$

Where:
C_1 = First measurement value
C_2 = Second measurement value

10.0 POLLUTION CONTROL

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

10.1 Waste Management

Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to UP-WM-001.

- Waste from this process goes into the "Corrosive Wastewater" wastestream.
- Single component standards should not be mixed into the waste streams unless approved by the Waste Coordinator. All standards with Hazardous constituents will be turned in to the waste technician for disposal.

11.0 METHOD PERFORMANCE CRITERIA

Refer to Sections 1, 6, 7 and 8.

12.0 REFERENCES

Refer to Section 1.0.

13.0 ATTACHMENTS

Attachment 1: Example: Instrument Maintenance Log
Attachment 2: Example: Data Review Checklist
Attachment 3: DoD QSM Version 4.1: Appendix F QC Requirements Summary (Table F-1 and Table F-7)

Appendix 1: Daily Operation of the HydraAA (HG5)
Appendix 2: Daily Operation of the Hydra II_{AA} (HG6)

14.0 REVISION HISTORY

- Revision 16 updated on 10/01/10
- Annual Review
- Removal of out-dated instrument-specific instructions from Section 7 and addition of work instructions in Appendices 1 and 2.
- Incorporation of SOP Change Forms.

Attachment 1:

Example: Instrument Maintenance Log (020-001)

TestAmerica Chicago
Hydra AA II - Leeman Mercury Analyzer – HG6
Instrument Maintenance Log

Page No._____

	Date/Initials	Date/Initials	Date/Initials	Date/Initials	Date/Initials	Date/Initials	Date/Initials
Daily Maintenance:							
Check / Clean and Refill Rinse Bottle							
Check / Clean Sample Tip							
Check/Clean Optical Cell (Clean when reference intensity is <300,000)							

Weekly Maintenance:							
Change Pump Tubing							
Check scheduled maintenance in computer software							

Comments:_____

_____

_____

Return to Control:

_____

_____

Any Maintenance/Repair/Part Replacement performed that is not listed above must be documented in the Comments sections

Reviewer Signature:_____ Date:_____

(020-001)

Attachment 2.

Example: Data Review Checklist (021-001 to 021-002)

TestAmerica Chicago
Mercury Data Review Checklist: Automated Cold Vapor

Instrument ID: HG5 HG6 **Analytical Batch:**_____

Preparation Batch:_____

_____

Copies: _____ _____

_____ _____

_____ _____

_____ _____

****

Method (circle): a. **EPA 245.1** b. **EPA 7470A** c. **EPA 7471A** d. **CLP ILM04.0**

Matrix (circle): a. Drinking Water b. Non-Potable Water c. Soil/Sediment/Waste d. Other_____

Circle: a. Total b. Solubles **QC Type (circle):** a. CLP b. Standard c. DoD QSM / AFCEE

I. Calibration:

NCM #	Analyst	Reviewer	
	☐	☐	1. Calibration is clearly documented. a. c.c.: 0.995 to 1.000 b. y-intercept: **Std. QC:** < RL; **CLP QC:** < CRDL
	☐	☐	2. Calibration Verification **CLP QC and Drinking waters:** Every 10 Sample Readings **Std. QC and TCLP QC:** Every 20 Sample Readings a. ICV/CCV **SW-846:** ± 10% (ICV); ± 20% (CCV); (Note: CCV Default of +/-10% in TALS **CLP:** ± 10% (ICV); ± 10% (CCV) **EPA 245.1:** ± 5% (ICV); ± 10% (CCV)
	☐	☐	b. ICB/CCB **Std. QC**: < RL; **CLP QC**: < CRDL **AFCEE/DoD**: <1/2 RL
	☐	☐	3. CRA **CLP QC**: At CRDL; Analyzed each Calibration; No Limit; **Std. QC**: At CRDL; Analyzed Daily; ± RL

II. Sample Analysis:

NCM #	Analyst	Reviewer	
	☐	☐	Each Preparation Batch: a. Must be clearly identified
	☐	☐	b. Contains a maximum of 20 samples
	☐	☐	c. 1 Prep Blank: **CLP:** < CRDL; **Std. QC:** < RL **AFCEE/DoD**: <1/2 RL
	☐	☐	d. 1 LCS: **SW 846/CLP:** 80-120% Rec. **EPA 245.1:** 85-115% Rec.
	☐	☐	e. 1 Matrix Spike: **Std. / CLP:** 75-125% Rec.; ***Unless*** the sample conc. >4X the spike conc. **TCLP:** > 50% Rec.; If <50%, MSA analysis is required **245.1:** 70-130% Rec.
	☐	☐	f. 1 Matrix Spike Duplicate: **Std. / SW 846:** 75-125% Rec.; ***Unless*** the sample conc. >4X the spike conc.; RPD/RSD limits are 20%
	☐	☐	g. 1 Matrix Duplicate: **Std.QC:** RPD/RSD limits are 20% ***Unless*** the sample conc. is <5x RL then ± RL **CLP:** RPD or RSD limits are 20%; ***Unless*** the sample conc. <5x CRDL then ± CRDL applies.

Mercury Data Review Checklist: Automated Cold Vapor

I. Data Documentation

Analyst | Reviewer

1. The instrument ID must be clearly documented.
2. If any curve data, or initial or continuing calibration data are outside of the control limits, **an NCM must be written** and the Manager or Supervisor must be notified that redigestion is required.
3. Matrix Spike/Matrix Spike Duplicate outside the control limits:
   a. **CLP QC**: Single spike only. No corrective action required, the sample is flagged appropriately.
   b. **Std. QC**: **An NCM must be written** and the Manager or Supervisor must make the decision as to whether re-digestion is required if no matrix spike duplicate was done.
   c. If MSA is performed; check the calculation.
4. Sample Duplicate outside the control limits:
   a. **CLP QC**: Normally no corrective action required; the result is flagged appropriately.
   b. **Std. QC**: **An NCM must be written** and the Manager or Supervisor must make the decision as to whether redigestion is required.
5. All unused data is clearly identified.
6. Standard Traceability is correctly documented.
7. Data Report accurately reflects the documentation in the Databook and LIMS.
8. The analyst's full signature is required on the following:
   a. Instrument Raw Data Report
   b. Data Review Checklist
9. Jobs needing copies are clearly marked
10. Proper Corrective Action Documentation for any out of control situation is clearly identified.

IV. TALS

Analyst | Reviewer

1. Samples Tab:
   a. LIMs Sample IDs/Containers are correct.
   b. Method and Matrix are correct.
   c. Date and Time match raw data.
   d. Dilutions are correct.
   e. Correct suffix designated (where applicable).
2. Worksheet Tab is complete and correct.
3. Reagent Tab is complete and correct.
4. QC Links Tab is correct.
5. Sample Results Tab:
   a. All unused data are designated Rejected or Acceptable.
   b. All reported analytes are designated Primary or Secondary.

1st | 2nd

6. Status set appropriate to review level.

Comments:_____

_____

_____

_____

Analyst Signature: _____ Date: _____.

Reviewer Signature:_____ Date: _____.

Attachment 3.

DoD QSM Version 4.1: Appendix F QC Requirements Summary
(Table F-1 and Table F-7)
(022-001 to 022-005)

TestAmerica Chicago

DoD QSM Version 4.1: Appendix F - Quality Control Requirements Summary

Table F-1 Summary of QC Check Definitions, Purpose, and Evaluation – Inorganics (Metals)

QC Check	Definition	Purpose	Evaluation
Calibration Blank	Reagent water containing no analytes of interest.	To determine the zero point of the calibration curve for all initial and continuing calibrations.	This is a required QC procedure. Continuing calibration blank responses above the LOD require corrective action
Continuing calibration verification (CCV)	This verification of the ICAL that is required during the course of analysis at periodic intervals. Continuing calibration verification applies to both external standard and internal standard calibration techniques, as well as to linear and non-linear calibration models	To verify that instrument response is reliable, and has not changed significantly from the current ICAL curve.	If the values for the analytes are outside the acceptance criteria, the ICAL may not be stable. Results associated with out-of-control CCV results require reanalysis or flagging
Demonstrate Acceptable Analytical Capability	QC samples are analyzed in series to verify ability to produce data of acceptable precision and bias.	To verify the ability to produce data of acceptable precision and bias for a specific instrument type, matrix, method, and analyst.	The average recovery of the spikes and standard deviation of the replicates must be within designated acceptance criteria. Analysis of field samples may not be conducted until this check is successful.
Dilution test (Metals only)	Analysis of a positive sample, which has been diluted to a concentration 1/5th of the original, to confirm that there is no interference in the original sample analysis. (Modified COE)	To assess matrix interference	Agreement within 10% between the concentration for the undiluted sample and 5x the concentration for the diluted sample indicates the absence of interferences, and such samples may be analyzed without using the MSA. Results outside acceptance limits indicate a possible matrix effect. For ICP a post-digestion spike must be run.
Duplicate Sample (replicate)	Two identical portions of material collected for chemical analysis, and identified by unique alphanumeric codes. The duplicate may be portioned from the same sample, or may be two identical samples taken from the same site. The two portions are prepared and analyzed identically. (modified QSM)	To provide information on the heterogeneity of the sample matrix or to determine the precision of the intralaboratory analytical process for a specific sample matrix	A duplicate sample will provide information on the heterogeneity of the sample matrix. The greater the heterogeneity of the matrix, the greater the relative percent difference between the sample and the sample duplicate. If the sample matrix is homogeneous (such as with drinking water) and the relative percent difference is high, this could indicate a problem in the analytical system.
Initial calibration for all analytes (ICAL)	Analysis of analytical standards at different concentrations that are used to determine and calibrate the quantitation range of the response of the analytical detector or method	To establish a calibration curve for the quantification of the analytes of interest	Statistical procedures are used to determine the relationship between the signal response and the known concentration of analytes of interest. The ICAL must be successful before any samples or other QC check samples can be analyzed.
Instrument Detection Limit (IDL) Study (6010 and 6020 only)	The process to determine the minimum concentration of a substance (analyte) that an instrument can differentiate from noise. The procedure for calculating varies by method.	To provide evaluation of instrument sensitivity	IDLs must be established before samples can be analyzed.
Interference check solutions (ICP and ICP/MS only)	A pair of solutions containing interfering elements that are used to verify the correction factors of analytes of concern.	To verify the established correction factors by analyzing the interference check solution at the beginning of the analytical sequence	No samples can be run if this check does not pass acceptance criteria.
Internal Standards	A substance that is introduced in known amount into each calibration standard and field and QC sample of the analyte.	The ratio of the analyte signal to the internal standard signal is then used to determine the analyte concentration.	Any sample associated with out-of-control results must be reanalyzed.
Laboratory control sample (LCS) containing all analytes to be reported	A sample matrix, free from the analytes of interest, spiked with known amounts of analytes or a material containing known and verified amounts of analytes.	Used to evaluate the performance of the total analytical system, including all preparation and analysis steps. Assesses the ability of the laboratory/analyst to successfully recover the target analytes from a control (clean) matrix. Control limits for LCS recovery, typically expressed as percent recovery, are used for the development of statistical control limits and serve as acceptance criteria for determining whether an analytical run is in control (batch acceptance).	This is a required QC Check. The inability to achieve acceptable recoveries in the LCS indicate problems with the precision and bias of the measurement system. Failure to achieve acceptable recoveries in a "clean" matrix is an indicator of possible problems achieving acceptable recoveries in field samples.

(10-22-001)

F-1 (cont.)			
QC Check	**Definition**	**Purpose**	**Evaluation**
Linear dynamic range or high-level check standards (ICP and ICP.MS only)	High-level check standard periodically analyzed to verify the linearity of the calibration curve at the upper end.	To verify quantitative accuracy of data up to the high=level standard.	The QC check establishes the upper linear range of the calibration.
Low-Level calibration check standard (ICP only)	A reference standard that contains a quantity of analyte equal to or less than the reporting limit.	To confirm the accuracy of measurements at or near the RL.	This QC check must be within acceptance criteria before any samples are analyzed.
Matrix Spike (MS)	A sample prepared by adding a know mass of target analyte to a specified amount of matrix sample for which an independent estimate of target analyte concentration is available.	To assess the performance of the method as applied to a particular matrix. Matrix spikes are used, for example, to determine the effect of the matrix on a method's recovery efficiency. The recovery of target analytes from the matrix spike sample is used to determine the bias of the method in the specific sample matrix.	The lack of acceptable recoveries in the matrix spike often points to problems with the sample matrix. One test of this is a comparison to the LCS recoveries. If the corresponding LCS recoveries are within acceptable limits, a matrix effect is likely. The lab should not correct for recovery; only report the results of the analyses and the associated MS results and indicate that the results from these analyses have increased uncertainty
Matrix Spike Duplicate (MSD)	A second replicate matrix spike prepared in the laboratory and analyzed to obtain a measure of the precision of recovery for each analyte	To assess the performance of the method as applied to a particular matrix and provide information on the homogeneity of the matrix. Also used to determine the precision of the intralaboratory analytical process for a specific sample matrix.	When compared to the MS, the MSD will provide information on the heterogeneity of the sample matrix. The greater the heterogeneity of the matrix, the greater the RPD between the matrix spike and the matrix spike duplicate. If the sample matrix is homogeneous, such as with drinking water, and the RPD is high, this could indicate a problem in the analytical system.
MB	A sample of a matrix similar to the batch of associated samples (when available) that is free from the analytes of interest and is processed simultaneously with an under the same conditions as samples through all steps of the analytical procedures, and in which no target analytes or interferences are present at concentrations that impact the analytical results for sample analyses.	To assess background interferences or contamination in the analytical system that might lead to high bias or false positive data. Results of method blanks provide an estimate of the within-batch variability of the blank response and an indication of bias introduced by the preparation and analytical procedure.	This is one of the QC samples used to measure lab accuracy/bias. The sample could indicate whether contamination is occurring during sample prep and analysis. If analytes are detected > ½ RL, reanalyze or qualify (B-flag) all results for the specific analytes(s) in all samples in the associated prep batch as appropriate. For common lab contaminants, no analytes detected > RL. See Section D.1.1.1 and Box D-1
MSA (ICP only)	A set of procedures adding one or more increments of a standard solution to sample aliquots of the same size in order to overcome inherent matrix effects. The procedures encompass the extrapolation back to obtain the sample concentration. (This process is also called spiking the sample.)	To compensate for a sample constituent that enhances or depresses the analyte signal, thus producing a different slope from that of the calibration standards. It will not correct for additive interferences that cause a baseline shift.	This is the method used when matrix interferences are present and do not allow determination of accurate sample results
Post digestion spike addition (ICP and ICP/MS only)	An analyte spike added to a portion of prepared sample to verify absence or presence of matrix effects	To confirm the presence of a matrix interference. Assess matrix effects based on: 1. the occurrence of new and unusual matrices included within the batch, or 2. contingency analysis based on SD or MS failures	To verify the absence of an interference, the spike recovery must be between 75%-125% Results outside the acceptance limits require MSA for all samples within the batch
Second source calibration verification (ICV)	A standard obtained or prepared from a source independent of the source of standards for the ICAL. Its concentration should be at or near the middle of the calibration range. It is done after the ICAL.	To verify the accuracy of the ICAL.	The concentration of the 2nd source calibration verification, determined from the analysis, is compared with the known value of the standard to determine the accuracy of the ICAL. This independent verification of the ICAL must be acceptable before sample analysis can begin.

Notes:
1. Project-specific requirements identified by the client supersede any requirements listed. The requirements are meant to be default, to be used when project-specific direction based on DQOs is not available.
2. If there is a contradiction between the method and the DoD tables, the requirements specified in the tables shall be followed.

TestAmerica Chicago

DoD QSM Version 4.1: Appendix F - Quality Control Requirements Summary

Table F-7: Inorganic Analysis by ICP and CVAA - Methods 6010 and 7000 Series

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Demonstrate acceptable analytical capability	Prior to using any test method and at any time there is a significant change in instrument type, personnel, test method, or sample matrix.	QC acceptance criteria published by DoD, if available; otherwise method- specified criteria.	Recalculate results; locate and fix problem, then rerun demonstration for those analytes that did not meet criteria (see Section C.1.f).	NA	This is a demonstration of analytical ability to generate acceptable precision and bias per the procedure in Appendix C. No analysis shall be allowed by analyst until successful demonstration of capability is complete.
LOD Determination and verification (See Box D-13)					
LOQ Establishment and verification (See Box D-14)					
Instrument detection limit (IDL) study (ICP only)	At initial set-up and after significant change in instrument type, personnel, test method, or sample matrix.	IDLs shall be $\leq$ LOD	NA	NA	Samples may not be analyzed without a valid IDL
Linear dynamic range (LRS) or high-level check standard (ICP only)	Every 6 months	Within $\pm$ 10% of expected value	NA	NA	
Initial Calibration (ICAL) for all analytes ICP: min 1 high std and a Calib.Blank CVAA: min 5 stds and a Calib. Blank	Daily ICAL prior to sample analysis	If more than one calibration standard is used $r \geq 0.995$.	Correct problem then repeat ICAL.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until ICAL has passed.
2nd Source calibration verification (ICV)	Once after each ICAL, prior to beginning a sample run.	Value of 2nd source for all analyte(s) within $\pm$ 10% of true value.	Correct problem and verify 2nd source standard. Rerun ICV. If that fails, correct problem and repeat ICAL.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until calibration has been verified.
Continuing Calibration verification (CCV)	After every 10 field samples and at the end of analysis sequence.	ICP: within $\pm$ 10% of true value. CVAA: within $\pm$ 20% of true value.	Correct problem, rerun CCV. If that fails, then repeat ICAL. Reanalyze all samples since the last successful CCV.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to all results for the specific analyte(s) in all samples since the last acceptable calibration verification.	Problem must be corrected. Results may not be reported without a valid CCV. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Low-level calibration check standard (ICP only)	Daily, after one-point ICAL.	Within $\pm$ 20% of true value	Correct problem, then reanalyze.	Flagging criteria are not appropriate.	No samples may be analyzed without a valid low-level calibration check standard. Low-level calibration check standard should be less than or equal to the reporting limit.

F-7 (cont.)					
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action**	**Flagging Criteria**	**Comments**
Method Blank (MB)	One per prep batch	No analytes detected > ½ RL and greater than 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater). Blank results must not otherwise affect sample results. For common lab contaminants, no analytes detected >RL (see Box D-1)	Correct problem, then see criteria in box D-1; If required, reprep and reanalyze MB and all samples processed with the contaminated blank.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply B-flag to all results for the specific analyte(s) in all samples in the associated prep batch.	Problem must be corrected. Results may not be reported without a valid method blank. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Calibration Blank (ICB / CCB)	Before beginning a sample run, after every 10 samples, and at the end of the analysis sequence	No analytes detected > LOD.	Correct problem. Reprep and reanalyze calibration blank. All samples following the last acceptable calibration blank must be reanalyzed.	Apply B-flag to all results for the specific analyte(s) in all samples associated with the blank.	
Interference check solutions (ICS) (ICP only)	At the beginning of an analytical run.	ICS-A: Absolute value of concentration for all non-spiked analytes < LOD (unless they are verified trace impurity from one of the spiked analytes) ICS-AB: ± 20% of true value.	Terminate analysis; locate and correct problem; reanalyze ICS, reanalyze all samples.	If corrective action fails, apply Q-flag to all results for specific analyte(s) in all samples associated with the ICS.	
LCS containing all analytes to be reported	One per prep batch	QC acceptance criteria specified by DoD, if available; see Box D-3 and Appendix G.	Correct problem., then reprep and reanalyze the LCS and all samples in the associated prep batch for failed analytes, if sufficient sample material is available (see full explanation in Appendix G).	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to specific analyte(s) in the associated prep batch.	Problem must be corrected. Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Matrix Spike (MS)	One per prep batch per matrix (see Box D-7)	For matrix evaluation, use QC acceptance criteria specified by DoD for LCS	Examine the project-specific DQOs. If the matrix spike fails outside of DoD criteria, additional quality control tests are required to evaluate matrix effects.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met.	For matrix evaluation only. If MS results are outside the LCS limits, the data shall be evaluated to determine the source of difference and to determine if there is a matrix effect or analytical error.
Matrix Spike Duplicate (MSD) or Sample Duplicate	One per prep batch per matrix (see Box D-7)	MSD: For matrix evaluation use QC acceptance criteria specified by DoD for LCS. MSD or sample duplicate: RPD ≤ 20% (between MS and MSD or sample and sample duplicate)	Examine the project-specific DQOs. Contact client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met.	The data shall be evaluated to determine the source of difference.
Dilution Test (ICP only)	One per prep batch .	Five-fold dilution must agree within ± 10% of the original measurement.	ICP: Perform post-digestion spike (PDS) addition.	Flagging criteria are not appropriate.	Only applicable for samples with concentrations > 50x LOQ.
Post-digestion spike (PDS) addition (ICP only)	When dilution test fails or analyte concentration in all samples < 50 x LOD	Recovery within 75-125% (see Table B-1)	Run all associated samples in the prep batch by MSA or see flagging criteria.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met.	Spike addition should produce a concentration of 10 – 100 x LOQ.

(400-022)

F-7 (cont.)					
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action**	**Flagging Criteria**	**Comments**
Method of Standard Addition (MSA)	When matrix interference is confirmed.	NA	NA	NA	Document use of MSA in the case narrative.
Results reported between DL and LOQ	NA	NA	NA	Apply J-flag to all results between LOD and LOQ	

Notes:
1. Project-specific requirements identified by the client supersede any requirements listed. The requirements are meant to be default, to be used when project-specific direction based on DQOs is not available.
2. If there is a contradiction between the method and the DoD tables, the requirements specified in the tables shall be followed.

(D22-005)

Appendix 1.

Daily Operation of the HydraAA (HG5) (023-001)

TestAmerica Chicago

HG5: Hydra AA Run Notes

To Start Run

Start - WinHg
Protocol - select water or soil
File - New Dataset - Today's Date - Enter
Sample - Today's Date - Enter

Main Tab
    Reset page #
Control Tab
    Lamp On * needs to warm up for at least 45 min. then
    Gas On
    Pump On
Standard Tab
    New Cal Reset - OK
    DB - Cal Curve - Reset Calib, New Cal, Update Coeffs, Spike Coeffs (Click then hit OK)
    Standard Tab - Click all 6 STD's and Rep 1
Sample Tab
    Click on Rack Icon at Top of page and add samples to 1st column
    Save as today's date
    Sample Tab - Select Rack and Start and Finish
Utility Tab
    DAQ - Do Action - Sample Measurement should be over 300,000
Standard Tab
    Click Stnd Auto to start calibration
Cal Curve Tab on DB
    Click Accept when calibration is finished
Sample Tab
    Click Run Auto to start sample run
Report Tab
    Watch run

When Run is Finished

File - Page Eject - Will not print automatically
Report Tab on DB
    Click on Run (make sure all samples in batch are checked)
    Click on Generate Report
    Format -check PRN File
    Output File Path - click on ... - Double click data folder - type in file name - Generate
    Print Intensities
        Report Spec - Edit - Absorbance - OK
        Generate Report - Generate Report
        Edit - Turn Off Absorbance

Send to LIMS

Data Folder - Find File - right click - send to HG5

Turn Off Instrument

Control Tab
    Lamp - Off
    Gas - Off
    Pump - Off
File – Exit

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(023-001)

Appendix 2.

Daily Operation of the Hydra II_{AA} (HG6) (024-001)

TestAmerica Chicago

HG6: Hydra II Run Notes

To Start Run

Start - Envoy
Method
- Instrument Control
  - Lamp - On (Needs to warm up for 20 min. before starting run)
- Calibration
  - Clear Calibration
- Sequence
  - Sequence – New – Enter/Scan in QC & Samples
  - **UPDATE**

Start Up (Green oval with arrow in it), starts the pump and gas
- Allow instrument to sip stannous and rinse for a few minutes

Run Sequence (Test tubes in a Rack icon)
Analysis
- Right click on first sample
- Insert Chapter – enter date

Method
- Calibration (watch curve)

Analysis
- Results (watch samples)

When Run is Finished

Analysis
- Report
  - GENERATE LIMS INPORT
  - Statistics – load – Tals import – OK
  - Check the chapter folder – Report – CSV File – Type date – Save
  - Envoy folder on main screen – Find file – Right Click – Send to TALS

IMPORT
- PRINT REPORT
- Detailed – load – Print – Report – Printer – Type Date – Print

Turn Off Instrument

Click Sleep (Blue oval with black circle inside)
Method
- Instrument Control
  - Lamp – Off

Method – Exit

CHI-22-14-087/A-1010

(024-001)

TITLE: Metals Analysis
Trace Inductively Coupled Argon Plasma by SW-846 6010C (Simultaneous Operation)

Approvals (Signature/Date):

Debbie Johnson 9/28/11
Debbie Johnson Date
Supervisor, Metals Supervisor

Diane L. Harper 9/27/11
Diane L. Harper Date
Inorganics Manager

Terese A. Preston 9/28/11
Terese A. Preston Date
Quality Assurance Manager

9/27/11
John D. Nagel Date
Env. Health & Safety Coor.

Michael J. Healy 9/27/11
Michael J. Healy Date
Laboratory Director

Facility Distribution No: _____ **Distributed To:** Uncontrolled

1.0 SCOPE / APPLICATION

This Standard Operating Procedure (SOP) outlines the guidelines for determining metal concentrations by Trace Inductively Coupled Argon Plasma (ICAP) Emission Spectrometry - Simultaneous Operation. This SOP was written using U.S. EPA SW-846 "Test Methods for Evaluating Solid Waste", Update IV, Method 6010C as a reference. The hardness calculation found in section 9.4 is from Standard Methods 18th and 20th Editions, method 2340 B.

On occasion, clients request slight modifications to this SOP. These modifications are addressed on a case-by-case basis with the range of accuracy (i.e., MDLs, linearity check or PT sample) verified prior to implementation. Any modifications would be written into a Quality Assurance Plan (QAP), authorized via laboratory signature approval, and mentioned in the data package's case narrative.

Specific requirements pertaining to the DOD QSM Version 4.2 are located in Appendix A. These requirements are additionally applicable to all NFESC projects. Any deviations from these procedures and/or variances from must be addressed appropriately in accordance with standard operating protocol and pre-approved on a project by project basis.

1.1 Method Sensitivity

1.1.1 Method Detection Limits

The method detection limit (MDL), referred to as the detection limit (DL) in NELAC and DOD QSM documents, is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to Appendix B of 40 CFR 136, "Guidelines Establishing Test Procedures for the Analysis of Pollutants" with additional details are provided in the TestAmerica Corporate SOP, *CA-QS-006, Detection Limits* and the TestAmerica Chicago SOP, *UP-QA-017, Method Detection Limit Studies.* MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; the MDL will be verified on a quarterly basis to meet the requirements of the DoD QSM version 4.2.

1.1.2 Demonstration of Capability

Before the laboratory may institute a new method and begin reporting results, the laboratory shall confirm that it can properly operate the method. A demonstration of capability is performed whenever there is a change in instrument type, method or personnel. An Initial Demonstration of Capability (IDOC) must be thoroughly documented and approved by the Department Manager/Supervisor and QA Manager prior to independently analyzing client samples. All associated documentation must be retained in the QA Department and in the Analyst Training files. For additional details on the demonstration of capability procedures followed, refer to the laboratory SOP, *UP-QA-QAM, Quality Assurance Manual, Sections 20.4.2 and 20.4.3.*

1.1.3 Instrument Detection Limits

Instrument Detection Limits (IDLs) are performed on a quarterly basis for each element and for each instrument (as specified in CLP). These limits are used to gauge instrument sensitivity and when routinely evaluated, instrument performance without the introduction of method variance can be determined. Refer to the IDL SOP, *UP-QA-010* for additional details.

1.1.4 Reporting Limits

Reporting Limits are defined as the lowest concentration of an analyte determined by a given method in a given matrix that the laboratory feels can be reported with acceptable quantitative error or client requirements, values specified by the EPA methods or other project and client requirements. The laboratory maintains reporting limits that are higher than the MDL. Wherever possible, reporting is limited to values ~3-5x the respective MDL to ensure confidence in the value reported. Client specific requests for reporting to the MDL are special circumstances not to be confused with the previous statement. Refer to Table 1 for element wavelength and reporting limits.

1.1.5 Definitions

Refer to Section 3.0 of the Laboratory's Quality Assurance Manual (UP-QA-QAM).

1.2 Summary of Method

ICAP is a technique for the analysis of soluble or digested samples for metal concentrations using atomic emission spectrometry. All matrices, including water, TCLP extracts, wastes, soils, sludges and sediments, require digestion prior to analysis. The iCAP 6500 is capable of analyzing simultaneously 29 different elements on a sample, and the Trace 61E analyzes 28 elements simultaneously.

2.0 INTERFERENCES

Spectral, Physical and Chemical Interferences are the three main interferences that are commonly present on the ICAP.

2.1 Spectral Interferences

Spectral interferences are mainly caused by continuous background wavelength, stray light from a high concentration element or overlap of a spectral line from another element. The ICAP can correct for the first two types of interferences by using background correction adjacent to the wavelength. Spectral overlap can be corrected by monitoring the interfering wavelength and computer correcting the results for the false concentration. The values used to correct are known as Inter-Element Correction Factors or IECs. See Section 6.2. Interferent Check Standard A (ICSA) is monitored to detect instrument drift requiring the IEC equations to be corrected.

2.2 Physical Interferences

Physical interferences are usually associated with the sample uptake and nebulization processes. These interferences can usually be eliminated by using a peristaltic pump which assures a constant sample uptake rate. If a sample is extremely viscous or contains a very high dissolved solids concentration, a dilution of the sample may be required to assure a constant and smooth nebulization rate.

2.3 Chemical Interferences

Normally there are not significant chemical interferences on the ICAP. These interferences include ionization effects and molecular compound formation. Chemical interferences are highly dependent on the sample matrix type and the element.

The ICP can have some ionization effects caused by torch positioning. To eliminate these effects, Cesium may be added to the internal standard solution (100 mLs / 1-Liter).

Most interference can be corrected by ensuring a constant sample uptake rate and by using the correcting abilities of the computer. If severe interferences are suspected, an alternate method such as ICP/MS can be used or to verify the ICAP results.

3.0 SAFETY

Employees must abide by the policies and procedures in the Corporate Safety Manual, Radiation Safety Manual, Lab Specific Addendum to the CSM, and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coat and closed-toe, nonabsorbent shoes are a minimum.

3.1 Specific Safety Concerns or Requirements

- The ICP plasma emits strong UV light and is harmful to vision. All analysts must avoid looking directly at the plasma.
- Parts of the instrument can be extremely hot. Care should be taken if the instrument needs to be adjusted internally.
- Proper ventilation is required due to sample fumes and extreme heat generation (RF generator and plasma) and plasma emissions. People with medical conditions that may respond to ozone emissions should exercise caution.

3.2 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and Symptoms of Exposure
Nitric Acid	Corrosive Oxidizer Poison	2 ppm-TWA 4 ppm-STEL	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Hydrochloric Acid	Corrosive Poison	5 ppm-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
1 – Always add acid to water to prevent violent reactions. 2 – Exposure limit refers to the OSHA regulatory exposure limit.			

4.0 EQUIPMENT AND SUPPLIES

4.1 Instrumentation

1) Thermo Fisher ICAP 61E Trace Analyzer. This simultaneous ICAP currently has 31 analytical wavelengths, with additional wavelengths available. It is equipped with a peristaltic pump for sample uptake and an autosampler, and uses TJA software version 6.2. TestAmerica Chicago Lab ID: ICP5

2) Thermo Fisher iCAP 6500. Duo, with Cetac ASX-520 autosampler, and Neslab Thermo Flex 900 water chiller. Currently has 32 analytical wavelengths with additional wavelengths available. The instrument employs iTEVA Analyst – TA 2009B (Revision 47) software. TestAmerica Chicago Lab ID: ICP6

4.2 Supplies

- Volumetric Flasks (Class A): 100 mLs; 200 mLs; 1000 mLs
- Eppendorf Pipettes, varying volumes

5.0 REAGENTS AND STANDARDS

5.1 Reagents

- Milli-Q Water
- *Concentrated Nitric Acid (HNO_3) - InstraPure
- *Concentrated Hydrochloric Acid (HCl) - InstraPure

*Purchased from a vendor.

5.2 Standards and QC Solutions

All stock standards and QC solutions are purchased from an outside supplier in aqueous form. The suppliers that are currently used are Inorganic Ventures, Hi Purity, CPI and Ultra. Two types of standards are used: single element and custom mixed standards. Single element standards are available for most elements at a 1,000 mg/L concentration. The shelf life of all purchased solutions are as stated by the manufacturer and are listed in TALS.

5.2.1 Calibration Standards

Prepared with Milli-Q water that has been acidified with 1% HNO_3 and 5% HCl. Internal standard is automatically mixed into all standards, QC solutions and samples at an approximate concentration of 1 ppm. The calibration standards are prepared daily as follows:

A. Calibration Blank

Add ~500 mLs of Milli-Q water to a 1-L Class A volumetric flask. Repipette 10 mLs conc. HNO_3 and 50 mLs conc. HCl into the flask. Dilute to volume with Milli-Q water and mix thoroughly.

B. Calibration Standards (Refer to Attachment 1 for element concentrations)

Note: A blank and a 3-standard curve is done for each element on the Trace 61E, but a blank and single standard are used to calibrate the iCAP 6500.

The trace 61E calibration curve must have a correlation coefficient of ≥ 0.998.

Trace 61E Calibration

Standard	Preparation
S1	• Add ~50 mLs of Milli-Q water to a 200 mL Class A volumetric flask. • Re-pipette 2 mLs of concentrated HNO_3 into the flask. • Re-pipette 10 mLs of concentrated HCl into the flask. • Using Eppendorf pipettes, add 2.0 mLs each of: RFW-ICPT-STD-1B RFW-ICPT-STD-1C RFW-ICPT-STD-1D • Dilute to volume with Milli-Q water and mix thoroughly.
S1A	• Add ~50 mLs of Milli-Q water to a 200 mL Class A volumetric flask. • Re-pipette 2 mLs of concentrated HNO_3 into the flask. • Re-pipette 10 mLs of concentrated HCl into the flask. • Using Eppendorf pipettes, add 0.8 mLs each of: RFW-ICPT-STD-1B RFW-ICPT-STD-1C RFW-ICPT-STD-1D • Dilute to volume with Milli-Q water and mix thoroughly.
S1B	• Add ~50 mLs of Milli-Q water to a 200 mL Class A volumetric flask. • Re-pipette 2 mLs of concentrated HNO_3 into the flask. • Re-pipette 10 mLs of concentrated HCl into the flask. • Using Eppendorf pipettes, add 1.0 mL each of RFW-ICPT-STD-1B RFW-ICPT-STD-1C RFW-ICPT-STD-1D • Dilute to volume with Milli-Q water and mix thoroughly.
S2	• Add ~50 mLs of Milli-Q water to a 200 mL Class A volumetric flask. • Re-pipette 2 mLs of concentrated HNO_3 into the flask. • Re-pipette 10 mLs of concentrated HCl into the flask. • Using Eppendorf pipettes, add 2.0 mLs each of: RFW-ICPT-STD-2A RFW-ICPT-STD-2B RFW-ICPT-STD-3 • Dilute to volume with Milli-Q water and mix thoroughly.
S2A	• Add ~50 mLs of Milli-Q water to a 200 mL Class A volumetric flask. • Re-pipette 2 mLs of concentrated HNO_3 into the flask. • Re-pipette 10 mLs of concentrated HCl into the flask. • Using Eppendorf pipettes, add 0.8 mLs each of: RFW-ICPT-STD-2A RFW-ICPT-STD-2B RFW-ICPT-STD-3. • Dilute to volume with Milli-Q water and mix thoroughly.
S2B	• Add ~50 mLs of Milli-Q water to a 200 mL Class A volumetric flask. • Re-pipette 2 mLs of concentrated HNO_3 into the flask. • Re-pipette 10 mLs of concentrated HCl into the flask. • Using Eppendorf pipettes, add 1.0 mL each of: RFW-ICPT-STD-2A RFW-ICPT-STD-2B RFW-ICPT-STD-3 • Dilute to volume with Milli-Q water and mix thoroughly.

ICAP 6500 Calibration

Standard	Preparation
S1	• Add ~50 mLs of Milli-Q water to a 100ml Class A volumetric flask • Re-pipette 1 mL of conc. HNO_3 into the flask. • Re-pipette 5 mLs of conc. HCl into the flask. • Using Eppendorf pipettes, add 1.0 mL each of: STDIL-STD-1 STDIL-STD-2 • Using an Eppendorf pipette, add 0.50 mL of RFW-ICPT-STD-2B • Using an Eppendorf pipette, add 0.250 mL of RFW-ICPT-STD-2A • Using Eppendorf pipettes, add 0.10 mL each of: Bi, Si and Ag from a 1000 ug/mL stock solution • Dilute to volume with Milli-Q water and mix thoroughly.
S2	• Add ~50 mLs of Milli-Q water to a 100ml Class A volumetric flask • Re-pipette 1 mL of conc. HNO_3 into the flask. • Re-pipette 5 mLs of conc. HCl into the flask. • Using Eppendorf pipettes, add 2.00 mL of RFW-ICPT-STD-2B • Using Eppendorf pipettes, add 1.00 mL of RFW-ICPT-STD-2A • Dilute to volume with Milli-Q water and mix thoroughly.

5.2.2 Instrument QC Solutions (Refer to Attachment 2 for element concentrations.)

All QC solutions are recorded in the TALS reagent module and are prepared as follows:

QC Solution	Preparation To a 1-L Class A volumetric flask filled w/ ~500 mLs of Milli-Q water, add the following for each QC Solution:
Reporting Limit Check Std. (ICVL/CCVL)	• 10 mLs of concentrated HNO_3. • 50 mLs of concentrated HCl. • 1.0 mL of TACHI-1 • 1.0 mL of TACHI-2 • 50 uLs Bi from a 1000 µg/mL stock • Dilute to volume with Milli-Q water and mix thoroughly.
Initial Calibration Verification (ICV)	• 10 mLs of concentrated HNO_3. • 50 mLs of concentrated HCl. • 8 mLs each of: CCV Solution A CCV Solution A1 CCV Solution B • 1.6 mLs of 10,000 µg/mL Fe • 1.6 mLs of 10,000 µg/mL Na • 1.68 mLs of 10,000 µg/mL Mg • 3.6 mLs of 10,000 µg/mL Al • 1.84 mLs of 10,000 µg/mL Ca • 3.6 mLs of 10,000 ug/mL K • Dilute to volume with Milli-Q water and mix thoroughly.
Continuing Calibration Verification (CCV)	• 10 mLs of concentrated HNO_3. • 50 mLs of concentrated HCl. • 10 mLs each of: CCV Solution A CCV Solution A1 CCV Solution B • 4.5 mLs of 10,000 µg/mL Al • 2.3 mLs of 10,000 µg/mL Ca • 2.0 mLs of 10,000 µg/mL Fe • 2.0 mLs of 10,000 µg/mL Na • 4.5 mLs of 10,000 µg/mL K • 2.1 mLs of 10,000 µg/mL Mg • Dilute to volume with Milli-Q water and mix thoroughly.
CRI [Contract Required Detection Limit (CRDL) Standard for ICAP]	• 10 mLs of concentrated HNO_3 . • 50 mLs of concentrated HCl. • 2.0 mL of TACHI-1 • 2.0 mL of TACHI-2 • 100 uLs of 1,000 µg/mL Bi • Dilute to volume with Mill-Q water and mix thoroughly.
ICSA (Interferent Check Standard)	• 10 mLs of concentrated HNO_3. • 50 mLs of concentrated HCl. • 100 mLs of CLPP-ICS-A. • Dilute to volume with Milli-Q water and mix thoroughly.
ICSAB (Interferent Check Standard)	• 10 mLs of concentrated HNO_3. • 50 mLs of concentrated HCl. • 100 mLs of CLPP-ICS-A • 10 mLs of CLPP-ICS-B4 • Dilute to volume with Milli-Q water and mix thoroughly.

QC Solution	Preparation To a 1-L Class A volumetric flask filled w/ ~500 mLs of Milli-Q water, add the following for each QC Solution:
Daily LRS Sample (Note: made in a 100mL Class A volumetric containing ~20 mL Milli-Q water) Required for DoD version 4.1	• 1 mL conc. HNO_3 • 5 mLs conc. HCl • 5 mLs of 10,000 µg/mL K *(ICP6 only)* • 4 mLs of 10,000 µg/ml Na *(ICP6 only)* • 1mL of 1,000 µg/mL Sb, B, Ba, Co, Cu, Cr, Mn, Mo, Ni, Pb, Si, V, Zn • 0.5 mLs of 1,000 µg/mL As, Be, Bi, Cd, Se, Sn, Ti, Tl • Dilute to volume with Milli-Q water and mix thoroughly

Note: Some of the volume amounts of the single-element spikes in the above table appear to be reduced from the obvious volume necessary to achieve the final desired concentration. This is to compensate for the presence of those metals in the mixed standards also added to the working standard.

6.0 CALIBRATION (NON-DAILY)

6.1 Linear Range Standard (LRS)

ICAP analysis involves a Linear Dynamic Range (LDR) and a Linear Calibration Range (LCR). At TestAmerica Chicago, the high standard of the LCR is set below the established upper limit of the LDR for all elements. In order to establish, verify, and document linearity at or near that upper limit, a Linear Range Study (LRS) is done every six months on each instrument. This is done by running a single standard at the upper limit of the anticipated linear range of measurement after the instrument has been calibrated in the usual daily manner. The acceptance limits for these single-element verification standards is 95-105%. During routine analysis, all results, target or non-target, that are above the LDR are flagged by the instrument software, aiding the analyst in making appropriate dilution decisions. All samples for which a target analyte or an interfering element result is found to be above the LDR are diluted and re-analyzed until the concentration falls within the instrument's linear range.

Note: For DoD projects a daily linear range verification standard must be run. At TestAmerica Chicago, this standard is at a lower level than the semi-annual linear range study and must be within the acceptance range of 90-110%. Target and non-target interferences must be diluted below the level of the daily linear range standard.

6.2 Inter-Element Correction (IEC)

Correction factors for spectral interference will be determined at least annually for all wavelengths used for each analyte reported, any time the ICAP is adjusted in any way that may affect the IECs, or as needed based on continuing observation of Interferent Check Standard A (ICSA) and Interferent Check Standard B (ICSAB). The correction factor is manually calculated from the result ratios between the affected analyte and a known interferent analyzed simultaneously at an appropriately high concentration. The factor, documented with the instrument records, is entered into the method at the instrument PC and becomes part of the software algorithm. See Section 9.5 for calculation details.

Correction factors for spectral interferences other than Al, Ca, Fe, and Mg are recommended and are performed as needed.

7.0 PROCEDURE

7.1 Batch Quality Control Checks

The following section summarizes the quality control (QC) samples associated with routine ICAP analysis.

QC Sample	Frequency[6]	Control Limit [1]
Method Blank (MB)	1 per 20 samples	≤ Reporting Limit (<1/2 LOD for DoD[8])
Lab Control Sample (LCS) [2]	1 per 20 samples	80 – 120%
Matrix Spike (MS) [3,6]	1 per 20 samples	75 – 125% (80-120% for DoD)
MS Duplicate (MSD) [3,5]	1 per 20 samples	75 – 125%; 20 RPD (80-120% for DoD)
Duplicates (DU) [4,6]	1 per 20 samples	20 RPD
Serial Dilution (SD) (5x dilution)	1 per 20 samples	± 10% of the original result if analyte conc. > 10X MDL[9]
Post Digestion Spike (PDS)	1 per 20 samples - required for DoD/AFCEE if SD or MS or MSD fails	75 - 125%[7]

[1] Refer to Section 8 for additional details.
[2] LCS Duplicate (LCSD) is run only when required by the client or project.
[3] If sample concentration is ≤ 4X spike level, 75-125%; if sample concentration is > 4X spike level, no control range. If TCLP matrix spike is < 50%, Standard Addition must be performed.
[4] If ≥ 5X reporting limit, 20 RPD; if < 5X reporting limit, ± reporting limit; if < reporting limit, no control range.
[5] The sample selection for matrix QC, if not specified by the client or on the chain-of-custody, is rotated among client samples so that various matrix problems may be noted and/or addressed…pre-determined by the digestion department.
[6] Some programs, including IDEM, require 1/10 frequency.
[7] For DoD, if PDS is out of control, MSA is required.
[8] Reporting Limit is referred to as LOD – Limit of Detection in DoD Version 4.2
[9] The Method Detection Limit (MDL) is referred to as Detection Limit (DL) in DoD Version 4.2

7.2 Sample Preservation and Storage

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

Matrix	Holding Time [1]	Preservation	Reference
Waters	180 days	HNO_3, pH < 2; Cool 4 ± 2°C	40 CFR Part 136.3
Soils	180 days	Cool 4 ± 2°C	N/A

[1] Inclusive of digestion and analysis.

7.3 Sample Preparation

The most commonly used digestion procedures are SW-846 Methods 3010A (waters) and 3050B (soils). Refer to UP-SP-3000 for details on sample digestion. The samples are received in the metals laboratory as 25, 50 or 100 mL final volumes.

7.4 Calibration / Standardization

7.4.1 Instrument Set Up

Trace 61E:

Set up the instrument with the proper operating conditions as defined in the instrument manual. The instrument must be allowed to become thermally stable (~1-hour) prior to profiling and calibration. The instrument is profiled using a 1-ppm Arsenic standard by aspiration and selecting the automatic profile feature from the Thermo Fisher software. The peak position reading should be within +/- 0.1. If the reading is acceptable, record the peak area in the logbook & rinse. If the reading is > +/- 0.1, set the micrometer to the adjusted vernier position given by the instrument and profile again to verify. Record the peak area in the logbook and rinse. The instrument is now ready to calibrate.

ICAP 6500:

Set up the instrument with the proper operating conditions as defined in the instrument manual. The instrument must be allowed to become thermally stable (~1/2-hour) prior to calibration. Alignment of the torch or sensor chip is done as needed. A 2ppm Zinc solution is aspirated to align the torch after cleaning. An autopeak adjust is run only if some part of the induction system is changed such as pump speed, nebulizer pressure, etc.

7.4.2 Standardization

Before any instrument is used as a measurement device, the instrument response to known reference materials must be determined. All sample measurements must be made within the linear range of the instrument.

The Trace 61E instrument is standardized using a calibration blank and 3 calibration standards, the 6500 uses a calibration blank and one standard. The results are given in intensities.

Standard	Frequency	Control Limit
Calibration Curve	Initially	Corr. Coeff. $\geq$ 0.998 (Trace only)
High Standards (S1, S2)	After the Calibration Curve	$\pm$ 5% of the Known Conc.
Initial Cal. Verif. (ICV)	After the Calibration Curve – mid-range	$\pm$ 10% of the Known Conc.
Low-level ICV (ICVL)	After mid-range ICV - RL level	$\pm$ 30% of Known Conc. (± 20% for DoD, replacing MRL)
Initial. Cal. Blank (ICB)	After the ICV	≤ Reporting Limit (≤ LOD[1] for DoD)
ICSA / ICSAB	Daily	*± 20% of the Known Conc. or absolute value <RL for all non-spiked elements. (< LOD for DoD)
Cont. Cal. Verif. (CCV)	Every 10 reading; End of each run – mid-range	$\pm$ 10% of the Known Conc.
Low-level CCV (CCVL)	After mid-range CCV – at RL	$\pm$ 30% of Known Conc.
Cont. Cal. Blank (CCB)	Every 10 readings; End of each run	≤ Reporting Limit (≤ LOD for DoD)

* Note: Samples with extremely high "A" concentrations and low "B" concentrations may warrant further evaluation.
The Low-level ICV and Low-level CCV are only required at the beginning and the end of a run, but may be run with every CCV since reported data must be successfully bracketed.
[1] RL is referred to as LOD in DoD version 4.2

7.5 Preventive Maintenance

The required preventive maintenance is listed in the preventive maintenance logbooks which are kept at the instrument. All maintenance is recorded along with the date and the signature of the analyst performing the maintenance. Documentation of "Return to Control" must be entered into the maintenance log book. The instrument is under a full service contract with the manufacturer for all major repairs and an annual PM services.

7.5.1 Daily Maintenance

Minimally includes changing the pump tubing for consistent sample uptake and a visible check of the waste container to make sure that it doesn't overflow.

7.5.2 Weekly Maintenance

Minimally includes checking the air filters on the back of the instrument for excessive dust buildup, and checking the tip of the torch for excessive buildup of material.

7.5.3 Monthly Maintenance

Includes cleaning and checking the water re-circulator for proper fluid level, cleaning the spray chamber. Check the air filters to be cleaned or replaced.

7.6 Sample Analysis

7.6.1 Analytical Run

After the instrument is standardized (Section 7.4.2), an analytical run is initiated. The first run of the day would proceed as follows:

- S1,S2 Reanalysis of calibration standard as a sample
- ICV Initial Calibration Verification
- ICVL Low-level ICV
- ICB Initial Calibration Blank
- ICSA Interferent Check Standard A
- ICSAB Interferent Check Standard B
- CCV Continuing Calibration Verification
- CCB Continuing Calibration Blank
- MB (1) Method Blank
- LCS (2) Laboratory Control Sample
- Sample (3)
- Sample (4) Serial Dilution (SD)
- Sample (5) Matrix Duplicate (DU)
- Sample (6) Matrix Spike (MS)
- Sample (7) Matrix Spike Duplicate (MSD)
- Sample (8)
- Sample (9)
- Sample (10)
- CCV Continuing Calibration Verification
- CCVL Low-level CCV
- CCB Continuing Calibration Blank

If the CCV, CCVL and CCB results are acceptable, the run may continue without restandardization. If any of the post-run QC is out of control, or close to being out of control, the instrument is restandardized before analyzing the next batch. Any samples with elements associated with an out of control CCV, CCVL or CCB will be reanalyzed.

7.7 Documentation

7.7.1 Instrument Run-Log

The analysis of each day's samples and standards is documented within the instrument run log (Attachment 3), and is supported by the instrument print-out.

7.7.2 Traceability of Standards

Custom made and single element stock standard solutions which are traceable to NIST or EPA are purchased. Upon receipt, each standard is entered into LIMS and is issued a unique source ID#. The manufacturer, lot #, date received, expiration date, date of verification and the initials of the recording analyst are also entered. The preparation data for all intermediate standards prepared in house are also entered into TALS. Unique bar-coded labels are generated that are affixed to the stock or standard containers. The unique standard IDs are scanned or manually entered into the TALS preparation and analytical batches along with the volumes used for spiking the various QC standards. The standard IDs appear in the TALS-generated raw data.

7.7.3 Data Review

Analytical data goes through a 200% review cycle. As results are generated, analysts review their work to ensure that the results generated meet QC requirements and relevant EPA methodologies. The analyst transfers the data into TALs in the Analyst Desktop module. Where non-compliance is observed, the analyst creates Non-Conformance Memos (NCMs) in TALs. Flags and data qualifiers can be method, project, program or QAPP specific. The analyst documents the initial review on a data review checklist (Attachment 4) and sets the batch status in LIMs to 1st level review. The second level or peer review of the data is conducted by another individual who has been trained on the review process. This secondary review is documented on the same checklist, making any necessary corrections to the data or additions to the NCMs as necessary. The batch is then set to 2nd level review. The raw data, including the checklist, instrument print-outs, and manual entries, and electronic files are retained for easy retrieval in accordance with the laboratory's record and retention policy outlined in the SOP, *UP-QA-QAM, Section 15.*

Examples of items included in the above reviews are as follows:

- QC data are outside the specified control limits for accuracy and precision
- Unusual detection limit changes are observed
- Samples having unusually high results
- Samples exceeding a known regulatory limit
- Raw data indicating some type of contamination or poor technique
- Transcription errors
- Results outside of calibration range

8.0 QUALITY CONTROL

8.1 QC Summary

NOTE: The following laboratory acceptance criteria are set at default control limits. Statistical limits are generated on an annual basis from cumulative LCS data and can be implemented when specified by the client, contract, or QAP.

8.1.1 Method Blank (MB)

At least one MB and one LCS will be included in each digestion batch of 20 samples. The MBs are analyzed to determine if contaminants are being introduced into the sample via the sample preparation procedures.

8.1.2 Laboratory Control Sample (LCS)

The LCS is analyzed to determine the accuracy of the digestion process.

Accuracy will be measured by the percent recovery (%R) of the LCS. The recovery must be within ±20% of the known concentration. If the LCS results are outside these control limits, all samples in the preparation set must be redigested and reanalyzed. Refer to Attachment E for element concentrations.

8.1.3 Matrix Duplicate (DU)

A duplicate sample will be prepared at a frequency of 5% (1 in 20 samples). A 20 RPD is set as the acceptance limits.

8.1.4 Matrix Spike (MS) / Matrix Spike Duplicate (MSD)

The MS / MSD will be prepared at a frequency of 5% (1 in 20 samples). The recovery must be within 75–125%. (Exception allowed if the sample concentration exceeds 4 times the spike added concentration.) DoD version 4.1 requires 80-120% recovery.

TCLP - If the MS recovery is <50% and the concentration does not exceed the regulatory limit or the sample concentration is within 20% of the regulation level, the Method of Standard Addition (MSA) is required. Three aliquots of the sample are spiked at 50%, 100% and 150% of the sample concentration or, if the sample concentration is < RL, the MSA is at 50%, 100% and 150% of the MS level. The data is subjected to linear regression whereas the concentration of the unknown is the x-intercept and the correlation coefficient value must be ≥ 0.995.

8.1.5 Serial Dilution

A Serial Dilution (5X) will be prepared from the digestate at a frequency of 5% (1 in 20 samples). If the concentration is >10 times the MDL, results should agree within +/- 10% of the original results.

8.1.6 Post Digestion Spikes

If the MS/MSD is not within limits, a Post Digestion Spike may be analyzed to compensate for the matrix effect. Post Digestion Spikes are analyzed on a project, program or client request basis. Recoveries must fall between 75-125%.

8.2 Corrective Action

When an out-of-control situation occurs, the analysts must use his/her best analytical judgment and available resources to determine the corrective action to be taken. The out- of-control situation may be caused by more than one variable. The analyst should seek the assistance of his/her supervisor, QA personnel, or other experienced staff if he/she is uncertain of the cause of the out-of-control situation. The analysis must not be resumed until the source of the problem and an in-control status is attained. All samples associated with the out-of-control situation should be reanalyzed. Out-of-control data must never be released without approval of the supervisor, or QA personnel.

The following steps that must be taken when an out-of-control situation occurs:

- demonstrate that all the problems creating the out-of-control situation were addressed;
- document the problem and the action which was taken to correct the problem on a NCM;
- document on the NCM that an in-control has been achieved; and
- receive approval (signature) of the supervisor or QA personnel prior to the release of any analytical data associated with the problem.

QC Indicator	Suggested Corrective Actions
Calibration Curve	• reanalyze the standard curve; • prepare a new stock and/or working standards; • check the reagents/solutions and prepare fresh if necessary.
ICV/ICVL	• repeat the ICV to verify proper preparation; • prepare a new ICV from original stock; • recalibrate with a new standard curve; • prepare a new stock and/or working standards; • check the reagents/solutions and prepare fresh if necessary.
ICB	• prepare a new ICB to verify proper preparation; • verify that the instrument base-line is stable and perform necessary maintenance, cleaning, etc.. to achieve stability; • determine the source of contamination by process of elimination, carryover from a previous analysis or reagent contamination and correct the problem; • check the reagents/solutions and prepare fresh if necessary; • correct for any contamination and reanalyze the ICB and any associated samples.
LCS	If the LCS is low: • reanalyze the LCS and all samples in the set for the failed analyte(s) to confirm that it is out of control. • If continued out of control, redigest and reanalyze the set. • Write an NCM. If the LCS is high: • reanalyze the LCS and all samples in the set for the failed analyte(s) to confirm that it is out of control. • check for contamination of reagents, LCS stock solution, or in the preparation area; • correct for contamination, redigest and re-analyze the set; • Write an NCM.

QC Indicator	Suggested Corrective Actions
MB	• reanalyze the MB to verify that it is beyond the reporting limit; • determine the source of contamination; • determine if a high value is due to contamination; • check for contamination of reagents or in the preparation area; • correct for contamination, reanalyze the set; • report results at least 10x > the MB or < RL where applicable; • in the extreme case where all samples in the set are at least 10x > the MB or < RL for all metals, reanalysis will not be required; however, an NCM will be written and approved by the supervisor or section manager.
DU	• an NCM will be written and approved by the supervisor or section manager.
MS / MSD	• an NCM will be written and approved by the supervisor or section manager.
Serial Dilution (SD)	• prepare a new serial dilution to verify proper preparation; • an NCM will be written and approved by the supervisor or section manager.
CCV/CCVL	• repeat the CCV to verify proper preparation; • prepare a new CCV from the original stock; • check for instrument base-line drift or a change in one or more of the reagents; • check the reagents/solutions and prepare fresh if necessary; • recalibrate with a new standard curve and repeat all samples since the previous in control CCV; • never dispose of any samples until you are sure that all QC are within the control limits.
CCB	• check reagents/solutions to verify proper preparation and prepare fresh if necessary; • verify that the instrument base-line is stable and/or perform necessary maintenance, cleaning, etc., to achieve stability; • correct for any contamination (carryover from a previous analysis or reagent contamination) and reanalyze the CCB and any associated samples; • never dispose of any samples until you are sure that all QC are within the control limits.
Additional CAs	• If any of the ICV, ICB, ICSA, ICSAB, CCV or CCB results are out-of-control for any element, the instrument is restandardized and the samples associated with the out-of-control elements are reanalyzed. • If the MB or LCS is out of control for any element, the samples are redigested. An exception is if the sample concentrations are ≥ 10X the MB contamination or < RL. In this case, the results are reported as is. • If any of the DU or MS/MSD results are out of control, the client is notified of the poor results via a case narrative that is sent with the data report. • NCMs can be created at any time in LIMS, most frequently as the data are initially reviewed in AD. They are automatically forwarded to the appropriate managers as emails and the text appears in the final report to the client.

9.0 DATA ANALYSIS AND CALCULATIONS

The sample results are stored in a data file on the desktop computer. The data is transferred over to LabNet and edited there. This system helps to eliminate transcription errors, since data is not entered by hand.

9.1 Accuracy

9.1.1 ICV / CCV, LCS % Recovery = $\frac{\text{observed concentration}}{\text{known concentration}} \times 100$

9.1.2 MS / MSD % Recovery = $\frac{\text{(spiked sample) - (unspiked sample)}}{\text{spiked concentration}} \times 100$

9.2 Precision (RPD)

9.2.1 Matrix Duplicate (MD) = $\frac{|\text{orig. sample value - dup. sample value}|}{[(\text{orig. sample value + dup. sample value})/2]} \times 100$

9.3 Concentration

mg/kg or L = $\frac{C \times V \times D}{W}$

Where:
C = sample concentration in extract (ppm)
V = Volume of extract (mL)
D = Dilution Factor
W = Weight/Volume of sample aliquot extracted (grams or mLs)

NOTE: All dry weight corrections are made in the TALs LIMs analytical batch at the time the data is processed.

9.4 Calculations for SiO2 and Hardness

Calculation of SiO2

(Si) Silicon Raw (mg/L) * 2.1392 * PF

PF=Prep Factor

Calculation of Magnesium Hardness

(Mg) Magnesium Raw (mg/L) * 4.118 * PF

Calculation of Calcium Hardness

(Ca) Calcium Raw (mg/L) * 2.497 * PF

Calculation of Total Hardness

(((Ca) Calcium Raw (mg/L) * 2.497) + ((Mg) Magnesium Raw (mg/L) * 4.118)) * PF

9.5 Calculation of Inter-element Correction Factors

Spike a blank with the known interfering element at an appropriate concentration, typically at the upper level of the linear range. For ICP5, divide the observed result of the affected element by the observed result of the interfering element. Since the IEC study was done with the previous IEC factor applied, add (or subtract if negative) this result to the current correction factor value in the method. The result is the new IEC for the affected element, equal to the adjustment made on 1 ppm of the affected element. ICP6 has an optional auto-IEC calculation feature in the software.

10.0 POLLUTION CONTROL

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

10.1 Waste Management

Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to UP-WM-001. The following waste streams are produced when this method is carried out.

- Waste from this procedure will enter the "Corrosive Wastewater" waste stream.

11.0 METHOD PERFORMANCE CRITERIA

Refer to Sections 1.0, 7.0 and 8.0.

12.0 REFERENCES

Refer to Section 1.0.

13.0 ATTACHMENTS

Table 1. Element and Reporting Limits
Attachment 1(A&B) Calibration Stock Solutions
Attachment 2. Stock QC Solutions
Attachment 3. Example: Analysis Run Log / Maintenance Log
Attachment 4. Example: Data Review Checklist
Attachment 5. Known Digested Quality Control
Appendix A: Requirements for DOD QSM Version 4.2 – Table F-1 and Table F-6

14.0 REVISION HISTORY

- Revision 02 updated on 09/27/11
- Added note about auto-IEC function on ICP6 to Section 9.5

Table 1
Element and Reporting Limits

Element	ICAP 61E (ICP5) Wavelength (nm)	ICAP 6500 (ICP6) Wavelength (nm)	Reporting Limit (RL)[1] (ug/L)	Soils (mg/kg)
Al	308.2	308.2	200	20.0
Sb	206.8	206.8	20	2.0
As	189.0	189.0	10	1.0
Ba	493.4	455.4	10	1.0
Be	313.0	234.8	4	0.40
Bi	N/A	223.0	20	2.0
B	249.6	208.9	50	5.0
Ca	317.9	317.9	200	20.0
Cd	226.5	228.8	2	0.20
Cr	267.7	267.7	10	1.0
Co	228.6	228.6	5	0.50
Cu	324.7	324.7	10	1.0
Fe	271.4	271.4	200	20.0
Pb	220.3	220.3	5	0.5
Mg	279.0	279.0	100	10.0
Mn	257.6	257.6	10	1.0
Mo	202.0	202.0	10	1.0
Ni	231.6	231.6	10	1.0
K	766.4	766.4	500	50.0
Se	196.0	196.0	10	1.0
Si	288.1	212.4	200	20.0
Ag	328.0	328.0	5	0.50
Na	588.9	589.5	1,000	100
Sr	421.5	421.5	5	0.50
Tl	190.8	190.8	10	1.0
Sn	189.9	189.9	40	4.0
Ti	334.9	334.9	5	0.50
V	292.4	292.4	5	0.50
Zn	206.2	206.2	20	2.0
Y[3]	371.0	224.3/360.0/371.0	N/A	N/A
In[3]	N/A	230.6	N/A	N/A

[1] These are routine ICAP reporting limits (RL). Lower RLs may be available to use per client request. Sample RLs will vary depending on sample volume, dilution factors, and changes in MDLs. Contact the laboratory for the most current RLs based on annual MDL determinations.
[3] Y, and In on the iCAP 6500, are used as internal standards and are introduced continuously to all samples (including standards and QC samples) via the peristaltic pump at an approximate concentration of 5 ppm.

Attachment 1A.

Trace 61E Calibration Stock Standard Solutions

Vendor	Stock Name	Element	Conc. (mg/L)	S1A	S1B	S1	S2A	S2B	S2
Inorganic Ventures	RFW-ICPT-STD-1B	Sb	100	0.4	0.5	1.0			
		Mo	100	0.4	0.5	1.0			
		Si	100	0.4	0.5	1.0			
		Sn	100	0.4	0.5	1.0			
		Ti	100	0.4	0.5	1.0			
Inorganic Ventures	RFW-ICPT-STD-1C	Al	1,000	---	5	10			
		Fe	1,000	---	5	10			
		K	1,000	---	---	---			
		Na	1,000	---	---	---			
		Li	800	3.2	4	8			
		Mg	800	---	4	8			
		Ca	400	---	---	---			
Inorganic Ventures	RFW-ICPT-STD-1D	As	100	0.4	0.5	1.0			
		Ba	100	0.4	0.5	1.0			
		Be	100	0.4	0.5	1.0			
		Bi	100	0.4	0.5	1.0			
		B	100	0.4	0.5	1.0			
		Cd	100	0.4	0.5	1.0			
		Cr	100	0.4	0.5	1.0			
		Co	100	0.4	0.5	1.0			
		Cu	100	0.4	0.5	1.0			
		Pb	100	0.4	0.5	1.0			
		Ni	100	0.4	0.5	1.0			
		Se	100	0.4	0.5	1.0			
		Ag	100	0.4	0.5	1.0			
		Sr	100	0.4	0.5	1.0			
		Tl	100	0.4	0.5	1.0			
		Zn	100	0.4	0.5	1.0			
Inorganic Ventures	RFW-ICPT-STD-2A	Al	10,000				40	50	100
		K	10,000				40	50	100
Inorganic Ventures	RFW-ICPT-STD-2B	Ca	5,000				20	25	50
		Fe	5,000				20	25	50
		Mg	5,000				20	25	50
		Na	5,000				20	25	50
Inorganic Ventures	RFW-ICPT-STD-3	Pb	2,000				8	10	20
		Mn	1,000				4	5	10
		V	1,000				4	5	10

Attachment 1B.

iCAP 6500 Calibration Stock Standard Solutions

Vendor	Stock Name	Element	Conc. (mg/L)	S1 mg/L	S2 mg/L
Inorganic Ventures	STDIL-STD-2	Sb	100	1	
		Mo	100	1	
		Sn	100	1	
		Ti	100	1	
Inorganic Ventures	STDIL-STD-1	As	100	1	
		Ba	100	1	
		Be	100	1	
		B-	100	1	
		Cd	100	1	
		Co	100	1	
		Cr	100	1	
		Cu	100	1	
		Mn	100	1	
		Ni	100	1	
		Pb	100	1	
		Se	100	1	
		Sr	100	1	
		Tl	100	1	
		V-	100	1	
		Zn	100	1	
Inorganic Ventures	CGBI1-1	Bi	1000	1	
	CGSI1-1	Si	1000	1	
	CGAG1-1	Ag	1000	1	
Inorganic Ventures	RFW-ICPT-STD-2A	Al	10,000		100
		K	10,000		100
Inorganic Ventures	RFW-ICPT-STD-2B	Ca	5,000		100
		Fe	5,000		100
		Mg	5,000		100
		Na	5,000		100

Note: Vendors and stock names may vary.

Attachment 2.

Example of Stock QC Solutions

Vendor	Stock Name	Element	Conc. (mg/L)	ICV (mg/L)	CCV (mg/L)
High Purity	CCV Solution A	As	50	0.4	0.5
		B	50	0.4	0.5
		Ba	50	0.4	0.5
		Be	50	0.4	0.5
		Bi	50	0.4	0.5
		Cd	50	0.4	0.5
		Co	50	0.4	0.5
		Cr	50	0.4	0.5
		Cu	50	0.4	0.5
		Ni	50	0.4	0.5
		Pb	50	0.4	0.5
		Se	50	0.4	0.5
		Fe	500	20	25
		Mn	500	4	5
		V	500	4	5
		Tl	50	0.4	0.5
		Zn	50	0.4	0.5
		Sr	50	0.4	0.5
High Purity	CCV Solution A1	Ca	200	20	25
		Li	400	---	---
		Na	500	20	25
		Al	500	40	50
		Mg	400	20	25
		K	500	40	50
High Purity	CCV Solution B	Ag	50	0.4	0.5
		Sb	50	0.4	0.5
		Mo	50	0.4	0.5
		Si	50	0.4	0.5
		Sn	50	0.4	0.5
		Ti	50	0.4	0.5
Ultra	Single Elements * spiked on top of custom mixes.	Al	10,000	40	50
		Ca	10,000	20	25
		Fe	10,000	20	25
		Na	10,000	20	25
		K	10,000	40	50
		Mg	10,000	20	25

Attachment 2.
(continued)
Examples of Stock QC Solutions

Vendor	Stock Name	Element	Conc. (mg/L)	Daily LR DoD (mg/L)
Inorganic	Aluminum	Al	*See ICSA*[1]	500 (ICSA)[1]
Ventures	Antimony	Sb	1,000	10
	Arsenic	As	1,000	5
	Barium	Ba	1,000	10
	Beryllium	Be	1,000	5
	Bismuth[2]	Bi	1,000	5
	Boron	B	1,000	10
	Cadmium	Cd	1,000	5
	Calcium	Ca	*See ICSA*[1]	500 (ICSA)[1]
	Chromium	Cr	1,000	10
	Cobalt	Co	1,000	10
	Copper	Cu	1,000	10
	Iron	Fe	*See ICSA*[1]	200 (ICSA)[1]
	Lead	Pb	1,000	10
	Magnesium	Mg	*See ICSA*[1]	500 (ICSA)[1]
	Manganese	Mn	1,000	10
	Molybdenum	Mo	1,000	10
	Nickel	Ni	1,000	10
	Potassium	K	*See S2*[1]*/10,000*	100 (S2)[1]/500
	Selenium	Se	1,000	5
	Silicon	Si	1,000	10
	Silver	Ag	*See S1*[1]	1 (S1)[1]
	Sodium	Na	*See S2*/10,000[3]	50 (S2)[1]/400[3]
	Strontium	Sr	*See S1*[1]	1 (S1)[1]
	Thallium	Tl	1,000	5
	Tin	Sn	1,000	5
	Titanium	Ti	1,000	5
	Vanadium	V	1,000	10
	Zinc	Zn	1,000	10

[1] The ICSA is used for the daily LR verification for Al, Ca, Fe, and Mg. The calibration read-back (S1) is used for the daily LR for Ag and Sr. The calibration read-back (S2) is used for the daily LR for K (optionally) and Na on the Trace (ICP5) and for K only on the iCAP 6500 (ICP6).
[2] Bismuth is run on ICP6 only.
[3] Na has a higher linear range on ICP6. The S2 read-back is used for the daily LR on ICP5, but a 400 mg/L is made for ICP6.

Attachment 2.
(continued)
Examples of Stock QC Solutions

Vendor	Stock Name	Element	Conc. (mg/L)	ICVL/CCVLConc. (mg/L)	CRI (mg/L)
Inorganic	TACHI-2	Al	200	0.200	0.400
Ventures	TACHI-1	Sb	20	0.020	0.040
	TACHI-2	As	10	0.010	0.020
	TACHI-2	Ba	10	0.010	0.020
	TACHI-2	Be	4	0.0040	0.008
	Bi Stock	Bi	1,000	0.020	0.040
	TACHI-2	B	50	0.050	0.100
	TACHI-2	Cd	2	0.0020	0.0040
	TACHI-2	Ca	200	0.200	0.400
	TACHI-2	Cr	10	0.010	0.020
	TACHI-2	Co	5	0.0050	0.010
	TACHI-2	Cu	10	0.010	0.020
	TACHI-2	Fe	200	0.20	0.40
	TACHI-2	Pb	5	0.0050	0.010
	TACHI-2	Mg	100	0.10	0.20
	TACHI-2	Mn	10	0.010	0.020
	TACHI-1	Mo	10	0.010	0.020
	TACHI-2	Ni	10	0.010	0.020
	TACHI-2	K	500	0.500	1.00
	TACHI-2	Se	10	0.010	0.020
	TACHI-1	Si	200	0.20	0.40
	TACHI-2	Ag	5	0.0050	0.010
	TACHI-2	Na	1000	1.00	2.00
	TACHI-2	Sr	5	0.0050	0.010
	TACHI-2	Tl	10	0.010	0.020
	TACHI-1	Sn	40	0.040	0.080
	TACHI-1	Ti	5	0.0050	0.010
	TACHI-2	V	5	0.0050	0.010
	TACHI-2	Zn	20	0.020	0.040

Attachment 2.
(continued)
Stock QC Solutions

Vendor	Stock Name	Element	Conc. (mg/L)	ICSA Conc. (mg/L)
Inorganic Ventures	CLP Interferent "A" Solution	Al	5,000	500
		Ca	5,000	500
		Mg	5,000	500
		Fe	2,000	200
				ICSAB Conc. (mg/L)
Inorganic Ventures	CLP Interferent "A" Solution	Al	5,000	500
		Ca	5,000	500
		Mg	5,000	500
		Fe	2,000	200
Inorganic Ventures	CLPP-ICS-B4	Cd	100	1
		Ni	100	1
		Zn	100	1
		Sb	60	0.6
		Ba	50	0.5
		Be	50	0.5
		Co	50	0.5
		Cr	50	0.5
		Cu	50	0.5
		Mn	50	0.5
		V	50	0.5
		Ag	20	0.2
		As, Tl	10	0.1
		Pb, Se	5	0.05

Attachment 3.

Example: Analysis Runlog / Maintenance Log
(026-001 to 026-002)

TestAmerica Chicago

TJA ICAP (6500 DUO) Analysis Log – ICP6

Note: Calibration standards are prepared daily per Method SOPs

Page No._____

Date	Initials	File Name	Dig. Set	Int. Std	Sample Nos.	Parameters	Comments
				Y =			
				Y =			
				Y =			
				Y =			
				Y =			
				Y =			
				Y =			
				Y =			
				Y =			
				Y =			
				Y =			
				Y =			
				Y =			
				Y =			
				Y =			
				Y =			
				Y =			
				Y =			
				Y =			
				Y =			
				Y =			

Reviewed by:_____ Date:_____

CHI-22-14-081/A-12/08

(C-2) C-320

(Sab-001)

TestAmerica Chicago
TJA ICAP 6500 DUO – ICP6
Instrument Maintenance Log

Page No._____

	Date/Initials	Date/Initials	Date/Initials	Date/Initials	Date/Initials	Date/Initials	Date/Initials
Daily Maintenance:							
Check/Change Pump Tubing							
Check Waste Container							
Check Torch for buildup (Note Cleaning)							

Monthly Maintenance:							
Check/Refill Recirculator							
Check Nebulizer/Spray Chamber							

Comments/Return to Control Documentation:

_____

_____

_____

_____

_____

Any Maintenance/Repair/Part Replacement performed that is not listed above must be documented in the Comments sections

Reviewer Signature:_____ Date:_____

CHI-22-14-082/A-12/08

(C-2) C-321

(2006-002)

Attachment 4.

Example: Data Review Checklist
(027-001 to 027-002)

TestAmerica Chicago
ICAP Metals Data Review Checklist

Instrument ID: ICP 5 ICP6 **Filename:**_____

Analyst Initial(s):_____ **LIMS Batch No.:**_____

Copies: _____

_____

****

Method (circle): a. **EPA 200.7** b. **SW-846 6010B** c. **SW-846 6010C** d. **CLP ILM04.0**
Matrix (circle): a. Drinking Water b. Non-potable water c. Soil/Sediment/Waste d. Other_____
Circle: a. Total b. Solubles **QC Type (circle):** a. CLP b. Standard c. DoD QSM d. Other

I. Calibration:

Analyst	Reviewer		NCM #
☐	☐	1. Verification of standard traceability and expiration (daily).	
		2. Calibration is clearly documented:	
☐	☐	a. Instrument is calibrated using a Blank and three Calibration Standards. The correlation coefficient must be ≥0.995 (6010B); ≥ 0.998 (6010C) | y-intercept | ≤ RL; (≤ ½ RL DoD QSM)	
☐	☐	b. Reanalysis of the top calibration standard as a sample. Control limits are 95 - 105%. (Run once daily prior to sample analysis).	
		3. Calibration Verification: (10% Frequency):	
☐	☐	a. ICV/CCV: **6010B/6010C/CLP** – 90-110% **EPA 200.7 (ICV)** – 95-105% ICVL/CCVL: **6010C** - 70 – 130%	
☐	☐	b. ICB/CCB: **Std. QC:** < RL; **CLP QC:** < CRDL; **SW-846 QC:** < 3x MDL. **AFCEE / DoD QSM**: <½ RL	
		4. **CLP QC:** Initial & Final each run:	
☐	☐	a. CRI - 2x RL; No Limit Set	
☐	☐	b. ICSA/ICSAB - 80-120%	
		5. **Std. QC:** Analyzed at the beginning of the day and every 8 hours thereafter:	
☐	☐	a. CRI: 2x CRDL; No Limit Set	
☐	☐	b. ICSA/ICSAB: 80-120%	
☐	☐	6a. MRL: **DoD QSM / AFCEE** 80-120% or per QAPP **6010C:** 10% frequency, 70-130% 6b. DLCK: **LCG** only 10 – 190%	

II. Sample Analysis:

Analyst	Reviewer		NCM #
		1. Each Prep Batch consists of a maximum of 20 samples of a similar matrix:	
☐	☐	a. Prep Batches must be clearly identified	
☐	☐	b. 1 Prep Blank **CLP** - < CRDL; **Std. QC** - < RL **TCLP** - < TCLP RL **AFCEE/DoD QSM** <½ RL	
☐	☐	c. 1 LCS **Std./CLP** - 80-120% Rec.; **EPA 200.7** - 85-115% Rec.	
☐	☐	d. 1 duplicate **Std.** - RPD or RSD limits are 20%; ***Unless*** the sample conc. is <5x RL the ± CRDL applies. EPA 200.7 – 10% Frequency	
☐	☐	e. 1 Matrix Spike **Std./CLP** 75-125% unless sample concentration exceeds MS conc. by 4x; **200.7** – 70-130% - 10% frequency; **DoD QSM** – 80-120%	
☐	☐	f. Analytical MS **TCLP** - >50% (MSA performed if <50% recovery)	
☐	☐	g. Serial Dilution 1 per 20 samples; 10% Difference Limit	
☐	☐	h. A post-digestion spike (PDS) must be performed for **CLP** (75-125%) and **200.7** (85-115%) if the above limits are not met, (CLP - except for Ag, Na, Ca, K, and Mg for waters and soils, and Al and Fe for soils only). Program specific criteria may apply.	
☐	☐	i. Turbidity Checked: **EPA 200.7 Drinking Water** (< 1 NTU; no prep required).	
☐	☐	j. **EPA 200.7/200.8**: Sample dilution performed for analytes > 90% of linear range. Sample dilution is performed for analytes or interferents > Linear Range Check	

TestAmerica Chicago
ICAP Metals Data Review Checklist

III. Data Documentation

Analyst	Reviewer	
		1. Raw Data:
		a. Unused data is clearly identified.
		b. All crossed out data is initialed and dated.
		c. Out of control QC is clearly identified.
		d. Any data that has a tick is commented on with appropriate action taken.
		e. The first page of the run must have the filename; instrument; and analyst's signature
		2. Run Log:
		a. Unused data is clearly identified.
		b. All cross outs are initialed and dated.
		c. Analyst's Signature is required.

IV. LIMS

Analyst	Reviewer	
		1. Samples Tab:
		a. LIMs Sample IDs/Containers are correct.
		b. Method and Matrix are correct.
		c. Date and Time match raw data.
		d. Dilutions are correct.
		e. Correct suffix designated (where applicable).
		2. Worksheet Tab is complete and correct.
		3. Reagent Tab is complete and correct.
		4. QC Links Tab is correct.
		5. Sample Results Tab:
		a. All unused data are designated Rejected or Acceptable.
		b. All reported analytes are designated Primary or Secondary.
		6. Batch Information Screen: Documentation is complete.
1st	2nd	7. Status set appropriate to review level.

Comments:

_____

_____

_____

_____

Analyst Signature: _____ Date: _____

Reviewer Signature: _____ Date: _____

Attachment 5.

Known Digested QC Values (mg/L)

Element	LCS/Matrix Spike	TCLP Spike
Al	2	---
Sb	0.5	---
As	0.1	5
Ba	2	100
Be	0.05	---
Bi	0.5	---
B	1	---
Cd	0.05	1
Ca	10	---
Cr	0.2	5
Co	0.5	---
Cu	0.25	0.25
Fe	1	---
Pb	0.10	5
Mg	10	---
Mn	0.5	---
Mo	1	---
Ni	0.5	0.5
P	0.5	---
K	10	---
Se	0.10	1
Si	5	---
Ag	0.05	1
Na	10	---
Sr	1	---
Tl	0.10	---
Sn	1	---
Ti	1	---
V	0.5	---
Zn	0.5	---

Default Control Limits
LCS: 80 - 120%
Matrix Spike: 75 - 125%
TCLP Spike: >50%

Appendix A:

Requirements for DOD QSM Version 4.2
Table F-1 and Table F-6
(029-001 to 029-005)

TestAmerica Chicago

DoD QSM Version 4.2: Appendix F - Quality Control Requirements Summary

Table F-1 Summary of QC Check Definitions, Purpose, and Evaluation – Inorganics (Metals)

QC Check	Definition	Purpose	Evaluation
Calibration Blank	Reagent water containing no analytes of interest.	To determine the zero point of the calibration curve for all initial and continuing calibrations.	This is a required QC procedure. Continuing calibration blank responses above the LOD require corrective action
Continuing calibration verification (CCV)	This verification of the ICAL that is required during the course of analysis at periodic intervals. Continuing calibration verification applies to both external standard and internal standard calibration techniques, as well as to linear and non-linear calibration models	To verify that instrument response is reliable, and has not changed significantly from the current ICAL curve.	If the values for the analytes are outside the acceptance criteria, the ICAL may not be stable. Results associated with out-of-control CCV results require reanalysis or flagging
Demonstrate Acceptable Analytical Capability	QC samples are analyzed in series to verify ability to produce data of acceptable precision and bias.	To verify the ability to produce data of acceptable precision and bias for a specific instrument type, matrix, method, and analyst.	The average recovery of the spikes and standard deviation of the replicates must be within designated acceptance criteria. Analysis of field samples may not be conducted until this check is successful.
Dilution test (Metals only)	Analysis of a positive sample, which has been diluted to a concentration 1/5th of the original, to confirm that there is no interference in the original sample analysis. (Modified COE)	To assess matrix interference	Agreement within 10% between the concentration for the undiluted sample and 5x the concentration for the diluted sample indicates the absence of interferences, and such samples may be analyzed without using the MSA. Results outside acceptance limits indicate a possible matrix effect. For ICP a post-digestion spike must be run.
Duplicate Sample (replicate)	Two identical portions of material collected for chemical analysis, and identified by unique alphanumeric codes. The duplicate may be portioned from the same sample, or may be two identical samples taken from the same site. The two portions are prepared and analyzed identically. (modified QSM)	To provide information on the heterogeneity of the sample matrix or to determine the precision of the intralaboratory analytical process for a specific sample matrix	A duplicate sample will provide information on the heterogeneity of the sample matrix. The greater the heterogeneity of the matrix, the greater the relative percent difference between the sample and the sample duplicate. If the sample matrix is homogeneous (such as with drinking water) and the relative percent difference is high, this could indicate a problem in the analytical system.
Initial calibration for all analytes (ICAL)	Analysis of analytical standards at different concentrations that are used to determine and calibrate the quantitation range of the response of the analytical detector or method	To establish a calibration curve for the quantification of the analytes of interest	Statistical procedures are used to determine the relationship between the signal response and the known concentration of analytes of interest. The ICAL must be successful before any samples or other QC check samples can be analyzed.
Instrument Detection Limit (IDL) Study (6010 and 6020 only)	The process to determine the minimum concentration of a substance (analyte) that an instrument can differentiate from noise. The procedure for calculating varies by method.	To provide evaluation of instrument sensitivity	IDLs must be established before samples can be analyzed.
Interference check solutions (ICP and ICP/MS only)	A pair of solutions containing interfering elements that are used to verify the correction factors of analytes of concern.	To verify the established correction factors by analyzing the interference check solution at the beginning of the analytical sequence	No samples can be run if this check does not pass acceptance criteria.
Internal Standards	A substance that is introduced in known amount into each calibration standard and field and QC sample of the analyte.	The ratio of the analyte signal to the internal standard signal is then used to determine the analyte concentration.	Any sample associated with out-of-control results must be reanalyzed.
Laboratory control sample (LCS) containing all analytes to be reported	A sample matrix, free from the analytes of interest, spiked with known amounts of analytes or a material containing known and verified amounts of analytes.	Used to evaluate the performance of the total analytical system, including all preparation and analysis steps. Assesses the ability of the laboratory/analyst to successfully recover the target analytes from a control (clean) matrix. Control limits for LCS recovery, typically expressed as percent recovery, are used for the development of statistical control limits and serve as acceptance criteria for determining whether an analytical run is in control (batch acceptance).	This is a required QC Check. The inability to achieve acceptable recoveries in the LCS indicate problems with the precision and bias of the measurement system. Failure to achieve acceptable recoveries in a "clean" matrix is an indicator of possible problems achieving acceptable recoveries in field samples.

(100-620)

F-1 (cont.)			
QC Check	**Definition**	**Purpose**	**Evaluation**
Linear dynamic range or high-level check standards (ICP and ICP.MS only)	High-level check standard periodically analyzed to verify the linearity of the calibration curve at the upper end.	To verify quantitative accuracy of data up to the high=level standard.	The QC check establishes the upper linear range of the calibration.
Low-Level calibration check standard (ICP only)	A reference standard that contains a quantity of analyte equal to or less than the reporting limit.	To confirm the accuracy of measurements at or near the RL.	This QC check must be within acceptance criteria before any samples are analyzed.
Matrix Spike (MS)	A sample prepared by adding a know mass of target analyte to a specified amount of matrix sample for which an independent estimate of target analyte concentration is available.	To assess the performance of the method as applied to a particular matrix. Matrix spikes are used, for example, to determine the effect of the matrix on a method's recovery efficiency. The recovery of target analytes from the matrix spike sample is used to determine the bias of the method in the specific sample matrix.	The lack of acceptable recoveries in the matrix spike often points to problems with the sample matrix. One test of this is a comparison to the LCS recoveries. If the corresponding LCS recoveries are within acceptable limits, a matrix effect is likely. The lab should not correct for recovery; only report the results of the analyses and the associated MS results and indicate that the results from these analyses have increased uncertainty
Matrix Spike Duplicate (MSD)	A second replicate matrix spike prepared in the laboratory and analyzed to obtain a measure of the precision of recovery for each analyte	To assess the performance of the method as applied to a particular matrix and provide information on the homogeneity of the matrix. Also used to determine the precision of the intralaboratory analytical process for a specific sample matrix.	When compared to the MS, the MSD will provide information on the heterogeneity of the sample matrix. The greater the heterogeneity of the matrix, the greater the RPD between the matrix spike and the matrix spike duplicate. If the sample matrix is homogeneous, such as with drinking water, and the RPD is high, this could indicate a problem in the analytical system.
MB	A sample of a matrix similar to the batch of associated samples (when available) that is free from the analytes of interest and is processed simultaneously with an under the same conditions as samples through all steps of the analytical procedures, and in which no target analytes or interferences are present at concentrations that impact the analytical results for sample analyses.	To assess background interferences or contamination in the analytical system that might lead to high bias or false positive data. Results of method blanks provide an estimate of the within-batch variability of the blank response and an indication of bias introduced by the preparation and analytical procedure.	This is one of the QC samples used to measure lab accuracy/bias. The sample could indicate whether contamination is occurring during sample prep and analysis. If analytes are detected > ½ RL, reanalyze or qualify (B-flag) all results for the specific analytes(s) in all samples in the associated prep batch as appropriate. For common lab contaminants, no analytes detected > RL. See Section D.1.1.1 and Box D-1
MSA (ICP only)	A set of procedures adding one or more increments of a standard solution to sample aliquots of the same size in order to overcome inherent matrix effects. The procedures encompass the extrapolation back to obtain the sample concentration. (This process is also called spiking the sample.)	To compensate for a sample constituent that enhances or depresses the analyte signal, thus producing a different slope from that of the calibration standards. It will not correct for additive interferences that cause a baseline shift.	This is the method used when matrix interferences are present and do not allow determination of accurate sample results
Post digestion spike addition (ICP and ICP/MS only)	An analyte spike added to a portion of prepared sample to verify absence or presence of matrix effects	To confirm the presence of a matrix interference. Assess matrix effects based on: 1. the occurrence of new and unusual matrices included within the batch, or 2. contingency analysis based on SD or MS failures	To verify the absence of an interference, the spike recovery must be between 75%-125% Results outside the acceptance limits require MSA for all samples within the batch
Second source calibration verification (ICV)	A standard obtained or prepared from a source independent of the source of standards for the ICAL. Its concentration should be at or near the middle of the calibration range. It is done after the ICAL.	To verify the accuracy of the ICAL.	The concentration of the 2nd source calibration verification, determined from the analysis, is compared with the known value of the standard to determine the accuracy of the ICAL. This independent verification of the ICAL must be acceptable before sample analysis can begin.

Notes:
1. Project-specific requirements identified by the client supersede any requirements listed. The requirements are meant to be default, to be used when project-specific direction based on DQOs is not available.
2. If there is a contradiction between the method and the DoD tables, the requirements specified in the tables shall be followed.

(0229-002)

TestAmerica Chicago

DoD QSM Version 4.2: Appendix F - Quality Control Requirements Summary

Table F-7: Inorganic Analysis by ICP and CVAA - Methods 6010 and 7000 Series

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Demonstrate acceptable analytical capability	Prior to using any test method and at any time there is a significant change in instrument type, personnel, test method, or sample matrix.	QC acceptance criteria published by DoD, if available; otherwise method- specified criteria.	Recalculate results; locate and fix problem, then rerun demonstration for those analytes that did not meet criteria (see Section C.1.f).	NA	This is a demonstration of analytical ability to generate acceptable precision and bias per the procedure in Appendix C. No analysis shall be allowed by analyst until successful demonstration of capability is complete.
LOD Determination and verification (See Box D-13)					
LOQ Establishment and verification (See Box D-14)					
Instrument detection limit (IDL) study (ICP only)	At initial set-up and after significant change in instrument type, personnel, test method, or sample matrix.	IDLs shall be $\leq$ LOD	NA	NA	Samples may not be analyzed without a valid IDL
Linear dynamic range (LRS) or high-level check standard (ICP only)	Every 6 months	Within $\pm$ 10% of expected value	NA	NA	
Initial Calibration (ICAL) for all analytes ICP: min 1 high std and a Calib.Blank CVAA: min 5 stds and a Calib. Blank	Daily ICAL prior to sample analysis	If more than one calibration standard is used $r \geq 0.995$.	Correct problem then repeat ICAL.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until ICAL has passed.
2nd Source calibration verification (ICV)	Once after each ICAL, prior to beginning a sample run.	Value of 2nd source for all analyte(s) within $\pm$ 10% of true value.	Correct problem and verify 2nd source standard. Rerun ICV. If that fails, correct problem and repeat ICAL.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until calibration has been verified.
Continuing Calibration verification (CCV)	After every 10 field samples and at the end of analysis sequence.	ICP: within $\pm$ 10% of true value. CVAA: within $\pm$ 20% of true value.	Correct problem, rerun CCV. If that fails, then repeat ICAL. Reanalyze all samples since the last successful CCV.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to all results for the specific analyte(s) in all samples since the last acceptable calibration verification.	Problem must be corrected. Results may not be reported without a valid CCV. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Low-level calibration check standard (ICP only)	Daily, after one-point ICAL.	Within $\pm$ 20% of true value	Correct problem, then reanalyze.	Flagging criteria are not appropriate.	No samples may be analyzed without a valid low-level calibration check standard. Low-level calibration check standard should be less than or equal to the reporting limit.

(DA9-003)

F-7 (cont.)					
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action**	**Flagging Criteria**	**Comments**
Method Blank (MB)	One per prep batch	No analytes detected > ½ RL and greater than 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater). Blank results must not otherwise affect sample results. For common lab contaminants, no analytes detected >RL (see Box D-1)	Correct problem, then see criteria in box D-1; If required, reprep and reanalyze MB and all samples processed with the contaminated blank.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply B-flag to all results for the specific analyte(s) in all samples in the associated prep batch.	Problem must be corrected. Results may not be reported without a valid method blank. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Calibration Blank (ICB / CCB)	Before beginning a sample run, after every 10 samples, and at the end of the analysis sequence	No analytes detected > LOD.	Correct problem. Reprep and reanalyze calibration blank. All samples following the last acceptable calibration blank must be reanalyzed.	Apply B-flag to all results for the specific analyte(s) in all samples associated with the blank.	
Interference check solutions (ICS) (ICP only)	At the beginning of an analytical run.	ICS-A: Absolute value of concentration for all non-spiked analytes < LOD (unless they are verified trace impurity from one of the spiked analytes) ICS-AB: ± 20% of true value.	Terminate analysis; locate and correct problem; reanalyze ICS, reanalyze all samples.	If corrective action fails, apply Q-flag to all results for specific analyte(s) in all samples associated with the ICS.	
LCS containing all analytes to be reported	One per prep batch	QC acceptance criteria specified by DoD, if available; see Box D-3 and Appendix G.	Correct problem., then reprep and reanalyze the LCS and all samples in the associated prep batch for failed analytes, if sufficient sample material is available (see full explanation in Appendix G).	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to specific analyte(s) in the associated prep batch.	Problem must be corrected. Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Matrix Spike (MS)	One per prep batch per matrix (see Box D-7)	For matrix evaluation, use QC acceptance criteria specified by DoD for LCS	Examine the project-specific DQOs. If the matrix spike fails outside of DoD criteria, additional quality control tests are required to evaluate matrix effects.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met.	For matrix evaluation only. If MS results are outside the LCS limits, the data shall be evaluated to determine the source of difference and to determine if there is a matrix effect or analytical error.
Matrix Spike Duplicate (MSD) or Sample Duplicate	One per prep batch per matrix (see Box D-7)	MSD: For matrix evaluation use QC acceptance criteria specified by DoD for LCS. MSD or sample duplicate: RPD ≤ 20% (between MS and MSD or sample and sample duplicate)	Examine the project-specific DQOs. Contact client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met.	The data shall be evaluated to determine the source of difference.
Dilution Test (ICP only)	One per prep batch .	Five-fold dilution must agree within ± 10% of the original measurement.	ICP: Perform post-digestion spike (PDS) addition.	Flagging criteria are not appropriate.	Only applicable for samples with concentrations > 50x LOQ.
Post-digestion spike (PDS) addition (ICP only)	When dilution test fails or analyte concentration in all samples < 50 x LOD	Recovery within 75-125% (see Table B-1)	Run all associated samples in the prep batch by MSA or see flagging criteria.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met.	Spike addition should produce a concentration of 10 – 100 x LOQ.

F-7 (cont.)					
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action**	**Flagging Criteria**	**Comments**
Method of Standard Addition (MSA)	When matrix interference is confirmed.	NA	NA	NA	Document use of MSA in the case narrative.
Results reported between DL and LOQ	NA	NA	NA	Apply J-flag to all results between LOD and LOQ	

Notes:
1. Project-specific requirements identified by the client supersede any requirements listed. The requirements are meant to be default, to be used when project-specific direction based on DQOs is not available.
2. If there is a contradiction between the method and the DoD tables, the requirements specified in the tables shall be followed.

(09-005)

TITLE: Gas Chromatography Mass Spectrometry - Volatiles SW-846 Method 8260B

Approvals (Signature/Date):

JoAnn Petruszak-Kmetty 8-1-11 JoAnn Petruszak-Kmetty Date Supervisor, GC/MS VOA	8-1-11 John D. Nagel Date Env. Health & Safety Coor.
Terese A. Preston 8/1/11 Terese A. Preston Date Quality Assurance Manager	Michael J. Healy 8/1/11 Michael J. Healy Date Laboratory Director

Facility Distribution No: _____ **Distributed To:** Uncontrolled

1.0 SCOPE / APPLICATION

To outline the guidelines for the analysis of Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS) using SW-846 Methods 8260B and 8000B as references. The preparation of all volatile samples is based on Methods 8000B and 5030B. Method 5035 is covered by a separate SOP (UP-SP-5035), but can also be found in this SOP.

On occasion, clients request slight modifications to this SOP. These modifications are addressed on a case-by-case basis with the range of accuracy (i.e., MDLs, linearity check or PT sample) verified prior to implementation. Any modifications would be written into a Quality Assurance Plan (QAP), authorized via laboratory signature approval, and mentioned in the data package's case narrative.

Specific requirements pertaining to the DOD QSM Version 4.2 are located in Attachment 10. These requirements are additionally applicable to all NFESC projects. Any deviations from these procedures and/or variances from must be addressed appropriately in accordance with standard operating protocol and pre-approved on a project by project basis.

1.1 Method Sensitivity

1.1.1 Method Detection Limits

The method detection limit (MDL), referred to as the detection limit (DL) in NELAC and DOD QSM documents, is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to Appendix B of 40 CFR 136, "Guidelines Establishing Test Procedures for the Analysis of Pollutants" with additional details are provided in the TestAmerica Corporate SOP, *CA-QS-006, Detection Limits* and the TestAmerica Chicago SOP, *UP-QA-017, Method Detection Limit Studies.* MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; the MDL will be verified on a quarterly basis to meet the requirements of the DoD QSM version 4.2.

1.1.2 Demonstration of Capability

Before the laboratory may institute a new method and begin reporting results, the laboratory shall confirm that it can properly operate the method. A demonstration of capability is performed whenever there is a change in instrument type, method or personnel. An Initial Demonstration of Capability (IDOC) must be thoroughly documented and approved by the Department Manager/Supervisor and QA Manager prior to independently analyzing client samples. All associated documentation must be retained in the QA Department and in the Analyst Training files. For additional details on the demonstration of capability procedures followed, refer to the laboratory SOP, *UP-QA-QAM, Quality Assurance Manual, Sections 20.4.2 and 20.4.3.*

1.1.3 Reporting Limits

Reporting Limits [a.k.a., Estimated Quantitation Limits (EQLs) as designated in the method] are defined as the lowest concentration of an analyte determined by a given method in a given matrix that the laboratory feels can be reported with acceptable quantitative error or client requirements, values specified by the EPA methods or other project and client requirements. The laboratory maintains reporting limits that are higher than the MDL. Wherever possible, reporting is limited to values approximately 3-5x the respective MDL to ensure confidence in the value reported.

Table 1 defines the reporting limits and analyte list for SW-846 Method 8260B.

1.1.4 Definitions

Refer to Section 3.0 of the Laboratory's Quality Assurance Manual (UP-QA-QAM).

1.2 Summary of Method

This method is used to determine volatile organic compounds in a variety of matrices. It is applicable to water, soil, sediment, sludge and waste drum samples.

This method can be used to quantify most volatile organic compounds that have a boiling point less than 200°F. It is also limited to those compounds that elute as sharp peaks from a capillary column. A listing of applicable compounds and their characteristic ions appears in Table 2.

A portion of sample, measured into a sample vessel, is purged with an inert gas. The volatile compounds are transferred to a trap, containing retarding materials. The trap is then backflushed with the inert gas and rapidly heated to effectively transfer the compounds to the GC column. The GC oven is then, temperature ramped to separate the compounds and introduce them to the source. The mass filter separates the ions, which are then detected by the analyzer. The data system then provides qualitative and quantitative information concerning the sample.

Instrument calibration occurs about every 12-hours, or prior to analysis. Instrument maintenance is performed as needed or daily basis.

2.0 INTERFERENCES

1. External interferences can be caused by contaminants from sample containers, preparative glassware and reagents, syringes and columns and manifest themselves as high background and/or discrete peaks. Some contaminants are also introduced through the sample vial seal and/or instrument sample connections. Proper glassware preparation including rinsing of all volumetric glassware and syringes, baking of syringes and proper sample handling and instrument maintenance should eliminate these sources. A laboratory method blank (MB) is analyzed prior to any analysis to show absence of any contaminants. Reagent (Milli-Q) water sampled in the lab and carried through all field operations is also analyzed to show absence of contaminants from field sampling.

2. Carryover is also another source of contamination. Any time a high-level sample is analyzed, the next sample in the batch is checked for carryover. If carryover is suspected, that sample is re-analyzed. If the carryover is excessive and continues into the next samples, the batch is aborted/paused, the column and trap baked, and/or blanks analyzed until all contamination is absent. If further response is required (i.e., trap replacement), it is documented in the maintenance logbook. Refer to Section 7.4 for information on preventive maintenance.

3. Internal interferences can be purged from the sample with the target compounds and appear as elevated baselines or distinct peaks. Internal interferences most often manifest themselves as low/high recoveries of surrogate/matrix spike compounds. Matrix interferences vary from sample to sample.

4. The volatile lab must be free of solvents. All analytes must be less than their RLs or < 3X the RL for Acetone and Methlyene Chloride. The volatile lab is under positive pressure to reduce lab contamination; however, intermittent low levels of acetone and methylene chloride may occasionally be detected. Refer to Section 8.2 (Corrective Action) for clarification for blank contamination.

3.0 SAFETY

Employees must abide by the policies and procedures in the Corporate Safety Manual, Radiation Safety Manual, Lab Specific Addendum to the CSM, and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coat and closed-toe, nonabsorbent shoes are a minimum.

3.1 Specific Safety Concerns or Requirements

- The GC contains zones that have elevated temperatures. The analyst needs to be aware of the locations of those zones, and must cool them to room temperature prior to working on them.
- There are areas of high voltage in the gas chromatograph. Depending on the type of work involved, either turn the power to the instrument off, or disconnect it from its source of power.
- All employees will adhere to the practices and policies in the TestAmerica Corporate Safety Manual (CSM) and will read the MSDS's for the materials used in this method before handling or using the material.

3.2 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material	Hazards	Exposure Limit (1)	Signs and symptoms of exposure
Methanol (MeOH)	Flammable Poison Irritant	200 ppm-TWA	A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness and dizziness. Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes.
1 – Exposure limit refers to the OSHA regulatory exposure limit.			

4.0 EQUIPMENT AND SUPPLIES

4.1 Current Hardware/Software

- 4 Hewlett-Packard 6890 GC interfaced with a 5973 MSD. Equipped with DB-624 column.
- 2 Hewlett-Packard 6890 GC interfaced with a 5975 MSD. Equipped with DB-624 column.
- 2 Tekmar 3000 concentrators, 1 Enchon concentrator, 5 OI Elcipse 4660 concentrator in connection with 8 Varian Archon Autosamplers for 8 systems.
- 2 Hewlett-Packard 5890 GC interfaced with a 5972 MSD. Equipped with DB-624 column used as Screeners.
- 8-Hewlett-Packard Chemstations and peripheral hardware.
- 1-Hewlett-Packard Chemserver 9000 series with Target/NT version 4.14
- Chrom version 1.2

The GC/MS has a temperature programmable chromatograph interfaced with a mass-selective detector capable of scanning from 35 - 260 amu every second or less using 70 volts of electron energy in the electron ionization mode. The system is capable of producing an acceptable spectrum of bromofluorobenzene when 50 ng is analyzed.

4.2 Data System

The analytical systems are interfaced with stand alone PC's which are Pentium based systems running Agilent Chemstation. This system is capable of continuous acquisition and storage of mass spectral data. The software allows plotting specific masses versus time or scan numbers (Extracted Ion Current Profile-EICP) and integration of that abundance. The system also stores the data. The system contains the latest NBS Library.

4.3 Data File Name/ Batch Directory Assignment

Tune, standard, blank, and laboratory control sample (LCS) data files are designated by specific letters unique to each instrument in conjunction with the appropriate month and day (example : 3b0318 = instrument #3, first 12 hour BFB tune, March 18). When a worklist is made in Chrom, a unique Chrom ID# is assigned to each file which aids in the transfer of data from chemstation to Chrom.

4.4 Miscellaneous

- assorted syringes (10, 25, 50, 100, 500 and 1000 uL)
- 5 mL luer-lock gas-tight syringes
- assorted purge vessels (water, 5/25 mL)
- top-loading balance, capable of weighing to $\pm$ 0.1 g, stainless steel spatula
- assorted amber and clear Teflon-lined screw-capped vials (1.5-2.0 mL, 3.5-5.0 mL)
- cleaned 40 mL vials w/Teflon-lined screw-caps
- assorted volumetrics (10 mL, 20 mL, 25 mL, 50 mL and 100 mL)

5.0 REAGENTS AND STANDARDS

The majority of the calibration standards are EPA certified, A2LA or second-source verified by the standard vendor in situations where suitable SRMs (Standard Reference Material) was available. For those compounds where standards must be made from neat material (due to instability) or some non-routine compounds, **where available**, a second-source is purchased and used in the LCS to verify the standard

Each time a new initial calibration is required, new standards are prepared and the standards are verified against a second-source LCS (ICV-Initial Calibration Verification standard) prior to any sample analysis. This holds for all routine compounds and those available as second-source material in the LCS (see page 11 for list of compounds and venders).

All neat standards received are entered into TALs (LIMS). A label is printed from TALs and placed on the bottle. All neat standards are then stored in a separate freezer at approximately –10°C until needed. The standard is issued a unique ID# [i.e., Neat Standards Reference Number (NSRN)] which is used to track all standards as they are used as is or in preparation of stock/working solutions. The format of the standards in TALs (LIMS) will prevent working or intermediate level solutions from being used past the expiration date of the neat or stock solutions. Lot # of Methanol reagent used should be recorded in the comment section of the prep batch in TALs. In addition, the preparation of the 20% Sodium Bisulfate should be recorded in TALs.

5.1.1 Reagent Water (Milli-Q)

1-Liter of water is continuously purged with pre-purified nitrogen. The reagent water is routinely demonstrated to be interference-free. All compounds are < EQL or 3x EQL for methylene chloride and acetone.

5.1.2 Methanol (MeOH)

All new lot numbers of P & T J.T.Baker Methanol are analyzed and verified to be free of contaminants. This information is available on the TestAmerica Oasis Web-Site that can be accessed by all analysts. The currently approved lot numbers are listed on this site.

5.2 Surrogate Spiking Solution

Working surrogates are purchased as custom mix solutions from Restek in 5.0 mL ampules. The following surrogates are used:

Compound	Concentration
4-Bromofluorobenzene	\
1,2-Dichloroethane-d_4	250 ppm
Toluene-d_8	/
Dibromofluoromethane	

Upon arrival to the laboratory, the standard is entered into TALs (LIMS). When the ampule is opened, the transfer to the surrogate vial on the Archon Autosampler is entered into TALs (LIMS). The standard issued is another unique ID# [i.e., SRN (Standard Reference Number)] which can be traced back to the parent ID# (i.e., NSRN with the date of receipt, date of opening, and the supplier).

- Life of Standard: 1-year unopened or manufacturers expiration; once opened, they are used for a period of 14 days or until used.
- Storage Requirements: Stored in a freezer at ~ –10°C in the dark and kept for a period of one year unopened. *

5.3 Internal Standard Spiking Solutions

Working internal standards are purchased as custom mix solutions from Restek in 5.0 mL ampules. The following internal standards are used:

Compound	Concentration
Pentafluorobenzene	\
Chlorobenzene-d_5	250 ppm
1,4-Difluorobenzene	/
1,4-Dichlorobenzene-d_4	

After opening, the mixture is transferred to the internal standard vial on the Archon autosampler. The transfer is entered into TALs (LIMS). The standard issued is another unique ID# [i.e., SRN (Standard Reference Number)] which can be traced back to the parent ID# (i.e., NSRN with the date of receipt, date of opening, and the supplier).

- Life of Standard: 1-year unopened or manufacturers expiration; once opened, they are used for a period of 14 days or until used.
- Storage Requirements: Stored in a freezer at ~ –10°C in the dark and kept for a period of 1-year unopened. *

Working Internal Standards/Surrogate mix are also prepared using the custom mixes above and diluting to 50 ppm as follows:

Custom Mix	Volume (mL)	MeOH	Concentration
250 ppm Internal Standard Mix	4	Dilute to 20 mLs	50 ppm each component
250 ppm Surrogate Mix	4		

For waters, 5uL injected into 5 mL of water/sample results in 50 ppb internal and surrogate concentrations.

- The mixture is transferred to and stored in 1.5-2.0 mL amber Teflon-lined screw-capped vials at ~ –10 °C in the dark. The transfer is entered into TALs (LIMS). The standard issued is another unique ID# [i.e., SRN (Standard Reference Number)] which can be traced back to the parent ID# (i.e., NSRN with the date of receipt, date of opening, and the supplier).

- Life of Standard: Working IS/SS solutions have an expiration date of 2-weeks.

Purgeables
Calibration Standards

VOC Custom Mix (No Gases)
100 ug/mL in MeOH

1,1,1,2-Tetrachloroethane	n-Propylbenzene
1,1,1-Trichloroethane	Methyl-tert-butyl ether
1,1,2-Trichlorotrifluoroethane	Naphthalene
1,1,2,2-Tetrachloroethane	o-Xylene, p-Xylene
1,1,2-Trichloroethane	sec-Butylbenzene
1,1-Dichloroethane	Styrene
1,1-Dichloroethylene	tert-Butylbenzene
1,1-Dichloropropylene	Tetrachloroethylene
1,2,3-Trichlorobenzene	Tetrahydrafuran
1,2,3-Trichloropropane	Toluene
1,2,4-Trichlorobenzene	trans-1,2-Dichloroethylene
1,2,4-Trimethylbenzene	trans-1,3-Dichloropropylene
1,2-Dibromo-3-chloropropane	Trichloroethylene
1,2-Dibromoethane	
1,2-Dichlorobenzene	**ICAL 2 STD Custom Mix**
1,2-Dichloroethane	100 ug/ml in MeOH
1,2-Dichloropropane	2-Methylnaphthalene
1,3,5-Trimethylbenzene	1,3,5-Trichlorobenzene
1,3-Dichlorobenzene	1,3-Butadiene
1,3-Dichloropropane	Isopropylether
1,4-Dichlorobenzene	Methyl Acetate
2,2-Dichloropropane	Hexane
2-Chlorotoluene	Heptane
4-Chlorotoluene	Cyclohexane
4-Isopropyltoluene	Ethyl ether
Benzene	Methyl Cyclohexane
Bromobenzene	
Bromochloromethane	**APIX Custom STD**
Bromodichloromethane	2000, 8000 & 10000 ug/mL
Bromoform	Allyl Chloride
Carbon tetrachloride	Ethyl Methacrylate
Chlorobenzene	Methyl Methacrylate
Chloroform	Methacrylonitrile
Chlorohexane	Pentachloroethane
cis-1,2-Dichloroethylene	Trans-1,4-Dichloro-2-Butene
cis-1,3-Dichloropropylene	Iodomethane
Dibromomethane	Isobutanol
Dibromochloromethane	Cyclohexanone
Dichloromethane	n-Butanol
Ethylbenzene	2-Nitropropane
Hexachlorobutadiene	Ethyl Acetate
Isopropylbenzene	
m-Xylene	**Chloroprene**
n-Butylbenzene	5000 ug/mL

Gaese Mix
100 ug/mL in MeOH
Chloroethane
Methyl Bromide (Bromomethane)
Methyl chloride (Chloromethane)
Trichlorofluoromethane
Dichlorodifluoromethane
Vinyl chloride
Dichlorofluoromethane

Vinyl Acetate
2000 ug/mL in MeOH

Volatile Custom Ketone Mix
Acetone
2-Hexanone
Methyl ethyl ketone
4- Methyl-2-pentanone
Carbon Disulfide
100 ug/mL in MeOH

Nitriles and Acrolein Custom Mix
Acetonitrile 800 ug/mL in MeOH
Acrylonitrile 800 ug/mL in MeOH
Propionitrile 800 ug/mL in MeOH
Acrolein 4000 ug/mL in MeOH

2-Chloroethylvinylether
2000 ug/mL in MeOH

5.4 Stock Purgeable Standards

These are obtained as custom mixes from Restek and Supelco. The contents of each solution and concentration appear on the previous page. Upon opening, all contents are transferred to 1.5-2.0 mL amber, Teflon-lined screw-capped vials. Listed are compounds in the EPA TCL and includes compounds done on a regular basis. Other standards, if needed, are either purchased as neat solutions or neat standards from Supelco, Chem Service or other certified supplier. See appropriate entries in TALs (LIMS).

* If the stock solution has manufacturers' expiration date, that is assigned. If the date is not evident, 1-year is assigned to un-opened ampules. This is applicable for all "neat" standards.

5.4.1.1 Main 8260 Mix

The 8260 Working Standard is a vial transfer:

Compound / TCL Mix	Volume[1] (mL)	Concentration
8260 Custom Mix Stock/Working Std	2	100 ppm each component

5.4.1.2 Gases

The Gas Working Standard is a vial transfer:

Compound / TCL Mix	Volume (mL)	Concentration
Gases Mix	2	100 ppm each component

5.4.1.3 Additional Compounds

The Ketone and Acrolein/Nitrile Working Standards are vial transfers:

Compound / TCL Mix	Volume[1] (mL)	Concentration
Ketone Custom Mix Stock/Working Std	2	100 ppm each component

Compound / TCL Mix	Volume[1] (mL)	Concentration
Nitriles/Acrolein Custom Mix Stock/Working Std	2	800 ppm Nitriles 4000 ppm Acrolein

The CEVE and Vinyl Acetate Working Standard are prepared as follows:

Stock Compound/Mix	Volume (uL)	MeOH	Concentration
2000 ppm CEVE	100	Dilute to 2 mLs	100 ppm each component
2000 ppm Vinyl Acetate	100		

5.4.1.7 Appendix IX

The Appendix IX and extra compounds are prepared as follows:

Compound / TCL Mix	Volume[1] (mL)	MeOH	Concentration
APIX Custom Stock Standard	250ul	Diluted to 2mLs	250/1250/10000 ppm

Compound/TCL Mix	Volume[1] (uL)	MeOH	Concentration
Chloroprene 5000 ug/mL	40ul	Diluted to 2mLs	100 ppm

The ICAL 2 Custom Working Standard is a vial transfer:

Compound / TCL Mix	Volume[1] (mL)	Concentration
ICAL2 Custom Mix Stock/Working Std	2	100 ppm each component

- Life of Standard: Unopened ampules are assigned the manufacturer's expiration date or 1 year from receipt. Working solutions have an expiration date of 1-week (Gases, 2-CEVE, Vinyl Acetate, Acrolein and nitriles) and 2-weeks for all others.
- Storage Requirements: These mixtures are stored in 1.5-2.0 mL amber Teflon-lined screw-capped vials at ~ –10 °C in the dark.

NOTE: All standard 'recipes' are listed here in this SOP for guidelines for standard preparation. These 'recipes' are subject to change. The standards listed here are standards that are regularly used. Some clients may request other compounds not listed here. Those compounds will be evaluated for accuracy via this method and analyzed for on a project basis.

5.4.1.8 Low Level Standard

A low level standard is prepared by making a 1/10 dilution of the stock standards of each of the above (nitriles and acrolein included). This standard is used to prepare the low points in the initial calibration. The low level standard may contain the Main 8260 Mix, gases, nitriles and acrolein, and any other required standard. A low-level standard for the Appendix IX compounds is also prepared separately due to duplication of some compounds.

A low level surrogate solutions is also prepared by a 1/10 dilution of the working for low points in the water curve. The calibration levels may vary with the compounds. See recipes in the calibration section for the levels. The low point in the calibrations is based on each compounds reporting limit.

All solutions are stored in a 1.5-2.0 mLs amber Teflon-lined screw-capped vials at –10°C in the dark. All standard preparation is recorded in the TALs (LIMS) system. Solutions are prepared every 2-weeks (1-week for the gases, 2-CEVE, Vinyl Acetate, Acrolein and Nitriles).

Purgeable Spike Standard Mixes
ACCUSTANDARD, o2si and Absolute Standard

VOC LIQUID MIXTURE
2000 ug/mL in MeOH

1,1,1,2-Tetrachloroethane	m-Xylene
1,1,1-Trichloroethane	n-Butylbenzene
1,1,2,2-Tetrachloroethane	n-Propylbenzene
1,1,2-Trichloroethane	Naphthalene
1,1-Dichloroethane	o-Xylene
1,1-Dichloroethylene	p-Xylene
1,1-Dichloropropylene	sec-Butylbenzene
1,2,3-Trichlorobenzene	Styrene
1,2,3-Trichloropropane	tert-Butylbenzene
1,2,4-Trichlorobenzene	Tetrachloroethylene
1,2,4-Trimethylbenzene	Toluene
1,2-Dibromo-3-chloropropane	trans-1,2-Dichloroethylene
1,2-Dibromoethane	trans-1,3-Dichloropropylene
1,2-Dichlorobenzene	Trichloroethylene
1,2-Dichloroethane	
1,2-Dichloropropane	**Bromochloromethane**
1,3,5-Trimethylbenzene	2000 ug/mL in MeOH
1,3-Dichlorobenzene	
1,3-Dichloropropane	**Heptane**
1,4-Dichlorobenzene	2000 ug/mL in MeOH
2,2-Dichloropropane	
2-Chlorotoluene	**MTBE**
4-Chlorotoluene	2000 ug/mL in MeOH
4-Isopropyltoluene	
Benzene	**1,3,5-Trichlorobenzene**
Bromobenzene	2000 ug/mL in MeOH
Bromoform	
Carbon tetrachloride	**Clorohexane**
Chlorobenzene	1000 ug/mL in MeOH
Chlorodibromomethane	
Chloroform	**Ethyl Ether**
cis-1,2-Dichloroethylene	1000 ug/mL in MeOH
cis-1,3-Dichloropropylene	
Dibromomethane	**Hexane**
Dichlorobromomethane	1000 ug/mL in MeOH
Dichloromethane	
Ethylbenzene	**Trichlorotrifluoroethane**
Hexachlorobutadiene	2000 ug/mL in MeOH
Isopropylbenzene	

Volatile Organic Compound Gas Spike
2000 ug/mL in MeOH
Chloroethane
Methyl Bromide (Bromomethane)
Methyl chloride (Chloromethane)
Trichlorofluoromethane
Dichlorodifluoromethane
Vinyl chloride

Volatile Mix Additional Spike Compounds
2000 ug/mL in MeOH
Acetone
2-Hexanone
Methyl ethyl ketone
4- Methyl-2-pentanone
Carbon Disulfide
Vinyl Acetate
2-Chloroethylvinylether
Iodomethane

THF
2000 ug/mL in MeOH

Methyl Acetate
2000 ug/mL in MeOH

Methyl Cyclohexane
2000 ug/mL in MeOH

Cyclohexane
2000 ug/mL in MeOH

5.5 Stock Matrix Spike Solution

The matrix spike compounds are obtained as solutions from a second source (i.e., Accustandard) in 1.5-2.0 mL ampules. These are listed on the previous page. A different analyst than the one who prepared the calibration solutions usually prepares matrix spike solutions. These are stored at ~ -10°C in the dark prior to use. Neat standards are kept for a period of 1-year un-opened or the manufacturer's expiration date. Once opened, the stock may be used for 3-months.

The matrix spike solutions are prepared as follows:

5.5.1 VOC Spike

Stock Compound/Mix	Volume (uL)	MeOH	Concentration
2000 ppm VOC Liquid Spike	50	Dilute to 2 mL	50 ppm each component
2000 ppm 8260 Additional Spike	50		
1000 ppm 1,3,5-Trichlorobenzene Spike	100		
2000 ppm Tetrahydrafuran	50		

5.5.2 Gas Spike

Mix	Volume (uL)	MeOH	Concentration
2000 ppm Gas Spike	50	Dilute to 2 mLs	50 ppm each component

5.5.3 Additional Spike Compound Mix

Compound	Volume[1] (uL)	MeOH	Concentration
2000 ppm Bromochloromethane	50	Dilute to 2 mL	50 ppm each component
1000 ppm Ethyl Ether	100		
1000 ppm Chlorohexane	100		
1000 ppm Hexane	100		
2000 ppm MTBE	50		
1000 ppm Heptane	100		
1000 ppm Trichlorotrifluoroethane	100		

Compound	Volume (uL)	MeOH	Concentration
2000 ppm Methyl Acetate	50	Dilute to 2 mL	50 ppm each component
2000 ppm Cyclohexane	50		
2000 ppm Methyl Cyclohexane	50		

For waters, addition of 5 uL of each solution results in all spike compounds at 50 ppb.
For soils, addition of 5 uL of each solution results in all compounds at 50 ppb.

These solutions are stored at ~ -10°C in several 1.5-2.0 mL amber Teflon-lined screw-capped vials. All standard preparation is recorded in the TALs (LIMS) system. Working matrix spike solutions have a 2-week/1-week (Gases) expiration date or until low recoveries of the matrix spike compounds indicate a new solution is needed. See above for label information.

5.6 Stock BFB Solution

The BFB standard is purchased as a neat solution from Supelco.

Stock	Amount	MeOH	Concentration
2000 ppm BFB	25 uL	Dilute to 2 mLs	25 ppm

- Life of Standard: This stock can be kept for a period of 1-year until opening. Upon opening, the solution is transferred to a 1.5 - 2.0 mL vial and assigned an SRN. Once opened, it is used for a period of 6-months.
- Storage Requirements: The standard is stored at ~ –10°C in the dark

Addition of 2 uL to 5 mLs results in a concentration of 50 ng/5 mLs.
All preparation is recorded in the TALs (LIMS) system. All labels are completed as above.

NOTE: Intermediate and Working Solutions are never assigned an expiration date exceeding the expiration date of the neat/stock standards/solutions.

6.0 CALIBRATION

Before an instrument is used as a measuring device, the instrument response to known reference materials must be determined. The manner in which various instruments are calibrated depends on the particular type of instrument and its intended use. All sample measurements must be made within the calibration range of the instrument. Preparation of all reference materials used for calibration is documented.

6.1 PFTBA Autotune or Manual Tune

The instrument is first tuned in one of two ways: autotune or manual tune. The ion abundances in the calibration gas are best monitored near the temperature of analysis of BFB. Monitoring at this temperature produces the most representative cal gas scan and therefore the best estimate of BFB response.

1. If an AUTOTUNE is to be performed, continue below. If not, skip to step 6. An autotune is not run before every initial calibration. If the instrument has been down for any reason previously listed or major difficulties in manual tune are encountered, an autotune is performed. Autotunes are generally NOT performed when an existing initial calibration is being met.

2. The Chemstation software has a menu driven tune program. Begin the autotune program. Key masses are 69, 219 and 502.

3. Follow instructions and retrieve a hardcopy of the autotune results. Check the following:

- passed/fail: in itself, not necessarily an indication of MS performance
- repeller and ion focus settings
- electron multiplier voltage

4. The repeller and EM voltages are good indicators of the sources' cleanliness. Generally, the lower the setting the cleaner the source. Other factors may however, supersede (i.e., the age of the multiplier) and a clean source will not always autotune these low. The EM is set by autotune program to produce a target abundance for mass 69 (varies depending on the tune program and instrument). The operator may plan on having to increase this by 100-200 to achieve normal analysis sensitivity (depends on the tune program and the instrument).

5. Observe peak shape, absence of lead-ons/tailing, the resolution between isotopes, peak width and mass axis. A hardcopy of the profile scan is desirable, and can be filed with the autotune results.

6. If an AUTOTUNE has just been performed, continue here. If not, skip to step 9. Enter MANUAL TUNE and read the autotune (which was automatically stored in a file). For volatiles, edit the scan parameters to monitor ions 69, 131 and 219.

7. Enter one of several methods available and adjust the parameters (usually the ion focus, entrance lens and amu gain) to achieve the following relative abundances:

Mass	Relative Abundance
69	100%
131	40-60%
219	50-70%

These will vary with the MS. Mass 219 is usually 5-9% greater than mass 131. If necessary, adjust the amu gain for peak shape and high-end isotope resolution. An overall peak-width of 0.500 to 0.550 is desirable.

Again, these adjustments and relative abundances may not guarantee that BFB will meet requirements, but is a good place to start.

8. A hardcopy the profile scan is desirable. This can be filed with the autotune results. This file can serve as a diagnostic tool and can also provide a starting point in the event the operator has trouble meeting the initial calibration.

Save the changes to the appropriate Tune File. Exit the program.

9. If an AUTOTUNE has not been performed, enter MANUAL TUNE and adjust any parameters, if need be. Adjustment may not be necessary, and not desirable, if problems in tuning or meeting the initial calibration have not been encountered. A hardcopy of profile scan can be printed and filed. Save the changes and exit the program.

6.2 BFB Analysis

Once the instrument is tuned, 50 ng of 4-Bromofluorobenzene must meet criteria. The BFB can be purged. The mass spectrum must meet the following criteria:

Mass	Ion Abundance
50	15-40% of mass 95
75	30-60% of mass 95
95	Base Peak, 100% rel. abund.
96	5 - 9% of mass 95
173	<2% of mass 174
174	>50% of mass 95
175	5 - 9% of mass 174
176	>95% but <101% of mass 174
177	5 - 9% of mass 176

The manner of acquiring the mass spectrum of BFB can be performed in only two specific ways with Chrom software:

- Three scans (the peak apex scan and the scans immediately preceding and following the apex) are acquired and averaged. Background subtraction is performed and must be accomplished using a single scan acquired no more than 20 scans prior to the elution of BFB. This procedure of averaging and subtracting is performed automatically by the Chrom software and evaluated by the criteria in the table below. If this procedure does not meet the acceptance criteria, then the analyst will use the following procedure:
- One scan of the BFB peak is acquired and a background peak is subtracted and evaluated by Chrom software using the criteria below. This procedure is performed manually by the analyst. No other manner of BFB acquisition and evaluation can be utilized.

The BFB is analyzed by one of the methods in Attachment 1. **(Method parameters listed in the appendices are examples only. This statement applies to all references made to these methods).** Typical Concentrator conditions also appear in Attachment 1. The EM voltage may be 100-200 volts above autotune. The abundances of the designated masses above MUST meet the criteria before analyses can begin. If necessary, enter MANUAL TUNE and adjust parameters. BFB analysis is completed about every 12-hours of analysis.

6.3 Description of Initial Calibration

An initial calibration may be completed:

- as needed - continuing calibration can not be met
- after a source cleaning and/or column change or any time a major repair or change has occurred with the instrument that affects calibration where a new calibration is indicated.

Confirm that the GC/MSD is stable and equilibrated. If at all possible, allow the instrument to equilibrate overnight at all operating temperatures if the source/column has been cleaned/changed. Prior to beginning initial calibration it is a good idea to:

- check the background of air/water levels and base ion by scanning for appropriate ions and also visually inspecting the spectrum scan for any other possible and undesirable background.
- recheck the multiplier settings, after a source is cleaned the EM can most often be dropped.

6.4 Initial Calibration

Each calibration standard is analyzed according to one of the methods in Attachment 1. These are examples. The actual number of points in the calibration is determined by the calibration and acceptance criteria table (Attachment 4). The EM voltage may be 100-200 volts above autotune.

Allow standards to come to ambient temperature.

Fill ten 5-mL luer-lock gas-tight syringes with reagent water to overflowing. Replace the plunger and invert. Adjust to 5-mL confirming the absence of any air bubbles. Pull back slightly on the plunger to allow addition of standards. Following the guides found in the Attachment 1, add the appropriate amount of standards.

Immediately add the standards to a clean 40-mL vial. Following the method parameters outlined in Attachment 1, analyze the 50 ppb standard. A normal standard will appear very similar to the ones in Figures 1 and 2. Quantitate the standard against the appropriate method file. Sufficient areas for the first internal standard will vary somewhat between instruments. Acceptable areas should be based on maintaining sufficient sensitivity for poor responders without saturating the detector at the upper end of the calibration range. Too low an area will almost guarantee poor/unsatisfactory responses of low-response compounds and too high an area will result in saturation of some compounds at higher levels, resulting in false low response factors at high concentrations.

It is helpful to analyze a medium level standard first and assess the areas before continuing with the low/high level standards.

Response factors are calculated by the data system as follows:

$$RF = \frac{A_x \times Q_s}{A_s \times Q_x}$$

Where:
A_x = ion abundance for analyte
A_s = ion abundance for its internal standard
Q_s = concentration of its internal standard
Q_x = concentration of analyte
(Response Factors have no units)
The appropriate quant ion must be in the method file. A listing of the target compounds with their appropriate internal standards appears in Attachment 2. Confirm the presence of all targets and the separation of non-co-eluting compounds. Note the response factors for the gasses. If necessary, prepare new standards.

If adjustments to the acquisition parameters are necessary, make them and re-analyze the 50 ppb standard.

When a standard is analyzed and processed on Cas part of the initial calibration the RF's are automatically updated in the daily method. After all initial calibration standards are processed, checked and confirmed as being accurate and passing method criteria, the initial calibration is locked and set as the most recent. This ensures that the correct initial calibration is used for each ensuing continuing calibration check. A hardcopy of the calibration report is generated. All method criteria are assessed for compliance. Confirm that:

1) all CCC's are below 30% (Vinyl Chloride, 1,1-Dichloroethene, Chloroform, 1,2-Dichloropropane, Toluene, Ethylbenzene)

2) the RF's for SPCC compounds are >0.300 (Chlorobenzene, 1,1,2,2-Tetrachloroethane) (Minimum RF for Chloromethane, Bromoform and 1,1-Dichloroethane is 0.100).

Calibration curves are evaluated following the "Evaluation and Acceptance Criteria" table (Attachment 4). For all compounds in the initial calibration with a %RSD > 15.0%, calibration curves of area ratio versus concentration using a first or higher order regression curve of the calibration curve points will be performed. Weighted linear curves may be used. Quadratic curves may only be used after evaluation of the curve plot and Supervisor approval (Note: The state of South Carolina does not allow the use of quadratic curves. Therefore, in those cases, only the weighted linear option is allowed).

Method 8000B/8260B specifies a minimum coefficient of determination (R^2) of 0.990. The methods also specify a minimum of 5 calibration points for a linear model and a minimum of 6 calibration points for a higher order regression. The laboratory, in order to meet AFCEE requirements, will analyze a minimum number of points to satisfy both the SW846 and AFCEE. All efforts will be made to meet the minimum COD of 0.990. However, there are some compounds that historically present a problem meeting this requirement (See attachment 9 for a list of poor purgers). These compounds are usually those listed in the analyte table of Method 8260B with qualifying remarks. Many of these have various known issues that would effect reproducibility (i.e., Acetone qualifier pp = poor purger). These typically include many of the Appendix IX compounds as well. The laboratory will take minimal action for these compounds.

The preparation instructions noted above will be modified to include the necessary calibration levels. These instructions are for guidance only and may change as needed.

An example of an acceptable initial calibration appears in Attachment 2. The BFB tune, and all standard raw data are filed. Each instrument has its own initial calibration.

Each time a new initial calibration is required, the standards are verified against a second-source LCS (ICV-Initial Calibration Verification standard) prior to any sample analysis. This holds for all routine compounds and those available as second-source material in the LCS (see page 11 for list of compounds and venders). The ICV must meet 25%D for all compounds or corrective action must be taken. Some client's require 20%D. For South Carolina, all compounds must meet 30%D unless there are compounds identified as poor purgers in the SOP. The poor purgers should still meet 40%D. South Carolina does not allow the use of marginal exceedances.

NOTE: The actual number of points in the calibration and the low point in the calibration may vary with client and project need. Clients may have additional requirements, which would be covered in a client-specific or regulatory/agency QAPP.

6.5 Daily or Continuing Calibration

Continuing calibration occurs prior to analysis.

If time remains after the initial calibration, and the 50 ppb standard meets continuing calibration criteria, samples can be analyzed up to the 12-hour tune limit. The samples are quantitated against the average RF or appropriate as per method. See later sections describing calculations.

After having satisfied BFB tune requirements, a continuing calibration standard must be analyzed. Analyze a 50 ppb standard following the procedure outlined above. Confirm Form 7 that:

1) all CCC's are below 20% (Vinyl Chloride, 1,1-Dichloroethene, Chloroform, 1,2-Dichloropropane, Toluene, Ethylbenzene)

2) the RF's for SPCC compounds are >0.300 (Chlorobenzene, 1,1,2,2-Tetrachloroethane) (Minimum RF for Chloromethane, Bromoform and 1,1-Dichloroethane is 0.100).

If continuing calibration can not be met, either new standards and/or a new calibration are needed.

NOTE: Method 8260B stipulates that if the CCC's are not part of the analyte list then all compounds being reported must be < 20% drift.

All internal standard areas and retention times are assessed immediately after calibration. Areas and times compared to the mid point of the initial calibration. Internal standard areas should not deviate by a factor of two or the retention times should not deviate by > 30 s. If the situation occurs, appropriate action is taken and the standard re-analyzed. All corrective action and return to control are documented in the Corrective Action section in the analysis logbook for the appropriate instrument.

7.0 PROCEDURE

7.1 Quality Control Checks

Quality Control is accomplished through:
1) daily tuning and calibration checks and
2) preparation QC traceable through individual batches.

7.1.1 Initial Calibration

PFTBA BFB TUNE 200 \ 150 | 100 | 50 | 20 | 5 | 2 | 1 | 0.5 /	Prior to Initial Cal Initial Cal need dependent on situation.	*limits in Section 6.2 *limits in Section 6.4

NOTE: As stated, the actual number of points in the calibration and the low point in the calibration may vary with client and project need. Minimum number of points for AFCEE and/or 3rd Edition SW-846 may be 6 or 7 depending on matrix. Other clients may have additional requirements, which would be covered in a client-specific QAPP.

7.1.2 Method Blank (MB)

Prior to any analysis, the reagent water must be shown to be free of interference's and target compounds.

A 5 mL portion of reagent water is analyzed using one of the methods in Attachment 1. Internal Standards (ISS) and Surrogates (SSS) will be added prior to analysis. Initial concentrations of both surrogate and internal standard solutions shall be such that "sample concentrations" of the analytes conform to the method and surrogate tables provided in this SOP. All target compounds must be less than the quantitation limit (See Section 2.0). Once the MB analysis is complete and acceptable, analysis can proceed.

7.1.3 Daily Analysis

BFB	Prior to continuing	* See above calibration
Daily Calibration Standard	Prior to samples	* See Section 6.5

Prep QC	Frequency
MB	Prior to analysis
LCS[1]	1 per analysis batch
MS/MSDs[2]	at least 1 set in 20
Surrogates	every blank, sample and QC Sample
Samples *	

***Any given 12-hour period contains a tune, standard, blank and LCS. Preparation QC is at a 5% frequency. Instrumental controls are outlined above and further discussed in the procedure section.**

[1] LCS Duplicate (LCD) is performed when insufficient sample is available for an MS/MSD.
[2] The sample selection for MS/MSD, if not specified by the client on the chain-of-custody, is rotated among client samples so that various matrix problems may be noted and/or addressed.

7.2 Sample Preservation and Storage

Sample containers, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance and/or specific contract or client requests. Listed below are the holding times and the references that include container and preservation requirements for compliance with the Resource Conservation and Recovery Act (RCRA).

Matrix	SW-846
All	14-days

All samples received for volatile analysis are refrigerated upon receipt at 4±2°C. Refrigeration is the only preservative for 5030 soil samples, while water samples are additionally preserved with 3 drops of 36% HCl to a pH <2. Water samples marked as un-preserved are analyzed within 7-days.

7.3 Sample Preparation / Screening /Analysis

Once the samples are logged into the TALs (LIMS) database upon receipt, a paperwork trail is initiated. The Supervisor or Analyst prints and prepares the necessary information (Sample Tracking Sheets) and places it in the appropriate file bin in the GC/MS VOA lab. The analysts take this information and subsequently screen the associated samples. Samples are screened by MSD prior to analysis. The actual screening procedures vary due to sample appearance, sample matrix, client history and analytical method. Once the samples are screened, the paperwork is transferred to a second file appropriately labeled. This file contains information about samples that have been screened but need to be reviewed. Once screened, an 'X' is placed on top of the vial, indicating both that the sample has been screened, and that the particular vial can not be used for subsequent analysis. The screened analysis can be reviewed on screen or hard-copy, as all screening data is collected and stored on the data system as with all GC/MS analyses. Upon review, the analyst makes decisions concerning the screen and indicates if an initial dilution is required. This information is physically recorded on the paperwork. Once reviewed, the paperwork is then placed in appropriate files that are broken down by matrix and method. The samples are now ready to be analyzed.

7.3.1 Waters

Allow samples and standards to come to ambient temperature. Observe all vials of sample and confirm the absence of any air bubbles. If a vial has an air bubble do not use that vial and notify the Supervisor of the situation. Make sure the vial used for screening is properly marked and do not use this vial for analysis.

Remove the plunger from a 5 mL luer-lock gas-tight syringe and fill to near over-flowing. Replace the plunger. The pH of all samples is verified at time of analysis. If the pH < 2, a check-mark is placed in the appropriate column on the sample tracking sheet. If the pH > 2, the actual estimated pH is written in the same column. pH checks and verification of hold-times are documented on the review form. Samples lacking preservation may be noted in the case narrative. Invert the syringe, and adjust the volume to 5 mLs. Confirm the absence of all air bubbles.

Add IS and SS to the 5 mL syringe. Initial concentrations of both internal standard and surrogate solutions shall be such that "sample concentrations" of the analytes conform to the method and surrogate tables provided in this SOP. Immediately add the sample to a clean 40 mL vial using the method described in attachment 2 to analyze the sample.

If a batch is going to be analyzed, which is usually the case, load all samples following the procedure above. After the batch is loaded, replace all samples and standards back in storage. Appropriate documentation is made on the ICOC page.

If a dilution is required as indicated from the screening results, the following guidelines are followed. If the dilution is > 1/100 (250 uL of sample) an initial dilution is made into a volumetric flask. If serial dilutions are required, no less than 1 mL is taken for further dilutions. The final sample aliquot taken for analysis from the volumetric is no less than 250 uL. If the dilution is < 1/100, the appropriate sample amount is added directly to the 5 mL syringe.

Opened sample vials are used only once unless: (a) any necessary dilutions/reruns are done the same day or (b) there are no other vials for that sample.

7.3.2 Soils

As some clients still request method 5030 at the present time, soils are still being analyzed as indicated below. As clients convert to Method 5035 completely, this section will be removed.

Before weighing any samples, check the balance using the appropriate class weights. Record the actual weights in the Balance Logbook. If a problem is noted, contact the QC department.

Allow samples and standards to come to ambient temperature.

Weight out 5 grams of the sample into a clean 40-mL vial. Add 5-mL reagent water into vial. IS/SS will be added though the septum. The autosampler will add an additional 10-mLs of reagent water. Initial concentrations of both surrogate and internal standard solutions shall be such that "sample concentrations" of the analytes conform to the method and surrogate tables provided in this SOP. Using the methods described in Attachment 1, analyze the sample. All soil samples are analyzed with a heated purge (40°C).

If a batch of samples is to be analyzed, prepare each as above. After the batch is loaded, replace all samples and standards to their appropriate storage location. Documentation is made on the ICOC page.

Any sample that based on screening results/historical data has shown to contain high concentrations of compounds is analyzed at an initial dilution. Any sample that after the initial run contains targets above the calibration range is diluted to accurately quantitate those compounds. If an initial analysis over-diluted the given sample it is re-analyzed as a low level soil. If the low-level analysis contains compounds above the calibration range, and the same compounds are within range in the dilution, both sets of data may be reported to the client.

If a 1/2 or 1/5 dilution is required, 2.5g/1.0g of sample is weighed into the purge vessel.

7.3.3 Medium-Level Soil Extracts

If a larger dilution is required, a medium-level soil extract is prepared as follows. Five grams of sample is weighed into a tarred vial. Five 5-mLs of MeOH is added to the vial and the vial sealed. After the 24-48 hour contact time, the MeOH portion is decanted and stored in a 1-1.5 mL Teflon-lined screw-capped vial for storage. A portion of the extract (100 uL maximum) is taken for analysis. ISS and SSS will be added prior to analysis. Initial concentrations of both surrogate and internal standard solutions shall be such that "sample concentrations" of the analytes conform to the method and surrogate tables provided in this SOP. Serial dilutions, if needed, are made from the extract and appropriate amounts taken for analysis.

If the sample upon which a medium-level prep as been performed also required an MS/MSD, the appropriate amount of MS solution is also added.

All samples prepared in this manner will be analyzed against a medium-level soil curve. The standards, blanks and LCS samples will contain 100 uL MeOH. The curve will be at ambient temperature.

NOTE: Some soils are analyzed initially at low levels due to increasing client requests for lower reporting limits. The same samples may then require large dilutions to bring compounds into the calibration range of the instrument. Some compounds, most notably the ketones, have very different responses when heated versus non-heated, despite the sample matrix. Traditionally, the lab heats soils. Therefore, the match between original analyses and dilutions for compounds such as these may not appear to correlate.

Dilution	Sample Weight	Vol. MeOH (1/2.5) Extract
1/2	2.5 grams	---
1/5	1.0 gram	---
1/50	5 grams / 5 mLs	100 uL
1/250	5 grams / 5 mLs	20 uL
1/500	5 grams / 5 mLs	10 uL

Using those parameters in Attachment 1, analyze all samples in the batch.

Sample vials/jars are only used once unless: (a) any dilutions/reruns are analyzed the same day or (b) there is only one jar for analysis.

7.3.4 Method 5035

NOTE: ICAL Standards are prepared with 5 mL milli-Q water.

Samples for low level VOA soil analysis may be received at the lab in one of three manners: First, as replicate 5 gram core samples in 40 mL vials containing organic free water and frozen within 48 hours of collection. Secondly, as replicate 5 gram core samples in 40 mL vials containing a Sodium Bisulfate preservative solution (refer to USP-5035 for collection/preservation). Thirdly, unpreserved 5 gram core samples may be received in Encore containers. These core samples must be placed in organic free water and frozen within 48-hours of collection. This time requirement is currently under review by appropriate regulatory agencies and may be extended beyond the 48-hours. Until such time, the laboratory will endeavor to "fix" the sample cores in preservative within 48-hours of collection. The laboratory may receive replicate 5 gram soil cores to be used for reanalysis if needed.

In addition to low level samples, an additional soil aliquot should be received for use as a screen and possible use as a mid-level extraction/analysis. This additional core must also be fixed in MeOH within 48-hours of collection. The amount of MeOH added must closely correspond to a soil to solvent ratio of 1:1. TestAmerica may adjust the methanol levels of any sample prepared in the laboratory from encore plugs with a corresponding 1:1 ratio of MeOH to soil. Any sample prepared in the field and fixed in methanol prior to arrival to the laboratory will not be adjusted but an NCM will document the discrepancies. Though not specified in the method, TestAmerica will pursue a goal of removing the MeOH from the soil within 24-48 hours after the initial extraction. A portion of the MeOH be removed and placed in an amber 1.5 - 2.0 mL Teflon-lined screw-capped vial for storage. This time limit should standardize the amount of time the MeOH comes in contact with the sample.

MeOH extracts of soils will be analyzed as stated above at ambient-temperature against a medium-level soil initial calibration. All surrogate and internal standard solutions will be added at time of analysis.

Low level soils will be analyzed using the Closed Purge and Trap Auto Sampler System. Surrogate and internal standard solutions will be added at the time of analysis through the septum by either a small gauge (10uL) syringe or automatically by the Archon autosampler. Initial concentrations of both surrogate and internal standard solutions shall be such that "sample concentrations" of the analytes conform to the method and the spike and surrogate tables provided in this SOP. The concentration of the solution and amounts spiked may vary depending on the precision obtained with a given solution/volume combination. However, the final concentrations of such compounds in the samples will follow the same guidelines as previously stated in this SOP for all other samples.

As with the internal standard and surrogate, all QC spike solutions must also be added to the closed sample container. This is accomplished by the addition of the spike solutions through the septum with a small gauge (10 uL) syringe just prior to the sample being placed on the instrument for analysis.

7.3.5 Drum/Waste Samples

- Non-MeOH Miscible
- MeOH Miscible

These samples are normally treated as medium level soils or waste dilutions. Waste dilutions normally consist of 5 grams of sample to 5 mLs of MeOH. If the sample is non-miscible with methanol, mix the sample and allow sample to separate. Draw off the methanol into a screw top vial. Use a portion of this methanol extract to screen. Continue to analyze the sample as high a high level methanol extract at a dilution based on the screening result.

If the sample is miscible with methanol, notify the PM and provide a detailed description of sample matrix and any matrix issues. The sample will automatically be given a 1:2 dilution factor, as a result of its miscibility with the methanol. Screen the sample and continue to analyze by the high level methanol extract procedure based on the screening results. Both ISS and SSS are added prior to analysis. The laboratory can also pre-spike the surrogates and spike compounds if client-specified to do so. In many cases, due to the high level dilutions required, the surrogates and matrix spike compounds may be diluted out. Therefore, unless specifically instructed, both solutions will be added at the time of analysis.

Drum/waste samples that are biphasic in nature require a discussion with the PM and client in order to determine how the samples will be prepared and analyzed (one phase, both phases?). The preparation and analysis of the sample will be documented.

7.4 Preventive Maintenance

Instrumental maintenance can be categorized as daily and "as required". Required maintenance may be performed for a variety of reasons. Certain trouble-flags will indicate what maintenance procedures may be required. A description of the situation, actions taken and follow-up must be documented in the instrument maintenance logbook on the day of maintenance and initialed / dated. An example Maintenance logbook page can be found in Attachment 3.

7.4.1 Daily Maintenance

The most routinely performed maintenance includes:

- Purge-line or sample transfer line rinses within the concentrator and vial autosampler
- Analysis of blanks after high level samples
- GC oven bake after high level samples

The Corrective Action/Qualification Report for GC/MS VOA is used to document that the instrument Preventive Maintenance as described in this SOP has been performed.

7.4.2 "As Required"

Most maintenance is done on an "as needed" basis, is operator determined and can be categorized as GC, Concentrator, or MS related.

7.4.2.1 GC Related

- change column; condition new column
- clean separator; change separator
- check helium flow rate
- change gas cylinders and moisture trap
- Replace liners

7.4.2.2 MS Related

- clean source/rods and anything associated with that activity
- replace electron multiplier
- change filaments

7.4.2.3 Concentrator Related

- change transfer line; clean transfer line
- replace trap; condition new trap
- run 20 ug/L Bromoform standard to check for formation of Chloromethane and Bromomethane.
- refurbish Concentrator
- check purge pressure and flow rate
- analysis of position blanks after high-level samples
- change bulk head fitting

7.4.2.4 Autosampler Related

- change sparge needle
- change pencil filters
- flush standard pickups
- calibrate standard valve
- run vial position calibration
- clean transfer rods
- oil bearings

7.5 Documentation/Tracking of Sample Analyses

7.5.1 The GC/MS VOA lab employs several forms that serve both a tracking and review function. The Sample Tracking Sheet is filled out for each job. It contains information the analyst needs as for method, QC requirements, special reporting requirements, screening results, methanol lot # etc.., in addition for space to track the analysis of every single sample in the job and the outcome of that analysis. The Sample Tracking Sheet can be found in Attachment 3.

7.5.2 In addition, all samples logged into the department appear on back-logs ordered by both Hold Time and Due Date. The back-logs are utilized by the analysts when making decisions as to methods and analyses that are needed for the day. As samples are analyzed and reviewed, the back-logs are updated to reflect those samples completely analyzed, those requiring dilutions and re-analyses.

7.5.3 In addition, an instrument sequence log is printed, reviewed, signed and bound in a log daily for each instrument.

7.6 Archival of Data

There are three full back-ups performed per week.

- Every Thursday a full back up of VOA data is performed.
- Every Friday a full back up of SVOA data is performed.
- Every Tuesday a system back up (minus the NBS Library) is performed. There are two tapes provided for this back up, and are rotated each week. Most current tapes are kept off site. Older tapes are in locked storage.

The system maintains a database, or logs, of each back-up session. Successful completion of each back-up can be verified each morning by accessing the job report logs in ARCServe. This is done each morning. Any missed jobs can be rescheduled and completed in the morning of the following day. As noted above, this database is re-archived after every normal back-up and can be retrieved at any time necessary. Back-ups are the responsibility of the IT Manager. Clarification of procedures can be found in the *UP-IS-014,* TestAmerica Chicago IT Procedures and Processes SOP.

7.7 Removal of Data

There is a substantial amount of space available to both BNAs and VOAs on the current data system. Data older than approximately six months is zipped and remains on the system. At the IT Manager's, Technical Manager's or GCMS Supervisor's discretion, previously archived data is purged from the system.

8.0 QUALITY CONTROL

8.1 QC Summary

The department will review the quality controls as follows:

8.1.1 Method Blank (MB) / Laboratory Control Standard (LCS)

At least one MB and LCS will be included in each laboratory batch. Regardless of the matrix being processed, the LCS and MB will be in an aqueous media.

The MB will be examined to determine if contamination is being introduced in the laboratory. The LCS will be examined to determine accuracy and precision.

8.1.2 Accuracy

Accuracy will be measured by the percent recovery (%R) of the LCS. Method 8260B refers to Method 8000 for guidance on initial demonstration performance criteria. Guidelines for LCS/LCSD and MS/MSD accuracy limits can also be found in Method 8000. The laboratory's current in-house statistical limits can be found in Table 1. The number of compounds being used for bench level control and the accuracy limits assigned to those compounds may vary with client, QAP, project, or state certifications etc. This information is transmitted to the bench via the COC, kick-off meetings, tech profiles, job note or NCM etc.., and indicated on one of the forms used at the bench. In-house generated limits are subject to change.

8.1.3 Precision

Precision will be measured by the reproducibility of the LCS and will be calculated as Relative Percent Difference (RPD). Current limits are listed in Table 1. RPD's are not used to assess bench level Corrective Action.

8.1.4 Surrogates

Surrogate Compounds will be added to every sample to measure performance of the analysis. Method guidance limits are listed in Attachment 2. In-house statistical limits are listed in Table 1. Guidelines for the generation of statistical surrogate limits can be found in Method 8000. As with LCS samples, surrogate recovery limits may vary with client, QAP, project, etc.., and the information transmitted to the bench in the same manner.

8.1.5 QC Charting/Generation of Statistical Limits

Precision and accuracy are monitored using LCS data. Review of QC Charts and generation of in-house statistical criteria, including surrogate limits, is completed on an annual basis. Additional data may be added at QA/QC discretion during the year for other purposes. Spike levels are 50 ppb. Only routine compounds are spiked and should be representative of the whole. The more non-routine compounds are not part of the spiking solutions. Other limitations (availability of second source) may also prevent adding these to spiking solutions.

8.2 Corrective Actions

Listed below are the steps to be taken when an out-of-control situation occurs. The analyst must address the following issues as described below in the individual sections.

- demonstrate that all of the problems creating the out-of-control situation were addressed;
- document the problem and the action that was taken to correct the problem;
- document that an in-control situation has been achieved; and
- receive approval (signature) of the supervisor, project manager, QC personnel or other qualified personnel prior to release of data associated with the problem.

Corrective Actions are documented on the Corrective Action/Qualification Report included in the instrument logbook. In addition, a sample tracking form, specific to a unique job, is attached to the sample tracking documentation. The corrective action/qualification report and sample tracking form are used to note all out-of-control events, the actions taken to try and correct the problem, the return to control.

Discussed below are the suggested and required courses of action when an out-of-control situation has occurred.

8.2.1 BFB Criteria

If BFB criteria can not be met, determine if the source of the problem is instrumental or tune related. Inspect overall sensitivity, possible excessive background, the proportionality of the masses, relative abundances of the target masses. If it seems tune-related, adjust the tune parameters in Manual Tune slightly, until acceptance is achieved. If the problem seems instrumental, perform suggested trouble-shooting to locate and correct the problem (Suggestions can be found in most of the manuals). NO analysis can proceed until criteria are met. Corrective action for BFB analysis is documented on the corrective action/qualification report in the logbook.

8.2.2 Initial Calibration

If initial calibration can not be met, determine if the problem is analytical or instrumental. Some suggested questions to ask would be:

- were the standards prepared correctly?
- was the proper amount analyzed?
- check the chromatogram - did something happen on one or two analyses; i.e., a leak
- check the response factors - is one concentration level very high or low? re-analyze
- how old are the standards?

All calibration criteria must be met (Section 6.4). If the ICAL does not meet specified criteria, at minimum, the appropriate levels must be re-analyzed. If necessary, new standards should be prepared and the levels re-analyzed. During analysis of an initial calibration, documentation of the re-analyses of specific levels is not required. See previous section outlining CA for minimum COD values as well. Refer to the TestAmerica Corporate Policy, CA-T-P-002, *Selection of Calibration Points* (Attachment 7), for further guidance.

8.2.3 Continuing Calibration

If continuing calibration can not be met, determine if the problem is analytical or instrumental. Some suggestions:

- check the chromatography
- is overall sensitivity low?
- excessive background?
- how old is the standard?
- need a new 5-point?
- has the tune shifted?

Compare the relative abundances of 69, 131 and 219 from that day's manual tune to those on the day the initial calibration was analyzed. Slight adjustments to the tune may bring the standard in. Certain compounds will help indicate what the problem is.

All calibration criteria must be met (Section 6.5). If the CCAL does not meet specified criteria, at minimum the standard should be re-analyzed. A new standard may be prepared and then re-analyzed. If necessary, a new ICAL must be run. All corrective action taken for CCAL's must be recorded on the corrective action/qualification report and included in the logbook.

8.2.4 Method Blank (MB)

If the MB is/appears to be contaminated, re-analyze. If contamination is still present, the problem may be in one of the common elements, such as the trap, transfer line, port valve or column. Baking the trap/column and running position blanks may be necessary. If contamination has occurred beyond that, and maintenance is required (i.e., replace trap) it is documented in the Maintenance logbook. All corrective action taken for Method Blanks must be recorded on the corrective action/qualification report and included in the logbook. Under extenuating circumstances, if analysis continues, qualification must be made as to the positive hits above the RL for the compounds in question. Any associated samples analyzed in the tune must be noted. Any samples containing positive hits must be noted. IF, the samples containing positive hits can not be re-analyzed (i.e., past hold-time), the positive hits are flagged with "B" and the situation and data noted and qualified in a case narrative and/or Non-Conformance Memo (NCM).

8.2.5 Surrogates

All surrogate recoveries are calculated. If ANY surrogates are outside limits in the MB, it must be re-analyzed. Analyses CAN NOT proceed until an in-control situation is demonstrated. Re-analyze the blank. If surrogates are still out, the instrument may need to be re-tuned (BFB) and/or another calibration standard analyzed. If the problem persists, further maintenance action may be required (i.e., trap replacement, clean instrument).

Before pursuing other measures, check to be sure that:

- calculations are correct
- concentrations of the surrogates in the spiking solution are correct
- the correct amount of ISS/SSS solution was added
- ISS/SSS areas are reasonable

If any surrogates in a sample are outside limits, check the above first. Any sample that has a surrogate out must be re-analyzed. The re-analysis can take the form of a dilution, if there is reasonable expectation that a high concentration of a target compound is causing a matrix effect. If the surrogate(s) is/are still outside limits, a matrix effect is demonstrated and both reports are submitted. Depending on the client, the best result may be reported and the other result narrated. If all surrogates are in-control on the re-analysis, only the second analysis is reported.

Every effort is made to complete the re-analysis within hold-time. If this is impossible (i.e., capacity hold-times preclude re-analyses within hold-time), both reports may be submitted. This is documented in the narrative.

If the sample with the out-of-control surrogates is the same sample on which the MS and MSD were performed, and the pattern is duplicated, then re-analysis is NOT required. Documentation of the similarities is required.

Surrogate corrective action is documented on the sample tracking form for samples.

8.2.6 Laboratory Control Sample (LCS)

As specified in Section 8.1.2, the number of compounds and the limits used to assess accuracy vary with client, QAP, project etc. The in-house generated limits are listed in Table 1. In-house limits are subject to change. The need and course of corrective action varies with the number of compounds being used for bench control and positive detected of compounds outside limits. The LCS limits are based on the mean recovery +/- 3 standard deviations; therefore, it is statistically quite likely that there will be exceedances of the limits for a few analytes when a large number of compounds are included in the LCS. Therefore a number of individual analytes are allowed to exceed the LCS control limit before the LCS as a whole is considered to have failed. The control limit for a marginal exceedance is mean +/- 4 standard deviations which results in a larger range of +/-10% outside the in-house generated limits. The number of marginal exceedances allowed is based on DOD guidance:

Number of analytes in LCS	Number of marginal exceedances allowed
> 90	5
71-90	4
51-70	3
31-50	2
11-30	1
< 11	0

(1) AFCEE or Specified Projects: All compounds are used for bench control. Samples may require re-analysis if contaminants of concern identified by the client or QAPP fail in the LCS, regardless of the number of marginal exceedences

- If any compound exhibits low recoveries, the LCS is re-analyzed. If the compound is still low a new spike may be prepared and the LCS re-analyzed. Analysis should not continue until the situation is taken care of. All corrective action is documented at the time and return to control demonstrated for low compounds. IF, in extenuating circumstances, analysis is continued, the data must be qualified for those compounds in a NCM.

- If any compound exhibits high recoveries, the LCS may be re-analyzed, and/or a new solution prepared, and/or a new standard prepared and calibrations repeated. All corrective action is recorded at the time on the corrective action/qualification report and included in the logbook and return to control documented if applicable. If positive detects are noted, and the samples are unable to be re-analyzed, the situation must be documented. AFCEE allows for high recoveries on a one time basis if the said compounds are not detected in the associated samples. Any high compounds, the associated samples, and presence of absence of those compounds must be addressed in a NCM. Following the first occurrence of high recoveries, the bench will take appropriate note and follow-up with appropriate Corrective Action within a reasonable amount of time.

(2) QAP's etc.., specifying five compounds.

- ALL five compounds must be within limits for analysis to proceed. The LCS samples may be re-analyzed. New spike solutions may be prepared. Or new standards or CCAL's may be analyzed. All corrective action and return to control must be documented at the time on the corrective action/qualification report and included in the logbook.

- The actual limits used for the five compounds may be QAP-specific (usually those listed in the table in the appendix) or in-house generated by matrix and method. In either case, the above corrective action and required documentation apply.

- For all other compounds in the full-list spike, all recoveries are assessed, although no immediate corrective action may be required. If the recoveries are low, in general another LCS may be re-analyzed. The spike solution and standard may be verified for correct concentrations. However, no corrective action is absolutely required by the bench unless an error is discovered. The recoveries may or may not be documented in a NCM, however, they are noted on the review form. The recoveries of the "un-controlled" compounds may be used for data interpretation.

- Although not strictly required to take immediate corrective action, the purpose of the full-spike is two-fold in that the bench should use it as an indicator of the status of the calibration standards, instrument conditions etc.., as well as a tool for data interpretation. Therefore, in keeping with good lab practice, the situation should be noted and assessed and any corrective action deemed necessary should be taken within a reasonable amount of time (Example: High recoveries on gases => new calibration standard may be needed).

- For South Carolina, all compounds in the LCS must meet 70-130% unless the compounds are identified as poor purgers in the SOP. The poor purgers should still be within 60-140%. South Carolina does not allow the use of mariginal exceedances.

8.2.7 Matrix Spikes (MS)

As specified in Section 8.1.2, the number of compounds and the limits used to assess accuracy vary with client, QAP, project, state certifications etc. In-house generated limits are listed in Table 1. In-house limits are subject to change. The need and course of corrective action varies with the number of compounds being used for bench control and recoveries of same compounds in the associated LCS samples. The following guidelines are used:

(1) AFCEE or Specified Projects: All compounds are used for bench level control.

- If the MS exhibits recoveries outside limits, AFCEE requires it to be re-analyzed as the MSD. No further action is required. Documentation is required and association made to the LCS for those compounds in the NCM. See above specifications for associated LCS samples.

(2) QAP's etc., specifying 5 compounds.

- ALL 5 compounds are assessed. If recoveries are outside limits, the LCS is reviewed for those compounds. If the recoveries are within limits in the associated LCS samples, no further action is required. See above section concerning LCS corrective action for further information and action required for recoveries outside limits in LCS samples.

- The actual limits used for the five compounds may be QAP-specific (usually those listed in the table in the appendix) or in-house generated by matrix and method. In either case, corrective action and required documentation apply.

- For all other compounds in the full-list spike, all recoveries are assessed, although no immediate corrective action may be required. The affected compounds may be compared to the same compounds in the associated LCS samples. See the above section for further information and action required for these compounds in the LCS samples. The recoveries may or may not be documented in the Job narrative, however, they are noted on the review form. The recoveries of the "un-controlled" compounds may be used for data interpretation.

8.2.8 Internal Standard Policy

Method 8260 does not require re-analyses of samples for low internal standard areas. However, it is TestAmerica's policy to monitor areas and retention times, therefore, the following guidelines apply.

Situations requiring re-analyses :

- If ALL areas are outside limits the sample will be re-analyzed.
- Any sample that has a positive hit associated with any internal standard outside limits will be re-analyzed.
- If ANY surrogates are outside limits the sample will be re-analyzed.

Situations NOT requiring re-analyses :

- If all surrogates are within limits and there are no positive hits associated with those internal that are outside limits, the sample does not have to be re-analyzed. Situation should be addressed in a NCM.
- If all surrogates are within limits, but there is an obvious matrix effect occurring, even if positive hits are noted, the sample does not need to be re-analyzed. This decision will be approved by the supervisor. The situation should be addressed in a NCM.

- If there is historical evidence that shows a repeated pattern for a certain client and site, and this can be documented by reviewing past projects, the samples do not have to be re-analyzed. This decision will be approved by the supervisor and documented in a NCM.
- Corrective action for internal standard areas for samples is documented on the sample tracking form.

Any sample showing retention times outside windows will be re-analyzed. This is documented in the appropriate manner as in the preceding paragraph.

9.0 DATA ANALYSIS AND CALCULATIONS

9.1 Computer Data Production/Reduction

The Target 3.5 software produces a Total Ion Chromatogram (TIC), header, quant report and background subtracted spectra. For those clients requiring it, a 5 tentatively identified compound (TIC) search is also performed. The data system will produce an integration listing and tentative identification of each hit found at the selected percentage of the largest peak present.

9.1.1 Quantitation of Target Compounds

Quantitation of the target compounds is performed by the data system can be accomplished as follows:

WATERs: concentration (mg/L) = $\frac{[A_x \times I_s]}{[A_{is} \times RF]} \times DF$

Where:
A_x = area of characteristic ion for target
I_s = concentration of internal standard (ng)
A_{is} = area of characteristic ion for int. std.
RF = response factor for target
DF = dilution factor (if any)

SOILs: concentration (mg/kg) = $\frac{[A_x \times I_s]}{[A_{is} \times RF \times D]} \times DF$

Where:
All variables are equal and
D = (100 - % moisture in sample/100) or 1 for wet weight. (As in the case of drum samples)

The target methods all contain calculations for waters and soils that allow automatic processing and calculations of concentrations to be completed. The user may enter some variables (Dilution Factor) and others are imported from LIMS. Sample prep info for VOA's is entered directly into LIMS. The sample volume is considered to be "constant" for calculation purposes. Less sample volume (in the case of waters) and soil weight (in the case of soils) are taken into account in the dilution factor entered by the user. For medium-level soils and waste/drum type samples medium level calculations are needed and actual weights are brought into LIMS.

NOTE: As noted previously, weights are recorded to 0.1 gram. It is SOP to weigh out 5.0 grams (or as appropriate for the dilution), however, to keep data entry and calculations simple. The same holds true for all water volumes.

9.1.2 Accuracy: $\%R = \frac{(A_T - A_0)}{A_F} \times 100$

Where:
A_T = Total amount recovered in fortified sample
A_0 = Amount recovered in unfortified sample
A_F = Amount added to sample

9.1.3 Precision: $\%D = \frac{|B_1 - B_2|}{B_1} \times 100$

$RPD = \frac{|B_1 - B_2|}{(B_1 + B_2)/2} \times 100$

Where:
B_1 = %Recovery MS (or LCS)
B_2 = %Recovered MSD (or LCS)

9.1.4 Modifications for 8260B quantitation

Initial Calibration Criteria

Methods 8000B/8260B require the use of linear or higher order calibration curves for those compounds exceeding 15%.

The following equations apply :

Linear Regression: $y = a_0 + a_1 * x$ Quadratic Curve: $y = a_o + (a1 * x) + (a2 * x^2)$
Weighted Linear Regression: $y = a_1 * x + a_0$

Where: $x = Area_{UNK}/Area_{ISTD}$
$y = Amount_{UNK}/Amount_{ISTD}$
a_1 = slope
a_0 = y-intercept

The equation for weighted linear regression follows the linear regression but introduces a weighting factor for the slope and y-intercept. The User manually enters the weighting factor into the method as: 1/Amt or $1/Amt^2$

Once the $Amount_{UNK}$ is solved, the value is adjusted for total solids, dilution factors etc.., to calculate a final concentration.

The quantitation of compounds using linear regressions and quadratic curves as performed automatically by the Target software has been confirmed to be accurate.

Method 8000B/8260B specifies a minimum COD(R^2). The corrective action regarding an initial calibration for method 8260B as it relates to the 0.990 correlation coefficient acceptance criteria is outlined. When a compound has a correlation coefficient less than 0.990, the occurrence is documented by the analyst in the Corrective Action section of the instrument's logbook. Any corrective action or data qualification is also documented on the corrective action/qualification report and included with the logbook. All corrective actions taken may include those listed below.

Samples may be analyzed against an initial calibration that have compounds with a correlation coefficient less than 0.990 and the corrective actions taken may also include some but not all of the following:

- The data for these samples may be reported without qualification if the compounds with a correlation coefficient less than 0.990 are not detected in the sample; therefore no further corrective action is required.
- If a compound is detected in the sample that has a correlation coefficient less than 0.990, the samples may be reanalyzed against an initial calibration with an acceptable correlation coefficient and only the reanalysis will be reported on the sample. If this reanalysis occurs beyond analysis hold times then both analyses on the sample will be reported.
- If a compound is detected in the sample that has a correlation coefficient less than 0.990, the decision to reanalyze or to reported the data without further corrective action is made on a case by case basis with the approval of the supervisor, the project manager and the client. The sample results may require qualification for this compound on the report and will be addressed in the case narrative.

Continuing Calibration Check

Prior to sample analysis a 50 ppb calibration check is completed. All minimum RF's must meet same limits. All CCC's must be less than 20% Drift as calculated below; the analysts may verify %DIFF and only calculate those that are close. (Error may only be made in favor of tighter control).

$$\%Drift = \frac{(Ci - Cc)}{Ci} \times 100$$

Where:
Ci = standard conc. (10/50)
Cc = measured conc. in cal check

9.1.5 Quantitation of TIC's (Tentatively Identified Compounds)

Quantitation of TIC's is performed by the Chrom processing software. The formulas above for waters and soils can be used with the following modifications. A_x and A_{is} should be taken from the total ion integration listing accompanying the TIC report produced by the data system. The nearest non-interfered with internal standard should be used. The RF is assumed to be one (1). The concentration is therefore an estimate and is flagged as such with a "J". Any TIC also found in the MB is flagged with a "JB". Any TIC identified with a CAS number is also flagged with an "N", indicating that the ID was based on the mass spectra. The operator should visually confirm that the integration is correct. If not, the peak in question must be manually integrated. The Chrom system automatically calculates the actual concentration of the TIC's, including dilutions and total solids, once that information is retrieved from TALs (LIMS).

9.2 Operator Data Reduction/Review

The operator does on-screen review of all data and

- makes judgments concerning the "realness" of those target compounds found and
- makes judgments concerning the identification of the tentatively identified compounds
- modifies the output to produce a data package reflective of those decisions

9.2.1 Initial Review

The GC/MS VOA area uses two kinds of corrective action documentation. The first consists of the Batch Information section in Analyst Desktop in TALs (LIMs). This area in TALs contains a comment section to report out-of-control situations, corrective action and return-to-control for documenting problems related to general QC: tune, ICAL, CCAL, internal standard areas from CCAL to ICAL, and LCS samples. The second are the sample tracking forms that refer to a single job. These are used to record events, corrective actions and final actions for surrogates, internal standard areas, carry-over situations, analyses past tune time, MS/MSD data etc.., for each sample in the batch. These forms are attached to the other sample documentation that accompanies the job through analysis. Both may be used during initial review of the data. See Section 8.2 of this SOP for details on Corrective Action.

All data is initially reviewed on-screen. The review is both a QC review and a general review as described below.

- The MB contains no interferences or target compounds at the RL.
- ALL surrogates are in control in the blank. Surrogate limits are listed in Table 1;
- ALL surrogates in samples are in control;
- LCS recoveries meet the limits listed in Table 1. See Section 8.2 concerning compounds and limits for LCS samples. In-house limits have been generated and are in use.
- Internal standard areas and retention times are checked and meet guidelines. Limits are listed in Attachment 2. Additional guidelines can be found in Section 8.2.
- The sample does not require any further dilutions or analysis at a more concentrated level. Dilutions are made to keep the target in the upper half of the calibration range. The MS and MSD are never diluted to get spiked or non-spiked compounds within range, as this would reduce the matrix affect assessment.
- Visually confirm complete integration for any large and/or saturated target compounds.
- The sample does not require re-analysis for any other reason (i.e., leak, analysis past tune time, ISTD areas low, etc.).

9.2.2 Identification of Targets

The following guidelines are used in the positive identification of target compounds.

1. "elution of component at the same relative retention time as the standard component."

The elution times should compare within +- 30 s. The standard must be run on the same 12 hour period as the sample. If co-eluting analytes interfere with the comparisons of retention times, other ions characteristic to that compound can be used to confirm relative retention times.

2. "correspondence of the sample component and standard component mass spectrum." Comparisons of sample spectra to standard spectra must be made using standard spectra obtained from the GC/MS system.

All ions present in the standard spectrum at a 10% relative intensity (most abundant ion being 100%) should be present in the sample.

The relative intensities of the above ions should agree within ±20%, between the standard and sample. If an ion is 50% intensity in the standard the corresponding ion must be between 30 and 70% in the sample.

Ions >10% in the sample but not present in the standard should be considered and accounted for.

3. Operator judgment. If a compound can not be verified by the above, but in the operators technical judgment the ID is correct, it is reported as such.

- Once all positive identification is made, the file is modified to reflect these decisions. At this time TIC's may also be reviewed and name. In each case where the file has been edited or manual integrations have taken place the operator must identify, initial and date the changes on the hardcopy (if such is generated).

9.2.3 Manual Integration Policy

In each case where the file has been edited or manual integrations have been performed the operator must identify, initial, and date the changes on the hardcopy report. The following guidelines apply:

- Manual integrations should be consistent between all files integrated.
- Manual integrations should not be performed to meet QC criteria.
- Manual integrations are automatically flagged with an 'M' on the raw data.
- Excessive manual integrations may reflect an instrumental or methodological problem that should be addressed.
- Manual integrations shall follow the TestAmerica Corporate SOP for Manual Integrations (CA-Q-S-002 – Attachment 8). Example integrations and documentation are provided within this attachment.

Manual integrations are most often performed for the following reasons:

- Assignment of correct peak that was mis-identified by the data system.
- Incomplete auto-integration due to high level of target compound detected.
- Incomplete auto-integration due to background interference.
- Incorrect auto-integration due to co-elution or near co-elution of compounds.
- Missed peaks.

All manual integrations are reviewed, initialed and dated. For those clients requiring full data packages, spectra and Extracted Ion Chromatography Profiles (EICP) are printed for all manually integrated compounds. Manual integrations are documented in the case narrative and a Manual Integration Summary is included in the data package.

9.2.4 Identification of TIC's

In general, up to as many as 5 non-target compounds are tentatively identified by the data system and operator. Compounds with responses >10% of the nearest ISTD are identified. The data system provides the operator with a SUB ADC C sample spectrum, spectra of the first three matches and a listing of two other possibilities. Molecular formulas, molecular weights and CAS #'s are included. The following guidelines are used:

Relative intensities of major ions in the reference spectrum should be present in the sample (ions >10%).

- Relative ions should agree within ±20%;
- Molecular ions in the reference should be in the samples;
- Review the possibility of background and/or co-eluting compounds for those ions present in the sample but not in the standard;
- If ions are present in the sample but not in the standard, review the possibility of the presence of background or co-eluting compounds;
- If ions are present in the standard but not in the sample, review the possibility that the ions were subtracted out because they are also common to the background or co-eluting compounds;
- In the event no valid interpretation can be made, the compound is called "unknown".
- Interpretation can be often narrowed down to a class of compounds, molecular formula or weight.

9.3 Final Review

9.3.1 Once (a) the analysis is determined to be acceptable and (b) the initial review and data reduction has occurred and (c) the analyst has entered sample prep info into TALs (LIMS), the following steps occur. The sample prep information, client ID information and some data applicable to fields in the forms is retrieved from TALs (LIMS).

9.3.2 All necessary forms are then generated using the TALs. The package is then assembled and ready for the first review. For level 2 data reports and similar deliverables other data may be generated for review purposes. In these cases, final packages with raw data and forms are not generated. Review of all reports and associated data is required regardless of data deliverable level.

9.3.3 Analytical data goes through a 200% review cycle. As results are generated, analysts review their work to ensure that the results generated meet QC requirements and relevant EPA methodologies. The analyst transfers the data into TALs in the Analyst Desktop module. Where non-compliance is observed, the analyst creates Non-Conformance Memos (NCMs) in TALs. Flags and data qualifiers can be method, project, program or QAPP specific. The analyst documents the initial review on a data review checklist (Attachment 6) and sets the batch status in LIMs to 2nd level. The peer/supervisor review of the data is conducted by another individual who has been trained on the review process or by the department supervisor. This secondary review is documented on the same checklist, making any necessary corrections to the data or additions to the NCMs as necessary. The batch is then set to lab complete. Any Spectra and all manual integrations are reviewed. For the organic instruments, manual integrations may also be electronically reviewed utilizing auditing software to help ensure compliance to the ethics and manual integration policies. The raw data, including the checklist, instrument print-outs, and manual entries, and electronic files are retained for easy retrieval in accordance with the laboratory's record and retention policy outlined in the SOP, *UP-QA-QAM, Section 15.*

Examples of items included in the above reviews are as follows:

- QC data are outside the specified control limits for accuracy and precision
- Unusual detection limit changes are observed
- Samples having unusually high results
- Samples exceeding a known regulatory limit
- Raw data indicating some type of contamination or poor technique

- Inconsistent peak integration (if applicable)
- Transcription errors
- Results outside of calibration range

Note: The complete analysis scheme can be summarized below (Section 7.1.1 & 7.1.2) and in Attachment 5. The entire sample tracking system can be summarized in Attachment 5.

10.0 POLLUTION CONTROL

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

10.1 Waste Management

Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to UP-WM-001.

The following waste streams are produced when this method is carried out.

- Waste from this procedure will enter the 'Flammable Vials' wastestream.

11.0 METHOD PERFORMANCE CRITERIA

Refer to Sections 1, 6, 7 and 8.

12.0 REFERENCES

Refer to Section 1.0

13.0 ATTACHMENTS

Table 1. Estimated Quantitation Limits for Volatile Analytes; Laboratory Statistical Control Limits/ Surrogate Recovery Guidelines
Table 2. Characteristic Mass for Purgeable Organics Compounds

Figure 1. Example: Total Ion Chromatogram for 5 mL Purge Water
Figure 2. Example: Total Ion Chromatogram for 5 mL Purge Soil

Attachment 1. Example: Initial Calibration Guides; Method Listings; Concentrator Conditions; Flow Settings
Attachment 2. Example: Target and Internal Standards; Internal Standard Guidelines; Initial Calibration (Form 6)
Attachment 3. Example: Sample Run Log; Corrective Action/Qualification Report; GC/MS VOA Maintenance Logbook; Sample Tracking Sheet; GC/MS VOA-ICOC Form
Attachment 4. Example: Continuing Calibration Evaluation and Acceptance Criteria (Form 7)
Attachment 5. Example: Analysis and Sample Tracking Flowcharts
Attachment 6. Example: Data Review Checklist
Attachment 7. CA-T-P-002: TestAmerica Corporate Policy for the Selection of Calibration Points
Attachment 8. CA-Q-S-002: TestAmerica Corporate SOP for Manual Integrations
Attachment 9. List of Poor Purging or Poorly Performing Compounds
Attachment 10 DOD QSM Version 4.2: Appendix F QC Requirements Summary (Table F-1; and Table F-4)

14.0 REVISION HISTORY

- Revision 21, was updated on 07/29/11
- Annual Review
- Reference to the Target software has been replaced with a Chrom software reference.
- Reference to 10 mL purge volume has been replaced with a 5 mL purge volume.
- Reference to the standard preparation logbook has been removed.
- Updated standard preparation tables to reflect current practices.
- General text clarifications.
- Sections 6.4 and 6.5 were updated to include the SPCCs and CCCs.
- Sections 6.4 and 8.2.6 were updated to include specific criteria for South Carolina sample analysis.
- Section 7.3.1 was updated to include SOP Change Form changes.

Table 1.

Example: Estimated Quantitation Limits for Volatile Analytes[a] Laboratory Statistical Control Limits and Surrogate Recovery Guidelines (039-001 to 039-009)

[a]Estimated Quantitation Limit (EQL) – The lowest concentration that can be reliably achieved within specified limits of precision and accuracy during routine laboratory operating conditions. The EQL is generally 3 to 5 times the MDL. However, it may be nominally chosen within these guidelines to simplify data reporting. For many analytes the EQL analyte concentration is selected for the lowest non-zero standard in the calibration curve. Sample EQLs are highly matrix-dependent. The EQLs listed herein are provided for guidance and may not always be achievable. See the following example information for further guidance on matrix-dependent EQLs.

[b]EQLs listed for soil/sediment are based on wet weight. Normally data is reported on a dry weight basis; therefore, EQLs will be higher, based on the percent dry weight in each sample.

[c]The number of compounds being used for bench level control and the accuracy limits assigned to those compounds may vary with client, QAP, project, State Certifications etc. This information is transmitted to the bench via job notes and NCMs attached to the projects.

MDL W GCMS VOA	Volatile Organic Compounds (GC/MS)	8260B												
WATER	Analyte Description	CAS Number	RL - Limit	LOD - Limit	MDL - Limit	Units	LCSREC LCL	LCSREC - UCL	LCSRPD	MSREC - LCL	MSREC - UCL	MSRPD	SUREC - LCL	SUREC - UCL
	1,1,1,2-Tetrachloroethane	630-20-6	1	0.5	0.31	ug/L	73	122	20	73	122	20		
	1,1,1-Trichloroethane	71-55-6	1	0.5	0.26	ug/L	66	128	20	66	128	20		
	1,1,2,2-Tetrachloroethane	79-34-5	1	0.5	0.35	ug/L	66	121	20	66	121	20		
	1,1,2-Trichloro-1,2,2-trifluoroethane	76-13-1	1	0.5	0.4	ug/L	43	126	20	43	126	20		
	1,1,2-Trichloroethane	79-00-5	1	0.5	0.3	ug/L	62	137	20	62	137	20		
	1,1-Dichloroethane	75-34-3	1	0.5	0.24	ug/L	64	117	20	64	117	20		
	1,1-Dichloroethene	75-35-4	1	0.5	0.29	ug/L	60	126	20	60	126	20		
	1,1-Dichloropropene	563-58-6	1	0.5	0.25	ug/L	71	112	20	71	112	20		
	1,2,3-Trichlorobenzene	87-61-6	1	0.5	0.36	ug/L	66	119	20	66	119	20		
	1,2,3-Trichloropropane	96-18-4	1	0.6	0.6	ug/L	68	124	20	68	124	20		
	1,2,4-Trichlorobenzene	120-82-1	1	0.5	0.22	ug/L	63	115	20	63	115	20		
	1,2,4-Trimethylbenzene	95-63-6	1	0.5	0.22	ug/L	76	117	20	76	117	20		
	1,2-Dibromo-3-Chloropropane	96-12-8	2	1.2	1.21	ug/L	54	119	20	54	119	20		
	1,2-Dibromoethane	106-93-4	1	0.5	0.45	ug/L	71	125	20	71	125	20		
	1,2-Dichlorobenzene	95-50-1	1	0.5	0.21	ug/L	80	110	20	80	110	20		
	1,2-Dichloroethane	107-06-2	1	0.5	0.28	ug/L	69	115	20	69	115	20		
	1,2-Dichloroethene, Total	540-59-0	2	1	0.47	ug/L	68	115	20	68	115	20		
	1,2-Dichloropropane	78-87-5	1	0.5	0.36	ug/L	68	123	20	68	123	20		
	1,3,5-Trimethylbenzene	108-67-8	1	0.5	0.23	ug/L	77	117	20	77	117	20		
	1,3-Dichlorobenzene	541-73-1	1	0.5	0.26	ug/L	79	110	20	79	110	20		
	1,3-Dichloropropane	142-28-9	1	0.5	0.27	ug/L	71	119	20	71	119	20		
	1,4-Dichlorobenzene	106-46-7	1	0.5	0.24	ug/L	79	109	20	79	109	20		
	1-Chlorohexane	544-10-5	1	0.5	0.32	ug/L	66	127	20	66	127	20		
	2,2-Dichloropropane	594-20-7	1	0.5	0.31	ug/L	50	127	20	50	127	20		
	2-Chloroethyl vinyl ether	110-75-8	2	1	0.65	ug/L	37	136	20	37	136	20		
	2-Chlorotoluene	95-49-8	1	0.5	0.21	ug/L	77	117	20	77	117	20		
	2-Hexanone	591-78-6	5	2.5	0.56	ug/L	55	138	20	55	138	20		
	4-Chlorotoluene	106-43-4	1	0.5	0.21	ug/L	75	114	20	75	114	20		
	Acetone	67-64-1	5	2.5	1.9	ug/L	43	153	20	43	153	20		
	Acetonitrile	75-05-8	20	10	7.7	ug/L			20					
	Acrolein	107-02-8	100	50	18.5	ug/L			20			20		
	Acrylonitrile	107-13-1	20	10	4.7	ug/L						20		
	Benzene	71-43-2	0.5	0.25	0.12	ug/L	74	113	20	74	113	20		
	Bromobenzene	108-86-1	1	0.5	0.31	ug/L	80	117	20	80	117	20		
	Bromochloromethane	74-97-5	1	0.5	0.5	ug/L	69	116	20	69	116	20		
	Bromodichloromethane	75-27-4	1	0.5	0.23	ug/L	73	120	20	73	120	20		
	Bromoform	75-25-2	1	0.5	0.45	ug/L	64	126	20	64	126	20		
	Bromomethane	74-83-9	1	0.5	0.49	ug/L	46	155	20	46	155	20		
	Carbon disulfide	75-15-0	5	2.5	0.44	ug/L	36	110	20	36	110	20		
	Carbon tetrachloride	56-23-5	1	0.5	0.28	ug/L	58	132	20	58	132	20		

(039-001)

Analyte Description	CAS Number	RL - Limit	LOD - Limit	MDL - Limit	Units	LCSREC LCL	LCSREC - UCL	LCSRPD	MSREC - LCL	MSREC - UCL	MSRPD	SUREC - LCL	SUREC - UCL
Chlorobenzene	108-90-7	1	0.5	0.24	ug/L	81	111	20	81	111	20		
Chloroethane	75-00-3	1	0.5	0.33	ug/L	54	149	20	54	149	20		
Chloroform	67-66-3	1	0.5	0.25	ug/L	71	116	20	71	116	20		
Chloromethane	74-87-3	1	0.5	0.24	ug/L	36	148	20	36	148	20		
cis-1,2-Dichloroethene	156-59-2	1	0.5	0.22	ug/L	66	111	20	66	111	20		
cis-1,3-Dichloropropene	10061-01-5	1	0.5	0.28	ug/L	65	114	20	65	114	20		
Dibromochloromethane	124-48-1	1	0.5	0.25	ug/L	73	118	20	73	118	20		
Dibromomethane	74-95-3	1	0.5	0.39	ug/L	73	115	20	73	115	20		
Dichlorodifluoromethane	75-71-8	1	0.5	0.26	ug/L	39	139	20	39	139	20		
Ethylbenzene	100-41-4	0.5	0.25	0.14	ug/L	79	114	20	79	114	20		
Hexachlorobutadiene	87-68-3	1	0.5	0.45	ug/L	62	124	20	62	124	20		
Isopropylbenzene	98-82-8	1	0.5	0.21	ug/L	65	110	20	65	110	20		
m&p-Xylene	179601-23-1	1	0.5	0.3	ug/L	77	117	20	77	117	20		
Methyl Ethyl Ketone	78-93-3	5	2.5	1	ug/L	42	152	20	42	152	20		
methyl isobutyl ketone	108-10-1	5	2.5	0.79	ug/L	56	138	20	56	138	20		
Methyl tert-butyl ether	1634-04-4	1	0.5	0.28	ug/L	57	119	20	57	119	20		
Methylene Chloride	75-09-2	5	2.5	0.63	ug/L	65	125	20	65	125	20		
Naphthalene	91-20-3	1	0.5	0.24	ug/L	62	122	20	62	122	20		
n-Butylbenzene	104-51-8	1	0.5	0.21	ug/L	72	120	20	72	120	20		
N-Propylbenzene	103-65-1	1	0.5	0.19	ug/L	76	116	20	76	116	20		
o-Xylene	95-47-6	0.5	0.25	0.13	ug/L	74	117	20	74	117	20		
p-Isopropyltoluene	99-87-6	1	0.5	0.24	ug/L	72	114	20	72	114	20		
Propionitrile	107-12-0	20	10	10	ug/L								
sec-Butylbenzene	135-98-8	1	0.5	0.19	ug/L	76	116	20	76	116	20		
Styrene	100-42-5	1	0.5	0.26	ug/L	76	118	20	76	118	20		
tert-Butylbenzene	98-06-6	1	0.5	0.24	ug/L	75	117	20	75	117	20		
Tetrachloroethene	127-18-4	1	0.5	0.22	ug/L	76	114	20	76	114	20		
Tetrahydrofuran	109-99-9	5	2.5	1.4	ug/L	47	134	20	47	134	20		
Toluene	108-88-3	0.5	0.25	0.15	ug/L	76	121	20	76	121	20		
trans-1,2-Dichloroethene	156-60-5	1	0.5	0.27	ug/L	67	120	20	67	120	20		
trans-1,3-Dichloropropene	10061-02-6	1	0.5	0.35	ug/L	60	119	20	60	119	20		
Trichloroethene	79-01-6	0.5	0.25	0.18	ug/L	75	116	20	75	116	20		
Trichlorofluoromethane	75-69-4	1	0.5	0.22	ug/L	60	141	20	60	141	20		
Vinyl acetate	108-05-4	2	1	0.48	ug/L	52	147	20	52	147	20		
Vinyl chloride	75-01-4	0.5	0.25	0.13	ug/L	47	138	20	47	138	20		
1,2-Dichloroethane-d4 (Surr)	17060-07-0				ug/L							77	124
Toluene-d8 (Surr)	2037-26-5				ug/L							80	121
4-Bromofluorobenzene (Surr)	460-00-4				ug/L							77	112
Dibromofluoromethane	1868-53-7				ug/L							78	119
Dichlorofluoromethane	75-43-4	1	0.5	0.23	ug/L			20			20		

MDL W GCMS VOA	Purge and Trap	5030B

(C-2) C-372

(039-002)

MDL W GCMS VOA (APIX)	Volatile Organic Compounds (GC/MS)	8260B												
	Analyte Description	CAS Number	RL - Limit	LOD - Limit	MDL - Limit	Units	LCSREC LCL	LCSREC - UCL	LCSRPD	MSREC - LCL	MSREC - UCL	MSRPD	SUREC - LCL	SUREC - UCL
	3-Chloropropene	107-05-1	2.5	1.2	1.1	ug/L								
	2-Chloro-1,3-butadiene	126-99-8	1	0.5	0.23	ug/L								
	Ethyl methacrylate	97-63-2	2.5	1.2	0.94	ug/L								
	Iodomethane	74-88-4	2.5	1.2	0.99	ug/L	55	144	20	55	144	20		
	Isobutanol	78-83-1	100	50	37.8	ug/L								
	Methacrylonitrile	126-98-7	2.5	1.2	0.88	ug/L								
	Methyl methacrylate	80-62-6	2.5	1.2	1.04	ug/L								
	Pentachloroethane	76-01-7	10	1.2	1.23	ug/L								
	trans-1,4-Dichloro-2-butene	110-57-6	10	1.2	1.1	ug/L								
	1,3,5-Trichlorobenzene	108-70-3	1	0.5	0.41	ug/L	63	131	20	63	131	20		
	Ethyl ether	60-29-7	1	0.5	0.38	ug/L	44	116	20	44	116	20		
	Hexane	110-54-3	1	0.5	0.28	ug/L	44	123	20	44	123	20		
	Isopropyl ether	108-20-3	1	0.5	0.27	ug/L								
	Ethyl acetate	141-78-6	10	5	4.37	ug/L								
	Heptane	142-82-5	1	0.5	0.41	ug/L	47	140	20	47	140	20		
	2-Methylnaphthalene	91-57-6	1	0.5	0.22	ug/L								
	Butadiene	106-99-0	1	0.5	0.42	ug/L								
	n-Butyl alcohol	71-36-3	100	50	46.1	ug/L								
	2-Nitropropane	79-46-9	100	50	31.8	ug/L								
	Cyclohexanone	108-94-1	100	50	51.3	ug/L								
	Cyclohexane	110-82-7	1	0.5	0.35	ug/L			20			20		
	Methylcyclohexane	108-87-2	1	0.5	0.32	ug/L			20			20		
	Methyl acetate	79-20-9	2	1	0.65	ug/L			20			20		
	2,2,4-Trimethylpentane	540-84-1	1	0.5	0.5	ug/L								
MDL W GCMS VOA	Purge and Trap	5030B												

(039-003)

MDL S MSVOA_Low Default List	Volatile Organic Compounds (GC/MS)	8260B												
Soil	Analyte Description	CAS Number	RL - Limit	LOD - Limit	MDL - Limit	Units	LCSREC LCL	LCSREC - UCL	LCSRPD	MSREC - LCL	MSREC - UCL	MSRPD	SUREC - LCL	SUREC - UCL
	1,1,1,2-Tetrachloroethane	630-20-6	5	2.5	0.85	ug/Kg	77	113	30	77	113	30		
	1,1,1-Trichloroethane	71-55-6	5	2.5	0.96	ug/Kg	67	115	30	67	115	30		
	1,1,2,2-Tetrachloroethane	79-34-5	5	2.5	0.68	ug/Kg	73	114	30	73	114	30		
	1,1,2-Trichloro-1,2,2-trifluoroethane	76-13-1	5	2.5	0.91	ug/Kg	47	129	30	47	129	30		
	1,1,2-Trichloroethane	79-00-5	5	2.5	0.67	ug/Kg	69	118	30	69	118	30		
	1,1-Dichloroethane	75-34-3	5	2.5	0.79	ug/Kg	70	113	30	70	113	30		
	1,1-Dichloroethene	75-35-4	5	2.5	0.79	ug/Kg	60	128	30	60	128	30		
	1,1-Dichloropropene	563-58-6	5	2.5	0.66	ug/Kg	69	111	30	69	111	30		
	1,2,3-Trichlorobenzene	87-61-6	5	2.5	0.71	ug/Kg	68	116	30	68	116	30		
	1,2,3-Trichloropropane	96-18-4	5	2.5	1.03	ug/Kg	73	114	30	73	114	30		
	1,2,4-Trichlorobenzene	120-82-1	5	2.5	0.53	ug/Kg	64	111	30	64	111	30		
	1,2,4-Trimethylbenzene	95-63-6	5	2.5	0.74	ug/Kg	78	112	30	78	112	30		
	1,2-Dibromo-3-Chloropropane	96-12-8	5	2.5	1.65	ug/Kg	61	112	30	61	112	30		
	1,2-Dibromoethane	106-93-4	5	2.5	0.59	ug/Kg	74	110	30	74	110	30		
	1,2-Dichlorobenzene	95-50-1	5	2.5	0.59	ug/Kg	80	112	30	80	112	30		
	1,2-Dichloroethane	107-06-2	5	2.5	0.51	ug/Kg	74	114	30	74	114	30		
	1,2-Dichloroethene, Total	540-59-0	5	2.5	1.35	ug/Kg	63	114	30	63	114	30		
	1,2-Dichloropropane	78-87-5	5	2.5	1.13	ug/Kg	77	116	30	77	116	30		
	1,3,5-Trimethylbenzene	108-67-8	5	2.5	1.04	ug/Kg	79	114	30	79	114	30		
	1,3-Dichlorobenzene	541-73-1	5	2.5	0.62	ug/Kg	78	112	30	78	112	30		
	1,3-Dichloropropane	142-28-9	5	2.5	0.62	ug/Kg	75	115	30	75	115	30		
	1,4-Dichlorobenzene	106-46-7	5	2.5	0.58	ug/Kg	78	112	30	78	112	30		
	1-Chlorohexane	544-10-5	5	2.5	1.15	ug/Kg	69	117	30	69	117	30		
	2,2-Dichloropropane	594-20-7	5	2.5	0.92	ug/Kg	51	117	30	51	117	30		
	2-Chloroethyl vinyl ether	110-75-8	5	2.5	2.11	ug/Kg	22	136	30	22	136	30		
	2-Chlorotoluene	95-49-8	5	2.5	0.59	ug/Kg	78	117	30	78	117	30		
	2-Hexanone	591-78-6	5	2.5	0.71	ug/Kg	58	138	30	58	138	30		
	4-Chlorotoluene	106-43-4	5	2.5	0.82	ug/Kg	75	114	30	75	114	30		
	Acetone	67-64-1	5	2.5	2.45	ug/Kg	43	149	30	43	149	30		
	Acetonitrile	75-05-8	80	40	12.7	ug/Kg			30			30		
	Acrolein	107-02-8	400	200	40	ug/Kg			30			30		
	Acrylonitrile	107-13-1	80	40	8	ug/Kg			30			30		
	Benzene	71-43-2	5	2.5	0.54	ug/Kg	74	112	30	74	112	30		
	Bromobenzene	108-86-1	5	2.5	1	ug/Kg	79	115	30	79	115	30		
	Bromochloromethane	74-97-5	5	2.5	0.83	ug/Kg	61	117	30	61	117	30		
	Bromodichloromethane	75-27-4	5	2.5	0.76	ug/Kg	76	108	30	76	108	30		
	Bromoform	75-25-2	5	2.5	0.81	ug/Kg	66	115	30	66	115	30		
	Bromomethane	74-83-9	5	2.5	1.07	ug/Kg	36	146	30	36	146	30		
	Carbon disulfide	75-15-0	5	2.5	0.71	ug/Kg	27	107	30	27	107	30		

(039-004)

Analyte Description	CAS Number	RL - Limit	LOD - Limit	MDL - Limit	Units	LCSREC LCL	LCSREC - UCL	LCSRPD	MSREC - LCL	MSREC - UCL	MSRPD	SUREC - LCL	SUREC - UCL
Carbon tetrachloride	56-23-5	5	2.5	1.09	ug/Kg	64	116	30	64	116	30		
Chlorobenzene	108-90-7	5	2.5	0.79	ug/Kg	80	110	30	80	110	30		
Chloroethane	75-00-3	5	2.5	1.05	ug/Kg	34	144	30	34	144	30		
Chloroform	67-66-3	5	2.5	0.92	ug/Kg	70	112	30	70	112	30		
Chloromethane	74-87-3	5	2.5	0.82	ug/Kg	48	136	30	48	136	30		
cis-1,2-Dichloroethene	156-59-2	5	2.5	0.73	ug/Kg	62	111	30	62	111	30		
cis-1,3-Dichloropropene	10061-01-5	5	2.5	0.57	ug/Kg	68	103	30	68	103	30		
Dibromochloromethane	124-48-1	5	2.5	0.69	ug/Kg	76	110	30	76	110	30		
Dibromomethane	74-95-3	5	2.5	0.72	ug/Kg	75	108	30	75	108	30		
Dichlorodifluoromethane	75-71-8	5	2.5	1.1	ug/Kg	34	140	30	34	140	30		
Ethylbenzene	100-41-4	5	2.5	0.75	ug/Kg	78	112	30	78	112	30		
Hexachlorobutadiene	87-68-3	5	2.5	0.95	ug/Kg	65	117	30	65	117	30		
Isopropylbenzene	98-82-8	5	2.5	0.79	ug/Kg	67	101	30	67	101	30		
m&p-Xylene	179601-23-1	10	5	1.45	ug/Kg	77	114	30	77	114	30		
Methyl Ethyl Ketone	78-93-3	5	2.5	1.08	ug/Kg	58	140	30	58	140	30		
methyl isobutyl ketone	108-10-1	5	2.5	0.85	ug/Kg	65	127	30	65	127	30		
Methyl tert-butyl ether	1634-04-4	5	2.5	0.75	ug/Kg	55	116	30	55	116	30		
Methylene Chloride	75-09-2	5	2.5	1.4	ug/Kg	49	125	30	49	125	30		
Naphthalene	91-20-3	5	2.5	0.43	ug/Kg	68	116	30	68	116	30		
n-Butylbenzene	104-51-8	5	2.5	0.71	ug/Kg	72	118	30	72	118	30		
N-Propylbenzene	103-65-1	5	2.5	0.72	ug/Kg	74	116	30	74	116	30		
o-Xylene	95-47-6	5	2.5	0.7	ug/Kg	77	114	30	77	114	30		
p-Isopropyltoluene	99-87-6	5	2.5	0.65	ug/Kg	72	111	30	72	111	30		
Propionitrile	107-12-0	80	40	8	ug/Kg								
sec-Butylbenzene	135-98-8	5	2.5	0.81	ug/Kg	78	114	30	78	114	30		
Styrene	100-42-5	5	2.5	0.63	ug/Kg	78	109	30	78	109	30		
tert-Butylbenzene	98-06-6	5	2.5	0.82	ug/Kg	78	114	30	78	114	30		
Tetrachloroethene	127-18-4	5	2.5	0.95	ug/Kg	76	114	30	76	114	30		
Tetrahydrofuran	109-99-9	5	2.5	1.01	ug/Kg	58	119	30	58	119	30		
Toluene	108-88-3	5	2.5	0.97	ug/Kg	77	113	30	77	113	30		
trans-1,2-Dichloroethene	156-60-5	5	2.5	0.71	ug/Kg	62	119	30	62	119	30		
trans-1,3-Dichloropropene	10061-02-6	5	2.5	1.13	ug/Kg	63	107	30	63	107	30		
Trichloroethene	79-01-6	5	2.5	0.81	ug/Kg	76	111	30	76	111	30		
Trichlorofluoromethane	75-69-4	5	2.5	0.81	ug/Kg	45	137	30	45	137	30		
Vinyl acetate	108-05-4	5	2.5	0.73	ug/Kg	66	129	30	66	129	30		
Vinyl chloride	75-01-4	5	2.5	0.7	ug/Kg	44	130	30	44	130	30		
1,2-Dichloroethane-d4 (Surr)	17060-07-0				ug/Kg							69	120
4-Bromofluorobenzene (Surr)	460-00-4				ug/Kg							67	120
Dibromofluoromethane	1868-53-7				ug/Kg							69	120
Toluene-d8 (Surr)	2037-26-5				ug/Kg							69	122

(039-005)

MDL S MSVOA_Low APIX	Volatile Organic Compounds (GC/MS)	8260B												
	Analyte Description	CAS Number	RL - Limit	LOD - Limit	MDL - Limit	Units	LCSREC LCL	LCSREC - UCL	LCSRPD	MSREC - LCL	MSREC - UCL	MSRPD	SUREC - LCL	SUREC - UCL
	1,3,5-Trichlorobenzene	108-70-3	5	2.5	0.75	ug/Kg	70	126	30	70	126	30		
	2-Chloro-1,3-butadiene	126-99-8	5	2.5	0.93	ug/Kg								
	2-Methylnaphthalene	91-57-6	5	2.5	1.15	ug/Kg								
	2-Nitropropane	79-46-9	400	200	112	ug/Kg								
	3-Chloropropene	107-05-1	10	5	2.23	ug/Kg								
	Butadiene	106-99-0	5	2.5	1.29	ug/Kg								
	Cyclohexane	110-82-7	5	2.5	0.95	ug/Kg			30			30		
	Cyclohexanone	108-94-1	400	200	189	ug/Kg								
	Ethyl acetate	141-78-6	50	25	16.2	ug/Kg								
	Ethyl ether	60-29-7	5	2.5	0.78	ug/Kg	50	119	30	50	119	30		
	Ethyl methacrylate	97-63-2	10	5	2.55	ug/Kg								
	Hexane	110-54-3	5	2.5	0.63	ug/Kg	50	113	30	50	113	30		
	Iodomethane	74-88-4	10	5	1.58	ug/Kg	56	143	30	56	143	30		
	Isobutanol	78-83-1	400	200	130	ug/Kg								
	Isopropyl ether	108-20-3	5	2.5	0.86	ug/Kg								
	Methacrylonitrile	126-98-7	10	5	3.35	ug/Kg								
	Methyl acetate	79-20-9	5	2.5	1.94	ug/Kg			30			30		
	Methyl methacrylate	80-62-6	10	5	3.93	ug/Kg								
	Methylcyclohexane	108-87-2	5	2.5	0.98	ug/Kg			30			30		
	n-Butyl alcohol	71-36-3	400	200	129	ug/Kg								
	Pentachloroethane	76-01-7	20	10	3.58	ug/Kg								
	trans-1,4-Dichloro-2-butene	110-57-6	10	5	2.68	ug/Kg								
	2,2,4-Trimethylpentane	540-84-1	5	2.5	2.5	ug/Kg								
MDL S MSVOA_Low APIX	Closed System Purge and Trap	5035A_LP												

(039-004)

* Prep Factor of 50x Needs to be applied to the RL, LOD, MDL Listed.

MDL S MSVOA Default List	Volatile Organic Compounds (GC/MS)	8260B												
Soil – MeOH (High Level)	Analyte Description	CAS Number	* RL - Limit	* LOD - Limit	* MDL - Limit	Units	LCSREC LCL	LCSREC - UCL	LCSRPD	MSREC - LCL	MSREC - UCL	MSRPD	SUREC - LCL	SUREC - UCL
	1,1,1,2-Tetrachloroethane	630-20-6	2	1	0.337	ug/Kg	76	117	30	76	117	30		
	1,1,1-Trichloroethane	71-55-6	1	0.5	0.262	ug/Kg	70	125	30	70	125	30		
	1,1,2,2-Tetrachloroethane	79-34-5	1	0.5	0.352	ug/Kg	73	119	30	73	119	30		
	1,1,2-Trichloro-1,2,2-trifluoroethane	76-13-1	2	1	0.476	ug/Kg	47	128	30	47	128	30		
	1,1,2-Trichloroethane	79-00-5	1	0.5	0.302	ug/Kg	63	136	30	63	136	30		
	1,1-Dichloroethane	75-34-3	1	0.5	0.244	ug/Kg	69	118	30	69	118	30		
	1,1-Dichloroethene	75-35-4	1	0.5	0.29	ug/Kg	60	123	30	60	123	30		
	1,1-Dichloropropene	563-58-6	1	0.5	0.254	ug/Kg	74	113	30	74	113	30		
	1,2,3-Trichlorobenzene	87-61-6	2	1	0.458	ug/Kg	72	116	30	72	116	30		
	1,2,3-Trichloropropane	96-18-4	2	1	0.595	ug/Kg	71	123	30	71	123	30		
	1,2,4-Trichlorobenzene	120-82-1	2	1	0.282	ug/Kg	68	111	30	68	111	30		
	1;2,4-Trimethylbenzene	95-63-6	2	1	0.298	ug/Kg	76	116	30	76	116	30		
	1,2-Dibromo-3-Chloropropane	96-12-8	2	1	1.27	ug/Kg	49	131	30	49	131	30		
	1,2-Dibromoethane	106-93-4	2	1	0.749	ug/Kg	74	124	30	74	124	30		
	1,2-Dichlorobenzene	95-50-1	2	1	0.397	ug/Kg	80	111	30	80	111	30		
	1,2-Dichloroethane	107-06-2	1	0.5	0.281	ug/Kg	66	120	30	66	120	30		
	1,2-Dichloroethene, Total	540-59-0	2	1	0.466	ug/Kg	70	114	30	70	114	30		
	1,2-Dichloropropane	78-87-5	1	0.5	0.356	ug/Kg	72	124	30	72	124	30		
	1,3,5-Trimethylbenzene	108-67-8	2	1	0.275	ug/Kg	78	117	30	78	117	30		
	1,3-Dichlorobenzene	541-73-1	2	1	0.285	ug/Kg	80	110	30	80	110	30		
	1,3-Dichloropropane	142-28-9	1	0.5	0.268	ug/Kg	76	117	30	76	117	30		
	1,4-Dichlorobenzene	106-46-7	2	1	0.29	ug/Kg	79	109	30	79	109	30		
	1-Chlorohexane	544-10-5	2	1	0.639	ug/Kg	68	126	30	68	126	30		
	2,2-Dichloropropane	594-20-7	1	0.5	0.305	ug/Kg	60	126	30	60	126	30		
	2-Chloroethyl vinyl ether	110-75-8	2	1	0.653	ug/Kg	46	137	30	46	137	30		
	2-Chlorotoluene	95-49-8	1	0.5	0.208	ug/Kg	78	116	30	78	116	30		
	2-Hexanone	591-78-6	5	2.5	0.559	ug/Kg	58	137	30	58	137	30		
	4-Chlorotoluene	106-43-4	1	0.5	0.205	ug/Kg	78	113	30	78	113	30		
	Acetone	67-64-1	5	2.5	1.92	ug/Kg	46	152	30	46	152	30		
	Acetonitrile	75-05-8	16	8	7.65	ug/Kg								
	Acrolein	107-02-8	40	20	12	ug/Kg								
	Acrylonitrile	107-13-1	8	4	2.41	ug/Kg								
	Benzene	71-43-2	0.25	0.12	0.08	ug/Kg	74	112	30	74	112	30		
	Bromobenzene	108-86-1	2	1	0.425	ug/Kg	79	117	30	79	117	30		
	Bromochloromethane	74-97-5	2	1	0.542	ug/Kg	68	120	30	68	120	30		
	Bromodichloromethane	75-27-4	2	1	0.275	ug/Kg	73	122	30	73	122	30		
	Bromoform	75-25-2	2	1	0.569	ug/Kg	62	119	30	62	119	30		
	Bromomethane	74-83-9	2	1	0.861	ug/Kg	38	157	30	38	157	30		
	Carbon disulfide	75-15-0	5	2.5	0.439	ug/Kg	38	112	30	38	112	30		
	Carbon tetrachloride	56-23-5	1	0.5	0.281	ug/Kg	63	127	30	63	127	30		

(039-007)

Analyte Description	CAS Number	RL - Limit	LOD - Limit	MDL - Limit	Units	LCSREC LCL	LCSREC - UCL	LCSRPD	MSREC - LCL	MSREC - UCL	MSRPD	SUREC - LCL	SUREC - UCL
Chlorobenzene	108-90-7	1	0.5	0.238	ug/Kg	80	110	30	80	110	30		
Chloroethane	75-00-3	2	1	0.496	ug/Kg	53	156	30	53	156	30		
Chloroform	67-66-3	1	0.5	0.249	ug/Kg	74	115	30	74	115	30		
Chloromethane	74-87-3	2	1	0.497	ug/Kg	44	148	30	44	148	30		
cis-1,2-Dichloroethene	156-59-2	1	0.5	0.224	ug/Kg	68	110	30	68	110	30		
cis-1,3-Dichloropropene	10061-01-5	1	0.5	0.279	ug/Kg	65	116	30	65	116	30		
Dibromochloromethane	124-48-1	2	1	0.377	ug/Kg	66	123	30	66	123	30		
Dibromomethane	74-95-3	2	1	0.478	ug/Kg	74	115	30	74	115	30		
Dichlorodifluoromethane	75-71-8	2	1	0.536	ug/Kg	51	144	30	51	144	30		
Ethylbenzene	100-41-4	0.25	0.12	0.14	ug/Kg	79	112	30	79	112	30		
Hexachlorobutadiene	87-68-3	2	1	0.663	ug/Kg	68	118	30	68	118	30		
Isopropylbenzene	98-82-8	2	1	0.325	ug/Kg	65	110	30	65	110	30		
m&p-Xylene	179601-23-1	0.5	0.25	0.296	ug/Kg	78	114	30	78	114	30		
Methyl Ethyl Ketone	78-93-3	5	2.5	1.04	ug/Kg	48	152	30	48	152	30		
methyl isobutyl ketone	108-10-1	5	2.5	0.79	ug/Kg	58	135	30	58	135	30		
Methyl tert-butyl ether	1634-04-4	2	1	0.478	ug/Kg	57	122	30	57	122	30		
Methylene Chloride	75-09-2	5	2.5	0.63	ug/Kg	67	126	30	67	126	30		
Naphthalene	91-20-3	2	1	0.478	ug/Kg	68	120	30	68	120	30		
n-Butylbenzene	104-51-8	1	0.5	0.206	ug/Kg	75	118	30	75	118	30		
N-Propylbenzene	103-65-1	2	1	0.336	ug/Kg	76	116	30	76	116	30		
o-Xylene	95-47-6	0.25	0.12	0.129	ug/Kg	74	114	30	74	114	30		
p-Isopropyltoluene	99-87-6	2	1	0.316	ug/Kg	73	113	30	73	113	30		
Propionitrile	107-12-0	16	8	2.9	ug/Kg								
sec-Butylbenzene	135-98-8	1	0.5	0.191	ug/Kg	77	116	30	77	116	30		
Styrene	100-42-5	1	0.5	0.261	ug/Kg	77	115	30	77	115	30		
tert-Butylbenzene	98-06-6	1	0.5	0.237	ug/Kg	76	116	30	76	116	30		
Tetrachloroethene	127-18-4	1	0.5	0.217	ug/Kg	76	112	30	76	112	30		
Tetrahydrofuran	109-99-9	4	2	1.41	ug/Kg	49	136	30	49	136	30		
Toluene	108-88-3	0.25	0.12	0.151	ug/Kg	78	116	30	78	116	30		
trans-1,2-Dichloroethene	156-60-5	1	0.5	0.271	ug/Kg	70	119	30	70	119	30		
trans-1,3-Dichloropropene	10061-02-6	1	0.5	0.351	ug/Kg	64	114	30	64	114	30		
Trichloroethene	79-01-6	0.25	0.12	0.151	ug/Kg	75	113	30	75	113	30		
Trichlorofluoromethane	75-69-4	2	1	0.367	ug/Kg	64	139	30	64	139	30		
Vinyl acetate	108-05-4	2	1	0.483	ug/Kg	52	155	30	52	155	30		
Vinyl chloride	75-01-4	0.25	0.12	0.126	ug/Kg	58	136	30	58	136	30		
1,2-Dichloroethane-d4 (Surr)	17060-07-0				ug/Kg							77	124
Toluene-d8 (Surr)	2037-26-5				ug/Kg							80	121
4-Bromofluorobenzene (Surr)	460-00-4				ug/Kg							77	112
Dibromofluoromethane	1868-53-7				ug/Kg							78	119
Dichlorofluoromethane	75-43-4	2	1	0.294	ug/Kg								

MDL S MSVOA Default List	Closed System Purge and Trap	5035A_M_Calc

(C-2) C-378

(039-088)

MDL S MSVOA APIX	Volatile Organic Compounds (GC/MS)	8260B												
	Analyte Description	CAS Number	RL - Limit	LOD - Limit	MDL - Limit	Units	LCSREC LCL	LCSREC - UCL	LCSRPD	MSREC - LCL	MSREC - UCL	MSRPD	SUREC - LCL	SUREC - UCL
	1,3,5-Trichlorobenzene	108-70-3	2	1	0.407	ug/Kg	66	126	30	66	126	30		
	2-Chloro-1,3-butadiene	126-99-8	2	1	0.231	ug/Kg								
	2-Methylnaphthalene	91-57-6	2	1	0.223	ug/Kg								
	2-Nitropropane	79-46-9	200	100	31.8	ug/Kg								
	3-Chloropropene	107-05-1	4	2	1.1	ug/Kg								
	Butadiene	106-99-0	2	1	0.417	ug/Kg								
	Cyclohexane	110-82-7	2	1	0.348	ug/Kg			30			30		
	Cyclohexanone	108-94-1	200	100	51.3	ug/Kg								
	Ethyl acetate	141-78-6	20	10	4.37	ug/Kg								
	Ethyl ether	60-29-7	2	1	0.377	ug/Kg	45	119	30	45	119	30		
	Heptane	142-82-5	2	1	0.411	ug/Kg	48	138	30	48	138	30		
	Hexane	110-54-3	2	1	0.275	ug/Kg	34	123	30	34	123	30		
	Iodomethane	74-88-4	4	2	0.985	ug/Kg	55	140	30	55	140	30		
	Isobutanol	78-83-1	200	100	37.8	ug/Kg								
	Isopropyl ether	108-20-3	2	1	0.267	ug/Kg								
	Methacrylonitrile	126-98-7	4	2	0.881	ug/Kg								
	Methyl acetate	79-20-9	2	1	0.653	ug/Kg			30			30		
	Methyl methacrylate	80-62-6	4	2	1.04	ug/Kg								
	Methylcyclohexane	108-87-2	2	1	0.317	ug/Kg			30			30		
	n-Butyl alcohol	71-36-3	200	100	46.1	ug/Kg								
	Pentachloroethane	76-01-7	16	2	1.23	ug/Kg								
	trans-1,4-Dichloro-2-butene	110-57-6	16	2	1.1	ug/Kg								
	2,2,4-Trimethylpentane	540-84-1	2	1	1	ug/Kg								
MDL S MSVOA APIX	Closed System Purge and Trap	5035A_M_Calc												

(039-009)

Table 2

Characteristic Mass (m/z) for Purgeable Organic Compounds

Analyte	Primary Characteristic Ion	Secondary Characteristic Ion
Benzene	78	
Bromobenzene	156	77,158
Bromochloromethane	128	49,130
Bromodichloromethane	83	85,127
Bromoform	173	175,254
Bromomethane	94	96
n-Butylbenzene	91	92,134
sec-Butylbenzene	105	134
tert-Butylbenzene	119	91,134
Carbon tetrachloride	117	119
Chlorobenzene	112	77,114
Chloroethane	64	66
Chloroform	83	85
Chloromethane	50	52
2-Chlorotoluene	91	126
4-Chlorotoluene	91	126
1,2-Dibromo-3-chloropropane	75	155,157
Dibromochloromethane	129	127
1,2-Dibromoethane	107	109,188
Dibromomethane	93	95,174
1,2-Dichlorobenzene	146	111,148
1,3-Dichlorobenzene	146	111,148
1,4-Dichlorobenzene	146	111,148
Dichlorodifluoromethane	85	87
1,1-Dichloroethane	63	65,83
1,2-Dichloroethane	62	98
1,1-Dichloroethene	96	61,63
cis-1,2-Dichloroethene	96	61,98
trans-1,2-Dichloroethene	96	61,98
1,2-Dichloropropane	63	112
1,3-Dichloropropane	76	78
2,2-Dichloropropane	77	97
1,1-Dichloropropene	75	110,77
Ethylbenzene	91	106
Hexachlorobutadiene	225	223,227
Isopropylbenzene	105	120
p-Isopropylbenzene	119	134, 91
Methylene chloride	84	86,49
Naphthalene	128	86, 49
n-Propylbenzene	91	120
Styrene	104	78
1,1,1,2-Tetrachloroethane	131	133, 119
1,1,2,2-Tetrachloroethane	83	131, 85
Tetrachloroethene	164	129,131,166

Table 2
(continued)
Characteristic Mass (m/z) for Purgeable Organic Compounds

Analyte	Primary Characteristic Ion	Secondary Characteristic Ion
Toluene	92	91
1,2,3-Trichlorobenzene	180	182, 145
1,2,4-Trichlorobenzene	180	182, 145
1,1,1-Trichloroethane	97	99, 61
1,1,2-Trichloroethane	83	97, 85
Trichloroethene	95	97, 130, 132
Trichlorofluoromethane	151	101,153
1,2,3-Trichloropropane	75	77
1,2,4-Trimethylbenzene	105	120
1,3,5-Trimethylbenzene	105	120
Vinyl chloride	62	64
o-Xylene	106	91
m-Xylene	106	91
p-Xylene	106	91
cis-1,3-Dichloropropene	75	77, 39
trans-1,3-Dichloropropene	75	77, 39
Internal Std./Surrogates		
4-Bromofluorobenzene (S)	95	174, 176
1,4-Dichlorobenzene-d_4 (IS)	152	115, 150
Pentafluorobenzene (IS)	168	
Chlorobenzene-d_5 (IS)	117	
1,4-Difluorobenzene (IS)	114	
1,2-Dichloroethane-d_4 (S)	65	
Toluene-d_8 (S)	98	
Dibromofluoromethane (S)	113	

*__NOTE:__ The primary and secondary ions listed here are taken directly from SW-846 Method 8260. The laboratory uses secondary ions in the cases of Ethylbenzene, Toluene, 1,1,2-Trichloroethane, Trichloroethene, 1,2,3-Trichloropropane and Xylenes due to interferences and/or to maintain consistency between methods.

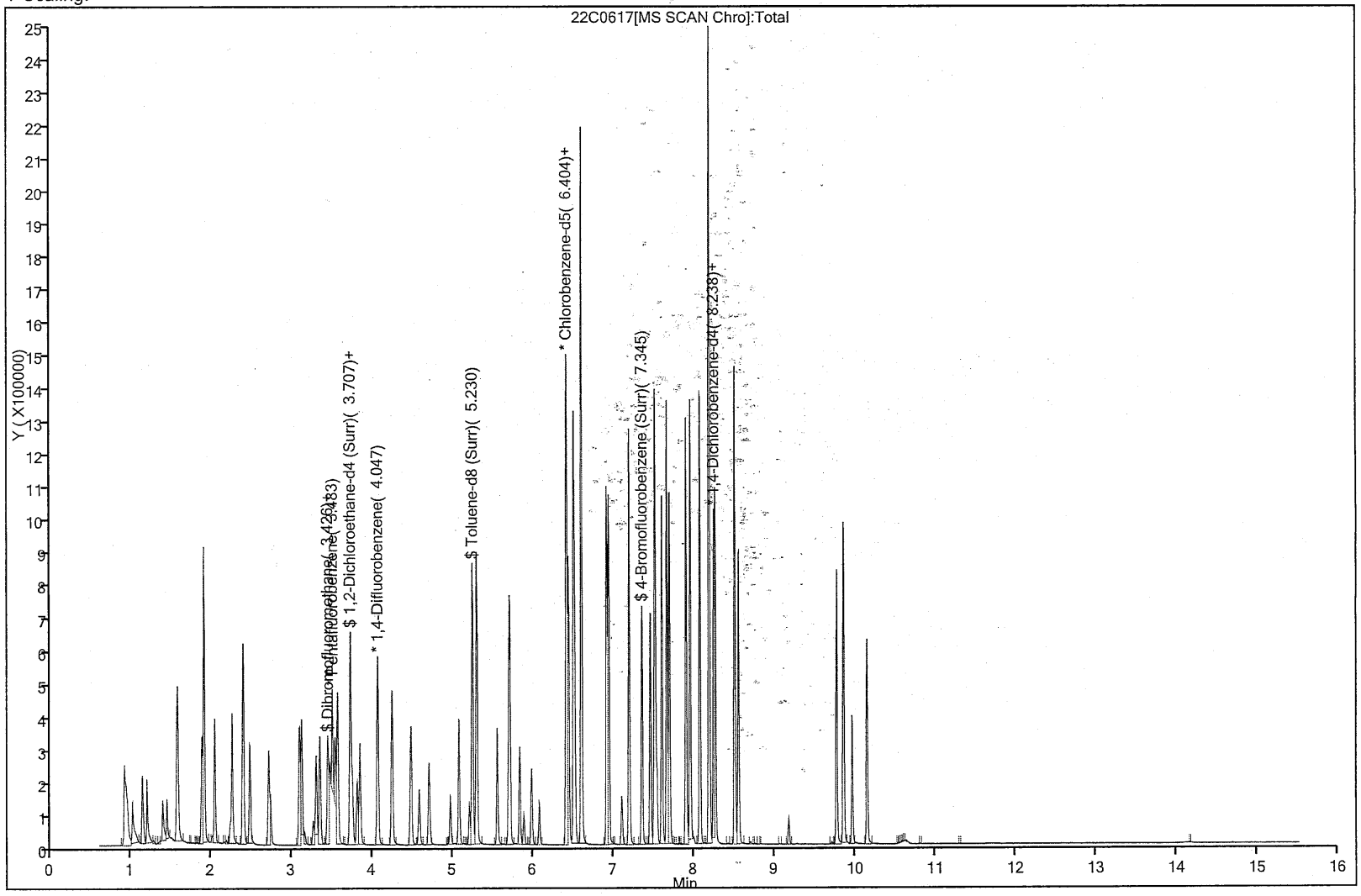
Figure 1.

Example: Total Ion Chromatogram for 5 mL Purge Water (042-001 to 042-002)

Report Date: 02-Aug-2011 12:34:45 Chrom Revision: 1.2 13-Jul-2011 10:43:06

Preliminary Report

Data File: \\chi-svr07\chromdata\CMS22\20110617-4051.b\22C0617.D
Injection Date: 17-Jun-2011 12:12:30 Limit Group: MSVOA_8260_ICAL_WATER
Client ID: Instrument ID: CMS22
Lims Batch ID: 4051 Lims Sample ID: 2
Operator ID: EA
Y Scaling:



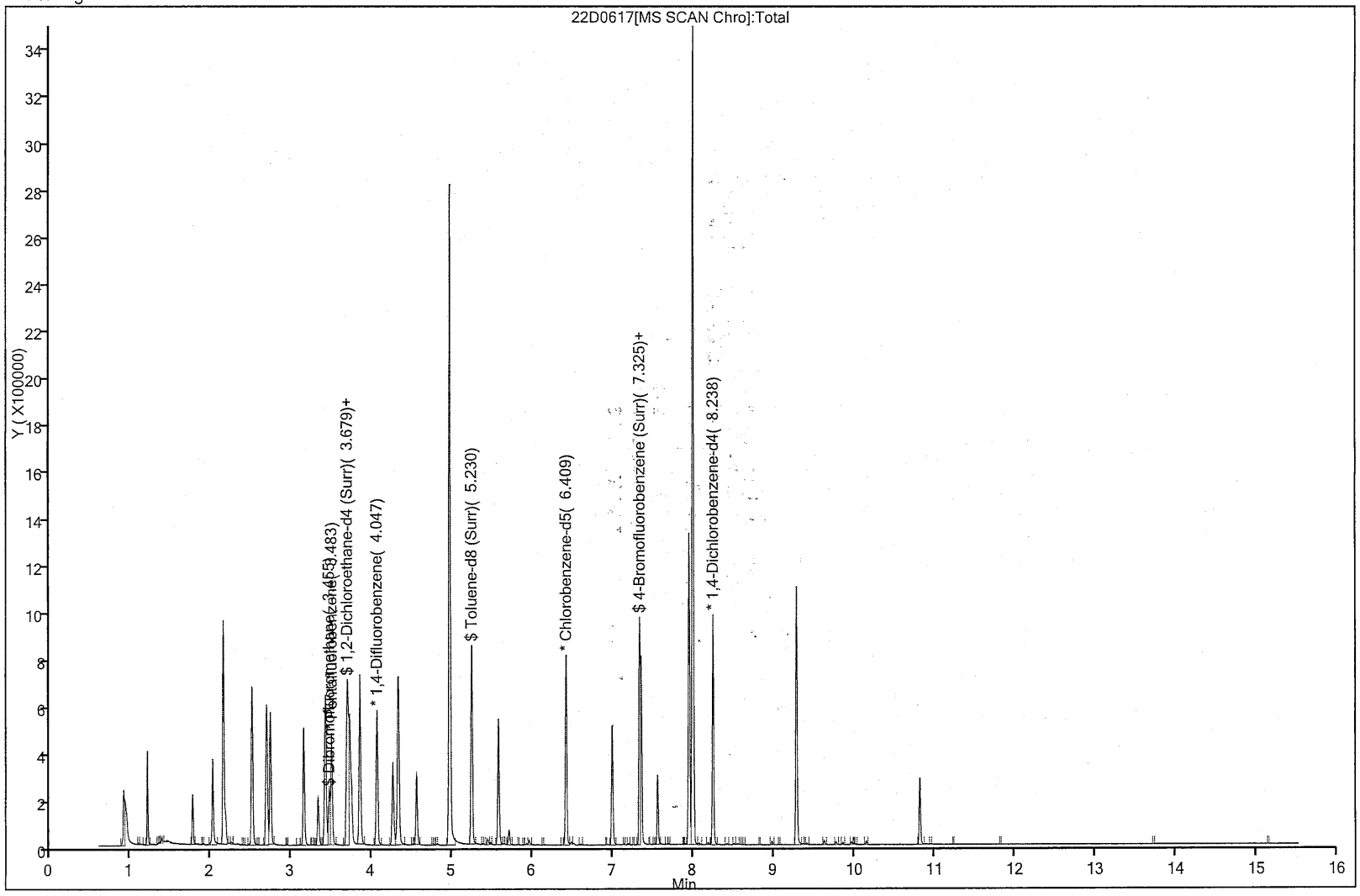
(C-2) C-383

(042-001)

Report Date: 02-Aug-2011 12:42:29 Chrom Revision: 1.2 13-Jul-2011 10:43:06

Preliminary Report

Data File: \\chi-svr07\chromdata\CMS22\20110617-4051.b\22D0617.D
Injection Date: 17-Jun-2011 12:35:30 Limit Group: MSVOA_8260_ICAL_WATER
Client ID: Instrument ID: CMS22
Lims Batch ID: 4051 Lims Sample ID: 3
Operator ID: EA
Y Scaling:



(C-2) C-384

(042-002)

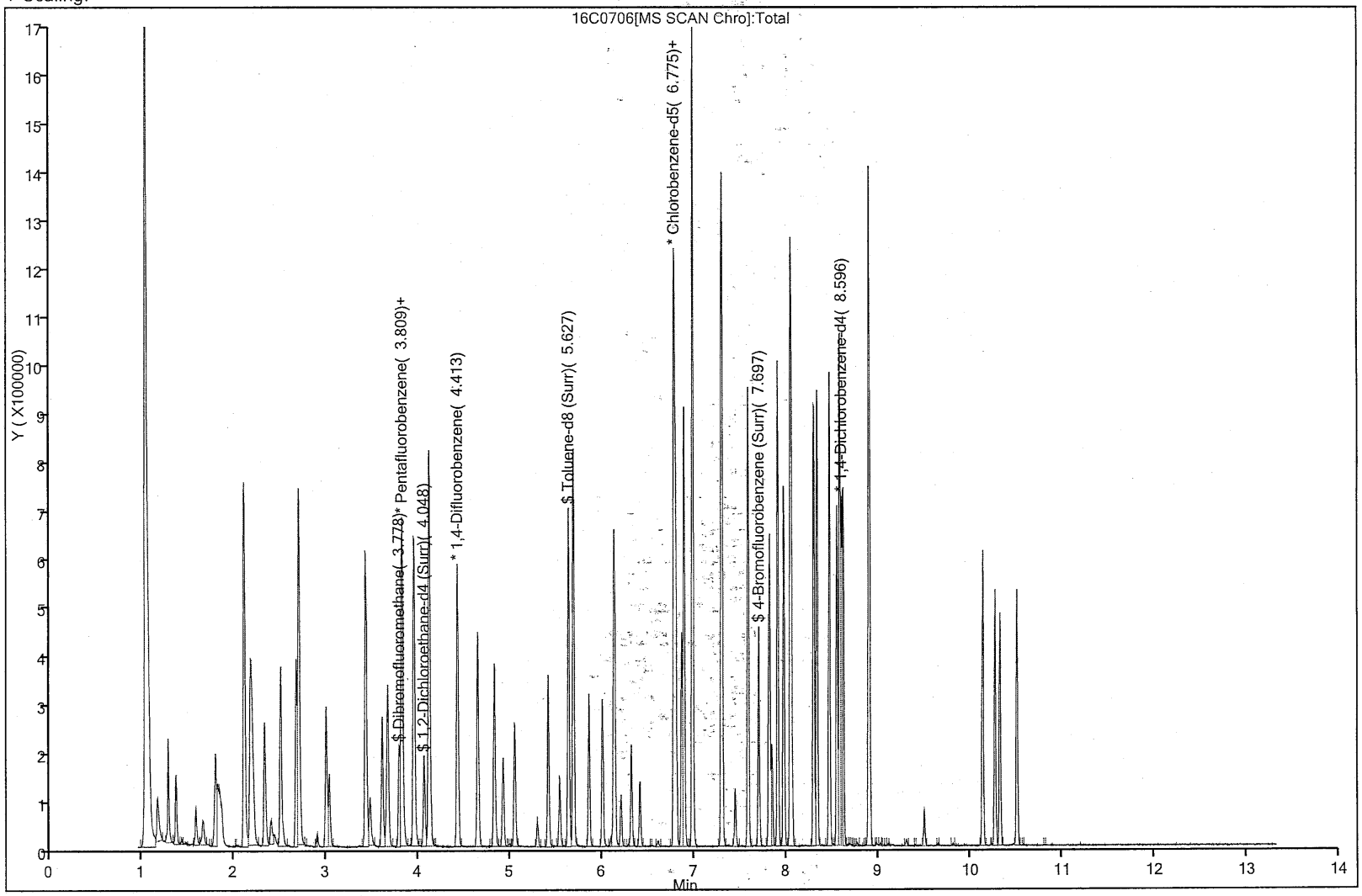
Figure 2.

Example: Total Ion Chromatogram for 5 mL Purge Soil (043-001 to 043-002)

Report Date: 02-Aug-2011 12:22:56 Chrom Revision: 1.2 13-Jul-2011 10:43:06

Preliminary Report

Data File: \\chi-svr07\chromdata\CMS16\20110706-4363.b\16C0706.D
Injection Date: 06-Jul-2011 07:42:30 Limit Group: MSVOA_8260_ICAL_SOIL_LOW
Client ID: Instrument ID: CMS16
Lims Batch ID: 4363 Lims Sample ID: 2
Operator ID: BDW
Y Scaling:



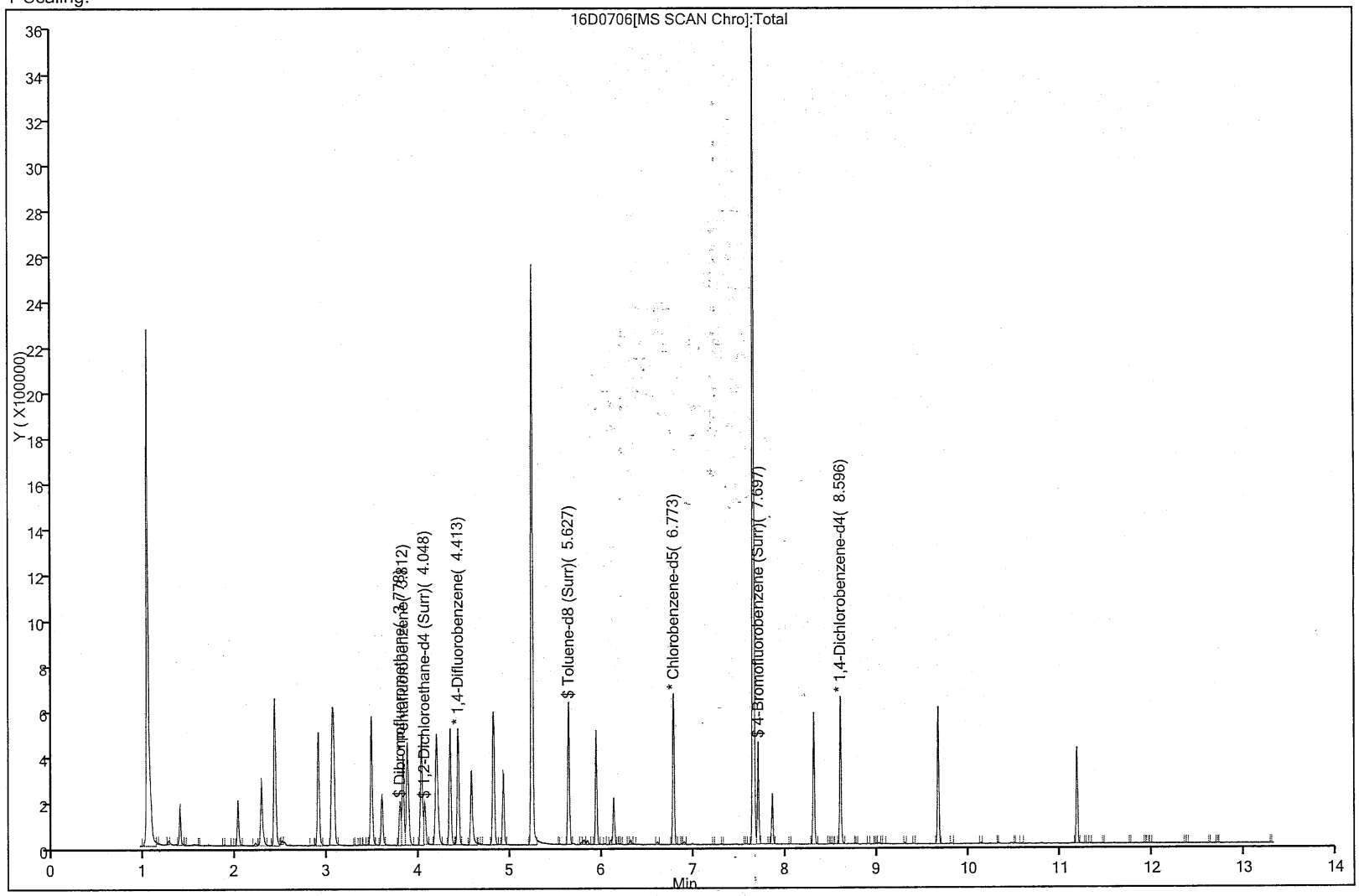
(C-2) C-386

(043-101)

Report Date: 02-Aug-2011 12:41:56 Chrom Revision: 1.2 13-Jul-2011 10:43:06

Preliminary Report

Data File: \\chi-svr07\chromdata\CMS16\20110706-4363.b\16D0706.D
Injection Date: 06-Jul-2011 08:08:30 Limit Group: MSVOA_8260_ICAL_SOIL_LOW
Client ID: Instrument ID: CMS16
Lims Batch ID: 4363 Lims Sample ID: 3
Operator ID: BDW
Y Scaling:



(C-2) C-387

(043-002)

Attachment 1.

Example: Initial Calibration Guides (044-001 to 044-004), Method Listings, Concentrator Conditions, Flow Settings

8260 5ml WATER/ HIGH LEVEL SOIL ICAL 1

TUNE FILE: _____ MATRIX: WATER AND HIGH LEVEL SOIL (NOT HEATED)

TUNE TIME: _____ INSTRUMENT NO: _____

LEVEL CHROM WATER	CONC WATER	FILE NAME	AMOUNT INJECTED	ADD MEOH
1 IC	0.25ppb	I A	5ul IS + 2.5ul LOW L	92.5ul
2 IC	1ppb	I B	5ul IS + 1 ul LOW 82	93.5ul
3 IC	2ppb	I C	5ul IS + 2 ul	92ul
4 IC	5ppb	I D	5ul IS + 5ul	87.5ul
5 IC	20ppb	I E	5ul IS + 2ul SS + 20ul	65ul
6 ICIS	50ppb	I F	GAS STD, 8260 STD, KET/CS2 STD 5ul IS + 5ul SS + 2.5ul @ VA/(	75ul
7 IC	100ppb	I G	5ul IS + 10ul SS+ 5ul	55ul
8 IC	150ppb	I H	5ul IS + 15ul SS+ 7.5ul	35ul
9 IC	200ppb	I I	5ul IS + 20ul SS+ 10ul	15ul

ICV 50ppb	S	5ul IS/SS + 5ul @GAS/DCFM SPK, VOC SPK, BCM++SPK, MEAC++SPK

STD NAME	CONC
8260 LOW IS	50ppm
8260 LOWSS	50ppm
GAS STD WK	100ppm
8260 STD WK	100ppm
KET / CS2 STDWK	100ppm
VA / 2CEVE STDWK	100ppm
ACROLEIN/NITRILES STDWK	4000/800ppm
DICHLOROFLOROMETHANE STD	100ppm
LOW 8260 STD WK	5ppm
LOW LOW 8260 STD WK	0.5ppm
LOW ACROLEIN/NITRILES STD WK	400/80ppm

CHI-22-20-059/C-10/09

(044-001)

8260 5ML WATER/ HIGH LEVEL SOIL ICAL2

TUNE FILE: _____ MATRIX: WATER AND HIGH LEVEL SOIL (NOT HEATED)

TUNE TIME: _____ INSTRUMENT NO: _____

LEVEL CHROM	CONC WATER ICAL2	CONC WATER APIX	FILE NAME	AMOUNT INJECTED	ADD MEOH
2 IC	1ppb	2.5ppb	J A	5ul IS + 1ul LOW ICAL2 + 0.5ul LOW APIX	93.5ul
3 IC	2ppb	5ppb	J B	5ul IS + 2ul LOW ICAL2 + 1ul LOW APIX	92ul
4 IC	5ppb	10ppb	J C	5ul IS + 5ul + 2ul	88ul
5 IC	20ppb	40ppb	J D	5ul IS + 20ul + 8ul	67ul
6 IC	50ppb	100ppb	J E	5ul IS + 2.5ul ICAL2 / 224TMP / CLPRENE + 2ul APIX	90.5ul
7 IC	100ppb	200ppb	J F	5ul IS + 5ul + 4ul	86ul
8 IC	150ppb	300ppb	J G	5ul IS + 7.5ul + 6ul	81.5ul
9 IC	200ppb	400ppb	J H	5ul IS + 10ul + 8ul	77ul

STD NAME	CONC
8260 LOW IS	50ppm
ICAL2 STD	100ppm
2,2,4 TRIMETHYLPENTANE	100ppm
CLHOROPRENE	100ppm
APIX STD	250/10000ppm
LOW ICAL2	5ppm
LOW APIX	25/1000ppm

CHI-22-20-060/C-10/09

(044-002)

8260 SOIL - ICAL 1

TUNE FILE: _____ MATRIX: SOIL (HEATED)

TUNE TIME: _____ INSTRUMENT NO: _____

IS ARCHON INJECTION

STD LEVELS	CONC SOIL	FILE NAME	AMOUNT INJECTED
1 IC	5ppb	I A	ARCH 1ul IS + 5ul LOW 8260 STD + 2.5ul LOW ARC/NIT STD
2 IC	20ppb	I B	+ 2ul 8260 LOWSS + 20ul + 10ul
3 ICIS	50ppb	I C	+ 5ul + 2.5ul @GAS, 8260, KET+CS2 ,VA+CEVE ,ACR/NIT STD
4 IC	100ppb	I D	+ 10ul + 5ul
5 IC	150ppb	I E	+ 15ul + 7.5ul
6 IC	200ppb	I F	+ 20ul + 10ul
ICV	50ppb	S	1ul IS/SS (ARCHON) + 5ul @ GAS SPK, VOC SPK, BDCM++SPK, 5ul MEAC++SPK + 2.5ul ACR/NIT STD

STD NAME	CONC
8260 IS	250ppm
8260 LOW SS	50ppm
GAS STD	100ppm
8260 STD	100ppm
KET + CS2	100ppm
VA + 2-CEVE	100ppm
ARC/NITS STD WK	4000ppm/ 800ppm
LOW 8260 STD	5ppm
LOW ACR/NITR	400ppm/ 80ppm

CHI-22-20-061/C-10/09

(044-003)

8260 SOIL - ICAL 2

TUNE FILE: _____ MATRIX: SOIL (HEATED)

TUNE TIME: _____ **INSTRUMENT NO:** _____

STD LEVELS	CONC SOIL		FILE NAME	AMOUNT INJECTED
	ICAL2	APIX		
1 IC	5ppb	10ppb	J___A	ARCH 1ul IS + 5ul LOW ICAL2 + 2ul LOW APIX
2 IC	20ppb	40ppb	J___B	1ul IS + 20ul + 8ul
3 IC	50ppb	100ppb	J___C	1ul IS + 2.5ul ICAL2 / 224TMP / CLPRENE + 2ul APIX
4 IC	100ppb	200ppb	J___D	1ul IS + 5ul + 4ul APIX
5 IC	150ppb	300ppb	J___E	1ul IS + 7.5ul + 6ul
6 IC	200ppb	400ppb	J___F	1ul IS + 10ul + 8ul

STD NAME	CONC
8260 IS	250ppm (ARCHON INJ)
ICAL2 STD	100ppm
2,2,4 TRIMETHYLPENTANE	100ppm
CLHOROPRENE	100ppm
APIX STD	250/10000ppm
LOW ICAL2	5ppm
LOW APIX	25/1000ppm

CHI-22-20-083/A-10/09

(044-004)

Example: Volatiles Method for 5972

GC Oven Parameters

Initial Temperature = 40 °C
Initial Time = 2.0 minutes
Detector A Temperature = 180 °C
Detector B Temperature = 250 °C
Oven Equib. Time = 0.50 min.

Ramp Rate (°C/min.)	Final Temp. (°C)	Final Time (min.)
7.0	65	0.00
12.0	165	0.00
20.0	212	5.00

Run Time = 21.25 min.

Inlet Pressure Program

Gas = Helium
Column length = 75 m
Column Diameter = 0.530 mm
Initial Pressure = 3 psi
Rate (psi/min) = 0.00
Initial Time = 7.0 min.
Oven Temp. 50 °C
Program Time = 7.0 min.

Scan Parameters

Mass Range = 35-260
Threshold = 150
Scans/sec = 1.9
EM Voltage = 1938
Solvent Delay (scan start time): before the elution of the first compound.
Run Time (scan stop time): until after the elution of last compound.

Example: Volatiles Method for 5973

GC Oven Parameters

Initial Temperature = 50 °C
Initial Time = 2.0 minutes
Aux Temperature = 250 °C
Oven Equib. Time = 0.50 min.

Ramp Rate (°C/min.)	Final Temp. (°C)	Final Time (min.)
15.0	220	0.00

Run Time = 13.33 min.

Inlet Pressure Program

Mode=split
Gas = Helium
Column length = 25 m
Column Diameter = 0.25 mm
Constant flow = 1.0 mL/min
Injection port temp. = 250 °C
Program Time = 7.0 min.
Split ratio = 80:1
Gas saver = off

Scan Parameters

Mass Range = 35-260
Threshold = 100
Scans/sec = 6
EM Voltage = 1938
Solvent Delay = 0.8 min. (scan start time): before the elution of the first compound.
Run Time (scan stop time): until after the elution of last compound.

Concentrator Conditions

Trap Temp. Prior to Purge	< 35
Desorb Preheat	250
Desorb	250
Bake	260
Purge Time	11 min
Desorb	0.5 - 2 min (inst. dependent)
Bake Time	4 min

Trap = Vocarb 3000

Flow Conditions

Purge Pressure	20 psi
Purge Flow Rate	~40 mLs/min

Flow Adjustment

Capillary Column: 5972/MSD's;

· Make-up gas off/separator pump on: flow through separator is 5-10 mLs/minutes.

· Open make-up gas: adjust until you achieve ~30 mLs/minute through the separator. (On MSD's - adjust to 0.5 torr on gauge)

(Flow into the Mass Spec is ≤ 1 mL/minute)

Approximate Vacuums

~5 x 10^{-6} torr

Example Archon Conditions

Transfer Line Temp	110 deg C
Soil Valve	95 deg C
Purge Pressure	25 psi
Purge Flow	~ 40 ml/min
Purge Time	11 min
Desorb	0.5 -2 min
SamplePre-heat(soils)	40 deg C ~ 2 min

Attachment 2.

**Example: Target and Internal Standards;
Initial Calibration (Form 6)
(050-001 to 050-006)**

Target and Internal Standards

Pentafluorobenzene
Acetone
Acrolein
Acrylonitrile
Bromochloromethane
Bromomethane
2-Butanone
Carbon disulfide
Chloroethane
Chloroform
Chloromethane
Dichlorodifluoromethane
1,1-Dichloroethane
1,1-Dichloroethene
cis-1,2-Dichloroethene
trans-1,2-Dichloroethene
2,2-Dichloropropane
Iodomethane
Methylene chloride
1,1,1-Trichloroethane
Trichlorofluoromethane
Vinyl acetate
Vinyl Chloride

Chlorobenzene-d_5
Bromoform
Bromofluorobenzene (surrogate)
Chlorodibromomethane
Chlorobenzene
1,3-Dichloropropane
Ethylbenzene
2-Hexanone
Styrene
1,1,1,2-Tetrachloroethane
Tetrachloroethene
Xylene

1,4-Difluorobenzene
Benzene
Bromodichloromethane
Carbon tetrachloride
2-Chloroethyl vinyl ether
1,2-Dibromoethane
Dibromomethane
1,2-Dichloroethane
1,2-Dichloroethane-d_4 (surrogate)
1,2-Dichloropropane
1,1-Dichloropropene
cis-1,3-Dichloropropene
trans-1,3-Dichloropropene
4-Methyl-2-pentanone
Toluene
Toluene-d_8 (surrogate)
1,1,2-Trichloroethane
Trichloroethene

1,4-Dichlorobenzene-d_4
Bromobenzene
n-Butylbenzene
sec-Butylbenzene
tert-Butylbenzene
2-Chlorotoluene
4-Chlorotoluene
1,2-Dibromo-3-chloropropane
1,2-Dichlorobenzene
1,3-Dichlorobenzene
1,4-Dichlorobenzene
Hexachlorobutadiene
Isopropyl benzene
p-Isopropyltoluene
Naphthalene
n-Propylbenzene
1,1,2,2-Tetrachloroethane
1,2,3-Trichlorobenzene
1,2,4-Trichlorobenzene
1,2,3-Trichloropropane
1,2,4-Trimethylbenzene
1,3,5-Trimethylbenzene

TestAmerica Laboratories
Initial Calibration Report

Method: \\chi-svr07\chromdata\CMS22\20110617-4051.b\8260W22.m
Instrument: CMS22 Lims Location: 500
Lock State: Initial Calib Locked Cpnd Order: Retention Time
Integrator: RTE Last Modified: 01-Aug-2011 13:00:22
No.Compounds:113

Initial Calibration Batches

Ical Batch: \\chi-svr07\chromdata\CMS22\20110616-4027.b
Inj Date : 16-Jun-2011 11:24:30, Sublist: chrom-8260W22*sub9

Limit Group: MSVOA_8260_ICAL_WATER

Detector 1: MS SCAN

Compound	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7	Level 8	Level 9	b	M1	M2	%RSD/R^2
1 Dichlorodifluoromethan		0.3448054	0.3636753	0.3267879	0.3184608	0.4474534	0.4652631	0.4241562	0.4735739	-0.2492235	0.4513377		Linr 0.994
2 Chloromethane		0.6345563	0.6592556	0.5922585	0.5973886	0.6896592	0.7107481	0.6659928	0.7392449		0.6611380		Avg 7.9
3 Vinyl chloride	0.3866341	0.5088963	0.4976517	0.4977575	0.5073222	0.5475229	0.5751149	0.5283971	0.5936724		0.5158855		Avg 12
4 Butadiene		0.3778217	0.3012005	0.2949981	0.2840799	0.3665462	0.3546022	0.3594207	0.3382813		0.3346188		Avg 11
5 Bromomethane		0.1664146	0.2160623	0.1752732	0.1855999	0.2046402	0.2206752	0.1965329	0.2277540		0.1991191		Avg 11
6 Chloroethane		0.2384502	0.2104836	0.1919896	0.1901999	0.1976438	0.2143892	0.1892001	0.2089205		0.2051596		Avg 8.1
7 Trichlorofluoromethane		0.5750332	0.6040889	0.5588969	0.5650665	0.5934872	0.6328143	0.5678700	0.6364212		0.5917098		Avg 5.1
8 Dichlorofluoromethane		0.7144568	0.7285018	0.6978920	0.7137255	0.7098388	0.7100411	0.7048012	0.7600744		0.7174165		Avg 2.7
9 Ethyl ether		0.2752457	0.2600588	0.2674498	0.2570127	0.2809414	0.2779645	0.2786019	0.2732204		0.2713119		Avg 3.3
10 1,1,2-Trichloro-1,2,2-		0.3884200	0.3831564	0.3636351	0.3858575	0.4022399	0.3796190	0.3721006	0.4008527		0.3844851		Avg 3.4
11 Acrolein		0.0284299	0.0246944	0.0231798	0.0264982	0.0271979	0.0283989	0.0287571	0.0314935		0.0273312		Avg 9.4
12 1,1-Dichloroethene		0.3524916	0.3432202	0.3262899	0.3341082	0.3365414	0.3235625	0.3127981	0.3391075		0.3335149		Avg 3.7
13 Acetone				0.0950345	0.0878418	0.0773947	0.0699036	0.0671195	0.0688032		0.0776829		Avg 15
14 Iodomethane		0.2728522	0.4980563	0.4431224	0.5185710	0.5635077	0.6212375	0.6050863	0.6139476	-0.9854506	0.6089204		Linr 0.999
15 Carbon disulfide		1.0976931	1.1625745	1.1022155	1.1207257	1.1899518	1.1294485	1.1075598	1.1834193		1.1366985		Avg 3.3
16 3-Chloro-1-propene		0.1957834	0.1990512	0.1897499	0.1852478	0.1862004	0.2014076	0.1947584	0.1982818		0.1938101		Avg 3.1
17 Methyl acetate		0.6034890	0.5609023	0.4230769	0.3822613	0.4092539	0.4244662	0.4256733	0.4362495	0.1489203	0.4248196		Linr 0.999
18 Acetonitrile		0.0595008	0.0649183	0.0389012	0.0417117	0.0364335	0.0352744	0.0352880	0.0380304	0.2639364	0.0363684		Linr 0.997
19 Methylene Chloride				0.3910215	0.3593230	0.3565553	0.3285145	0.3224283	0.3461580		0.3506668		Avg 7.0
20 Methyl tert-butyl ethe		0.6663735	0.6664282	0.6286434	0.6739851	0.6948007	0.6604216	0.6387891	0.6859405		0.6644228		Avg 3.3
21 trans-1,2-Dichloroethe		0.4381120	0.4193733	0.3744474	0.4093002	0.4055817	0.3895106	0.3766683	0.4032166		0.4020262		Avg 5.3
22 Acrylonitrile		0.0917649	0.0868013	0.0790339	0.0834269	0.0774412	0.0768360	0.0779081	0.0845106		0.0822154		Avg 6.5
23 Hexane		0.8334302	0.9486701	0.7629405	0.7330040	0.8318369	0.8124114	0.8205620	0.7855698		0.8160531		Avg 7.9
24 Isopropyl ether		1.5198348	1.4645931	1.5194392	1.4724697	1.5725229	1.5889400	1.5945324	1.5743091		1.5383301		Avg 3.4
25 1,1-Dichloroethane		0.7536027	0.7648074	0.7218284	0.7469714	0.7945722	0.7466963	0.7334080	0.7993555		0.7576552		Avg 3.6
26 Vinyl acetate			0.6136523	0.5616711	0.5996603	0.5464851	0.5566959	0.5602980	0.6005769		0.5770056		Avg 4.6
27 2-Chloro-1,3-butadiene		0.8971983	0.9090455	0.8851080	0.8671464	0.9410604	0.9373789	0.9467755	0.9067328		0.9113057		Avg 3.1
28 2,2-Dichloropropane		0.6048841	0.6138294	0.5735148	0.6115843	0.6359404	0.6070490	0.5981805	0.6423632		0.6109182		Avg 3.5

(C-2) C-399

(050-001)

Method: \\chi-svr07\chromdata\CMS22\20110617-4051.b\8260W22.m

Compound	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7	Level 8	Level 9	b	M1	M2	%RSD/R^2
29 cis-1,2-Dichloroethene		0.4052223	0.4419536	0.4126461	0.4255508	0.4472473	0.4148869	0.4094422	0.4411336		0.4247604		Avg 3.9
30 Ethyl acetate		0.2106365	0.1768328	0.1840508	0.1700333	0.1703304	0.1892346	0.1867169	0.1976439		0.1856849		Avg 7.5
31 2-Butanone (MEK)				0.1754511	0.1226901	0.1165282	0.1057701	0.1005733	0.1067446	0.3846857	0.1025929		Linr 0.999
32 Propionitrile		0.0239840	0.0237921	0.0228426	0.0231864	0.0215711	0.0212771	0.0207179	0.0220029		0.0224218		Avg 5.4
33 Chlorobromomethane		0.1692746	0.1838300	0.1759846	0.1896626	0.1969188	0.1830200	0.1797807	0.1924593		0.1838663		Avg 4.9
34 Tetrahydrofuran			0.1215793	0.0823016	0.0736273	0.0763017	0.0656532	0.0639362	0.0676105	0.1114618	0.0661099		Linr 0.997
35 Methacrylonitrile		0.2083661	0.1826301	0.1733422	0.1534376	0.1525745	0.1668448	0.1652959	0.1723737		0.1718581		Avg 10
36 Chloroform		0.6116766	0.6634175	0.6202852	0.6705540	0.7035932	0.6496401	0.6291247	0.6846907		0.6541227		Avg 4.9
37 Cyclohexane		0.8975402	0.9110981	0.8568776	0.8563231	0.9378420	0.9295460	0.9367152	0.9009189		0.9033576		Avg 3.6
38 1,1,1-Trichloroethane		0.6231164	0.6279974	0.6190759	0.6555382	0.6821586	0.6467308	0.6368358	0.6870921		0.6473181		Avg 4.0
$ 39 Dibromofluoromethane					0.4117792	0.3789504	0.3854324	0.3794664	0.3993557		0.3909968		Avg 3.6
* 40 Pentafluorobenzene	299508	279723	282326	281161	265221	271711	281502	282928	267037				
41 Carbon tetrachloride		0.5019187	0.4151984	0.4022968	0.4270041	0.4552176	0.4362897	0.4369028	0.4623853		0.4421517		Avg 7.0
42 1,1-Dichloropropene		0.5628783	0.5859361	0.5378057	0.5526429	0.5691452	0.5357298	0.5284478	0.5707627		0.5554185		Avg 3.6
43 Isooctane		1.4646623	1.5429129	1.4807595	1.4307366	1.5727975	1.5546876	1.5517458	1.4817617		1.5100080		Avg 3.4
45 Benzene	1.0821015	1.1046866	1.1194686	1.0580876	1.0979331	1.1578601	1.0886296	1.0399001	1.1231873		1.0968727		Avg 3.2
44 Isobutyl alcohol		0.0134706	0.0112310	0.0136970	0.0119461	0.0119616	0.0127978	0.0125788	0.0123855		0.0125085		Avg 6.5
$ 46 1,2-Dichloroethane-d4					0.2561730	0.2421173	0.2411008	0.2303487	0.2412454		0.2421971		Avg 3.8
47 1,2-Dichloroethane		0.2845758	0.2937857	0.2886651	0.2958857	0.3150230	0.2959763	0.2805600	0.2983656		0.2941046		Avg 3.6
48 n-Heptane		0.7585663	0.7348218	0.6837269	0.6875087	0.7433152	0.7453592	0.7442227	0.7110702		0.7260739		Avg 3.9
* 49 1,4-Difluorobenzene	411052	386716	392463	389240	373117	381534	395878	405805	383341				
50 Trichloroethene	0.3658904	0.3533601	0.3683787	0.3546912	0.3589290	0.3827313	0.3577100	0.3420165	0.3680137		0.3613023		Avg 3.2
51 n-Butanol		0.0057766	0.0043059	0.0057458	0.0050162	0.0046952	0.0052107	0.0050097	0.0048890		0.0050811		Avg 9.8
52 Methylcyclohexane		0.5766857	0.5675794	0.5282136	0.5208568	0.5716122	0.5594909	0.5529452	0.5270999		0.5505604		Avg 4.0
53 1,2-Dichloropropane		0.2845758	0.2932124	0.2834498	0.2852121	0.3098911	0.2902978	0.2786724	0.2988612		0.2905216		Avg 3.5
54 Methyl methacrylate		0.2020896	0.1793551	0.1889143	0.1644462	0.1760751	0.1916632	0.1906713	0.1971685		0.1862979		Avg 6.6
55 Dibromomethane		0.1184332	0.1206483	0.1235228	0.1218184	0.1332122	0.1237705	0.1168246	0.1241127		0.1227928		Avg 4.0
56 Dichlorobromomethane		0.2951779	0.3188199	0.3037971	0.3190286	0.3508547	0.3280101	0.3145041	0.3375480		0.3209676		Avg 5.6
57 2-Chloroethyl vinyl et			0.1133865	0.1078255	0.1113123	0.1153868	0.1111150	0.1119076	0.1190839		0.1128597		Avg 3.2
58 2-Nitropropane		0.0399555	0.0329319	0.0408363	0.0440113	0.0515814	0.0585222	0.0583168	0.0610176	-4.4237156	0.0587071		Linr 0.996
59 cis-1,3-Dichloropropen		0.3655137	0.3604161	0.3625270	0.3679275	0.4139107	0.3856529	0.3669653	0.3946128		0.3771907		Avg 5.1
60 4-Methyl-2-pentanone (			0.2028217	0.1721560	0.1690220	0.1888193	0.1697417	0.1578229	0.1698657		0.1757499		Avg 8.5
$ 61 Toluene-d8 (Surr)					1.2105720	1.1327693	1.1490118	1.1055466	1.1651421		1.1526084		Avg 3.4
62 Toluene	0.8251997	0.7799005	0.7869532	0.7409824	0.7708574	0.8259290	0.7713563	0.7357138	0.7875860		0.7804976		Avg 4.0
63 trans-1,3-Dichloroprop		0.3078487	0.2990091	0.2824222	0.3053399	0.3380878	0.3224882	0.3051728	0.3266439		0.3108766		Avg 5.6
64 Ethyl methacrylate		0.2423470	0.2194831	0.2283321	0.2087311	0.2257594	0.2449153	0.2401247	0.2490383		0.2323414		Avg 6.0
65 1,1,2-Trichloroethane		0.1737709	0.1688057	0.1692786	0.1791395	0.1926067	0.1815585	0.1706887	0.1795914		0.1769300		Avg 4.5
66 Tetrachloroethene		0.4383284	0.4609048	0.4329544	0.4322221	0.4629933	0.4392196	0.4220308	0.4431013		0.4414693		Avg 3.2
67 1,3-Dichloropropane		0.2891671	0.3079917	0.2877098	0.3003706	0.3304497	0.3083761	0.2969924	0.3100208		0.3038848		Avg 4.5
68 2-Hexanone			0.1376148	0.1206702	0.1269313	0.1368171	0.1229603	0.1184664	0.1237389		0.1267427		Avg 6.0
69 Chlorodibromomethane		0.2328267	0.2561591	0.2282140	0.2511901	0.2866819	0.2743254	0.2672862	0.2804564		0.2596425		Avg 8.3
70 Ethylene Dibromide		0.1654961	0.1882343	0.1689703	0.1776118	0.1983781	0.1840643	0.1735423	0.1852965		0.1801992		Avg 6.0
71 1-Chlorohexane		0.8077634	0.7474515	0.6443995	0.6702335	0.7180497	0.6805493	0.6618904	0.7097003		0.7050047		Avg 7.6
* 72 Chlorobenzene-d5	376594	354985	357883	363387	347038	356381	366541	371326	355935				
73 Chlorobenzene		0.9414482	1.0095478	0.9127734	0.9516033	1.0163533	0.9566461	0.9162102	0.9624440		0.9583783		Avg 4.0

(C-2) C-400

(650-002)

Method: \\chi-svr07\chromdata\CMS22\20110617-4051.b\8260W22.m

Compound	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7	Level 8	Level 9	b	M1	M2	%RSD/R^2
74 Ethylbenzene	0.5746241	0.5479105	0.5627537	0.5355998	0.5495364	0.5948353	0.5557414	0.5357161	0.5697957		0.5585015		Avg 3.4
75 1,1,1,2-Tetrachloroeth		0.3245208	0.3443164	0.3171275	0.3336378	0.3696802	0.3527573	0.3381781	0.3550388		0.3419071		Avg 5.0
76 m-Xylene & p-Xylene	1.3502605	1.2708988	1.3502667	1.2426834	1.3085455	1.4026884	1.3261340	1.2809230	1.3582195		1.3211800		Avg 3.8
77 o-Xylene	1.3606165	1.3101962	1.3753098	1.2346617	1.3329304	1.4237403	1.3521489	1.3071273	1.3820606		1.3420880		Avg 4.1
78 Styrene		1.0170852	1.0971463	0.9906794	1.0673327	1.1673462	1.1005195	1.0631763	1.1111769		1.0768078		Avg 5.2
79 Bromoform		0.1242306	0.1233643	0.1192943	0.1384503	0.1585186	0.1560521	0.1544824	0.1606192		0.1418765		Avg 12
80 Isopropylbenzene		3.0034121	3.1787679	2.9648557	3.1409295	3.2937611	3.1193751	3.0223549	3.2368773		3.1200417		Avg 3.7
81 Cyclohexanone		0.0179089	0.0199278	0.0171068	0.0138927	0.0173496	0.0165842	0.0157128	0.0173496		0.0169790		Avg 10
$ 83 4-Bromofluorobenzene (					0.5296826	0.4514045	0.4489593	0.4477521	0.4793944		0.4714386		Avg 7.4
84 Bromobenzene		0.6821867	0.7734990	0.7158998	0.7603914	0.8025629	0.7524522	0.7216337	0.7673518		0.7469972		Avg 5.1
85 1,1,2,2-Tetrachloroeth		0.3116908	0.3415773	0.3122557	0.3467619	0.3696528	0.3455249	0.3363888	0.3519781		0.3394788		Avg 5.8
86 N-Propylbenzene		3.4027055	3.6685479	3.4646555	3.6756832	3.8649327	3.6609979	3.5478009	3.7955433		3.6351084		Avg 4.3
87 1,2,3-Trichloropropane		0.0832466	0.1161908	0.1057855	0.1050009	0.1137189	0.1082425	0.1033429	0.1069325		0.1053076		Avg 9.4
88 trans-1,4-Dichloro-2-b		0.0745779	0.0526246	0.0622580	0.0516661	0.0593201	0.0661633	0.0662145	0.0674431		0.0625334		Avg 12
89 2-Chlorotoluene		1.9647654	2.0341696	1.9239240	2.0009424	2.1297373	2.0026388	1.9577367	2.0808988		2.0118516		Avg 3.4
90 1,3,5-Trimethylbenzene		2.4519033	2.6178507	2.4443184	2.6481587	2.7664112	2.6022715	2.5590352	2.7292623		2.6024014		Avg 4.5
91 4-Chlorotoluene		1.9233841	2.1092194	1.9174406	2.0996065	2.2171955	2.0698823	2.0489015	2.1976403		2.0729088		Avg 5.3
92 tert-Butylbenzene		2.3483290	2.5358057	2.3499743	2.4835268	2.5824438	2.4327751	2.3788052	2.5407814		2.4565552		Avg 3.8
93 Pentachloroethane		0.2781949	0.0303268	0.2577667	0.2375503	0.2639007	0.2833950	0.2830503	0.2639937	-0.3929025	0.2739023		Linr 0.996
94 1,2,4-Trimethylbenzene		2.4637611	2.6329081	2.4814077	2.6560578	2.7924251	2.6026472	2.5467299	2.7247553		2.6125865		Avg 4.4
95 sec-Butylbenzene		3.3814099	3.7169212	3.4342881	3.6374499	3.7616365	3.5438396	3.5045090	3.7037326		3.5854733		Avg 3.9
96 1,3-Dichlorobenzene		1.4873073	1.6022479	1.4897695	1.5783014	1.6729253	1.5461723	1.4995402	1.5870488		1.5579141		Avg 4.2
97 4-Isopropyltoluene		3.0614912	3.2810870	3.0827025	3.2427433	3.3931275	3.2030891	3.1609140	3.3608705		3.2232531		Avg 3.7
* 98 1,4-Dichlorobenzene-d4	216576	206615	210860	209764	200022	210695	215627	220041	207846				
99 1,4-Dichlorobenzene		1.6213731	1.6234705	1.4895788	1.5361685	1.6150027	1.5038237	1.4764506	1.5506084		1.5520595		Avg 3.9
100 n-Butylbenzene		2.4081020	2.6984729	2.4910375	2.7476978	2.8972591	2.7381195	2.7406529	2.8995350		2.7026096		Avg 6.5
101 1,2-Dichlorobenzene		1.3058103	1.3806554	1.2992220	1.3523762	1.4433897	1.3196585	1.2976112	1.3611328		1.3449820		Avg 3.7
102 1,2-Dibromo-3-Chloropr			0.0526416	0.0472436	0.0514318	0.0546097	0.0531775	0.0534779	0.0551646		0.0525352		Avg 5.0
103 1,3,5-Trichlorobenzene		1.2159639	1.5094551	1.0781092	1.1234704	1.3057944	1.2739525	1.2941542	1.2232315		1.2530164		Avg 10
104 1,2,4-Trichlorobenzene		0.6652470	0.7960258	0.7505101	0.8878398	0.9976981	0.9344910	0.9523604	1.0048137		0.8736232		Avg 14
105 Hexachlorobutadiene		0.6787987	0.7685194	0.7117046	0.7742398	0.7655189	0.7135748	0.7347767	0.7519293		0.7373828		Avg 4.6
106 Naphthalene		0.6722648	0.8229394	0.8480483	1.0208377	1.2104796	1.1617910	1.1894435	1.2507157	-0.8397964	1.2101444		Linr 0.998
107 1,2,3-Trichlorobenzene		0.4980277	0.5985014	0.5877081	0.6902491	0.7747597	0.7358285	0.7357932	0.7764463		0.6746642		Avg 15
108 2-Methylnaphthalene		0.0237420	0.9884307	0.0895424	0.3182819	0.5419540	0.5684555	0.6101933	0.6085117	-1.5761079	0.5572036	0.0003234	Quad 0.998

(C-2) C-401

050-003

TestAmerica Laboratories
Initial Calibration Report

Method: \\chi-svr07\chromdata\CMS16\20110706-4363.b\8260S16.m
Instrument: CMS16 Lims Location: 500
Lock State: Initial Calib Locked Cpnd Order: Retention Time
Integrator: RTE Last Modified: 07-Jul-2011 07:57:53
No.Compounds:112

Initial Calibration Batches

Ical Batch: \\chi-svr07\chromdata\CMS16\20110621-4088.b
Inj Date : 21-Jun-2011 08:43:30, Sublist: chrom-8260S16*sub2

Limit Group: MSVOA_8260_ICAL_SOIL_LOW

Detector 1: MS SCAN

Compound	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	b	M1	M2	%RSD/R^2
4 Dichlorodifluoromethan	0.3229729	0.3288985	0.4027211	0.3852392	0.3681478	0.3645036		0.3620805		Avg 8.6
5 Chloromethane	0.4968122	0.5126591	0.5976566	0.5907727	0.5188563	0.5181331		0.5391484		Avg 8.1
6 Vinyl chloride	0.4291714	0.4150502	0.4613975	0.4505522	0.4258974	0.4158015		0.4329784		Avg 4.4
7 Butadiene	0.2652957	0.2540822	0.2437645	0.2565636	0.2328427	0.2233131		0.2459770		Avg 6.4
8 Bromomethane	0.1474546	0.1312416	0.1578128	0.1546536	0.1431501	0.1362935		0.1451010		Avg 7.1
9 Chloroethane	0.1440366	0.1594246	0.1840069	0.1854268	0.1760882	0.1688215		0.1696341		Avg 9.3
10 Trichlorofluoromethane	0.6374308	0.5808306	0.6612707	0.6647948	0.6370990	0.5963994		0.6296376		Avg 5.4
11 Ethyl ether	0.5357019	0.5264639	0.4573148	0.5145459	0.4870350	0.4484513		0.4949188		Avg 7.4
12 Acrolein	0.0739327	0.0753225	0.0776729	0.0836552	0.0803243	0.0795812		0.0784148		Avg 4.5
13 1,1-Dichloroethene	0.5067065	0.4329607	0.4470838	0.4762735	0.4439303	0.4330446		0.4566666		Avg 6.4
14 1,1,2-Trichloro-1,2,2-	0.4655703	0.4115936	0.4260953	0.4451628	0.4199360	0.4143086		0.4304444		Avg 4.9
15 Acetone	0.2207921	0.1423921	0.1320174	0.1434690	0.1351860	0.1287388	0.4124755	0.1303817		Linr 0.998
16 Iodomethane	0.7015117	0.7310731	0.7089625	0.7799701	0.7150667	0.7180778		0.7257770		Avg 3.9
17 Carbon disulfide	1.4225560	1.3222844	1.4232783	1.5210276	1.4270896	1.3632196		1.4132426		Avg 4.8
18 Acetonitrile	0.0521173	0.0373107	0.0430353	0.0454032	0.0445769	0.0443358		0.0444632		Avg 11
19 3-Chloro-1-propene	0.2509190	0.2629018	0.2571425	0.2873535	0.2627813	0.2659134		0.2645019		Avg 4.7
20 Methyl acetate	0.7323150	0.7221745	0.6963136	0.7705775	0.7261585	0.7051149		0.7254423		Avg 3.6
21 Methylene Chloride	0.7126272	0.5601882	0.5124827	0.5330997	0.4949856	0.4777108	1.2540315	0.4875558		Linr 0.998
22 Acrylonitrile	0.1479193	0.1572694	0.1608097	0.1770805	0.1687439	0.1725213		0.1640573		Avg 6.6
23 trans-1,2-Dichloroethe	0.5152815	0.5080874	0.5186639	0.5468783	0.5196775	0.4918322		0.5167368		Avg 3.5
24 Methyl tert-butyl ethe	1.2186142	1.1457856	1.2020654	1.2813392	1.2252768	1.2011081		1.2123649		Avg 3.6
25 Hexane	1.1653580	1.1613035	0.9838072	1.0918489	1.0123892	0.9408190		1.0592543		Avg 8.9
26 1,1-Dichloroethane	1.0511911	0.9622759	1.0151078	1.0852697	1.0231896	0.9956446		1.0221131		Avg 4.2
27 Vinyl acetate	0.7292376	0.7831010	0.8039073	0.9587746	0.9256979	0.9092669		0.8516642		Avg 11
28 Isopropyl ether	1.9410279	2.0490771	1.9680167	2.2084163	2.0365170	1.9079558		2.0185018		Avg 5.3
29 2-Chloro-1,3-butadiene	1.0299022	1.0938611	1.0657790	1.2164591	1.1156450	1.0404354		1.0936803		Avg 6.2
31 cis-1,2-Dichloroethene	0.6046898	0.5319634	0.5551381	0.6084898	0.5622650	0.5546836		0.5695383		Avg 5.4
32 2,2-Dichloropropane	0.4583745	0.4058510	0.4341385	0.4808605	0.4663665	0.4446453		0.4483727		Avg 5.9
33 2-Butanone (MEK)	0.2294871	0.2313036	0.2172745	0.2262296	0.2099279	0.2037535		0.2196627		Avg 5.1
34 Propionitrile	0.0332027	0.0347446	0.0330137	0.0367824	0.0349489	0.0357073		0.0347333		Avg 4.2
35 Ethyl acetate	0.3142159	0.3326875	0.3072491	0.3407564	0.3325651	0.3339848		0.3269431		Avg 4.0
36 Methacrylonitrile	0.2699850	0.2726185	0.2594667	0.2805510	0.2669930	0.2675743		0.2695314		Avg 2.6
37 Chlorobromomethane	0.2457377	0.2343560	0.2351457	0.2627239	0.2502657	0.2419978		0.2450378		Avg 4.3
38 Tetrahydrofuran	0.1385198	0.1248719	0.1301731	0.1414228	0.1350312	0.1347031		0.1341203		Avg 4.4
39 Chloroform	0.8580430	0.7698598	0.8186374	0.8858097	0.8371090	0.8063535		0.8293021		Avg 4.9
$ 40 Dibromofluoromethane		0.4888388	0.5187293	0.5240857	0.4859927	0.5330135		0.5101320		Avg 4.2
41 1,1,1-Trichloroethane	0.6290956	0.5989780	0.6416624	0.7087247	0.6889794	0.6634401		0.6551467		Avg 6.1
* 42 Pentafluorobenzene	172750	185803	174126	174910	176203	175378				
43 Cyclohexane	1.0679781	1.1071515	1.0687122	1.2020460	1.0718364	1.0025536		1.0867130		Avg 6.1
44 1,1-Dichloropropene	0.6517625	0.6324157	0.6640311	0.7170517	0.6769744	0.6547308		0.6661610		Avg 4.3
45 Carbon tetrachloride	0.3350484	0.3030940	0.3261012	0.3624026	0.3615406	0.3495620		0.3396248		Avg 6.8
46 Isobutyl alcohol	0.0090251	0.0111358	0.0113173	0.0135638	0.0119929	0.0123827		0.0115696		Avg 13
$ 47 1,2-Dichloroethane-d4		0.2850331	0.3021348	0.3096504	0.2924809	0.3050994		0.2988797		Avg 3.3

(C-2) C-402

(4-06-05)

Compound	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	b	M1	M2	%RSD/R^2
48 Benzene	1.2352849	1.0980973	1.1615157	1.2566391	1.1698816	1.1216875		1.1738510		Avg 5.3
49 1,2-Dichloroethane	0.3741557	0.3490793	0.3537040	0.3949624	0.3742769	0.3563742		0.3670921		Avg 4.7
50 Isooctane	1.4466599	1.5117198	1.3885765	1.5920231	1.4258181	1.3017946		1.4444320		Avg 6.9
51 n-Heptane	0.5850791	0.6185899	0.5644893	0.6718325	0.6007726	0.5571843		0.5996580		Avg 7.0
* 52 1,4-Difluorobenzene	282899	314023	289422	294419	297576	299528				
53 n-Butanol	0.0049539	0.0058557	0.0067928	0.0083168	0.0072876	0.0075492	-1.4344918	0.0076643		Linr 0.995
54 Trichloroethene	0.3320824	0.2994983	0.3126505	0.3387410	0.3168917	0.3031047		0.3171614		Avg 4.9
55 Methylcyclohexane	0.5338526	0.5695717	0.5217969	0.6058659	0.5422037	0.5001368		0.5455713		Avg 6.9
56 1,2-Dichloropropane	0.3148356	0.2973788	0.3165948	0.3423650	0.3221018	0.3131437		0.3177366		Avg 4.6
58 Methyl methacrylate	0.2054307	0.2279140	0.2094597	0.2410001	0.2231580	0.2245527		0.2219192		Avg 5.8
57 Dibromomethane	0.1658398	0.1429949	0.1512857	0.1670299	0.1547199	0.1508598		0.1554550		Avg 6.0
59 Dichlorobromomethane	0.3097092	0.3039929	0.3189356	0.3672018	0.3477472	0.3384629		0.3310083		Avg 7.4
60 2-Nitropropane	0.0573789	0.0754235	0.0817410	0.0930729	0.0940403	0.0928538	-18.83876	0.0938656		Linr 0.999
61 2-Chloroethyl vinyl et	0.0387777	0.0368146	0.0387842	0.0409337	0.0386196	0.0388902		0.0388034		Avg 3.4
62 cis-1,3-Dichloropropen	0.3676378	0.3591242	0.3857951	0.4321155	0.4161444	0.4031557		0.3939955		Avg 7.2
63 4-Methyl-2-pentanone (	0.2426263	0.2350232	0.2338973	0.2489307	0.2431510	0.2301800		0.2389681		Avg 3.0
$ 64 Toluene-d8 (Surr)		0.9506160	1.0560652	1.0712781	0.9845835	1.0501151		1.0225316		Avg 5.1
65 Toluene	0.7887734	0.6999895	0.7264350	0.7617033	0.7163139	0.6889423		0.7303596		Avg 5.2
66 trans-1,3-Dichloroprop	0.3172157	0.2788561	0.3148338	0.3470833	0.3364556	0.3299976		0.3207403		Avg 7.4
67 Ethyl methacrylate	0.3478624	0.3802500	0.3573669	0.4209663	0.3930518	0.3921032		0.3819334		Avg 6.9
68 1,1,2-Trichloroethane	0.3425816	0.3189049	0.3343536	0.3490588	0.3333244	0.3219829		0.3333677		Avg 3.5
69 Tetrachloroethene	0.3942070	0.3544296	0.3539221	0.4003961	0.3811273	0.3739968		0.3763465		Avg 5.2
70 1,3-Dichloropropane	0.4567984	0.4523465	0.4468174	0.5170766	0.4800803	0.4754654		0.4714308		Avg 5.5
71 2-Hexanone	0.2159047	0.2190273	0.2113345	0.2305688	0.2194500	0.2183553		0.2191067		Avg 2.9
72 Chlorodibromomethane	0.2608373	0.2651217	0.2617316	0.3247142	0.3192239	0.3212945		0.2921539		Avg 11
73 Ethylene Dibromide	0.1845146	0.1780117	0.1927079	0.2003021	0.1898514	0.1856604		0.1885080		Avg 4.1
* 74 Chlorobenzene-d5	217635	230128	224910	210535	211581	206920				
75 1-Chlorohexane	0.6556003	0.6217948	0.6584152	0.6474005	0.6112196	0.5936868		0.6313528		Avg 4.2
76 Chlorobenzene	1.0934222	0.9967355	1.0304559	1.1336914	1.0642739	1.0438800		1.0604098		Avg 4.6
77 1,1,1,2-Tetrachloroeth	0.3010100	0.3103721	0.3229110	0.3860534	0.3714512	0.3734887		0.3442144		Avg 11
78 Ethylbenzene	0.5875026	0.5279814	0.5430518	0.5878014	0.5476026	0.5357085		0.5549414		Avg 4.7
79 m-Xylene & p-Xylene	1.3957412	1.3145011	1.3321823	1.4489118	1.3457130	1.3242600		1.3602182		Avg 3.8
80 o-Xylene	1.3982401	1.3784436	1.3658487	1.5154247	1.3921572	1.3830331		1.4055246		Avg 3.9
81 Styrene	1.0793332	1.0730119	1.0697806	1.1609032	1.0734947	1.0646573		1.0868635		Avg 3.4
82 Bromoform	0.1343217	0.1307997	0.1348545	0.1641961	0.1647111	0.1672186		0.1493503		Avg 12
84 Isopropylbenzene	3.8151127	3.3931614	3.5263984	3.8323331	3.6994578	3.5307562		3.6328699		Avg 4.9
85 Cyclohexanone	0.1305161	0.1470714	0.1257227	0.1347247	0.1155142	0.1152018		0.1281251		Avg 9.5
$ 87 4-Bromofluorobenzene (		0.4493981	0.4806749	0.4897861	0.4461314	0.4933692		0.4718719		Avg 4.8
88 1,1,2,2-Tetrachloroeth	0.6329563	0.5953526	0.6042302	0.6530069	0.6470052	0.6150967		0.6246080		Avg 3.7
89 Bromobenzene	0.8690501	0.8069909	0.8185718	0.8641847	0.8481408	0.8100397		0.8361630		Avg 3.3
90 1,2,3-Trichloropropane	0.2249535	0.2003849	0.1885681	0.2038842	0.2067568	0.1924894		0.2028395		Avg 6.3
91 trans-1,4-Dichloro-2-b	0.0829571	0.0771183	0.0797527	0.0990125	0.0998753	0.1025801		0.0902160		Avg 13
92 N-Propylbenzene	4.3124429	4.1757505	4.2625825	4.5411627	4.4513978	4.2238777		4.3278690		Avg 3.3
93 2-Chlorotoluene	2.5957034	2.3669753	2.4376969	2.6153058	2.5738527	2.4422869		2.5053035		Avg 4.1
94 1,3,5-Trimethylbenzene	3.0243517	2.8050848	2.9393804	3.1355083	3.0586640	2.8994218		2.9770685		Avg 4.0
95 4-Chlorotoluene	3.0089188	2.8037747	2.8265701	3.0258731	2.9311472	2.7897640		2.8976746		Avg 3.6
96 tert-Butylbenzene	2.7238687	2.5946865	2.5851508	2.8795589	2.7861587	2.6438275		2.7022085		Avg 4.3
97 Pentachloroethane	0.1664754	0.1861983	0.1899003	0.2112976	0.1789066	0.1956912		0.1880782		Avg 8.1
98 1,2,4-Trimethylbenzene	3.1953409	2.8563918	2.9589022	3.2246338	3.1187193	2.9616182		3.0526010		Avg 4.9
99 sec-Butylbenzene	4.0652087	3.7220828	3.7758116	4.1231317	3.9697138	3.8293616		3.9142184		Avg 4.2
100 1,3-Dichlorobenzene	1.8497083	1.5900948	1.6045410	1.7343897	1.7085712	1.6099268		1.6828720		Avg 6.0
101 4-Isopropyltoluene	3.4578036	3.2425159	3.2963776	3.5597916	3.5121268	3.3438555		3.4020785		Avg 3.7
* 102 1,4-Dichlorobenzene-d4	101355	110693	104058	100486	95951	98054				
103 1,4-Dichlorobenzene	1.8238504	1.5929256	1.6249289	1.7293693	1.6763232	1.6110335		1.6764051		Avg 5.2
104 n-Butylbenzene	3.2346899	3.0264090	3.0580742	3.3443975	3.2294732	3.0609174		3.1589935		Avg 4.1
105 1,2-Dichlorobenzene	1.6477510	1.4928151	1.4558527	1.6206929	1.5764553	1.4980162		1.5485972		Avg 5.0
106 1,2-Dibromo-3-Chloropr	0.0796178	0.0757790	0.0862503	0.1075373	0.1167782	0.1086726	-0.3048327	0.1105908		Linr 0.992
107 1,3,5-Trichlorobenzene	1.1450556	1.1234796	1.1398383	1.1351917	1.2024551	1.1625427		1.1814272		Avg 6.0
108 1,2,4-Trichlorobenzene	1.1532826	1.0671287	1.1041716	1.2177284	1.1838358	1.1169938		1.1405235		Avg 4.8
109 Hexachlorobutadiene	0.5300358	0.5022608	0.5405280	0.5881742	0.5600963	0.5495909		0.5451143		Avg 5.3
110 Naphthalene	2.2378833	2.1133182	2.1657854	2.5147064	2.4905173	2.3789654		2.3168627		Avg 7.3

(C-2) C-403

(050-005)

Compound	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	b	M1	M2	%RSD/R^2
111 1,2,3-Trichlorobenzene	0.9894478	0.8997786	0.9504262	1.0669349	1.0574455	0.9937420		0.9929625		Avg 6.4
112 2-Methylnaphthalene	0.8793647	1.0807246	1.3194568	1.6404114	1.3987064	1.3672873	-3.4203451	1.4472586		Linr 0.992

(C-2) C-404

(050-004)

Attachment 3.

Example: Sample Run Log; Corrective Action/Qualification Report; GC/MS VOA Maintenance Logbook; Sample Tracking Sheet; GC/MS VOA- ICOC Form (051-001 to 051-005)

TestAmerica Laboratories
Worklist Run Log Report

Worklist Name: 070611SOIL16 Worklist Num: 4363
Instrument: CMS16 Method: 8260S16
Batch Directory: \\chi-svr07\chromdata\CMS16\20110706-4363.b
Anaylysis Type: VOA Creator: Werner, Brian D
Inj Volume: 5.00 Inj Vol Units: mL
Run Reagents:
INST16 IS/SS_00119, Amount Added: 5.00 , Units: mL

Lab ID	Worklist ID	Sample Type	Cal Lvl	Inj Date/Time	File Name	Vial	Dil Fact	Client ID	Fraction
# 1 BFB	500-0004363-001	BFB		06-Jul-2011 07:08:30	16B0706.D	1	1.0		voaSoilLL
# 2 CCV	500-0004363-002	CCV		06-Jul-2011 07:42:30	16C0706.D	2	1.0		voaSoilLL
# 3 CCV	500-0004363-003	CCV		06-Jul-2011 08:08:30	16D0706.D	3	1.0		voaSoilLL
# 4 MB	500-0004363-004	MB		06-Jul-2011 08:33:30	16M0706.D	4	1.0		voaSoilLL
# 5 LCS	500-0004363-005	LCS		06-Jul-2011 08:59:30	16S0706.D	5	1.0		voaSoilLL
# 8 LCS	500-0004363-008	LCS		06-Jul-2011 09:37:30	16S0706a.D	6	1.0		voaSoilLL
# 6 500-36082-B-6-A	500-0004363-006	Client		06-Jul-2011 10:03:30	36082-06a.D	7	1.0	SS-18	voaSoilLL
# 7 500-35987-A-19-A	500-0004363-007	Client		06-Jul-2011 10:29:30	35987-19.D	8	1.0	E1519B03 (0-2)	voaSoilLL
# 9 500-35987-A-20-A	500-0004363-009	Client		06-Jul-2011 10:54:30	35987-20.D	9	1.0	E1519B03 (8-10)	voaSoilLL
#10 500-35987-A-21-A	500-0004363-010	Client		06-Jul-2011 11:20:30	35987-21.D	10	1.0	E1519B04 (4-6)	voaSoilLL
#11 500-35987-A-22-A	500-0004363-011	Client		06-Jul-2011 11:46:30	35987-22.D	11	1.0	E1519B04 (8-10)	voaSoilLL
#12 500-35987-A-24-A	500-0004363-012	Client		06-Jul-2011 12:12:30	35987-24.D	12	1.0	E1519B05 (4-6)	voaSoilLL
#13 500-36004-A-1-A	500-0004363-013	Client		06-Jul-2011 12:38:30	36004-01.D	13	1.0	SB-06-0-2	voaSoilLL
#14 500-36004-A-2-A	500-0004363-014	Client		06-Jul-2011 13:04:30	36004-02.D	14	1.0	SB-05-4-6	voaSoilLL
#15 500-36004-A-3-A	500-0004363-015	Client		06-Jul-2011 13:30:30	36004-03.D	15	1.0	SB-02-4-6	voaSoilLL
#16 500-36004-A-4-A	500-0004363-016	Client		06-Jul-2011 13:56:30	36004-04.D	16	1.0	SB-01-4-6	voaSoilLL
#17 500-36004-A-5-A	500-0004363-017	Client		06-Jul-2011 14:22:30	36004-05.D	17	1.0	SB-03-4-6	voaSoilLL
#18 500-36004-A-6-A	500-0004363-018	Client		06-Jul-2011 14:48:30	36004-06.D	18	1.0	SB-04-0-2	voaSoilLL
#19 500-36168-A-1-A	500-0004363-019	Client		06-Jul-2011 15:13:30	36168-01.D	19	1.0	0735-11 / RUD-1	voaSoilLL
#20 500-36168-A-2-A	500-0004363-020	Client		06-Jul-2011 15:40:30	36168-02.D	20	1.0	0736-11 / RUD-2	voaSoilLL
#21 500-36168-A-3-A	500-0004363-021	Client		06-Jul-2011 16:06:30	36168-03.D	21	1.0	0737-11 / RUD-3	voaSoilLL
#22 500-36168-A-4-A	500-0004363-022	Client		06-Jul-2011 16:31:30	36168-04.D	22	1.0	0738-11 / RUD-4	voaSoilLL
#23 500-36168-A-5-A	500-0004363-023	Client		06-Jul-2011 16:58:30	36168-05.D	23	1.0	0739-11 / RUD-5	voaSoilLL
#24 500-36168-A-6-A	500-0004363-024	Client		06-Jul-2011 17:24:30	36168-06.D	24	1.0	0740-11 / RUD-6	voaSoilLL
#25 500-35981-A-1-A	500-0004363-025	Client		06-Jul-2011 17:50:30	35981-01a.D	25	1.0	TNH-DRUMS	voaSoilLL
#26 500-36163-A-1-A	500-0004363-026	Client		06-Jul-2011 18:15:30	36163-01.D	26	1.0	SP-1/SP-2 1-3'	voaSoilLL
#27 500-36163-A-2-A	500-0004363-027	Client		06-Jul-2011 18:41:30	36163-02.D	27	1.0	SP-3/SP-4 1-3'	voaSoilLL
#28 LCSD	500-0004363-028	LCSD		06-Jul-2011 19:07:30	16T0706.D	28	1.0		voaSoilLL

(051-001)

Instrument ID# _____

TestAmerica Chicago
Corrective Action/Qualification Report GC/MS VOA and GC VOA

CHI-22-20-082/B-08/10

Analytical Methods
___SW846 8260
___40CFR 624
___8015B
___8015C

Tune Name:_____
__Other_____
__WI_GRO
__OA1_GRO

Instrument Preventative Maintenance was performed per SOP_____

Tune Criteria
Description of Situation:_____
_____
Action Taken:_____
_____
Demonstration of Control:_____
_____
_____

Initial Calibration Criteria
Description of Situation:_____
_____
Action Taken:_____
_____
Demonstration of Control_____
_____

Continuing Calibration Criteria
Description of Situation:_____
_____
Action Taken:_____
_____
Demonstration of Control:_____
_____

Internal Standards (CCV)
Description of situation:_____
_____
Action Taken:_____
_____
Demonstration of Control:_____
_____

Method Blank
Description of situation:_____
_____
Action Taken:_____
_____
Demonstration of Control:_____
_____

LCS
Description of Situation:_____
_____
_____
Action Taken:_____
_____
_____
Demonstration of Control:_____
_____

Qualification of Data
Data Affected (Client/Sample #)_____
_____
_____
_____
Qualification:_____
_____
_____
Associated samples reanalyzed: Yes No (see below)
Explanation for no reanalysis/data MUST be qualified and narrated:_____
_____
_____
_____
_____
_____

Client contacted/NCM # _____
_____

Analyst Signature/date_____/_____
Reviewer Signature/date_____/_____

(C-2) C-407

(051-002)

TestAmerica Chicago
GC/MS VOA Maintenance Logbook
Instrument No. 16

Page No._____

Date of Maintenance:_____ Entry No.:_____

Analyst:_____

Description:_____

Follow-Up:_____

Demonstration of Return to Control: Original Entry No: _____

Analyst:_____ Date:_____

Date of Maintenance:_____ Entry No.:_____

Analyst:_____

Description:_____

Follow-Up:_____

Demonstration of Return to Control: Original Entry No: _____

Analyst:_____ Date:_____

Reviewer Signature or Initials / Date:_____ CHI-22-20-051/B-02/08

(051-003)

TestAmerica Chicago

GC/MS Volatiles: Sample Tracking Sheet Screener: _____ Job Number:

Sample File Name	Dilution	pH	Tune	Action	Prep Batch	MeOH Lot #	Misc. Info

(C-2) C-409

Reviewed by: _____ Date: _____

CHI-22-20-075/C-07/10

(015-040)

TestAmerica Chicago

GC/MS Volatiles: Internal Chain of Custody Form

ICOC Job Number:

Date	Time Out	Time In	ICOC Initials	Misc. Info

CHI-22-20-074/A-01/08

(C-2) C-410

(051-005)

Attachment 4.

Example: Continuing Calibration Evaluation and Acceptance Criteria (Form 7) (052-001 to 052-010)

Report Date: 02-Aug-2011 12:34:06 Chrom Revision: 1.2 13-Jul-2011 10:43:06

Preliminary Report

TestAmerica Laboratories
CCV, Cal Verification Report

Data File: \\chi-svr07\chromdata\CMS22\20110617-4051.b\22C0617.D
Lims ID: CCV Client ID:
Inject. Date: 17-Jun-2011 12:12:30 Dil. Factor: 1.0000
Sample Type: CCV
Sample ID: CCV
Misc. Info.: 500-0004051-002 =500-0004051-002
Operator: EA Instrument ID: CMS22
Vol. Injected: 1.0000 ALS Bottle#: 2
Lims Batch ID: 4051 Lims Sample ID: 2
Sublist: chrom-8260W22*sub9
Detector: MS SCAN

Method: \\chi-svr07\chromdata\CMS22\20110617-4051.b\8260W22.m
Method Label: TAL CHICAGO VOA REPORT SW846 8260B
Last Update: 01-Aug-2011 13:00:22 Calib Date: 16-Jun-2011 18:31:30
Quant Method: Internal Standard Quant By: Initial Calibration
Last ICal File: \\chi-svr07\chromdata\CMS22\20110616-4027.b\22J0616H.D
Limit Group: MSVOA_8260_ICAL_WATER
Integrator: RTE ID Type: RT Order ID
Process Host: CHI-MS-OFF9A

First Level Reviewer: alikpalae Date: 17-Jun-2011 12:40:39

Start Cal Date: 16-Jun-2011 11:24:30
End Cal Date: 16-Jun-2011 18:31:30

Compound	Standard RRF/Amount	DLT RT	Ccal Amount	Ccal RF	Min. RRF	%D	Max. %D
1 Dichlorodifluorometha	50.0	0.000	50.3	0.449045	0.010	0.6	50.0
2 Chloromethane	0.661138	-0.003		0.721403	0.100	9.1	50.0
3 Vinyl chloride	0.515885	-0.003		0.571985	0.010	10.9	20.0
5 Bromomethane	0.199119	-0.003		0.216772	0.010	8.9	50.0
6 Chloroethane	0.205160	-0.003		0.217856	0.010	6.2	50.0
7 Trichlorofluoromethan	0.591710	-0.003		0.611231	0.010	3.3	50.0
8 Dichlorofluoromethane	0.717416	-0.003		0.677314	0.010	-5.6	50.0
10 1,1,2-Trichloro-1,2,2	0.384485	0.000		0.361287	0.010	-6.0	50.0
11 Acrolein	0.027331	-0.002		0.027311	0.001	-0.1	50.0
12 1,1-Dichloroethene	0.333515	-0.003		0.312768	0.010	-6.2	20.0
13 Acetone	0.077683	0.002		0.076611	0.010	-1.4	50.0
15 Carbon disulfide	1.136699	0.000		1.133315	0.010	-0.3	50.0
18 Acetonitrile	400.0	-0.001	384.9	0.035656	0.001	-3.8	50.0
19 Methylene Chloride	0.350667	-0.003		0.307037	0.010	-12.4	50.0
20 Methyl tert-butyl eth	0.664423	-0.003		0.629349	0.010	-5.3	50.0
21 trans-1,2-Dichloroeth	0.402026	-0.003		0.376478	0.010	-6.4	50.0
22 Acrylonitrile	0.082215	-0.003		0.075836	0.001	-7.8	50.0
25 1,1-Dichloroethane	0.757655	0.000		0.716351	0.100	-5.5	50.0

(052-001)

Report Date: 02-Aug-2011 12:34:06 Chrom Revision: 1.2 13-Jul-2011 10:43:06

Preliminary Report

Data File: \\chi-svr07\chromdata\CMS22\20110617-4051.b\22C0617.D

Compound	Standard RRF/Amount	DLT RT	Ccal Amount	Ccal RF	Min. RRF	%D	Max. %D
26 Vinyl acetate	0.577006	-0.003		0.512152	0.010	-11.2	50.0
28 2,2-Dichloropropane	0.610918	-0.003		0.590549	0.010	-3.3	50.0
29 cis-1,2-Dichloroethen	0.424760	-0.003		0.405925	0.010	-4.4	50.0
31 2-Butanone (MEK)	50.0	0.000	56.5	0.123684	0.010	13.1	50.0
32 Propionitrile	0.022422	0.000		0.021475	0.001	-4.2	50.0
33 Chlorobromomethane	0.183866	0.000		0.173351	0.010	-5.7	50.0
34 Tetrahydrofuran	50.0	0.000	49.6	0.067759	0.010	-0.9	50.0
36 Chloroform	0.654123	0.000		0.623457	0.010	-4.7	20.0
38 1,1,1-Trichloroethane	0.647318	0.000		0.616676	0.010	-4.7	50.0
$ 39 Dibromofluoromethane	0.390997	0.000		0.381290	0.010	-2.5	50.0
41 Carbon tetrachloride	0.442152	0.000		0.422945	0.010	-4.3	50.0
42 1,1-Dichloropropene	0.555419	-0.003		0.517284	0.010	-6.9	50.0
45 Benzene	1.096873	0.000		1.046656	0.010	-4.6	50.0
$ 46 1,2-Dichloroethane-d4	0.242197	-0.003		0.238625	0.010	-1.5	50.0
47 1,2-Dichloroethane	0.294105	0.000		0.284765	0.010	-3.2	50.0
50 Trichloroethene	0.361302	0.000		0.354230	0.010	-2.0	50.0
53 1,2-Dichloropropane	0.290522	-0.003		0.280418	0.010	-3.5	20.0
55 Dibromomethane	0.122793	0.000		0.120418	0.010	-1.9	50.0
56 Dichlorobromomethane	0.320968	0.000		0.313847	0.010	-2.2	50.0
57 2-Chloroethyl vinyl e	0.112860	0.000		0.112227	0.010	-0.6	50.0
59 cis-1,3-Dichloroprope	0.377191	0.000		0.376209	0.010	-0.3	50.0
60 4-Methyl-2-pentanone	0.175750	0.000		0.188969	0.010	7.5	50.0
$ 61 Toluene-d8 (Surr)	1.152608	0.000		1.147123	0.010	-0.5	50.0
62 Toluene	0.780498	0.000		0.748312	0.010	-4.1	20.0
63 trans-1,3-Dichloropro	0.310877	0.000		0.316394	0.010	1.8	50.0
65 1,1,2-Trichloroethane	0.176930	0.000		0.179778	0.010	1.6	50.0
66 Tetrachloroethene	0.441469	0.003		0.434668	0.010	-1.5	50.0
67 1,3-Dichloropropane	0.303885	-0.001		0.311866	0.010	2.6	50.0
68 2-Hexanone	0.126743	-0.001		0.143386	0.010	13.1	50.0
69 Chlorodibromomethane	0.259642	0.000		0.270972	0.010	4.4	50.0
70 Ethylene Dibromide	0.180199	-0.003		0.180854	0.010	0.4	50.0
71 1-Chlorohexane	0.705005	0.000		0.665336	0.010	-5.6	50.0
73 Chlorobenzene	0.958378	-0.003		0.964623	0.300	0.7	50.0
74 Ethylbenzene	0.558501	0.000		0.552290	0.010	-1.1	20.0
75 1,1,1,2-Tetrachloroet	0.341907	-0.003		0.354225	0.010	3.6	50.0
76 m-Xylene & p-Xylene	1.321180	0.000		1.321147	0.010	0.0	50.0

(052-002)

Report Date: 02-Aug-2011 12:34:06 Chrom Revision: 1.2 13-Jul-2011 10:43:06

Preliminary Report

Data File: \\chi-svr07\chromdata\CMS22\20110617-4051.b\22C0617.D

Compound	Standard RRF/Amount	DLT RT	Ccal Amount	Ccal RF	Min. RRF	%D	Max. %D
77 o-Xylene	1.342088	-0.001		1.343253	0.010	0.1	50.0
78 Styrene	1.076808	0.000		1.110846	0.010	3.2	50.0
79 Bromoform	0.141876	-0.001		0.158450	0.100	11.7	50.0
80 Isopropylbenzene	3.120042	0.000		3.216305	0.010	3.1	50.0
$ 83 4-Bromofluorobenzene	0.471439	0.000		0.477787	0.010	1.3	50.0
84 Bromobenzene	0.746997	0.000		0.793190	0.010	6.2	50.0
85 1,1,2,2-Tetrachloroet	0.339479	0.000		0.384016	0.300	13.1	50.0
86 N-Propylbenzene	3.635108	-0.003		3.783177	0.010	4.1	50.0
87 1,2,3-Trichloropropan	0.105308	0.000		0.117184	0.010	11.3	50.0
89 2-Chlorotoluene	2.011852	0.000		2.093599	0.010	4.1	50.0
90 1,3,5-Trimethylbenzen	2.602401	0.000		2.721596	0.010	4.6	50.0
91 4-Chlorotoluene	2.072909	0.000		2.180047	0.010	5.2	50.0
92 tert-Butylbenzene	2.456555	0.000		2.544641	0.010	3.6	50.0
94 1,2,4-Trimethylbenzen	2.612587	0.000		2.729876	0.010	4.5	50.0
95 sec-Butylbenzene	3.585473	0.000		3.749303	0.010	4.6	50.0
96 1,3-Dichlorobenzene	1.557914	0.000		1.667342	0.010	7.0	50.0
97 4-Isopropyltoluene	3.223253	0.000		3.390230	0.010	5.2	50.0
99 1,4-Dichlorobenzene	1.552060	0.000		1.616558	0.010	4.2	50.0
100 n-Butylbenzene	2.702610	0.000		2.920679	0.010	8.1	50.0
101 1,2-Dichlorobenzene	1.344982	0.000		1.427155	0.010	6.1	50.0
102 1,2-Dibromo-3-Chlorop	0.052535	0.003		0.058705	0.010	11.7	50.0
104 1,2,4-Trichlorobenzen	0.873623	-0.001		1.018662	0.010	16.6	50.0
105 Hexachlorobutadiene	0.737383	0.002		0.776512	0.010	5.3	50.0
106 Naphthalene	50.0	0.000	49.4	1.179642	0.010	-1.1	50.0
107 1,2,3-Trichlorobenzen	0.674664	0.002		0.771544	0.010	14.4	50.0
S 109 1,2-Dichloroethene, T	100.0		94.6			-5.4	50.0
S 110 Xylenes, Total	150.0		150.0			0.0	50.0
S 111 1,3-Dichloropropene,			100.8				50.0
S 112 Trimethylbenzene, Tot			104.5				50.0
S 113 Trihalomethanes, Tota			204.6				50.0

(052-003)

Report Date: 02-Aug-2011 12:42:29 Chrom Revision: 1.2 13-Jul-2011 10:43:06

Preliminary Report

TestAmerica Laboratories
CCV, Cal Verification Report

Data File: \\chi-svr07\chromdata\CMS22\20110617-4051.b\22D0617.D
Lims ID: CCV Client ID:
Inject. Date: 17-Jun-2011 12:35:30 Dil. Factor: 1.0000
Sample Type: CCV
Sample ID: CCV
Misc. Info.: 500-0004051-003 =500-0004051-003
Operator: EA Instrument ID: CMS22
Vol. Injected: 1.0000 ALS Bottle#: 3
Lims Batch ID: 4051 Lims Sample ID: 3
Sublist: chrom-8260W22*sub8
Detector: MS SCAN

Method: \\chi-svr07\chromdata\CMS22\20110617-4051.b\8260W22.m
Method Label: TAL CHICAGO VOA REPORT SW846 8260B
Last Update: 01-Aug-2011 13:00:22 Calib Date: 16-Jun-2011 18:31:30
Quant Method: Internal Standard Quant By: Initial Calibration
Last ICal File: \\chi-svr07\chromdata\CMS22\20110616-4027.b\22J0616H.D
Limit Group: MSVOA_8260_ICAL_WATER
Integrator: RTE ID Type: RT Order ID
Process Host: CHI-MS-OFF9A

First Level Reviewer: petruszakj Date: 02-Aug-2011 12:42:27

Start Cal Date: 16-Jun-2011 11:24:30
End Cal Date: 16-Jun-2011 18:31:30

Compound	Standard RRF/Amount	DLT RT	Ccal Amount	Ccal RF	Min. RRF	%D	Max. %D
4 Butadiene	0.334619	-0.003		0.340411	0.010	1.7	50.0
9 Ethyl ether	0.271312	0.000		0.273355	0.010	0.8	50.0
14 Iodomethane	100.0	-0.005	85.8	0.512804	0.010	-14.2	50.0
16 3-Chloro-1-propene	0.193810	-0.003		0.183023	0.010	-5.6	50.0
17 Methyl acetate	50.0	0.002	46.7	0.400136	0.010	-6.5	50.0
23 Hexane	0.816053	-0.006		0.809896	0.010	-0.8	50.0
24 Isopropyl ether	1.538330	0.000		1.553785	0.010	1.0	50.0
27 2-Chloro-1,3-butadien	0.911306	-0.003		0.945774	0.010	3.8	50.0
30 Ethyl acetate	0.185685	0.000		0.169040	0.010	-9.0	50.0
35 Methacrylonitrile	0.171858	0.003		0.152708	0.010	-11.1	50.0
37 Cyclohexane	0.903358	0.000		0.926651	0.010	2.6	50.0
$ 39 Dibromofluoromethane	0.390997	-0.003		0.381126	0.010	-2.5	50.0
43 Isooctane	1.510008	-0.002		1.572067	0.010	4.1	50.0
44 Isobutyl alcohol	0.012509	-0.009		0.013212	0.001	5.6	50.0
$ 46 1,2-Dichloroethane-d4	0.242197	-0.003		0.239538	0.010	-1.1	50.0
48 n-Heptane	0.726074	-0.002		0.746777	0.010	2.9	50.0
51 n-Butanol	0.005081	0.014		0.005291	0.001	4.1	50.0
52 Methylcyclohexane	0.550560	0.000		0.572834	0.010	4.0	50.0

(052-004)

Report Date: 02-Aug-2011 12:42:29 Chrom Revision: 1.2 13-Jul-2011 10:43:06

Preliminary Report

Data File: \\chi-svr07\chromdata\CMS22\20110617-4051.b\22D0617.D

Compound	Standard RRF/Amount	DLT RT	Ccal Amount	Ccal RF	Min. RRF	%D	Max. %D
54 Methyl methacrylate	0.186298	0.000		0.175848	0.010	-5.6	50.0
58 2-Nitropropane	4000.0	0.001	3800.7	0.054676	0.010	-5.0	50.0
$ 61 Toluene-d8 (Surr)	1.152608	0.000		1.146942	0.010	-0.5	50.0
64 Ethyl methacrylate	0.232341	0.000		0.236783	0.010	1.9	50.0
81 Cyclohexanone	0.016979	0.003		0.018886	0.010	11.2	50.0
$ 83 4-Bromofluorobenzene	0.471439	0.000		0.464202	0.010	-1.5	50.0
88 trans-1,4-Dichloro-2-	0.062533	0.000		0.066489	0.010	6.3	50.0
93 Pentachloroethane	100.0	0.001	107.1	0.289385	0.010	7.1	50.0
103 1,3,5-Trichlorobenzen	1.253016	0.000		1.397076	0.010	11.5	50.0
108 2-Methylnaphthalene	50.0	0.000	48.8	0.527950	0.010	-2.4	50.0

(052-005)

Report Date: 02-Aug-2011 12:19:10 Chrom Revision: 1.2 13-Jul-2011 10:43:06

Preliminary Report

TestAmerica Laboratories
CCV, Cal Verification Report

Data File: \\chi-svr07\chromdata\CMS16\20110706-4363.b\16C0706.D
Lims ID: CCV Client ID:
Inject. Date: 06-Jul-2011 07:42:30 Dil. Factor: 1.0000
Sample Type: CCV
Sample ID: CCV
Misc. Info.: 500-0004363-002
Operator: BDW Instrument ID: CMS16
Vol. Injected: 1.0000 ALS Bottle#: 2
Lims Batch ID: 4363 Lims Sample ID: 2
Sublist: chrom-8260S16*sub2
Detector: MS SCAN

Method: \\chi-svr07\chromdata\CMS16\20110706-4363.b\8260S16.m
Method Label: TAL Chicago VOA Report SW846 8260B
Last Update: 07-Jul-2011 07:57:53 Calib Date: 21-Jun-2011 13:55:30
Quant Method: Internal Standard Quant By: Initial Calibration
Last ICal File: \\chi-svr07\chromdata\CMS16\20110621-4088.b\16J0621F.D
Limit Group: MSVOA_8260_ICAL_SOIL_LOW
Integrator: RTE ID Type: RT Order ID
Process Host: CHI-MS-OFF9A

First Level Reviewer: wernerb Date: 06-Jul-2011 08:17:03

Start Cal Date: 21-Jun-2011 08:43:30
End Cal Date: 21-Jun-2011 13:55:30

Compound	Standard RRF/Amount	DLT RT	Ccal Amount	Ccal RF	Min. RRF	%D	Max. %D
S 1 Trimethylbenzene, Tot			104.0				50.0
S 2 1,3-Dichloropropene,			97.3				50.0
S 3 Trihalomethanes, Tota			195.7				50.0
4 Dichlorodifluorometha	0.362081	0.002		0.431522	0.010	19.2	50.0
5 Chloromethane	0.539148	0.000		0.634081	0.100	17.6	50.0
6 Vinyl chloride	0.432978	0.003		0.475562	0.010	9.8	20.0
8 Bromomethane	0.145101	0.002		0.146993	0.010	1.3	50.0
9 Chloroethane	0.169634	0.003		0.192984	0.010	13.8	50.0
10 Trichlorofluoromethan	0.629638	0.000		0.677803	0.010	7.6	50.0
12 Acrolein	0.078415	0.003		0.062447	0.001	-20.4	50.0
13 1,1-Dichloroethene	0.456667	0.000		0.449996	0.010	-1.5	20.0
14 1,1,2-Trichloro-1,2,2	0.430444	0.000		0.434774	0.010	1.0	50.0
15 Acetone	50.0	-0.003	48.2	0.133860	0.010	-3.7	50.0
17 Carbon disulfide	1.413243	0.000		1.338084	0.010	-5.3	50.0
18 Acetonitrile	0.044463	0.006		0.037286	0.001	-16.1	50.0
21 Methylene Chloride	50.0	0.003	47.9	0.491988	0.010	-4.2	50.0
22 Acrylonitrile	0.164057	0.000		0.142802	0.001	-13.0	50.0
23 trans-1,2-Dichloroeth	0.516737	-0.003		0.532013	0.010	3.0	50.0

(052-006)

Report Date: 02-Aug-2011 12:19:10 Chrom Revision: 1.2 13-Jul-2011 10:43:06

Preliminary Report

Data File: \\chi-svr07\chromdata\CMS16\20110706-4363.b\16C0706.D

Compound	Standard RRF/Amount	DLT RT	Ccal Amount	Ccal RF	Min. RRF	%D	Max. %D
24 Methyl tert-butyl eth	1.212365	0.000		1.149611	0.010	-5.2	50.0
26 1,1-Dichloroethane	1.022113	0.000		0.983863	0.100	-3.7	50.0
27 Vinyl acetate	0.851664	0.003		0.768155	0.010	-9.8	50.0
S 30 1,2-Dichloroethene, T	100.0		99.9			-0.1	50.0
31 cis-1,2-Dichloroethen	0.569538	0.000		0.551470	0.010	-3.2	50.0
32 2,2-Dichloropropane	0.448373	0.000		0.442485	0.010	-1.3	50.0
33 2-Butanone (MEK)	0.219663	-0.003		0.190751	0.010	-13.2	50.0
34 Propionitrile	0.034733	0.000		0.029280	0.001	-15.7	50.0
37 Chlorobromomethane	0.245038	0.003		0.236741	0.010	-3.4	50.0
38 Tetrahydrofuran	0.134120	0.008		0.125595	0.010	-6.4	50.0
39 Chloroform	0.829302	-0.001		0.817668	0.010	-1.4	20.0
$ 40 Dibromofluoromethane	0.510132	0.003		0.455690	0.010	-10.7	50.0
41 1,1,1-Trichloroethane	0.655147	0.002		0.682278	0.010	4.1	50.0
44 1,1-Dichloropropene	0.666161	0.000		0.689047	0.010	3.4	50.0
45 Carbon tetrachloride	0.339625	0.000		0.342979	0.010	1.0	50.0
$ 47 1,2-Dichloroethane-d4	0.298880	0.003		0.243094	0.010	-18.7	50.0
48 Benzene	1.173851	0.000		1.158893	0.010	-1.3	50.0
49 1,2-Dichloroethane	0.367092	0.000		0.348804	0.010	-5.0	50.0
54 Trichloroethene	0.317161	-0.003		0.314828	0.010	-0.7	50.0
56 1,2-Dichloropropane	0.317737	-0.003		0.320759	0.010	1.0	20.0
57 Dibromomethane	0.155455	0.002		0.148707	0.010	-4.3	50.0
59 Dichlorobromomethane	0.331008	0.000		0.327818	0.010	-1.0	50.0
61 2-Chloroethyl vinyl e	0.038803	0.003		0.044150	0.010	13.8	50.0
62 cis-1,3-Dichloroprope	0.393995	0.000		0.390320	0.010	-0.9	50.0
63 4-Methyl-2-pentanone	0.238968	-0.003		0.210377	0.010	-12.0	50.0
$ 64 Toluene-d8 (Surr)	1.022532	-0.006		0.901365	0.010	-11.8	50.0
65 Toluene	0.730360	0.003		0.724492	0.010	-0.8	20.0
66 trans-1,3-Dichloropro	0.320740	0.000		0.306593	0.010	-4.4	50.0
68 1,1,2-Trichloroethane	0.333368	0.002		0.318507	0.010	-4.5	50.0
69 Tetrachloroethene	0.376346	0.000		0.378979	0.010	0.7	50.0
70 1,3-Dichloropropane	0.471431	-0.003		0.465212	0.010	-1.3	50.0
71 2-Hexanone	0.219107	0.003		0.198604	0.010	-9.4	50.0
72 Chlorodibromomethane	0.292154	0.000		0.281754	0.010	-3.6	50.0
73 Ethylene Dibromide	0.188508	0.000		0.186530	0.010	-1.0	50.0
75 1-Chlorohexane	0.631353	0.003		0.674515	0.010	6.8	50.0
76 Chlorobenzene	1.060410	0.000		1.043879	0.300	-1.6	50.0

(052-007)

Report Date: 02-Aug-2011 12:19:10 Chrom Revision: 1.2 13-Jul-2011 10:43:06

Preliminary Report

Data File: \\chi-svr07\chromdata\CMS16\20110706-4363.b\16C0706.D

Compound	Standard RRF/Amount	DLT RT	Ccal Amount	Ccal RF	Min. RRF	%D	Max. %D
77 1,1,1,2-Tetrachloroet	0.344214	0.000		0.337548	0.010	-1.9	50.0
78 Ethylbenzene	0.554941	0.000		0.561023	0.010	1.1	20.0
79 m-Xylene & p-Xylene	1.360218	0.000		1.384070	0.010	1.8	50.0
80 o-Xylene	1.405525	0.000		1.409381	0.010	0.3	50.0
81 Styrene	1.086863	0.000		1.095296	0.010	0.8	50.0
82 Bromoform	0.149350	0.003		0.145505	0.100	-2.6	50.0
S 83 Xylenes, Total	150.0		151.9			1.3	50.0
84 Isopropylbenzene	3.632870	0.000		3.770386	0.010	3.8	50.0
$ 87 4-Bromofluorobenzene	0.471872	-0.003		0.398743	0.010	-15.5	50.0
88 1,1,2,2-Tetrachloroet	0.624608	0.000		0.636779	0.300	1.9	50.0
89 Bromobenzene	0.836163	0.003		0.848520	0.010	1.5	50.0
90 1,2,3-Trichloropropan	0.202839	0.000		0.193509	0.010	-4.6	50.0
92 N-Propylbenzene	4.327869	0.000		4.690454	0.010	8.4	50.0
93 2-Chlorotoluene	2.505304	0.000		2.557700	0.010	2.1	50.0
94 1,3,5-Trimethylbenzen	2.977069	0.000		3.102417	0.010	4.2	50.0
95 4-Chlorotoluene	2.897675	0.002		2.979118	0.010	2.8	50.0
96 tert-Butylbenzene	2.702209	0.003		2.747062	0.010	1.7	50.0
98 1,2,4-Trimethylbenzen	3.052601	0.000		3.169827	0.010	3.8	50.0
99 sec-Butylbenzene	3.914218	0.000		4.176373	0.010	6.7	50.0
100 1,3-Dichlorobenzene	1.682872	0.000		1.759467	0.010	4.6	50.0
101 4-Isopropyltoluene	3.402079	0.000		3.582315	0.010	5.3	50.0
103 1,4-Dichlorobenzene	1.676405	0.000		1.721865	0.010	2.7	50.0
104 n-Butylbenzene	3.158994	0.002		3.363054	0.010	6.5	50.0
105 1,2-Dichlorobenzene	1.548597	0.000		1.552027	0.010	0.2	50.0
106 1,2-Dibromo-3-Chlorop	50.0	0.005	47.8	0.099719	0.010	-4.3	50.0
108 1,2,4-Trichlorobenzen	1.140523	0.000		1.156843	0.010	1.4	50.0
109 Hexachlorobutadiene	0.545114	-0.003		0.575289	0.010	5.5	50.0
110 Naphthalene	2.316863	0.000		2.241002	0.010	-3.3	50.0
111 1,2,3-Trichlorobenzen	0.992962	0.000		0.968517	0.010	-2.5	50.0

(052-008)

Report Date: 02-Aug-2011 12:37:12 Chrom Revision: 1.2 13-Jul-2011 10:43:06

Preliminary Report

TestAmerica Laboratories
CCV, Cal Verification Report

Data File:	\\chi-svr07\chromdata\CMS16\20110706-4363.b\16D0706.D		
Lims ID:	CCV	Client ID:	
Inject. Date:	06-Jul-2011 08:08:30	Dil. Factor:	1.0000
Sample Type:	CCV		
Sample ID:	CCV		
Misc. Info.:	500-0004363-003		
Operator:	BDW	Instrument ID:	CMS16
Vol. Injected:	1.0000	ALS Bottle#:	3
Lims Batch ID:	4363	Lims Sample ID:	3
Sublist:	chrom-8260S16*sub4		
Detector:	MS SCAN		
Method:	\\chi-svr07\chromdata\CMS16\20110706-4363.b\8260S16.m		
Method Label:	TAL Chicago VOA Report SW846 8260B		
Last Update:	07-Jul-2011 07:57:53	Calib Date:	21-Jun-2011 13:55:30
Quant Method:	Internal Standard	Quant By:	Initial Calibration
Last ICal File:	\\chi-svr07\chromdata\CMS16\20110621-4088.b\16J0621F.D		
Limit Group:	MSVOA_8260_ICAL_SOIL_LOW		
Integrator:	RTE	ID Type:	RT Order ID
Process Host:	CHI-MS-OFF9A		
First Level Reviewer: wernerb		Date:	06-Jul-2011 08:26:28
Start Cal Date:	21-Jun-2011 08:43:30		
End Cal Date:	21-Jun-2011 13:55:30		

Compound	Standard RRF/Amount	DLT RT	Ccal Amount	Ccal RF	Min. RRF	%D	Max. %D
7 Butadiene	0.245977	0.003		0.240185	0.010	-2.4	50.0
11 Ethyl ether	0.494919	0.000		0.433470	0.010	-12.4	50.0
16 Iodomethane	0.725777	0.000		0.654672	0.010	-9.8	50.0
19 3-Chloro-1-propene	0.264502	0.000		0.238059	0.010	-10.0	50.0
20 Methyl acetate	0.725442	0.000		0.640256	0.010	-11.7	50.0
25 Hexane	1.059254	0.000		0.964921	0.010	-8.9	50.0
28 Isopropyl ether	2.018502	0.003		1.907320	0.010	-5.5	50.0
29 2-Chloro-1,3-butadien	1.093680	0.000		1.046910	0.010	-4.3	50.0
35 Ethyl acetate	0.326943	0.000		0.266071	0.010	-18.6	50.0
36 Methacrylonitrile	0.269531	0.003		0.224147	0.010	-16.8	50.0
$ 40 Dibromofluoromethane	0.510132	0.003		0.442521	0.010	-13.3	50.0
43 Cyclohexane	1.086713	0.000		1.038183	0.010	-4.5	50.0
46 Isobutyl alcohol	0.011570	-0.003		0.010594	0.001	-8.4	50.0
$ 47 1,2-Dichloroethane-d4	0.298880	0.003		0.241739	0.010	-19.1	50.0
50 Isooctane	1.444432	-0.011		1.419391	0.010	-1.7	50.0
51 n-Heptane	0.599658	0.000		0.582694	0.010	-2.8	50.0
53 n-Butanol	4000.0	-0.003	3522.1	0.006390	0.001	-11.9	50.0
55 Methylcyclohexane	0.545571	0.003		0.528624	0.010	-3.1	50.0

(052-009)

Report Date: 02-Aug-2011 12:37:12 Chrom Revision: 1.2 13-Jul-2011 10:43:06

Preliminary Report

Data File: \\chi-svr07\chromdata\CMS16\20110706-4363.b\16D0706.D

Compound	Standard RRF/Amount	DLT RT	Ccal Amount	Ccal RF	Min. RRF	%D	Max. %D
58 Methyl methacrylate	0.221919	0.002		0.194732	0.010	-12.3	50.0
60 2-Nitropropane	4000.0	-0.003	3222.2	0.070904	0.010	-19.4	50.0
$ 64 Toluene-d8 (Surr)	1.022532	-0.006		0.923979	0.010	-9.6	50.0
67 Ethyl methacrylate	0.381933	0.000		0.329464	0.010	-13.7	50.0
85 Cyclohexanone	0.128125	0.000		0.129708	0.010	1.2	50.0
$ 87 4-Bromofluorobenzene	0.471872	0.000		0.398797	0.010	-15.5	50.0
91 trans-1,4-Dichloro-2-	0.090216	0.000		0.072782	0.010	-19.3	50.0
97 Pentachloroethane	0.188078	0.000		0.135858	0.010	-27.8	50.0
107 1,3,5-Trichlorobenzen	1.181427	0.000		1.151802	0.010	-2.5	50.0
112 2-Methylnaphthalene	50.0	0.002	44.1	1.208652	0.010	-11.8	50.0

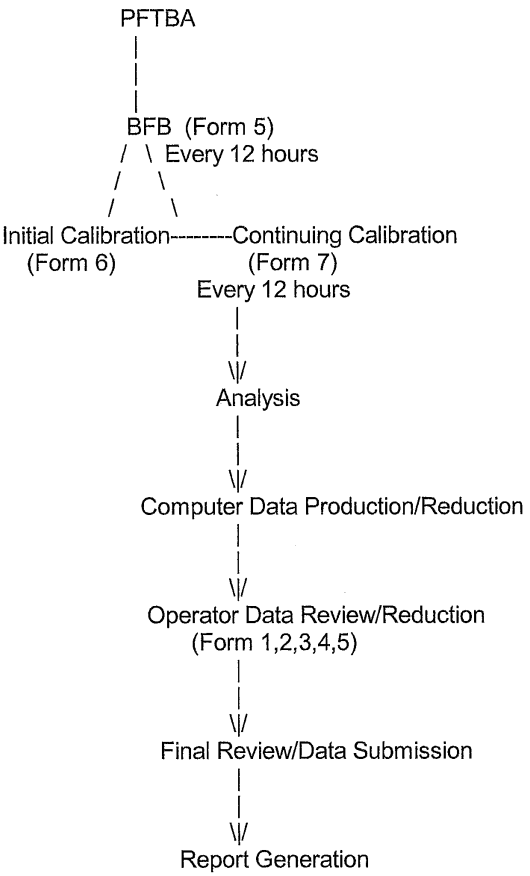
(052-010)

Attachment 5.

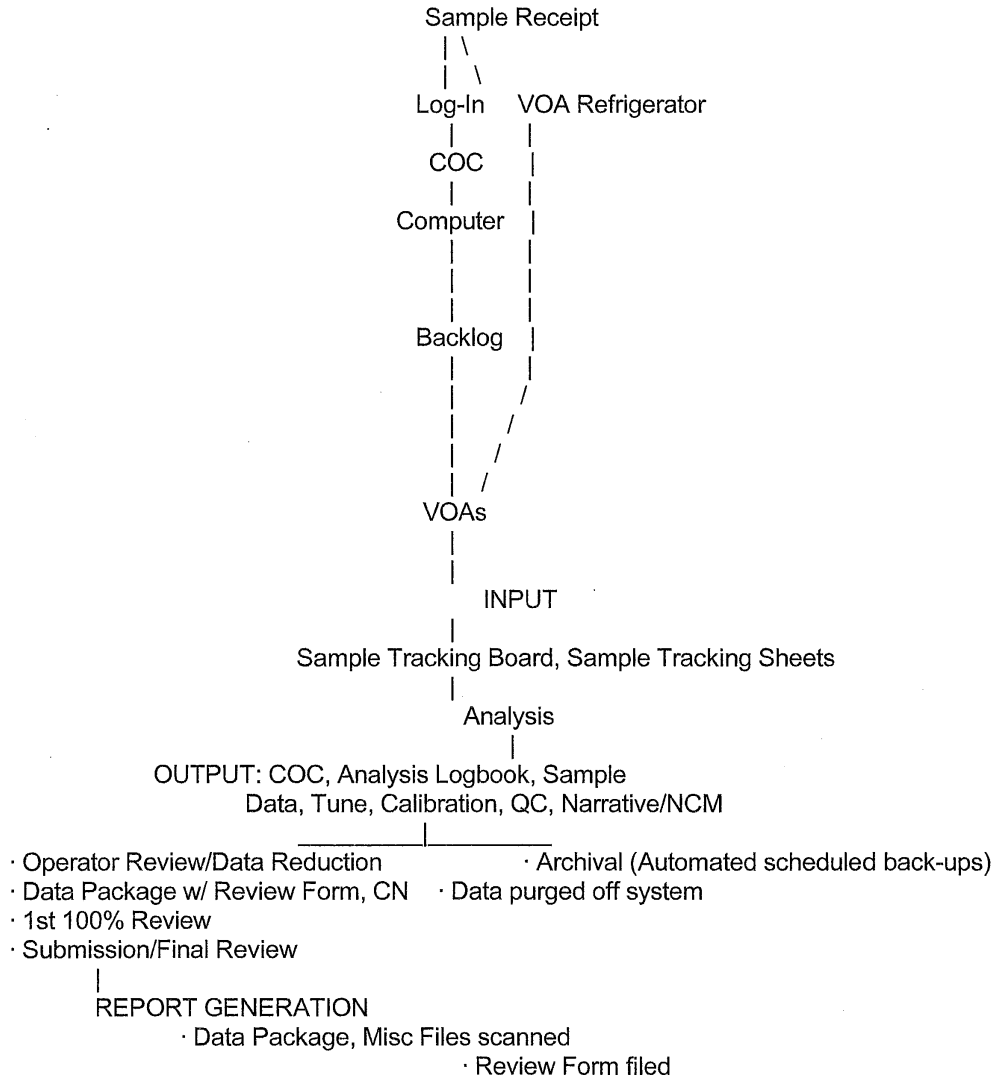
Example: Analysis and Sample Tracking Flowcharts

Analysis Scheme Flowchart

(Terms defined in the Section 9)



Sample Tracking Flowchart



Attachment 6.

Example: Data Review Checklist (056-001 to 056-003)

GC/MS Initial Calibration Review Checklist

Method: _____
Instrument: _____
Worklist Number or Batch: _____
Analysis Date: _____

Item for Review	Level 1	Level 2
TUNE:		
Tune passes, all points within the 12 hour clock?		
Reports:		
p-Flag reports reviewed and accepted?		
ICAL Pass / Fail reviewed and accepted?		
Average/Short Form 6 reviewed?		
Compounds:		
Linearity: CCC's ≤ 30% RSD		
Linear or Quadratic Regression curve fit for all >15% RSD R^2 >0.990		
Plots for all Linear Regressions reviewed?		
y-intercept < RL (<1/2 RL DoD QSM)		
Isomeric Pairs reviewed and accepted?		
Responses: SPCCs all pass minimum response factors?		
All manual integrations reviewed were found to be necessary, were performed correctly and have documented reasons?		
Method:		
Method saved to source/locked?		
Method set as most recent?		
Method uploaded to TALs?		
Calibration events correct in TALs?		
Initial Calibration Verification (ICV):		
ICV performed and evaluated against 75-125% (25%D) criteria?		
QAPP Specific ICV 80-120% (20%D) criteria?		
Comments:		

Note: By signing below I agree that I have reviewed the data as indicated on this checklist and according to the acceptance criteria found in the applicable SOP.

Primary Reviewer: _____ Date _____

Secondary Reviewer: _____ Date _____

(056-001)

TestAmerica Chicago
GCMS Volatiles DATA REVIEW CHECKLIST

Site Name:_____ Primary Reviewer:_____ Review Date:_____

JOB Number: _____ Secondary Reviewer:_____ Review Date:_____

Method: a) 8260B_____ 624 _____ 5030_____ Encores: 5035–High _____ 5035-Low_____ TCLP _____

Matrix: WATER / SOIL / SPLP-TCLP / Other (_____) Report Type: Level 1 2 3 4

TASK	PRI REV	SEC REV	COMMENTS
Inst# Date Worklist Analytical Batch _____ _____ _____ _____ _____			Associated Samples: Calib. ID: _____ _____ _____ _____ _____ _____
Sample Hold Time: _____ NCM Ref Number: _____			Date Analyzed: _____
Data Summaries: Executive Summary Are all flags correctly assigned? Y N Sample Data Sheets present? Y N Surrogate page present? Y N QC Data Summaries all present? Y N QC Associated Summary All samples associated? Y N Are 5035 samples linked to prep batch? Y N NA Are all dilutions appearing on summary? Y N NA			
FORM 1: IF **original** and **re-run** are to be reported in LIMS: Appropriate suffixes present Re-Analyzed (RA) Re-Extracted (RE) Dilution (DL) NCM Ref Number: _____			Smp # Original Dilution Comments
FORM 2: Surrogate Recoveries Within Limits Statistical Limits _____ AFCEE/LCG/DOD/QAPP:_____ NCM Ref Number: _____			List sample numbers and surrogates:
FORM 3: MS/MSD Recoveries Acceptable Statistical Limits _____ AFCEE/LCG/DOD/QAPP:_____ NCM Ref Number: _____			Sample _____ _____ MS MS MSD MSD RPD RPD
FORM 3: LCS Recoveries Acceptable (LCD if no MS/MSD) Statistical Limits _____ AFCEE/LCG/DOD/QAPP:_____ NCM Ref Number: _____			Control Compounds: Full / Other Batch # _____ Batch # _____ Batch # _____ Batch # _____

(056-002)

TASK	PRI REV	SEC REV	COMMENTS
Method Blank Detection Limits Met (< 1/2 RL for AFCEE / DoD QSM) NCM Ref Number: _____			
FORM 5: Tuning Criteria Met/Matches LIMS Analysis Batches ICAL Form 5 OK? Yes No Tunes: _____ _____ _____ _____ _____ _____ _____ _____ _____ NCM Ref Number: _____			Tune time met? Yes No
FORM 8: Internal Standards Criteria Met MPIS or CCVIS NCM Ref Number: _____			List sample numbers:
RAW DATA: 1) Raw Data Verified/Complete			
2) Screening Data match analysis data?			
Form 6: Initial Calibration Criteria met and Complete?			
Form 7: CCV criteria met method criteria NCM Ref Number: _____			
ICV (ICAL Spike Required): Yes No Control Limit applied: _____ NCM Ref Number: _____			
QC Raw data present and complete 1) Tune Yes No 2) Blank Yes No 3) LCS/LCSD Yes No 4) MS/MSD Yes No NA MRL Check Required: Yes No Control Limit Applied: _____			
Manual Integration reports (before and afters) present, analyst initials are present and the reasons for the MI are correctly documented and approved?			
Prep Log page Present / Verified			
NCM's reviewed and verified? Yes No			
ICOC Required/Properly Documented Yes No			
Additional Comments:			
Manual Calculation of On Column result: Response Factor (Smp) x Concentration of IS IS Response Factor (Smp) Cmpd. RRF (Cont.Calib)			Sample : _____ Compound: _____

CHI-22-20-081/C-05/11

(056-003)

Attachment 7.

CA-T-P-002: TestAmerica Corporate Policy for the Selection of Calibration Points (057-001 to 057-004)

Title: Selection of Calibration Points

Approvals (Signature/Date):

4/13/2011	4/1/2011
Richard Burrows	Dr. Charles W. Carter
Date	Date
Director of Technical Services	VP, Quality & Technical Services

Controlled Source: Intranet **Facility Distribution No.** _____

(057-001)

1.0 PURPOSE

This policy describes TestAmerica's requirements for determination of the number of points, and removal of points from calibration curves.

2.0 SCOPE

This Policy applies to all multi-level initial calibrations.

3.0 SAFETY

There are no specific safety hazards associated with this SOP.

During the course of performing this procedure it may be necessary to go into laboratory areas to consult with appropriate staff members, therefore employees performing this procedure must be familiar with the Laboratory Health & Safety Plan, and take appropriate precautions and wear appropriate attire and safety glasses.

4.0 DEFINITIONS

4.1 **Data Quality Objectives (DQOs)** are qualitative and quantitative statements used to ensure the generation of the type, quantity, and quality of environmental data that will be appropriate for the intended application.

5.0 PROCEDURE

5.1 If the number of data points required for an initial calibration is defined in the method, Quality Assurance (QA) plan, published report, or previously approved Standard Operating Procedure (SOP) that is what will be used.

5.2 In the cases not defined in Section 5.1, the number of data points will be determined by the technical manager based on the Data Quality Objectives (DQOs) for precision and accuracy to be met by the method.

5.2.1 When used for regulatory purposes, the minimum number of calibration points determined by the technical manager shall be three (3), except in cases where reference methods using similar technology use a single point and blank (ICP and ICP/MS primarily), or where the need is to demonstrate that the result is above or below a specific concentration limit.

5.2.2 Non-detects may use a simple point at the reporting level.

Examples:

A. Need to analyze a new pesticide in water and a published method does not exist. The data will be used to screen samples by UV-HPLC at a waste site for further remediation, using DQOs that require precision/accuracy of ± 50%.

The technical manager selects 2 data points to represent the range of the expected concentration of pesticide and based on 4 Laboratory Control Samples (LCS), the

recoveries ranged from 78-104%. Therefore, 2 data points are sufficient for initial calibration for this method.

Note: Calibration curves with less than 3 points should only be used after discussion with the client that the data quality objectives will be met. There must be indication in the final report to the client to reflect the calibration and/or analysis limitations.

B. Same compound as above but being measured in laboratory for meeting regulatory limit of 0.05 mg/L in water. Precision and accuracy of ± 20% required.

A five-point calibration is used, based on similar requirements in published methods with similar objectives and the high level of precision and accuracy required.

As noted above for methods where technical manager selects the number of data points to meet DQOs for precision and accuracy, the 4 LCS used in the demonstration of capability will be used to assure those DQOs are met. The SOP will then be approved by the Quality Assurance (QA) Manager.

5.3 Removal of Points from a Calibration Curve

5.3.1 Removal or replacement of levels from the middle of a calibration (i.e., levels other than the highest or lowest) is not permitted unless an injection or instrument problem confined to that point can be clearly documented as described below. The failed standard must be re-run within 24 hours and before any samples and inserted into the initial calibration. If not useful, recalibration is required. Removal of points for individual analytes from levels other than the highest and lowest is not permitted in any event.

5.3.2 If the analyst can document that a level is not valid because of an injection or instrument problem confined to that run (refer to Sec. 5.3.3), the level may be excluded if the curve still has sufficient levels, or the run may be repeated once only. The whole level (all compounds) must be removed or replaced. The curve is evaluated with the level removed or replaced. If the curve still fails to meet criteria, then corrective action must be taken and the whole curve reanalyzed. Corrective action may include, but is not limited to, instrument maintenance and/or re-preparation of standards.

5.3.3 One of the following conditions must be satisfied to allow removal or replacement of a level:

- The data file is corrupted and unusable or the run is interrupted before completion.
- The analyst observes and documents a problem such as leaking of a purge vessel.
- For internal standard methods, the recovery of the internal standard is less than 70% or greater than 130% of the recovery in the other standards (all internals show the same bias in the standard in question), or the amount of analyte recovered is less than 70% or greater than 130% of the expected values; indicating an improperly made up standard (all analytes in a spike mix must show the bias).
- For external standard methods, the unit response of the analyte is less than 70% or greater than 130% of the average unit response for the analyte in the other calibration standards; indicating an improperly prepared standard or bad injection. (all analytes in spike mix must

show the bias to demonstrate a standard is bad, all analytes in calibration standard must show bias to demonstrate bad injection)

5.3.4 When using autosamplers with discrete sample pathways for different samples (such as 16 port purge and trap autosamplers) the level to be replaced must be reanalyzed on the same port or that port must be excluded from sample analysis until corrective action is performed and verified by successful analysis of a continuing calibration standard on that port.

5.3.5 The reason for replacing the level **must** be documented in the run log. The fact that the curve passes criteria with the level removed is **not** alone sufficient evidence to document an injection or instrument problem confined to the level.

5.3.6 Removal of the highest or lowest levels is permitted, but the calibration range must be adjusted accordingly. If the lowest level is removed then the reporting limit is raised to be equivalent to the lowest level used in the calibration curve. In any event the number of levels remaining in the calibration must be at least that required by the method.

5.3.7 Removal of the highest or lowest point is permitted on a compound specific basis. This may be necessary when strongly responding and poorly responding analytes are included in the same standard mix at the same level. Each compound must have at least the minimum number of calibration levels required by the method.

6.0 RESPONSIBILITIES

All TestAmerica associates utilizing methods involving multi-point calibrations are required to follow this policy.

7.0 REFERENCES/CROSS-REFERENCES

None.

8.0 ATTACHMENTS

None.

9.0 REVISION HISTORY

- Revision 0, dated 20 November 2007
  - Initial Release. Previously Policy No. P-T-001.
- Revision 1, dated 23 June 2009.
  - No Changes.
- Revision 2, dated 1 April 2011.
  - Secs. 5.2.1-5.2.2: Section added to address the minimum number of calibration points as cited by the 2009 TNI Standard.

Attachment 8.

CA-Q-S-002: TestAmerica Corporate SOP for Manual Integrations (058-001 to 058-014)

Title: Acceptable Manual Integration Practices

Approvals (Signature/Date):

5/11/2011
Dr. Charles W. Carter — Date
Vice President of Quality & Technical Services

4/21/2011
Raymond J. Frederici — Date
Director of Quality and Client Advocacy

4/21/2011
Dr. Richard Burrows — Date
Director of Technical Services

Controlled Source: Intranet **Facility Distribution No.** _____

(058-001)

1.0 PURPOSE

Computerized data systems provide the analyst with the ability to re-integrate raw instrument data in order to optimize data quality. Though manual integration of data is an invaluable tool for resolving variations in instrument performance and some sample matrix problems, on the down side, the technique could also be improperly used to make unacceptable data appear to meet quality control acceptance limits. Improper re-integrations lead to legally indefensible data, a poor reputation, possible laboratory decertification, and potential legal consequences.

Guidelines for re-integration of data are not provided in the methods as most methods were written prior to widespread implementation of computerized data systems. This document describes TestAmerica's procedure for proper manual peak integrations and the requirements for documentation and review of manual peak integrations performed during the course of analyses. Attachment 2 highlights those improper procedures that must never be used.

2.0 SCOPE

This procedure applies to all TestAmerica laboratories for any analytical procedure involving identification or quantitation based on peak analysis (e.g., GC, GC/MS, HPLC, IC, and alpha or gamma spectroscopy). Each laboratory may have supplemental manual integration procedures or an addendum to this policy that describes additional details necessary to implement and enforce this policy. Any supplemental procedure or addendum must adhere to the requirements set forth in this policy.

Appropriate and consistent manual integration is a critical quality control requirement. Appropriate manual integration is integration that can be technically justified. Manual integrations shall not be used to manipulate analytical results for the sole purpose of meeting quality control (QC) acceptance criteria. Inappropriate and inconsistent chromatographic peak integration to meet quality requirements is never allowed, nor is it to be used as a substitute for corrective action on the chromatographic system. Willful failure to follow this policy shall result in disciplinary action, up to and including termination.

Analysts and data reviewers using peak integration techniques must be familiar with this policy and the applicable data processing system. This is to ensure that integration parameters are used in a manner that minimizes the need for manual integrations and that circumstances requiring manual integrations are performed in accordance with acceptable practices, documented and reviewed by a peer or supervisor.

3.0 SAFETY

There are no specific safety hazards associated with this SOP.

During the course of performing this procedure it may be necessary to go into laboratory areas to consult with appropriate staff members, therefore employees performing this procedure must be familiar with the Corporate Safety Plan, and take appropriate precautions and wear appropriate attire and safety glasses.

4.0 DEFINITIONS

4.1 Integration: The determination of the area or height under a curve (peak).

4.2 Manual integrations: Any manual changes to automated peak integration settings. This can include changes to integration start times, integration stop times, baseline changes, on-the-fly changes to retention time (RT) windows assigned to target analytes, manual peak height or area measurements, or changes to automated mass spectrometer tuning algorithms. This does not include re-centering of RT windows following routine instrument maintenance.

4.3 Chromatograms: In the context of this policy, chromatograms are not necessarily limited to the output of chromatography instruments or automated data systems. They can include strip charts, integrator printouts, computer screen dumps, or any graphic display of a continuous signal from an analytical detector.

4.4 Chromatography: A separation technique involving differential retention of components between stationary and mobile phases.

4.5 Baseline: The chromatographic signal plotted as a function of time in the absence of signal construction from components of interest.

4.6 Peak: An increase in signal from baseline to a maximum and then back to baseline.

4.7 Coelution: The concurrent elution of two or more compounds to the detector at the same time.

4.8 Elution: The process of movement of compounds from the chromatographic system.

4.9 "Wavy" Baselines: A "wavy" baseline can be caused by minor impurities in carrier gas or solvent.

4.10 Negative Spikes: Negative spikes in chromatograms are sudden upsets in chromatographic signal below the normal baseline.

4.11 Sudden Rise in Baseline: A rise in the baseline is caused by several factors including high concentration of components in the sample and by normal column bleed.

4.12 Carry-over: Carry-over results from system contamination from previous analyses and results in signal unrelated to the current analysis.

4.13 Peak Tailing: Peak tailing is a delayed return of a peak to chromatographic baseline and could be related to a delay of compound elution from the chromatographic system by adsorption or dead volume effects.

4.14 Interference Peaks or Fused Peaks: Peaks that partially or totally coelute with the peak of interest.

5.0 PROCEDURE

5.1 General Requirements

5.1.1 Audit trails or tracking systems MUST BE activated when the chromatographic system has this feature.

5.1.2 In some situations, manual integrations are necessary to compensate for imperfect chromatography, but manual integration must be performed ONLY when necessary. Baseline upsets, coelution, RT shifts, and peak shape variation can sometimes complicate automatic integration and analyst intervention through manual integration may be required to assure consistency between area assignment for standards and samples.

5.1.3 The same integration technique must be applied consistently to any chromatogram, i.e., field samples, calibration standards, calibration verification standards, and other applicable QC, etc., within the same analytical batch affected by the manual integration. Consistency in integration between standards and samples is one of the most important considerations in quantitative chromatographic analysis.

Examples of appropriate and improper integration can be found in Attachments 1 and 2.

5.2 Training Requirements

5.2.1 Initial manual integration training must conducted for all new analysts and data reviewers using methods involving peak analysis.

5.2.2 In addition, on-going training must be conducted annually.

5.3 Reasons to Manually Integrate

5.3.1 Undetected Peak

- A shift in RTs can result in undetected peaks or false positive identification of compounds. A common cause of RT shifts is analysis of highly contaminated samples. If significant RT shifts are observed in surrogate or internal standard compounds, then the potential of undetected peaks in samples must be carefully reviewed.

- Mass tuning changes that favor the light or heavy end of the mass spectra, or following highly contaminated samples, can sometimes cause the relative abundances of ions of compounds present to deviate from reference criteria, causing the peak to go undetected by the data system.

5.3.2 Incorrect Peak Integration

- Peak has small amount of splitting and the whole peak area was not integrated.

- Peaks close in retention times utilizing the same quantitation ions often integrate together as one peak, for example: ethylbenzene, xylenes; dichlorobenzenes, benzo(b)fluoranthene and benzo(k)fluoranthene.

5.3.3 Baseline Correction

- Matrix interferences caused by contaminated samples may interfere with calibrated compounds.

5.3.4 Other Examples of Events Requiring Manual Integration

- Incorrectly identified peak, where the wrong peak is chosen by the data system. This can occur both with the primary and the secondary ions.
- To remove a shoulder from a peak or to integrate a peak that only appears as a shoulder.

5.4 Data Systems

Ensure audit trails are turned on.

The chromatography system's method integration parameters must be optimized to the greatest extent possible so compounds are properly identified and integrated with minimal operator intervention. Ensure that all analytes in a midpoint standard have sufficient separation prior to calibration (e.g., minimum of a 25% peak/valley ratio, but there may be minor exceptions made on confirmation columns).

NOTE: **Even when integration parameters work properly for calibration standards, the analyst must ensure the integration is appropriate on all samples. The analyst must not assume that the chromatography system will automatically apply the correct integration**.

The following steps are required when manual integrations are necessary to identify peaks as targets (determine the RT), the calibration standards must be reprocessed after updating the retention times to demonstrate that the data system integration parameters are set properly:

5.4.1 Process the file using the current data system parameters for the mid-level standard.

5.4.2 Identify all of the target compounds and assign the correct RT to each target compound and the method (Calibration) file and save it.

5.4.2.1 The RTs may be updated daily by using the RTs in the ICV or daily CCV. If updating the RT is a daily procedure (in the method SOP), it need not be recorded, as it is a standard procedure.

5.4.2.2 If the RTs are only updated as needed (compounds not identified correctly), record in the instrument maintenance logbook or run log.

5.4.2.3 If the RTs are shifting on any frequent basis (use analytical judgment) within a calibration, instrument problems may be indicated. Perform maintenance and recalibrate the instrument. This **does not** include shifts due to column trimming, adjustments to gas pressure, or instrument maintenance.

5.4.3 Reprocess the data and confirm that all of the targets can be identified and properly integrated. If the targets cannot be identified and properly integrated, adjust the integration parameters and reprocess the data. This is an important step. If the data system cannot reliably detect and integrate the targets in the mid-level standard, the probability of properly identifying and quantifying the targets in the remaining standards and samples are low.

5.4.4 Process the remaining calibration standards and confirm that the data system can routinely and properly identify the target compounds at each calibration level. Pay particular attention to the lowest level standard because this standard typically defines the reporting limit (RL). The method integration parameters must allow for detection of the target compounds down to the quantitation limit or reporting limit.

5.4.5 Manual integration is not to be used solely to meet QC criteria, nor is it to be used as a substitute for corrective action on the chromatographic system.

5.4.6 The integration parameters as well as major method parameters (those that pertain to calculations/quantitation, e.g., changes to curve fit type, quant ion, internal standard assignment) that are set at the initial calibration must remain in use until the next calibration is performed (no changes without recalibration), except as noted below.

NOTE: An individual sample may need to utilize a different quantitation ion in the case of matrix interference. This would require clear documentation on why the change was made.

5.4.7 Any manual integration of a chromatographic peak or group of peaks must be documented. In all instances where the data system report has been edited or where manual integration has been performed, the chromatographic system operator must clearly identify such edits or manual procedures as listed below:

5.4.7.1 Manual integrations indicated on expanded scale "after" chromatograms. That is, the after chromatogram must be presented at sufficient scale expansion to allow data reviewers to independently evaluate the manual integration. Expanded scale "before" chromatograms are also required for all manual integrations on QC parameters (calibrations, calibration verifications, laboratory control samples, internal standards, surrogates, etc.) unless the laboratory has another documented corporate approved procedure in place that can demonstrate an active process for detection and deterrence of improper integration practices.

5.4.7.2 Re-integration technique marked, if available, on the data system.

5.4.7.3 Technical justification for manual integration.

- Either entered in the instrument audit trail for electronic review.
- Clearly marked on the "after" chromatogram, manually or electronically. The table below contains some example abbreviations that could be used to simplify documentation for the reason for manual integrations. Others means may be used.

Reasons for Manual Integration	Acronym/Code
Baseline	BL
Co-elution	CE
Contamination	CON
Wrong Retention Time	WRT
Tailing	T
Matrix Interference	MI
Splitting	S
No Spectral Match (for deletions)	NSM

5.4.7.4 Analyst's initials and date. (Data system application of analyst name or initials is acceptable so long as the data system allows the analyst to log in as himself or herself and has password control.)

5.4.8 Project specific, client, program or TestAmerica laboratory specific requirements for manual integration may exceed the requirements of this policy. In those instances, the more specific requirements will apply or written approval allowing a deviation from the requirement must be received. For example, the following requirements apply to all Department of Defense (DoD) projects:

5.4.8.1 Requiring the before and after printing of all chromatograms.

5.4.8.2 Handwritten or electronic initialing and dating the changes made to the report.

5.4.8.3 Hardcopy printout of the EICP of the quantitation ion displaying the manual integration included in the raw data for all standards and samples, this applies to internal standards and surrogates as well.

5.4.8.4 For DoD, manual integrations need to be documented in the raw data ***and*** in the case narrative.

5.4.9 Data reviewers shall confirm that documentation of manual integration is complete and that each manual integration is appropriate. This inspection shall be documented. At a minimum, the information required in Section 5.4.7 must be reviewed, and for DoD projects all of the elements in 5.4.8 must be reviewed. Any deficiencies must be resolved with the analyst or their supervisor before the results are approved and released from the analytical department.

5.4.10 If integration indicates problems with analytical instrumentation, investigate the problem and take action to correct it. If poor chromatography routinely interferes with the ability to identify and quantify components; e.g., is a result of delayed system maintenance; and is not inherent in the system, such as (benzoic acid for Method 8270; benzo(b)fluoranthene and benzo(k)fluoranthene merging with column age; and dichlorobutenes merging with column age), then instrument maintenance must be performed. In the case of isomeric pairs, if resolution does not meet method criteria, it may be more appropriate to report as totals instead of individual peaks.

5.4.11 Manual integrations on some data systems (e.g., Multichrom) may consist of adjusting integration parameters on a sample-by-sample basis. Changes such as these must be well documented.

5.5 Data Miner Software

5.5.1 Electronic data surveillance is performed using automated data mining software, such *Mint Miner*™ (works with EnviroQuant, Target, and Turbochrom) and *Audit Miner* (works with Chrom). This software allows the user to connect to a workstation or archived data source and evaluate the audit trails in the electronic data file for manual integrations. It identifies any changes that are made to raw data files so they can be reviewed to ensure integrations were performed in accordance with this SOP.

5.5.2 Any questions arising from review of the documentation must be investigated in the electronic record, when available, prior to data acceptance.

NOTE: Some integrations are not readily obvious on the printouts; in these cases the electronic record must be reviewed. Any failures to complete these requirements shall be described in a non-conformance report.

5.5.3 Any concerns about violations of this policy must be reported to the laboratory Quality Manager, Laboratory Director or the Corporate Quality Director.

6.0 RESPONSIBILITIES

6.1 Analysts are responsible for following this SOP and the TestAmerica Ethics and Data Integrity Policy.

6.2 Data reviewers are responsible to ensure that all documentation is correct and the decisions to report the data are in accordance with this SOP.

7.0 REFERENCES / CROSS-REFERENCES

7.1 Acceptable Manual Integration Training Presentation (CA-Q-T-001).

7.2 Practical Use of Mint Miner and Audit Miner Training Presentation (CA-Q-T-018).

8.0 ATTACHMENTS

Attachment 1. Examples of Peak Shape and Proper Integration Documenting Manual Integrations.

Attachment 2. Examples of Improper Integration and Inflection Point Guidance.

9.0 REVISION HISTORY

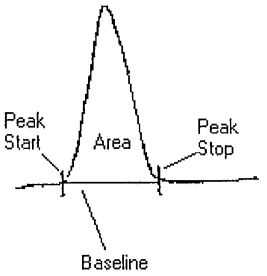
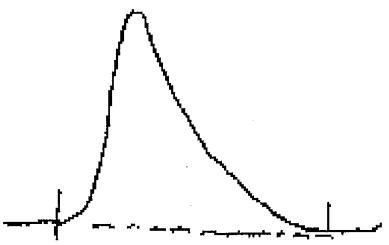
- Revision 0; dated 3 October 2007.
  - Initial Release.
- Revision 0.1; dated 30 November 2007.
  - Moved the first sentence from Sec. 7.2 to be the last sentence in Sec. 6.3.

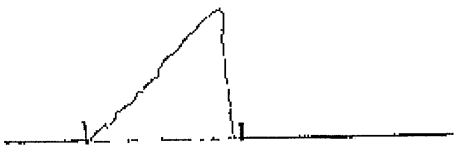
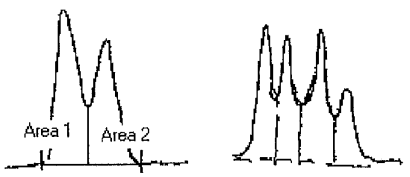
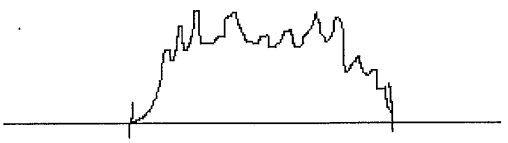
- Revision 0.2, dated 20 February 2008.
  - Section 2.3: Deleted the statement '..integration below the baseline is never acceptable.'
  - Section 4.2: Clarified that adjusting the actual retention time and not the RTW is a Manual Integration.
  - Section 6.2.2.1: Clarified that the RTs may also be updated by the daily CCV.
  - Section 6.2.4: Clarified reintegration of the low calibration point.
  - Section 6.2.7, Bullet Item 3.2: Clarified that the reason for the MI can documented either manually or electronically.
- Revision 1, dated 2 November 2009.
  - Section 2.1: Clarified annual training.
  - Section 5.2.10: Clarified Manual Integration documentation in the raw data & case narrative for DoD work.
- Revision 2, dated 20 April 2011.
  - Clarifications to separate policy from procedure and training requirements.
  - Simplified definition of manual integration.
  - Expanded discussion of undetected peaks to include all chromatography systems, rather than GC/MS only.
  - Expanded section on data miner software to include Chrom Audit Miner
  - Included reference to data miner training presentation.

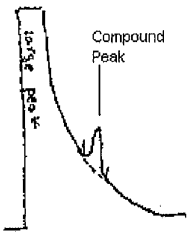
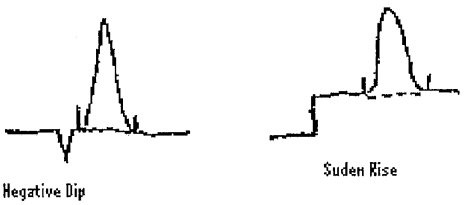
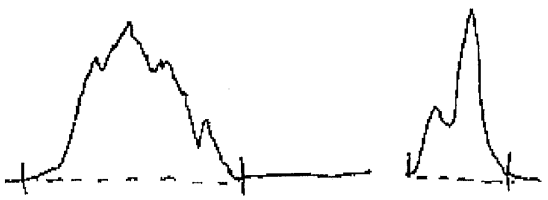
(058-009)

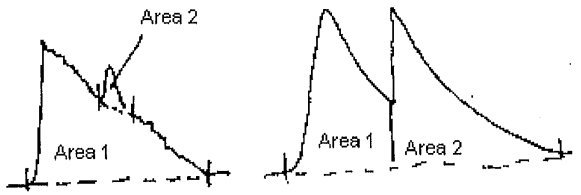
Attachment 1.

Examples of Peak Shape and Proper Integration Documenting Manual Integrations

Ideal peak **(Gaussian Shape)** The start of integration is where the peak begins to rise from the background, and the end is where the peak returns to background.	
Tailing Peak Tailing peaks result from surface adsorption effects or dead volume in the instrument. When integrating tailing peaks, include only and all area that can be attributed to compound. If the baseline rises, the integration line should rise with the baseline.	

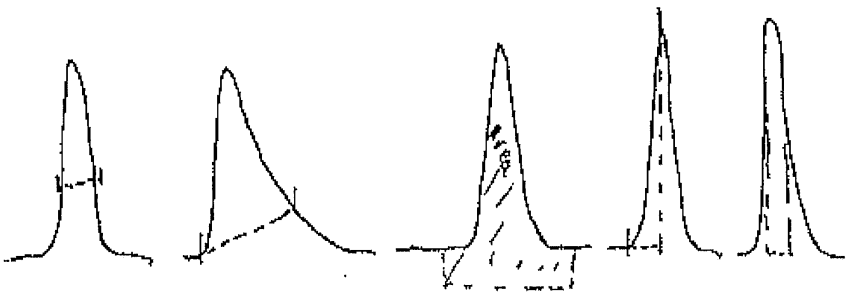
Fronting Peak Fronting peaks generally result from column overload and/or overcapacity. An example is benzoic acid on a 5% phenyl phase. In some cases, it is inherent in the analysis, in others, it is indicative of problems in the system. The analyst must have knowledge of their system. To integrate these peaks, it is important to have prior knowledge of how the compound acts when overloading versus co-eluting with a contaminate peak.	
Fused Peaks Fused peaks are near coeluters with some separation. They will occur in calibration mixes as well as real samples. To integrate, split the components with a vertical drop from the valley to the baseline.	
Hydrocarbon Envelope For semivolatile hydrocarbon analysis, most fuels produce "humpograms" like this one. Integration shall be performed in accordance with method, program or client specific requirements.	
Peak on a Baseline Rise Aside from hydrocarbon analysis, which uses the total area within a window for quantitation, baseline variations are not integrated. Therefore, skim the baseline rise to integrate the peak properly.	

Skimmed Peak Use the tangent skimming functions of the integration software to correctly integrate small peaks on the tails of big peaks. Dropping vertical integration lines to the baseline of the big peak adds extra area which is not a part of the small peak, and results in high bias.	
Peak near a baseline upset Negative dips are common in ECD analysis, and the sudden rises in baseline are common with programmable fluorescence detectors where gain/wavelength switching occurs. Often, the default integration will go from the base of the negative peak to the far side of the peak of interest. This is results in high bias and is incorrect. Similarly, a default integration using the low part of the baseline rise and the far side of the peak of interest will also result in high bias and must be corrected.	
Split Peak Particularly in GC/MS analysis, low-level peaks may appear jagged or split. To integrate these properly, the analyst must have prior knowledge of the peak/compound chromatography.	

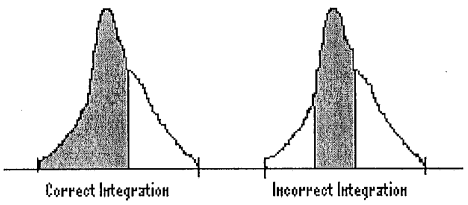
Fused tailing peaks These are very difficult to accurately quantify. It is best to do maintenance on the chromatography system to avoid this situation. To integrate these properly, the analyst should have prior knowledge of the compounds of interest. It is important to be consistent when integrating standards and samples.	

Attachment 2.

Examples of Improper Integration and Inflection Point Guidance



An inflection point must be apparent when and if an integration point is drawn. For example:



When MS data has an inflection point in a peak, as shown in the example above, checking the underlying spectrum may be necessary to correctly manually integrate. On some chromatographic columns, for example, allyl chloride and carbon disulfide elute very close to each other, and they both share the primary characteristic ion mass 76. For both compounds mass 76 is the quantification mass. If two compounds can not be separated and a fused peak is going to be split at the inflection point, this can be correctly verified by looking at the spectra for each scan. In this example, the 76 peak may be fused, but the secondary ions will indicate which compound is present (carbon disulfide with secondary ion 78 and allyl chloride with secondary ions 41, 39 and 78).

Attachment 9.

List of Poor Purging or Poorly Performing Compounds

Acetone	Ethyl Acetate
Acetonitrile	2-Hexanone
Acrolein	Isobutyl Alcohol
Acrylonitrile	Methacrylonitrile
Bromomethane	Methyl Acetate
n-Butanol	4-Methyl-2-pentanone
2-Butanone (MEK)	2-Nitropropane
Carbon Disulfide	Propionitrile
Chloroethane	Tetrahydrafuran
2-Chloroethylvinyl ether	Trans-1,4-dichloro-2-butene
Chloromethane	1,1,2-Trichloro-1,2,2-trifluoromethane
Dichlorodifluoromethane	Trichlorofluoromethane
1,2-Dibromo-3-chloropropane	Vinyl Acetate

Attachment 10.

DOD QSM Version 4.2: Appendix F QC Requirements Summary
(Table F-1; and Table F-4)
(060-001 to 060-005)

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DoD QSM Version 4.2: Appendix F - Quality Control Requirements Summary

Table F-1 Summary of QC Check Definitions, Purpose, and Evaluation – Organics (GC/MS)

QC Check	Definition	Purpose	Evaluation
Breakdown Check 8081: Endrin, DDT 8270: DDT	Analysis of s standard solution containing Endrin and DDT. Area counts of these compounds and their breakdown products are evaluated to assess instrument conditions.	To verify the inertness of the injection port because DDT and Endrin are easily degraded in the injection port.	If degredation of either DDT or Endrin exceeds method–specified criteria, corrective action must be taken before proceeding with calibration
Confirmation of positive results (organics only)	Use of alternative analytical techniques (another method, dissimilar column, or different detector such as MS detector) to validate the presence of target analytes identified	To verify the identification of an analyte	All positive results must be confirmed.
Continuing calibration verification (CCV)	This verification of the ICAL that is required during the course of analysis at periodic intervals. Continuing calibration verification applies to both external standard and internal standard calibration techniques, as well as to linear and non-linear calibration models	To verify that instrument response is reliable, and has not changed significantly from the current ICAL curve.	If the values for the analytes are outside the acceptance criteria, the ICAL may not be stable. Results associated with out-of-control CCV results require reanalysis or flagging
Demonstrate Acceptable Analytical Capability	QC samples are analyzed in series to verify ability to produce data of acceptable precision and bias.	To verify the ability to produce data of acceptable precision and bias for a specific instrument type, matrix, method, and analyst.	The average recovery of the spikes and standard deviation of the replicates must be within designated acceptance criteria. Analysis of field samples may not be conducted until this check is successful.
Duplicate Sample (replicate)	Two identical portions of material collected for chemical analysis, and identified by unique alphanumeric codes. The duplicate may be portioned from the same sample, or may be two identical samples taken from the same site. The two portions are prepared and analyzed identically. (modified QSM)	To provide information on the heterogeneity of the sample matrix or to determine the precision of the intralaboratory analytical process for a specific sample matrix	A duplicate sample will provide information on the heterogeneity of the sample matrix. The greater the heterogeneity of the matrix, the greater the relative percent difference between the sample and the sample duplicate. If the sample matrix is homogeneous (such as with drinking water) and the relative percent difference is high, this could indicate a problem in the analytical system.
Initial calibration for all analytes (ICAL)	Analysis of analytical standards at different concentrations that are used to determine and calibrate the quantitation range of the response of the analytical detector or method	To establish a calibration curve for the quantification of the analytes of interest	Statistical procedures are used to determine the relationship between the signal response and the known concentration of analytes of interest. The ICAL must be successful before any samples or other QC check samples can be analyzed.
Internal Standards	A substance that is introduced in known amount into each calibration standard and field and QC sample of the analyte.	The ratio of the analyte signal to the internal standard signal is then used to determine the analyte concentration.	Any sample associated with out-of-control results must be reanalyzed.
Laboratory control sample (LCS) containing all analytes to be reported	A sample matrix, free from the analytes of interest, spiked with known amounts of analytes or a material containing known and verified amounts of analytes.	Used to evaluate the performance of the total analytical system, including all preparation and analysis steps. Assesses the ability of the laboratory/analyst to successfully recover the target analytes from a control (clean) matrix. Control limits for LCS recovery, typically expressed as percent recovery, are used for the development of statistical control limits and serve as acceptance criteria for determining whether an analytical run is in control (batch acceptance).	This is a required QC Check. The inability to achieve acceptable recoveries in the LCS indicate problems with the precision and bias of the measurement system. Failure to achieve acceptable recoveries in a "clean" matrix is an indicator of possible problems achieving acceptable recoveries in field samples.
MS	A sample prepared by adding a know mass of target analyte to a specified amount of matrix sample for which an independent estimate of target analyte concentration is available.	To assess the performance of the method as applied to a particular matrix. Matrix spikes are used, for example, to determine the effect of the matrix on a method's recovery efficiency. The recovery of target analytes from the matrix spike sample is used to determine the bias of the method in the specific sample matrix.	The lack of acceptable recoveries in the matrix spike often points to problems with the sample matrix. One test of this is a comparison to the LCS recoveries. If the corresponding LCS recoveries are within acceptable limits, a matrix effect is likely. The lab should not correct for recovery; only report the results of the analyses and the associated MS results and indicate that the results from these analyses have increased uncertainty

Table F-1 Cont.			
QC Check	**Definition**	**Purpose**	**Evaluation**
MSD	A second replicate matrix spike prepared in the laboratory and analyzed to obtain a measure of the precision of recovery for each analyte	To assess the performance of the method as applied to a particular matrix and provide information on the homogeneity of the matrix. Also used to determine the precision of the intralaboratory analytical process for a specific sample matrix.	When compared to the MS, the MSD will provide information on the heterogeneity of the sample matrix. The greater the heterogeneity of the matrix, the greater the RPD between the matrix spike and the matrix spike duplicate. If the sample matrix is homogeneous, such as with drinking water, and the RPD is high, this could indicate a problem in the analytical system.
MB	A sample of a matrix similar to the batch of associated samples (when available) that is free from the analytes of interest and is processed simultaneously with an under the same conditions as samples through all steps of the analytical procedures, and in which no target analytes or interferences are present at concentrations that impact the analytical results for sample analyses.	To assess background interferences or contamination in the analytical system that might lead to high bias or false positive data. Results of method blanks provide an estimate of the within-batch variability of the blank response and an indication of bias introduced by the preparation and analytical procedure.	This is one of the QC samples used to measure lab accuracy/bias. The sample could indicate whether contamination is occurring during sample prep and analysis. If analytes are detected > ½ RL, reanalyze or qualify (B-flag) all results for the specific analytes(s) in all samples in the associated prep batch as appropriate. For common lab contaminants, no analytes detected > RL. See Section D.1.1.1 and Box D-1
RT window position establishment for each analyte (and surrogate) (all chromatographic methods only)	Determination of the placement of the RT window (i.e. start/stop time) of each analyte or group of analytes as it elutes through the chromatographic column so that analyte identification can be made during sample analysis. This is done during the ICAL.	To idendify analytes of interest	Incorrect window position may result in false negatives, require additional manual integrations, or cause unnecessary reanalysis of samples when surrogates or spiked compounds are erroneously not identified.
Second source calibration verification (ICV)	A standard obtained or prepared from a source independent of the source of standards for the ICAL. Its concentration should be at or near the middle of the calibration range. It is done after the ICAL.	To verify the accuracy of the ICAL.	The concentration of the 2nd source calibration verification, determined from the analysis, is compared with the known value of the standard to determine the accuracy of the ICAL. This independent verification of the ICAL must be acceptable before sample analysis can begin.
Surrogate spike (organic analysis only)	A substance with properties that mimic the analyte of interest. It is unlikely to be found in environmental samples and is added to them for quality control purposes.	To assess the ability of the method to successfully recover specific non-target analytes from an actual matrix. Because surrogates are generally added to each sample in a batch, they can be used to monitor recovery on a sample-specific, rather than batch-specific basis.	Whereas the MS is normally done on a batch-specific basis, the surrogate spike is done on a sample-specific basis. Taken with the information derived from other spikes (LCS; MS), the bias in the analytical system can be determined.
Tuning (MS methods only)	The analysis of a standard compound to verify that the mass spectrometer meets standard mass spectra abundance criteria prior to sample analysis. (COE)	To verify the proper working of the mass spectrometer	Proper tuning of the mass spectrometer must be verified prior to sample analysis

Notes:
1. Project-specific requirements identified by the client supersede any requirements listed. The requirements are meant to be default, to be used when project-specific direction based on DQOs is not available.
2. If there is a contradiction between the method and the DoD tables, the requirements specified in the tables shall be followed.

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DoD QSM Version 4.2: Appendix F - Quality Control Requirements Summary

Table F-4: Organic Analysis by GC/MS - Methods 8260 and 8270

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Demonstrate acceptable analytical capability	Prior to using any test method and at any time there is a significant change in instrument type, personnel, test method, or sample matrix.	QC acceptance criteria published by DoD, if available; otherwise method- specific criteria.	Recalculate results; locate and fix problem, then rerun demonstration for those analytes that did not meet criteria (see Section C.1.f).	NA	This is a demonstration of analytical ability to generate acceptable precision and bias per the procedure in Appendix C. No analysis shall be allowed by analyst until successful demonstration of capability is complete.
LOD Determination and verification (See Box D-13)					
LOQ Establishment and verification (See Box D-14)					
Tuning	Prior to ICAL and at the beginning of each 12- hour period.	Refer to method specific ion criteria	Retune instrument and verify. Rerun affected samples	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be accepted without a valid tune.
Breakdown check DDT (8270 only)	At the beginning of each 12-hour period, prior to analysis of samples	Degradation $\leq$ 20% for DDT Benzidine & PCP should be present at their normal responses and should not exceed a tailing factor of 2.	Correct problem then repeat breakdown check.	Flagging criteria are not appropriate.	No samples shall be run until degradation $\leq$ 20%.
Minimum five-point initial calibration (ICAL) for all analytes	ICAL prior to sample analysis.	1. Average response factor (RF) for SPCCs: VOCs $\geq$ 0.30 for Chlorobenzene and 1,1,2,2-tetrachloroethane, $\geq$ 0.1 for chloromethane, bromoform, and 1,1-dichloroethane SVOCs $\geq$ 0.050 2. RSD for RFs for CCCs: VOCs and SVOCs $\leq$ 30% and one option below. **Option 1:** RSD for each analyte $\leq$ 15% **Option 2:** linear least squares regression: $r \geq 0.995$ **Option 3:** non-linear regression-coefficient of determination (COD) $r^2 \geq 0.99$ (6 points shall be used for 2nd order, 7 points shall be used for 3rd order)	Correct problem then repeat initial calibration	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until ICAL has passed. Calibration may not be forced through the origin.
2nd Source calibration verification (ICV)	Once after each ICAL	All project analytes within $\pm$ 20% of true value	Correct problem and verify 2nd source standard. Rerun, 2nd source verification. If that fails, correct problem and repeat ICAL.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until calibration has been verified.

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
RT window position establishement for each analyte and surrogate	Once per ICAL	Position shall be set using midpoint standard of the ICAL curve when ICAL is performed. On days when ICAL is not performed, the initial CCV is used.	NA	NA	
Evaluation of relative retention times (RRT)	With each sample	RRT of each target analyte within ± 0.06 RRT units.	Correct problem, then rerun ICAL.	Flagging criteria are not appropriate.	Labs may update the RTs based on the CCV to account for minor performance fluctuations or after routine system maintenance (such as column clipping). With each sample, the RRT shall be compared with the most recently updated RRT. If the RRT has changed by more than ± 0.06 RRT units since the last update, this indicates a significant change in system performance and the lab must take appropriate corrective actions as required by the method and rerun the ICAL to reestablish the RTs.
Continuing calibration verification (CCV)	Daily before sample analysis and every 12 hours of analysis time.	1. Average RF for SPCCs: VOCs ≥ 0.30 for Chlorobenzene and 1,1,2,2-tetrachloroethane, ≥ 0.1 for chloromethane, bromoform, and 1,1-dichloroethane SVOCs ≥ 0.050 2. %Difference/Drift for all target compounds and surrogates: VOCs and SVOCs ≤ 20% D (Note: D = difference when using RFs or drift when using least squares regression or non-linear calibration).	DoD project level approval must be obtained for each of the failed analytes or corrective action must be taken. Correct problem, then rerun calibration verification. If that failes, then repeat ICAL. Reanalyze all samples since last acceptable CCV.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to all results for the specific analyte(s) in all samples since the last acceptable CCV.	Problem must be corrected. Results may not be reported without a valid CCV. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Internal Standards verification	Every field sample, standard, and QC sample.	RT ± 30 seconds from RT of the midpoint standard in the ICAL EICP area within -50% to +100% of ICAL midpoint standard.	Inspect mass spectrometer and GC for malfunctions. Reanalysis of samples analyzed while system was malfunctioning is mandatory.	If corrective action fails in field samples, apply Q-flag to analytes associated with the non-compliant IS. Flagging criteria are not appropriate for failed standards.	Sample results are not acceptable without a valid IS verification.
Method Blank (MB)	One per prep batch	No analytes detected > ½ RL and > 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater). Blank result must not otherwise affect sample results. For common lab contaminants, no analytes detected > RL (see Box D-1).	Correct problem, then see criteria in box D-1; if required, reprep and reanalyze MB and all samples processed with the contaminated blank.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply B-flag to all results for the specific analyte(s) in all samples in the associated prep batch.	Problem must be corrected. Results may not be reported without a valid MB. Flagging is only appropriate in cases where the samples cannot be reanalyzed.

(C-2) C-454

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QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
LCS containing all analytes to be reported, including surrogates	One per prep batch	QC acceptance criteria specified by DoD, if available. Otherwise, use in-house control limits. In-house control limits may not be greater than ± 3 times the standard deviation of the mean LCS recovery. See Box D-3 and Appendix G.	Correct problem, then reprep and reanalyze the LCS and all samples in the associated prep batch for failed analytes, if sufficient sample material is available (see full explanation in Appendix G).	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to specific analyte(s) in all samples in the associated prep batch.	Problem must be corrected. Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Matrix Spike (MS)	One per prep batch per matrix (see Box D-7).	For matrix evaluation, use LCS acceptance criteria specified by DoD, if available. Otherwise, use in-house LCS control limits.	Examine the project-specific DQOs. Contact the client as to additional measures to be taken.	For specific analytes(s) in the parent sample, apply J-flag if acceptable criteria are not met.	For matrix evaluation only. If MS results are outside the LCS limits, the data shall be evaluated to determine the source of difference and to determine if there is a matrix effect or analytical error.
Matrix spike duplicate MSD or Sample Duplicate	One per prep batch per matrix (see Box D-7).	MSD: For matrix evaluation, use LCS acceptance criteria specified by DoD, if available. Otherwise, use in-house LCS control limits. MSD or sample duplicate: RPD ≤ 30% (between MS and MSD or sample and sample duplicate).	Examine the project-specific DQOs. Contact client as to additional action measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met.	The data shall be evaluated to determine the source of difference.
Surrogate spike	All field and QC samples	QC acceptance criteria specified by DoD, if available. Otherwise use in-house control limits.	For QC and field samples, correct problem then reprep and reanalyze all failed samples for the failed surrogates in the associated prep batch, if sufficient sample material is available. If obvious chromatographic interferences with surrogate is present, reanalysis may not be necessary.	Apply Q-flag to all associated analytes if acceptance criteria are not met.	Alternative surrogates are recommended when there is obvious chromatographic interferences.
Results reported between DL and LOQ	NA	NA	NA	Apply J-flag to all results between DL and LOQ.	

Notes:
1. Project-specific requirements identified by the client supersede any requirements listed. The requirements are meant to be default, to be used when project-specific direction based on DQOs is not available.
2. If there is a contradiction between the method and the DoD tables, the requirements specified in the tables shall be followed.

(060-005)

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TITLE: SAMPLE PREPARATION
Toxicity Characteristic Leaching Procedure (TCLP)

Approvals (Signature/Date):

Diane L. Harper 2/11/11
Diane L. Harper — Date
Inorganics Manager

Debbie Johnson 2/11/11
Debbie Johnson — Date
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John D. Nagel 2/11/11
John D. Nagel — Date
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Terese A. Preston 2/11/11
Terese A. Preston — Date
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Michael J. Healy — Date
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Facility Distribution No: _____ **Distributed To:** Uncontrolled

COMPANY CONFIDENTIAL AND PROPRIETARY

1.0 SCOPE / APPLICATION

This Standard Operating Procedure (SOP) outlines the guidelines for the Toxicity Characteristic Leaching Procedure (TCLP). This SOP was written using 40 CFR 261 (Appendix II) and SW-846, 3rd Edition, Method 1311 as reference.

TCLP is designed to determine the mobility of both organic and inorganic contaminants present in liquid, solids and multi-phasic wastes.

On occasion, clients request slight modifications to this SOP. These modifications are addressed on a case-by-case basis with the range of accuracy (i.e., MDLs, linearity check or PT sample) verified prior to implementation. Any modifications would be written into a Quality Assurance Plan (QAP), authorized via laboratory signature approval, and mentioned in the data package's case narrative.

1.1 Method Sensitivity

1.1.1 Method Detection Limits

Not Applicable. Refer to the analytical SOPs.

1.1.2 Reporting Limits

Not Applicable. Refer to the analytical SOPs.

1.1.3 Definitions

Refer to Section 3.0 of the Laboratory's Quality Assurance Manual (UP-QA-QAM).

1.2 Summary of Method

Two distinct methods are utilized depending on whether volatile organics or other organic and metal constituents will be analyzed. A special zero-headspace extractor (ZHE) is used for volatile sample preparation and 2.0-Liter HDPE plastic or Teflon bottles are used for the other constituents.

- For solid wastes or wastes that contain significant amounts of solid material, the particle size is reduced and the liquid phase (if any) is separated from the solid phase and stored for later analysis. The solid phase is extracted with an amount of extraction fluid that is equal to 20 times the weight of the solid material.

- A portion of the extract for metals analysis only is spiked by the TCLP analyst with the analytes of concern (at the regulatory level) and acidified with nitric acid to a pH < 2 (refer to Attachment 1).

The TCLP sample is then analyzed by the appropriate method for organic and inorganic constituents. Refer to Figure 1 for the TCLP Flowchart; Table 1 for a listing of the Toxicity Characteristic Constituents and Regulatory Levels; and Table 2 for the maximum sample holding times.

2.0 INTERFERENCES

- Since this is a preparation procedure, interferences will only become apparent at the spiking and analysis stage. Interferences for spiking and for instrumentation are discussed in the analytical SOPs.
- A physical interference may occur for pH readings if the waste material is high in organic material (such as an oil). The waste may coat the pH probe, which affects the ability to obtain an accurate reading. When this type of interference occurs, pH paper is used instead of a meter for the final pH measurement. The use of pH paper is documented in the laboratory logbook.

3.0 SAFETY

Employees must abide by the policies and procedures in the Corporate Safety Manual, Radiation Safety Manual, Lab Specific Addendum to the CSM, and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coat and closed-toe, nonabsorbent shoes are a minimum.

3.1 Specific Safety Concerns or Requirements

- The standards contain potentially harmful elements. Care should be taken to avoid contact with the stock solutions. In case of contact, rinse with cold water for 15 minutes.
- If contact occurs with a standard containing Hydrofluoric Acid, flush with water and apply Calcium Gluconate Gel (located in standards cabinet) immediately. Seek medical attention.

4.0 EQUIPMENT AND SUPPLIES

- The extractor is a custom made rotary-type design that meets the specifications of tumbling the samples at a rate of 30±2 RPMs, which is checked monthly and documented in the extraction logbook.
- 2-Liter plastic bottles (HDPE for inorganics).
- 2-liter Teflon bottles [For organics (BNA, Herb/Pest)].
- pH meter and paper - pH meter accurate to ±0.05 pH units at 25°C. Refer to Appendix 3 for details on meter calibration.
- Filtering apparatus - pressure filter using compressed Nitrogen as the purge gas.
- Zero Headspace extraction vessel (ZHE) - purchased unit for volatiles.
- 9.5 mm Sieve.
- Filter paper - glass fiber, 0.7 um pore size.

NOTE: Filters shall be made of borosilicate glass fiber. When evaluating the mobility of metals, filters shall be acid-washed prior to use by rinsing with 1 N nitric acid followed by 3 consecutive rinses with deionized (DI) water (a minimum of 1 L per rinse is recommended).

NOTE: After a single use, Teflon tumbler jars and ZHEs shall be completely disassembled and all the parts washed in hot soapy water, followed by 3 rinses in tap water and three rinses in DI water, prior to re-assembly for the next sample. If there has been contamination with an oily substance, a solvent rinse may be necessary, followed by oven drying (ZHEs only) to evaporate the solvent.

- Lab balance capable of reading ± 0.01 g
- Tedlar Bags®
- ZHE Extraction Fluid Transfer Device - any device capable of transferring the extraction fluid to the ZHE without changing the nature of the extraction fluid is acceptable (e.g., a positive displacement or a peristaltic pump, a gas tight syringe, pressure filtration unit).

5.0 REAGENTS AND STANDARDS

All purchased acids, reagents, and dry chemicals used in this protocol must be ACS Reagent grade or better.

5.1 Hydrochloric Acid (HCl), 1.0 N

To a 1-L Class A volumetric flask containing ~500 mLs of DI water, carefully add 83 mLs of concentrated hydrochloric acid. Swirl the flask to mix. Dilute to volume with DI water.

- Life of Reagent: 1 year
- Storage Requirements: None

5.2 Nitric Acid (HNO_3), 1.0 N

To a 1-L Class A volumetric flask containing ~500 mLs of DI water, carefully add 64 mLs of concentrated nitric acid. Swirl the flask to mix. Dilute to volume with DI water.

- Life of Reagent: 1 year
- Storage Requirements: None

5.3 Sodium Hydroxide (NaOH), 1.0 N

To a 1-L Class A volumetric flask containing ~500 mLs of DI water, add 40.0 g of sodium hydroxide pellets. Swirl the flask to mix. This is an **EXOTHERMIC** reaction. The flask should be placed in a cool water bath when mixing. Dilute to volume with DI water.

- Life of Reagent: 1 year
- Storage Requirements: None

5.4 Glacial Acetic Acid, Reagent Grade

Purchased.

- Life of Reagent: 1 year
- Storage Requirements: None

5.5 Extraction Fluid #1

To a 1-L Class A volumetric flask containing ~500 mLs of DI water, carefully add 5.7 mLs of glacial acetic acid. Swirl the flask to mix. Then add 64.3 mLs of 1.0 N sodium hydroxide solution (Rgt. 5.3) and swirl to mix once again. Dilute to volume with DI water. The pH of this extraction fluid should be 4.93 ± 0.05.

Note: For convenience, TestAmerica Chicago typically prepares this fluid in 20 liter batches, using a 20 liter carboy, a class A graduated cylinder for adding the NaOH, and a re-pipettor to dispense the proportional amount of acetic acid.

- Life of Reagent: 1 day
- Storage Requirements: None

5.6 Extraction Fluid #2

To a 1-L Class A volumetric flask containing ~500 mLs of DI water, carefully add 5.7 mL of glacial acetic acid. Swirl the flask to mix. Dilute to volume with DI water. The pH of this Extraction Fluid should be 2.88 ± 0.05.

Note: For convenience, TestAmerica Chicago typically prepares this fluid in 20 liter batches, using a 20 liter carboy and a re-pipettor to dispense the proportional amount of acetic acid. Any volume can be prepared as long as the components are added proportionally and the pH is acceptable.

- Life of Reagent: 1 day
- Storage Requirements: None

6.0 CALIBRATION (NON-DAILY)

Not Applicable.

7.0 PROCEDURE

7.1 Quality Control Checks

Refer to Section 8.1.

7.2 Sample Preservation and Storage

Parameter	**From:** Field Collection **To:** TCLP Extraction	**From:** TCLP Extraction **To:** Preparative Extraction	**From:** Preparative Extraction **To:** Determinative Analysis	**Total Elapsed Time**
Volatiles	14 days	NA	14 days	28 days
Semi-Volatiles [1]	14 days	7 days	40 days	61 days
Mercury	28 days	NA	28 days	56 days
Metals (except Hg)	180 days	NA	180 days	360 days

[1] BNAs, Pesticides and Herbicides
NA = Not Applicable

7.3 Sample Preparation / Size

7.3.1 Inorganics & Semi-Volatiles (BNAs, Pesticides and Herbicides)

Type of Sample	Sample Size
Samples containing 100% solids	100g solid
Samples containing 0.5% - 99.9% solids	100 g solid ideally, 75.0 g solid minimum
Samples containing < 0.5% solids	Refer to Section 7.6.1.14

7.3.2 Volatiles (ZHE)

Type of Sample	Sample Size
Samples containing 100% solids	25 g solid
Samples containing 0.5% - 99.9% solids	25 g solid
Samples containing <0.5% solids	Refer to Section 7.6.2.7

7.4 Calibration / Standardization

Refer to Attachment 3 for instructions on calibrating the pH meter.

7.5 Preventive Maintenance

- The main preventive maintenance required is keeping the area and all equipment clean and free of contaminants.
- The pH probe should be checked periodically for bubbles. The probes are replaced when needed.
- The ZHE's shall be checked for leaks after every use. When a malfunctioning ZHE is discovered, that particular ZHE or part will be identified with an 'Out-of-Service' tag or equivalent and removed from service.

7.6 Sample Extraction

7.6.1 Procedure when Volatiles are Not Involved

Although a minimum sample size of 100 grams is required, a larger sample size may be necessary, depending on the percent solids of the waste sample. Enough waste sample should be collected such that at least 75 grams of the solid phase of the waste (as determined using glass fiber filter filtration) is extracted. This will ensure that there is adequate extract for the required analyses (semivolatiles, metals, pesticides and herbicides).

The determination of which extraction fluid to use (Sec. 7.6.1.12) may also be conducted at the start of this procedure. This determination shall be on the solid phase of the waste (as obtained using glass fiber filter filtration).

7.6.1.1 If the waste will obviously yield no free liquid when subjected to pressure filtration, weigh out a representative 100.0 g portion of the sample and proceed to 7.6.1.11.

7.6.1.2 If the sample is liquid that contains any visible sediment, even if logged in as a water, or if it is multi-phasic, liquid/solid separation is required. The only exception will be when a client has specifically agreed that the sample is to be analyzed as a water after a documented discussion with the laboratory.

This involves the filtration device outlined in Sections 7.6.1.3 through 7.6.1.9.

7.6.1.3 Pre-weigh the filter and the container which will receive the filtrate.

7.6.1.4 Assemble the filter holder and filter.

7.6.1.5 Weigh out a representative 100 g sub-sample of the waste and record the weight.

7.6.1.6 Allow slurries to stand to permit the solid phase to settle. Wastes that settle slowly may be centrifuged prior to filtration.

7.6.1.7 Transfer the waste sample to the filter holder.

NOTE: If waste material has obviously adhered to the container used to transfer the sample to the filtration apparatus, determine the weight of this residue and subtract it from the sample weight determined in Sec. 7.6.1.5 to determine the weight of the waste sample which will be filtered.

Gradually apply pressure of 10 psi, until gas moves through the filter. If this point is not reached under 10 psi, and if no additional liquid has passed through the filter in any two minute interval, slowly increase the pressure in 10 psi increments to a maximum of 50 psi. After each incremental increase of 10 psi, if the pressurizing gas has not moved through the filter in any two minute interval, proceed to the next 10 psi increment. When the pressurizing gas begins to move through the filter, or when liquid flow has ceased at 50 psi, filtration is stopped.

7.6.1.8 The material in the filter holder is defined as the solid phase of the waste, and the filtrate is defined as the liquid phase.

NOTE: Some wastes, such as oily wastes and some paint wastes will obviously contain some material that appears to be a liquid - but even after applying pressure filtration this material may not filter. In this case, the material within the filtration device is defined as a solid and is carried through the extraction as a solid.

7.6.1.9 Determine the weight of the liquid phase by subtracting the total weight of the filtrate container (Sec. 7.6.1.3) from the total weight of the filtrate-filled container. The liquid phase may now be either analyzed (Sec. 7.6.1.15) or stored at 4 $\pm$ 2°C until it is checked for compatibility with the rotated extract (Sec. 7.6.1.15).

The weight of the solid phase of the waste sample is determined by subtracting the weight of the liquid phase from the weight of the total waste sample, as determined in Sec. 7.6.1.5 or 7.6.1.7. Record the weight of the liquid and solid phases.

NOTE: If the weight of the solid phase of the waste is <75 g. Review the beginning of Section 7.3 about sample sizes.

7.6.1.10 The sample will be handled differently from this point, depending on whether it contains more or less than 0.5% solids. If the sample obviously has >0.5% solids, go to Sec. 7.6.1.11. If the sample is a liquid and contains any visible sediment, even if it is logged in as a water, the percent solids will be determined as follows. The only exception will be when a client has specifically agreed that the sample is to be analyzed as a water after a documented discussion with the laboratory.

- Remove the solid phase and filter from the filtration apparatus.
- Dry the filter and solid phase at 100 ± 20°C until two successive weighings yield the same value. Record the final weight.
- Calculate the percent solids as follows:

$$\frac{\text{(weight of dry waste \& filters) - (tared weight of filters)}}{\text{initial weight of waste}} \times 100 = \%\ \text{dry solids}$$

- If the dry solid phase comprises <0.5% of the waste, it is discarded and the liquid phase is defined as the TCLP extract. Proceed to Sec. 7.6.1.14.
- If the dry solid phase is ≥0.5% of the waste, return to Sec. 7.6.1.1 and begin the procedure with a new sample of waste. Do not extract the solid that has been dried.

7.6.1.11 If the sample has more than 0.5% solids, it is now evaluated for particle size. If the solid material is capable of passing through a 9.5 mm sieve, proceed to sec. 7.6.1.12. If the particle size is larger than 9.5 mm, the solid material is prepared for extraction by crushing, cutting, or grinding until it is < 9.5 mm. Note: It is not necessary to cut up filamentous material such as gloves or waste filters for all the pieces to fit through the sieve if the surface area per gram is ≥3.1 cm^2. It is not necessary or recommended to attempt to quantify this, but the analyst should use discretion.

7.6.1.12 This step describes the determination of the appropriate extracting fluid to use.

- Weigh out a small sub-sample of the solid phase of the waste, reduce the solid (if necessary) to a particle size of approximately 1 mm in diameter or less, and transfer a 5.0 g portion to a 250 mL beaker.
- Add 96.5 mL DI water, cover with watch glass, and stir vigorously for five minutes using a magnetic stirrer. Measure and record the pH. If the pH is ≤ 5.0, extraction fluid # 1 is used. Proceed to sec. 7.6.1.13.
- If the pH is >5.0, add 3.5 mL 1.0 N hydrochloric acid, stir for 30 seconds and heat to 50+/-5°C. Continue heating at 50+/-5°C for ten minutes.
- Let the solution cool to room temperature and record the pH. If pH is ≤ 5.0, use extraction fluid #1. If the pH is > 5.0, use extraction fluid #2.

7.6.1.13 Transfer the solid material into the extractor vessel, including the filter used to separate the initial liquid from the solid phase.

NOTE: If any of the solid phase remains adhered to the walls of the filter holder, or the container used to transfer the waste, its weight shall be determined, subtracted from the weight of the solid phase of the waste, as determined above, and this weight is used in calculating the amount of extraction fluid to add into the extractor bottle.

Slowly add an amount of the appropriate extraction fluid into the extractor bottle equal to 20 times the weight of the solid phase that has been placed into the extractor bottle. Close the extractor bottle tightly, and place in the rotary extractor and rotate for 18 ± 2 hours. The ambient room temperature shall be maintained at 23 ± 2°C during the extraction period. Note: If pressure builds up in the extractor bottle, especially initially, it is permissible to vent the bottle.

7.6.1.14 Following the 18 hour extraction, the material in the extractor vessel is separated into its component liquid and solid phases by filtering through a new glass fiber filter as outlined in Sec. 7.6.1.7.

7.6.1.15 The TCLP extract is now prepared as follows:

- If the waste contained no initial liquid phase, the filtered liquid material obtained from Sec. 7.6.1.14 is defined as the TCLP extract. Proceed to Sec. 7.6.1.16.

- If compatible (e.g., will not form a precipitate or has multiple phases), the filtered liquid is combined with the initial liquid phase of the waste. This combined liquid is defined as the TCLP extract.

- If the initial liquid phase of the waste, as obtained from Sec. 7.6.1.9 is not compatible with the filtered liquid resulting from Sec. 7.6.1.14, the liquids are not combined. The liquids are collectively defined as the TCLP extract and are analyzed separately. The results may be combined mathematically.

7.6.1.16 Measure and record the pH of the extracts and initiate an Internal Chain of Custody (ICOC) for those extracts which require it. TCLP extracts, accompanied by the extracted blank, are ready for transfer to metals digestion and/or organic extractions for further preparation according to the procedures for the particular analysis (organics or metals). *Note: For BP samples that require LaMP protocols, each analytical or prep group must also be supplied with a sufficient volume of extraction fluid to prepare an LCS.* If metals analysis is required, immediately remove and acidify (nitric acid to pH <2) an appropriate portion and reserve for analysis. Refrigerate the remaining aliquots at 4 ± 2°C.

7.6.2 Procedure for Volatiles by ZHE

The ZHE device has approximately a 500 mL internal capacity. Although a minimum sample size of 100 grams is required in Section 7.6.1, the ZHE can only accommodate a maximum 100% solids sample of 25 grams. This is due to the need to add an amount of extraction fluid equal to 20 times the weight of the solid phase. Sec. 7.6.2.4 provides the means by which to determine the approximate sample size for the ZHE device. Although the following procedure allows for particle size reduction during the procedure, this could result in the loss of volatile compounds. If possible, any particle size reduction (see Sec. 7.6.2.5) should be conducted on the sample as it is being taken. Particle size reduction should only be conducted during the procedure if there is no other choice.

In carrying out the following steps, do not allow the waste to be exposed to the atmosphere for any more time than is absolutely necessary.

7.6.2.1 Pre-weigh the (evacuated) container which will receive the filtrate, and set it aside.

7.6.2.2 Place the ZHE piston within the body of the ZHE (it may be helpful to first moisten the piston o-rings slightly with extraction fluid). Secure the gas inlet/outlet flange (bottom flange) onto the ZHE body in accordance with the manufacturer's instructions. Secure the glass fiber filter between the support screens and set it aside. Set liquid inlet/outlet flange (top flange) aside.

7.6.2.3 If the waste will obviously yield no free liquid when subjected to pressure filtration, weigh out a representative 25 g sample of the waste, record the weight, and proceed to Sec. 7.6.2.5.

7.6.2.4 This sec. provides the means by which to determine the approximate sample size for the ZHE device. If the waste is liquid or multi-phasic, follow the procedure outlined in Steps 7.6.1.2 to 7.6.1.9 (using the Section 7.6.1 filtration apparatus), and obtain the percent solids by dividing the weight of the solid phase of the waste by the original sample size used. If it appears that the solid may comprise <0.5% of the waste, see below.

- Determine the percent solids by using the procedure outlined in Sec. 7.6.1.10. If the waste contains <0.5% solids, proceed to Sec. 7.6.2.7 and follow until the liquid phase of the waste is filtered using the ZHE device (Sec. 7.6.2.8). This liquid filtrate is defined as the TCLP extract and is analyzed directly.

- If the sample is $\geq$ 0.5% solids, the maximum amount of sample the ZHE can accommodate is determined by dividing 25 grams by the percent solids obtained from Sec. 7.6.2.4. Weigh out a new representative sample of the determined size by the following calculation:

$$\text{weight of waste to change ZHE} = \frac{25}{\text{percent solids}} \times 100$$

7.6.2.5 After a representative sample of the waste has been weighed out and recorded, the sample is now evaluated for the particle size (see beginning of Procedure 7.6.2). If the solid material within the waste will obviously pass through a 9.5 mm sieve, proceed immediately to Sec. 7.6.2.6. If the particle size is larger than that described above, the solid material which does not meet the above criteria is separated from the liquid phase by sieving, and the solid is prepared for extraction by crushing, cutting, or grinding if possible until the particle size is < 9.5 mm. Note: It is not necessary to cut up filamentous material such as gloves or waste filters for all the pieces to fit through the sieve if the surface area per gram is $\geq$3.1 cm^2. It is not necessary or recommended to attempt to quantify this, but the analyst should use discretion.

NOTE: Wastes and appropriate equipment should be refrigerated, if possible, to 4$\pm$2°C prior to particle size reduction. If reduction of the solid phase of the waste is necessary, exposure of the waste to the atmosphere should be avoided to the furthest extent possible.

When particle size has been appropriately altered, the solid is re-combined with the rest of the waste.

7.6.2.6 Waste slurries should not be allowed to stand to permit the solid phase to settle. Wastes that settle slowly shall not be centrifuged prior to filtration. Again, this is to minimize the loss of volatile compounds to the atmosphere.

7.6.2.7 Transfer the entire sample (liquid and solid phases) quickly to the ZHE. If there is no solid/liquid separation, proceed to sec. 7.6.2.11.
Secure the filter and support screens into the top flange of the device and secure the top flange to the ZHE body in accordance with the manufacturer's instructions. Tighten all ZHE fittings and place the device in the vertical position (gas inlet/outlet flange on the bottom). Do not attach the extract collection device to the top plate.

NOTE: If waste material has obviously adhered to the container used to transfer the sample to the ZHE, determine the weight of this residue and subtract it from the sample weight determined in Sec. 7.6.2.4, to determine the weight of the waste sample which will be filtered.

Attach a gas line to the gas inlet/outlet valve (bottom flange), and with the liquid inlet/outlet valve (top flange) open, begin applying gentle pressure of 1-10 psi (more if necessary) to slowly force all headspace out of the ZHE device.

At the first appearance of liquid from the liquid inlet/outlet valve, quickly close the valve and discontinue pressure.

7.6.2.8 Attach the evacuated pre-weighed filtrate collection container to the liquid inlet/outlet valve and open valve. Begin applying gentle pressure of 1 - 10 psi to force the liquid phase into the filtrate collection container. If no additional liquid has passed through the filter in any two-minute interval, slowly increase the pressure in 10 psi increments to a maximum of 50 psi.

After each incremental increase of 10 psi, if no additional liquid has passed through the filter in any two-minute interval, proceed to the next 10 psi increment. When liquid flow has ceased, such that continued pressure filtration at 50 psi does not result in any additional filtrate within any two-minute period, filtration is stopped. Close the liquid inlet/outlet valve, discontinue pressure to the piston, and disconnect the filtrate collection container.

NOTE: Instantaneous application of high pressure can degrade the glass fiber filter and may cause premature plugging.

7.6.2.9 The material in the ZHE is defined as the solid phase of the waste, and the filtrate is defined as the liquid phase.

NOTE: Some wastes, such as oily wastes and some paint wastes, will obviously contain some material which appears to be a liquid - but even after applying pressure filtration this material will not filter. If this is the case, the material within the filtration device is defined as a solid, and is carried through the TCLP extraction as a solid.

If the original waste contained <0.5% solids (see Sec. 7.6.2.4) this filtrate is defined as the TCLP extract, and is analyzed directly - proceed to Sec. 7.6.2.13.

7.6.2.10 Determine the weight of the liquid phase by subtracting the weight of the filtrate container (see Sec. 7.6.2.1) from the total weight of the filtrate-filled container. The liquid phase may now be either analyzed or stored at 4 $\pm$ 2°C until time of analysis. The weight of the solid phase of the waste sample is determined by subtracting the weight of the liquid phase from the weight of the total waste sample (see Sec. 7.6.2.4). Record the final weight of the liquid and solid phases.

7.6.2.11 The following details how to add the appropriate amount of extraction fluid to the solid material within the ZHE and agitation of the ZHE vessel.

Extraction fluid #1 is used in all cases.

- With the ZHE in the vertical position, attach a connector for the extraction fluid syringe to the liquid inlet/outlet valve. Release gas pressure on the ZHE piston (from the gas inlet/outlet valve), open the liquid inlet/outlet valve, and begin transferring extraction fluid into the ZHE. Apply pressure to the plunger to add extraction fluid into the ZHE until the amount of fluid introduced into the device equals 20 times the weight of the solid phase of the waste that is in the ZHE.

- After the extraction fluid has been added, immediately close the liquid inlet/outlet valve and disconnect the syringe & connector. Check the ZHE to make sure that all valves are in their closed positions. Pick up the ZHE and physically rotate the device in an end-over-end fashion two or three times. Reposition the ZHE in the vertical position with the liquid inlet/outlet valve on top.

  Put 5-10 psi behind the piston and slowly open the liquid inlet/outlet valve to bleed out any headspace (into a hood) that may have been introduced due to the addition of extraction fluid. This is a check to show that the piston moves under 15 psi and that the o-rings are ok. This bleeding shall be done quickly and shall be stopped at the first appearance of liquid from the valve. Re-pressurize the ZHE with 10 ± 5 psi and check all ZHE fittings to ensure that they are closed. Document the pressure in the TCLP logbook.
- Place the ZHE in the rotary extractor apparatus and rotate the ZHE for 18 ± 2 hours. The temperature of the room shall be maintained at 23 ± 2°C during agitation.

7.6.2.12 Following the 18 hour extraction, check the pressure behind the ZHE piston by looking at the gas pressure gauge. If the pressure has not been maintained (i.e., no gas release is observed or the pressure is not within 5 psi of the initial pressure) the device is leaking. Replace ZHE o-rings or other fittings, as necessary, and re-do the extraction with a new sample of waste. The original extract can not be used. If the pressure within the device has been maintained and the final pressure is within 5 psi of the initial pressure, the material in the extractor vessel is once again separated into its component liquid and solid phases. If the waste contained an initial liquid phase, the liquid may be filtered directly into the same filtrate collection container holding the initial liquid phase of the waste, unless doing so would create multiple phases, or unless there is not enough volume left within the filtrate collection container. A separate filtrate collection container must be used in these cases. Filter through the glass fiber filter, using the ZHE device as discussed in Sec. 7.6.2.8.

7.6.2.13 If the waste contained no initial liquid phase, the filtered liquid material obtained from Sec. 7.6.2.12 is defined as the TCLP extract. If the waste contained an initial liquid phase the filtered liquid material obtained from Sec. 7.6.2.12 and the initial liquid phase (Sec. 7.6.2.8) are collectively defined as the TCLP extract.

7.6.2.14 Extracts are then transferred to GC/MS Volatiles and stored in the GC/MS Volatiles cooler until analysis. Internal Chain of Custody (ICOC) procedures will be initiated for those extracts which require it.

7.7 Documentation

7.7.1 Analysis Logbook

Important extraction information including dates, temperatures, filter lot numbers, container identification numbers, etc., are recorded in the TCLP extraction logbook and completed for each day's analysis (see Attachment 2).

8.0 QUALITY CONTROL

NOTE: All quality control measures described in the appropriate analytical methods shall be followed.

8.1 QC Summary

8.1.1 A minimum of one blank (using the same extraction fluid as used for the samples) must be analyzed for every 20 extractions that have been conducted in an extraction vessel. The extraction fluid is to be made up daily and the pH determined and recorded within the acceptable limits.

8.1.2 A blank extraction fluid must be prepared for each type of fluid used per batch. If both extraction fluids are used, two blanks must be analyzed. The blank for the volatile analysis is the ZHE vessel filled with the extraction fluid and run through the procedure.

8.1.3 A matrix spike shall be performed for each waste type (e.g. wastewater treatment sludge, contaminated soil, etc.) unless the result exceeds the regulatory level and the data is being used solely to demonstrate that the waste property exceeds the regulatory level. A minimum of one matrix spike must be analyzed for each analytical batch. At a minimum, follow the matrix spike addition guidance provided in each analytical method.

8.1.4 Matrix spikes are to be added after filtration of the TCLP extract and before preservation. Matrix spikes should not be added prior to TCLP extraction of the sample.

8.1.5 In most cases, matrix spikes should be added at a concentration equivalent to the corresponding regulatory level. If the analyte concentration is less than one half the regulatory level, the spike concentration may be as low as one half of the analyte concentration, but may not be less than 5x the method detection limit. In order to avoid differences in matrix effects, the matrix spikes must be added to the same nominal volume of TCLP extract as that which was analyzed for the unspiked sample.

8.1.6 The purpose of the matrix spike is to monitor the performance of the analytical methods used, and to determine whether matrix interferences exist. Use of other internal calibration methods, modification of the analytical methods, or use of alternate analytical methods may be needed to accurately measure the analyte concentration of the TCLP extract when the recovery of the matrix spike is below the expected analytical method performance.

8.2 Corrective Action

Since this is a preparation step, problems will not be known until the filtrates are analyzed. Corrective action for poor blank results will require all samples in the set to be re-prepared. Refer to the analytical SOPs for corrective actions.

9.0 Data Analysis and Calculations

Since this is a preparatory procedure, refer to the analytical SOPs for matrix and method QC calculations.

9.1 Multiphasic Wastes with Non-compatible Liquid Phases

Determine the volume of the individual phases, analyze as appropriate, and combine the results mathematically by using a volume weighted average:

$$\text{Final Analyte Conc.} = \frac{(V_1)(C_1) + (V_2)(C_2)}{V_1 + V_2}$$

Where:
V_1 = Volume in first phase (L)
V_2 = Volume in second phase (L)
C_1 = Conc. in first phase (mg/L)
C_2 = Conc. in second phase (mg/L)

10.0 POLLUTION CONTROL

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

10.1 Waste Streams Produced by the Method

Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to UP-WM-001.

Waste from this procedure will enter the "Waste Water" waste stream.

The following waste streams are produced when this method is carried out.

- Aqueous waste from the extraction step will be turned into the waste technician after the analysis has been completed on digestate. The concentration of, if present, heavy metals will dictate the disposal procedure.
- Aqueous waste that has heavy metal levels below regulatory levels will be turned into the waste technician for disposal in the "Waste Water" waste stream.
- Aqueous waste that has heavy metal levels above regulatory limits should be marked appropriately and turned into the waste technician for disposal into the "Heavy Metal Corrosive Waste Water" waste stream.

11.0 METHOD PERFORMANCE CRITERIA

Refer to section 1, 6, 7 and 8.

12.0 REFERENCES

Refer to Section 1.0

13.0 ATTACHMENTS

Figure 1: TCLP Flowchart
Table 1: TCLP Constituents and Regulatory Levels
Attachment 1: TCLP Metals Spiking
Attachment 2: TCLP Extraction Log
Attachment 3: pH Calibration Work Instruction

14.0 REVISION HISTORY

- Revision 16, was updated on 02/11/11
- Annual Review
- Added notes to sections 5.5 and 5.6 providing for extraction fluids to be prepared in larger volume.
- Added text to 5.0 requiring ACS Reagent grade reagents.
- Revised Attachment 3 to address new pH meter.

Figure 1.

TCLP Flowchart

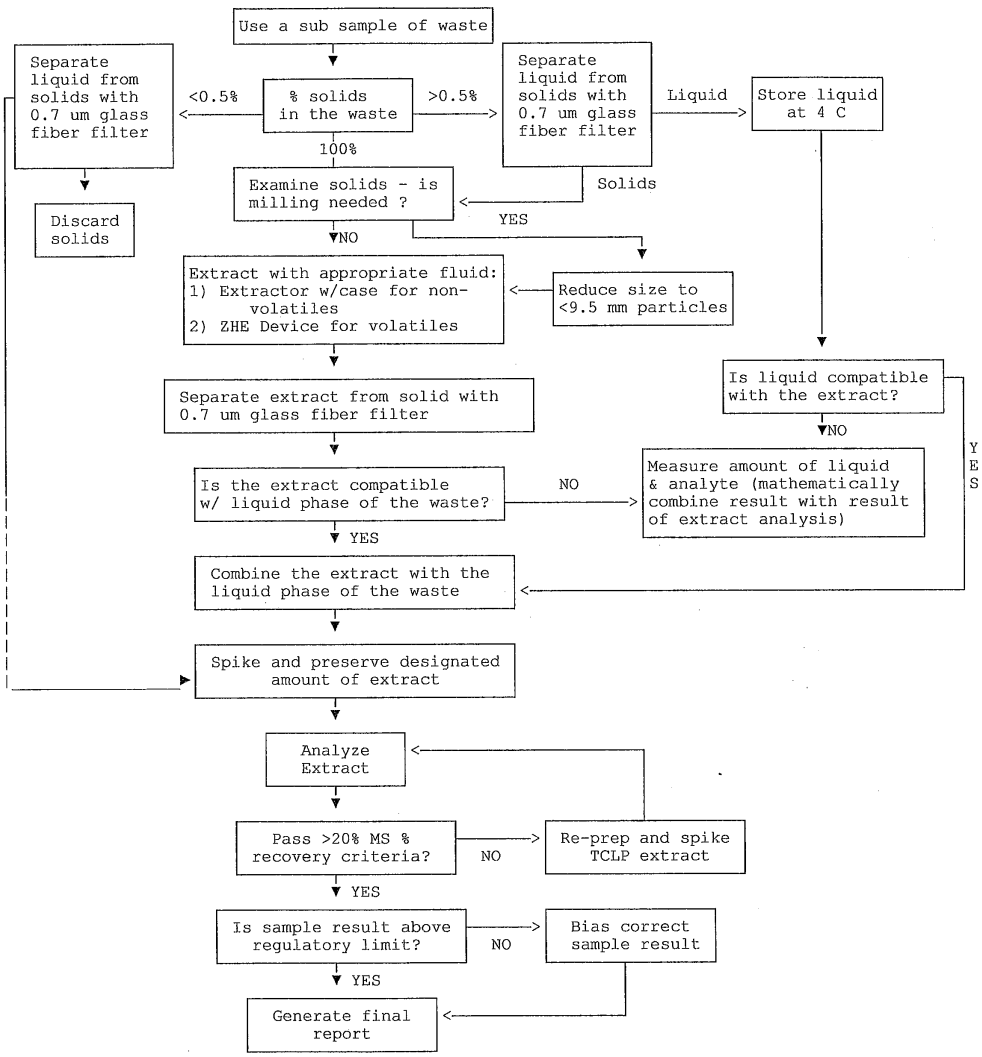

Table 1.

TCLP Constituents and Regulatory Levels

EPA HW Number	Constituent	CAS No.	Regulatory Level (ug/L)
D004	Arsenic	7740-38-2	5,000
D005	Barium	7440-39-3	100,000
D018	Benzene	71-43-2	500
D006	Cadmium	7440-43-9	1,000
D019	Carbon Tetrachloride	56-23-5	500
D020	Chlordane	57-74-9	30
D021	Chlorobenzene	108-90-7	100,000
D022	Chloroform	67-66-3	6,000
D007	Chromium	7440-47-3	5,000
D023	o-Cresol	95-48-7	[1]200,000
D024	m-Cresol	108-39-4	[1]200,000
D025	p-Cresol	108-44-5	[1]200,000
D026	Cresol		[1]200,000
D016	2,4-D	94-75-7	10,000
D027	1,4-Dichlorobenzene	106-46-7	7,500
D028	1,2-Dichloroethane	107-06-2	500
D029	1,1-Dichloroethylene	75-35-4	700
D030	2,4-Dinitrotoluene	121-14-2	130
D012	Endrin	72-20-8	20
D013	Heptachlor (& its epoxides)	76-44-8	8
D032	Hexachlorobenzene	118-74-1	130
D033	Hexachloro-1,3-butadiene	87-68-3	500
D034	Hexachloroethane	67-72-1	3,000
D008	Lead	7439-92-1	5,000
D013	Lindane	58-89-9	400
D004	Mercury	7439-97-6	200
D014	Methoxychlor	72-43-5	10,000
D035	Methyl Ethyl Ketone (2-Butanone)	78-93-3	200,000
D036	Nitrobenzene	98-95-3	2,000
D037	Pentachlorophenol	87-86-5	100,000
D038	Pyridine	110-86-1	5,000
D010	Selenium	7782-49-2	1,000
D011	Silver	7740-22-4	5,000
D039	Tetrachloroethylene	127-18-4	700
D015	Toxaphene	9001-35-2	500
D040	Trichloroethylene	79-01-6	500
D041	2,4,5-Trichlorophenol	95-95-4	400,000
D042	2,4,6-Trichlorophenol	88-06-2	2,000
D017	2,4,5-TP (Silvex)	93-72-1	1,000
D043	Vinyl Chloride	75-01-4	200

[1] If o-, m-, p-cresol concentration cannot be differentiated, the total cresol (D026) concentration is used. The regulatory level for total cresol is 200, 000 ug/L.

Attachment 1.

TCLP Metals Spiking

The purpose of the matrix spike is to monitor the performance of the analytical methods used and to determine whether matrix interferences exist.

Matrix spikes are to be added after filtration of the TCLP extract and before preservation. Matrix spikes should not be added prior to the TCLP extraction of the sample.

In order to avoid differences in matrix effects, the matrix spikes must be added to the same nominal volume of the TCLP extract as that which was analyzed for the unspiked sample.

The following steps detail the TCLP metals spiking procedure:

- Measure out 100 mLs of TCLP extract and transfer it into a small container.
- Using an eppendorf pipet, dispense 1 mL of each standard, STL-TCLP-1A and TCLP-2 (a.k.a., CGBA 10-A), into the TCLP extract.
- Preserve the TCLP spiked extract with 2 mLs of concentrated nitric acid.
- Store at 4 $\pm$ 2°C.

NOTE:

TCLP Stock Spike Solution Concentration:
Ba = 1000 ppm; As, Cr, Pb = 500 ppm; Cd, Se, Ag = 100 ppm.

Element Concentrations in Spiked Samples:
Ba = 100 ppm; As, Cr, Pb = 5 ppm; Cd, Se, Ag = 1 ppm

Attachment 2.

Example: TCLP Extraction Logbook

TestAmerica Chicago
TCLP Extraction Logbook

Page Number:_____

Rotator RPM : 30 ± 2 RPMs
Group Number:_____
LIMS Batch No.:_____
Sample Size Specifications:____<9.5 mm__
Sample Type (Circle): TCLV SPLP TCLP ASTM

Extraction Start Date / Time / Temp: _____/_____/_____ °C
Extraction End Date / Time / Temp: _____/_____/_____ °C
Min./Max.Temp: _____/_____ °C
Control Limits: 10 ± 5 PSI; 23 ± 2 °C

Filtration Start Time: _____
Filtration End Time: _____
Filter Paper Lot #: _____
Thermometer ID: _____

Sample Number					
Sample Description					
Sample Weight (g)					
Liquid-Solid Separation (Yes/No)					
Volume of Mother Liquid (mLs)					
Solid Extraction Material (g)					
Extraction Fluid Selection pH of Initial Solution: If <5.0, use Extraction Fluid #1					
pH of Acid/Heat Treated Solution: If <5.0, use Extraction Fluid #1 If >5.0, use Extraction Fluid #2					
Extraction Fluid Type (1 or 2)					
Extraction Vessel Type / Pressure Check					
Extraction Fluid Volume (mLs)					
Extract Filtered (Yes or No)					
Mother Liquid Added (mLs)					
Combined Filtrate Volume (mLs)					
Final pH Reading					
Spike Source ID # / Volume Added (mLs)					
Filtrate Preserved					
ZHE: Initial psi / Final psi					

Comments:_____

Extraction Vessel Codes: T = Teflon Organics/Metals; ZHE = Zero Headspace VOA's; HDPE = High Density Polyethylene Metals

Analyst: _____ Date:_____

Reviewer:_____ Date:_____

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(C-2) C-476

Attachment 3.

Example: pH Calibration Work Instruction

Calibration Instructions

Hanna Instruments HI-98183

C-2380

The meter must be re-calibrated daily prior to use.

1. Bring buffers pH 4.0, 7.0 and 10.0 to room temperature and pour fresh daily.
2. Immerse the electrode and ATC to a depth of ~1.5" in the pH 7.0 buffer and press the CAL key. The measured pH, the first expected buffer value, 7.0, and the temperature are displayed. Use the arrow keys to change the expected buffer if necessary.
3. Stir gently. Wait until the flashing hourglass symbol on the LCD screen stops flashing, indicating the reading is stable, and then press the CMF key to confirm the first point.
4. The calibrated value and the 2nd expected buffer value of 4.0 should now show on the screen. Again use the arrow keys if necessary to change buffer.
5. Rinse the probes with DI water, then immerse them ~1.5 "in the pH 4.0 buffer, again pressing the CAL key and waiting until the hourglass stops flashing.
6. Press CMF to confirm the 2nd buffer.
7. The calibrated value and the 3rd expected buffer value of 10.0 should now show on the screen.
8. Rinse the electrode again and continue with the pH 10.0 buffer, pressing CFM when the reading is stable.
9. Press the CAL or ESC to exit the calibration mode. The calibration will automatically be stored.
10. Verify calibration by reading back the three buffers, and bracket every 10 sample readings with an alternate-source pH 7.0 QC buffer.

Note: An error will show on the screen if there is a problem with the calibration. For additional information, more detailed instructions, and a trouble-shooting guide, see the Instruction Manual C-2380 stored near the pH meter.

CHI-22-15-022/B-02/11

TITLE: SAMPLE PREPARATION
Metals Digestion by SW-846 3000 Series

Approvals (Signature/Date):

Debbie Johnson 3/1/11
Debbie Johnson Date
Supervisor, Metals

John D. Nagel 3/1/11
John D. Nagel Date
Env. Health & Safety Coordinator

Terese A. Preston 3/1/11
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Quality Assurance Manager

Michael J. Healy 3/1/11
Michael J. Healy Date
Laboratory Director

Facility Distribution No: _____ **Distributed To:** Uncontrolled

1.0 SCOPE / APPLICATION

This Standard Operating Procedure (SOP) outlines the guidelines for the preparation of wastewaters, extracts, wastes and soil samples for metals analysis by Trace Inductively Couples Argon Plasma (ICP) and Inductively Coupled Argon Plasma / Mass Spectrometry (ICP-MS). This SOP was written using the following methods of SW-846, Third Edition:

Method	Description
3005A	Surface and ground waters for analysis by Trace ICP, ICP-MS
3010A	Waters and extracts for analysis by Trace ICP
3030C	See note below.
3050B	Soil and waste samples for analysis by Trace ICP, ICP-MS
E 821/R-91-100	Acid Volatile Sulfide and Simultaneously Extracted Metals

On occasion, clients request slight modifications to this SOP. These modifications are addressed on a case-by-case basis with the range of accuracy (i.e., MDLs, linearity check or PT sample) verified prior to implementation. Any modifications would be written into a Quality Assurance Plan (QAP), authorized via laboratory signature approval, and mentioned in the data package's case narrative.

Note: The state of North Carolina requires the use of Standard Methods 3030C for the preparation of wastewater samples.

1.1 Method Sensitivity

1.1.1 Method Detection Limits

Not Applicable. Refer to the analytical SOPs.

1.1.2 Reporting Limits

Not Applicable. Refer to the analytical SOPs.

1.1.3 Definitions

Refer to Section 3.0 of the Laboratory's Quality Assurance Manual (UP-QA_QAM).

1.2 Summary of Method

Water and soil samples are digested with nitric acid, hydrochloric acid and/or hydrogen peroxide to produce digestates that are in the correct acid media for analysis by the Trace ICP or ICP-MS.

2.0 INTERFERENCES

Matrix interferences are usually not present for the digestion process. Analytical matrix interferences may be apparent during the instrumental analysis of the digestates. The types of interferences for the instruments are discussed in the appropriate SOPs.

3.0 SAFETY

Employees must abide by the policies and procedures in the Corporate Safety Manual, Radiation Safety Manual, Lab Specific Addendum to the CSM, and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coat and closed-toe, nonabsorbent shoes are a minimum.

3.1 Specific Safety Concerns or Requirements

- Samples that contain high concentrations of carbonates or organic material or samples that are at elevated pH can react violently when acids are added.
- Acid vapor can be dangerous. Work in a well ventilated area (i.e., fume hood).
- Hydrogen peroxide (H_2O_2) is a strong oxidizer and is corrosive. The digestion must be cooled sufficiently before the addition of H_2O_2 to avoid a reaction and possible violent effervescence, or boiling over of the digestion. A splash/splatter hazard is possible and a face shield should be worn

3.2 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **NOTE:** This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table. A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Hydrochloric Acid	Corrosive Poison	5 ppm-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Hydrogen Peroxide	Oxidizer Corrosive	1 ppm-TWA	Vapors are corrosive and irritating to the respiratory tract. Vapors are very corrosive and irritating to the eyes and skin.
Nitric Acid	Corrosive Oxidizer Poison	2 ppm-TWA 4 ppm-STEL	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.

1 – Always add acid to water to prevent violent reactions.
2 – Exposure limit refers to the OSHA regulatory exposure limit.

4.0 EQUIPMENT AND SUPPLIES

- Top loading balance
- Hot plate (w/ thermometer)
- Hot Block w/ digestion vessels (w/ thermometer)
- 250 mL beakers
- 100 mL graduated cylinders
- Whatman No. 541 filter paper
- Funnels
- Fume hood(s)
- Eppendorf Pipettes
- Watch glasses (ribbed & non-ribbed)
- Filters and plunger apparatus[1]
- 100 & 50 mL digestate vessels[1] (which are checked to ensure volume markings are within 2.5% Tolerance).
- 100 mL Snap-Cap containers for digestates (which are checked to ensure volume markings are within 2.5% Tolerance).

[1]Each time the metals digestions staff opens a new case/box of filters or digestion tubes, the certificate must be removed and the lot number compared with previous lot numbers. If the case/box has a new lot number, the filters or digestion tubes must be verified as free of metals contamination to the lowest reporting level by in-house testing. This will be accomplished as follows:

1. Prep analyst identifies new lot and documents the prep batch number for the first ICP and ICPMS MB created on the vendor supplied quality certificate.
2. The lot number will be documented in the comments section of the LIMs prep batch.
3. The certificate will be forwarded to the instrument analyst attached to the metals digestion sheet.
4. After the analysis of the MB, the raw data will be reviewed to determine if the acceptance criteria of results falling below ½ the current RL are met.
5. If the acceptance criteria are met, the analyst or supervisor will initial and date the certificate to indicate approval and document the analysis run and batch where the results may be found.
6. If the criteria are not met, the analyst must notify their supervisor immediately who will then initiate corrective action measures.
7. The certificate will be maintained on file in the Metals Department.

5.0 REAGENTS AND STANDARDS

5.1 Reagents

- Concentrated Nitric Acid (Instra Pure)
- Concentrated Hydrochloric Acid (Instra Pure)
- 30% Hydrogen Peroxide Solution

Purchased from a chemical vendor.

- Life of Reagent: Specified by the Manufacturer, usually 1 year.
- Storage Requirements: Acid Cabinet

5.2 Standards

5.2.1 ICP and ICP-MS Intermediate Standards

These standards are prepared from multi-element solutions purchased from vendors. Single element spikes may be used if needed. These solutions expire 1-year from the date of receipt.

Standard	Preparation
ICP Spike Solution ICP-MS Spike Solution	• Add ~400 mLs of Milli-Q water to a 1-L Class A volumetric flask. • Add 100 mLs each of HP1381-A-500, HP1381-B-500 and HP1381-C-500 (ICP); HP2930-A-500, HP2930-B-500, HP2930-C-500 (ICP-MS) • Add 9 mLs of 1,000 ppm Se; • Add 8 mLs of 1,000 ppm Pb; • Add 6 mLs of 1,000 ppm As; • Add 5 mLs of 1,000 ppm Tl; and • Add 40 mLs of InstraPure nitric acid. • Swirl to mix; Dilute to volume with Milli-Q water. Life of Standard: Expiration date of the earliest expiring standard. Storage Requirements: None.

Refer to Appendix A for the individual element concentrations within the spiking solutions. Matrix spikes for TCLP extracts are added after filtration of the TCLP extract and before preservation. Refer to SOP No. UP-SP-1311.

6.0 CALIBRATION (NON-DAILY)

Not Applicable.

7.0 PROCEDURE

7.1 Quality Control Checks

QC Indicator	Preparation	Frequency
Method Blank (MB)	For soil sample batches, use 5 - 10 mLs of Milli-Q water. [1]	1 per 20 or fewer samples.
	For water sample batches, use 50 mLs of Milli-Q water.	1 per 20 or fewer samples.
Matrix Duplicate (DU) [2]	Aliquot of the same field sample that is digested independently.	1 per 20 or fewer samples.
Laboratory Control Sample (LCS) [3]	For soil sample batches, use 5 - 10 mLs of Milli-Q water and spike as listed below. [1 4]	1 per 20 or fewer samples.
	For water sample batches, use 50 mLs of Milli-Q water and spike as listed below. [4]	1 per 20 or fewer samples.
Matrix Spike (MS); MS Duplicate (MSD) [2]	Aliquot of the same field sample that is spiked as listed below [3] and digested independently.	1 per 20 or fewer samples.

[1] For those programs, clients, and projects mandating the use of a 'soil' matrix for the preparation of Soil MB's and LCS's (including all DoD, AFCEE and NFESC projects), Teflon Strands will be incorporated.
[2] The sample selection for MS/MSD/DU is rotated among client samples so that various matrix problems may be noted and/or addressed.
[3] LCS Duplicate (LCSD) is performed when requested by the client, contract or QAP.
[4] The LCS and MS/MSD are spiked with a known amount of analyte and processed through the digestion procedure. The spiking procedure is as follows:

Instrument	Waters Spike Volume	Soils Spike Volume
Trace ICP / ICP-MS	0.5 mL of ICP/ICP-MS Intermediate Spiking Solution.	1 mL of ICP/ICP-MS Intermediate Spiking Solution.

Refer to Appendix A for the individual element concentrations within the spiking solutions. Matrix spikes for TCLP extracts are added after filtration of the TCLP extract and before preservation. Refer to UP-SP-1311.

7.2 Sample Preservation and Storage

Matrix	Holding Time	Preservation
Waters	180 days	HNO_3, pH <2; Cool 4 $\pm$ 2°C
Soils	180 days	Cool 4 $\pm$ 2°C

Note: If the metals water samples are not preserved, they will be preserved in login and a sticker will be placed over the lid of the sample bottle. This pre-printed sticker will document "Sample preserved in login, do not analyze for 24 hours".

7.2.1 Sample Handling Procedures (Other than Soils / Waters)

Matrix	Description
Wipes	The entire wipe is digested with results reported as ug/wipe.
Paint Chips	Care is taken to remove the paint from the substrate. The chips are then cut and ground to provide a uniform matrix from which to take a sample aliquot. Sample size is approximately 1.0 grams.
Solids *	Dried and ground with a mechanical crusher.

*Bricks, wood, etc.

7.3 Sample Preparation

- Since the pH is checked by the sample custodian at sample receipt, the digestion analysis will check the pH at random and/or if the analyst has a reason to suspect that the sample may not be preserved.
- The start and end temperature of the hot plate or hot block digestion is documented in TALS.

NOTE: The LCS and MB must be filtered when analyzed with dissolved metals that are filtered in the laboratory (unpreserved samples).

7.4 Calibration / Standardization

Not Applicable.

7.5 Preventive Maintenance

- To minimize contamination during sample preparation, the fume hoods and counter areas must be kept clean and free of dust.
- The digestion hoods are cleaned on a regular basis (a minimum of once a month) and documented within the hood maintenance log.

7.6 Sample Digestion

7.6.1 Method 3005A

- Transfer 50 mLs of the well-mixed (homogenized) sample into a 50 mL digestion vessel.
- Add 1 mL of InstraPure nitric acid and 2.5 mLs of InstraPure hydrochloric acid.
- Cover the vessel with a ribbed watch glass and heat on a preheated hot block at 90-95°C until the volume has been reduced to 10-15 mLs. Do not allow the samples to boil.
- Remove the vessels from the hot block and allow to cool.
- Fill to a 50 mL final volume in the digestion vessel with Milli-Q water and filter using the plunger apparatus.
- The sample is now ready for analysis.

NOTE: When using the Hot Plates, all volumes remain the same in the 250 mL beaker. Transfer the digestate to a digestion vessel, washing down the sides of the beaker with Milli-Q water as needed. Dilute the sample to a 50 mL final volume using Milli-Q water and filter using the plunger apparatus.

7.6.1.1 Method 3005A-Modified ICP-MS

- Transfer 50 mLs of the well-mixed (homogenized) sample into a 50 mL digestion vessel.
- Add 1 mL of InstraPure nitric acid and ***0.5 mLs**** of InstraPure hydrochloric acid.
- Cover the vessel with a ribbed watch glass and heat on a preheated hot block at 90-95°C until the volume has been reduced to 10-15 mLs.
- Remove the vessels from the hot block and allow to cool.
- Fill to a 50 mL final volume in the digestion vessel with Milli-Q water and filter using the plunger apparatus.
- The sample is now ready for analysis.

***NOTE:** Method 3005A was modified for ICP-MS to matrix match the preparation with the analytical standards. In this regard, a volume of 0.5 mL of InstraPure hydrochloric acid is added to the digestion vessel in place of 2.5 mLs. Hydrochloric acid is a known interferent on the ICP-MS.

7.6.2 Method 3010A

- Transfer 50 mLs of the well-mixed (homogenized) sample into a 50 mL digestion vessel.
- Add 1.5 mLs of InstraPure nitric acid.
- Cover the vessel with a ribbed watch glass and place on a preheated hot block set at 90-95°C.
- Evaporate the sample down to a low volume (approximately 20 mLs) – just enough to cover the bottom of the vessel. **The sample should not boil or evaporate completely on any portion of the bottom of the vessel. If this should happen, a re-digestion must be done.**
- Remove the vessel from the hot block and allow to cool.
- Add another 1.5 mLs portion of InstraPure nitric acid.

- Cover the vessel with a non-ribbed watch glass and return to the hot block to allow a gentle reflux to occur.
- Continue to add InstraPure nitric acid as necessary, until the digestion is complete (no change in appearance with continued refluxing).
- Uncover and evaporate to a low volume, not allowing any part of the vessel to go dry.
- Remove the vessels from the hot block and allow to cool.
- Add 2.5 mLs of InstraPure hydrochloric acid.
- Warm the vessel for another 15 minutes to dissolve any precipitate.
- Remove from hot block and allow to cool.
- Fill to a 50 mL final volume in the digestion vessel with Milli-Q water and filter using plunger apparatus.
- The sample is now ready for analysis.

NOTE: When using the Hot Plate, the volume remains the same in a 250 mL beaker. Transfer the digestate to a digestion vessel, washing down the sides of the beaker with Milli-Q water as needed. Dilute the sample to a 50 mL final volume using Milli-Q water and filter using the plunger apparatus.

7.6.3 Method 3030C

- Transfer 50 mLs of well mixed (homogenized) sample into a digestion vessel.
- Add 1.25 mLs of InstraPure hydrochloric acid.
- Heat 15 minutes on hot block
- Cool sample
- Filter through a 0.45 um membrane filter.
- Bring sample up to volume of 50 mL and mix.
- Sample is now ready for analysis.

7.6.4 Method 3050B

- Weigh out a portion of homogenized sample, generally 1.0 – 1.2 grams, into a 100 mL vial. The exact weight is recorded in the TALS digestion spreadsheet. More sample weight may be used if the liquid content is high, as long as the digestion is complete.

NOTE: When using the hot plate/beaker combination, soils are generally weighed to 1.00-2.00 grams. All other volumes are the same as for digestions.

- Add 5 mLs of InstraPure nitric acid and 5 mLs of Milli-Q water.
- Cover the vial with a non-ribbed watch glass and place on a preheated hotplate set at 90-95°C for 15 minutes without boiling.
- Remove the vial from the hot block and allow to cool.
- Add 5 mLs of InstraPure nitric acid and reflux for 30 minutes with a non-ribbed watch glass.
- If brown fumes are generated, repeat this last step until no brown fumes are generated indicating complete reaction with the nitric acid.
- Allow the solution to evaporate to a low volume – just enough to cover the bottom of the vial. **Do not allow the sample to boil.**
- Remove the vial from the hot block and allow to cool.
- Add 2 mLs of Milli-Q water and 3 mLs of 30% hydrogen peroxide.
- Cover the vial with a non-ribbed watch glass and heat until the reaction is complete.
- Remove the vial from the hot block and allow to cool.

- Continue to add 30% hydrogen peroxide in 1 mL aliquots with warming until the effervescence is minimal or until the general sample appearance in unchanged. **Do not add more than a total of 10 mLs of hydrogen peroxide.**
- Cover the sample with a ribbed watch glass and heat until the volume has been reduced to ~5mLs or heat at 90-95°C for 2-hours without boiling.
- Maintain a covering of solution on the bottom of the vial (or beaker) at all times.

If the sample is being analyzed by ICP:

- Allow the sample to cool.
- Add 10 mLs of InstraPure hydrochloric acid.
- Place the vial on the hot block and heat for 15 minutes without boiling.
- Remove the vial from the hot block and allow to cool.
- Wash down the sides of the vial with Milli-Q water and filter into a 100 mL snap-cap container through Whatman 541 filter paper.
- Dilute the sample to the 100 mL mark in a snap-cap container.
- The sample is now ready for analysis.

If the sample is being analyzed by the ICP-MS:

- Allow the sample to cool.
- Add ***1.0 mL**** of InstraPure hydrochloric acid.
- Place the vial on the hot block and heat for 15 minutes without boiling.
- Remove the vial from the hot block and allow to cool.
- Wash down the sides of the vial with Milli-Q water and filter into a 100 mL snap-cap container through Whatman 541 filter paper.
- Dilute the sample to the 100 mL mark in a snap-cap container.
- The sample is now ready for analysis.

***NOTE:** Method 3050B was modified for ICP-MS to matrix match the preparation with the analytical standards. In this regard, a volume of 1.0 mL of InstraPure hydrochloric acid is added to the digestion vessel in place of 10 mL. Hydrochloric acid is a known interferent on the ICP-MS.

7.6.5 AVS-SEM

The SEM extracts are prepared and spiked in the wet chemistry section using 2.5 mL of the Trace (3010) spiking solution, and when requested, 2.5 mL of the 100 ug/L mercury stock, both provided by the metals digestion section. Upon receipt from Wet Chemistry, the extracts still include the solid fraction, and will need to be filtered and brought to 250 mL. The extracts are then ready for the instruments, as no digestion is required.

7.7 Documentation

7.7.1 TALS Digestion Spreadsheets

Sample digestion and standard traceability are documented within the TALS ADII spreadsheets. The spreadsheets must be completed for each day's work. The following information must be recorded in the Batch Notes when applicable: Filter Lot #, HCl Lot #, HNO_3 Lot #, H_2O_2 Lot #, Hot Block ID # and Temperature, Thermometer ID #, Start Time, and Digestion Tube Lot #. Refer to Appendix B for examples of digestion spreadsheets.

7.7.2 Traceability of Standards

Custom made and single element stock standard solutions which are traceable to NIST or EPA are purchased. Upon receipt, each standard is entered into the TALs LIMS database and is issued unique source ID #. The manufacturer, lot #, date received, expiration date, and the initials of the analyst are also entered.

8.0 QUALITY CONTROL

8.1 QC Summary

QC Standard	Indicator
Method Blank (MB)	Examined to determine if there was any contamination introduced during the digestion process.
Laboratory Control Sample (LCS)	Used to determine the completeness of the digestion process. The accuracy is measured by the percent recovery (%R) of each standard.
Matrix Duplicate (DU)	Demonstrate analytical precision and is reported as Relative Percent Difference (RPD).
Matrix Spike (MS) / MS Duplicate (MSD)	Used to demonstrate analytical accuracy and is reported as % recovery.

8.2 Corrective Action

A Non-Conformance Memo (NCM) will be initiated by the analyst in TALS anytime there is a deviation from the routine preparation procedures, as outlined within this SOP. This includes, but is not limited to: limited sample volume, sample loss due to spills, extremely vigorous reactions, spiking issues or any other issues that may occur during the preparatory procedure. The Section Manager will review the NCM and document any corrective action needed at that time. All analytical 'out-of-control' situations are identified as indicated in the supporting analytical SOP's proper.

9.0 DATA ANALYSIS AND CALCULATIONS

Not Applicable.

10.0 POLLUTION CONTROL

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

10.1 Waste Management

Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to UP-WM-001.

- Waste from this procedure will enter the "Corrosive Wastewater" wastestream.

11.0 METHOD PERFORMANCE CRITERIA

Refer to sections 1, 6, 7 and 8.

12.0 REFERENCES

Refer to Section 1.0.

13.0 ATTACHMENTS

Appendix A. Metals Digestion Standard Spike Concentrations
Appendix B. Example: ICP; ICP-MS TALs LIMS Digestion Spreadsheets

14.0 REVISION HISTORY

- Revision 20 was updated on 03/01/11
- Annual Review
- Added Section 7.6.5 for handling AVS-SEM extracts
- Added AVS-SEM LCS table to Attachment 3

Appendix A.

Metals Digestion Standard Spike Concentration

ICP

Vendor	Stock Name	Elements	Conc. (mg/L)
Environmental Express	HP1381-A-500	Al, Ba	2,000
		Ca, Mg, K, Na	10,000
	HP1381-B-500	Se	10
		Pb	20
		As	40
		Tl, Be, Cd	50
		Cr	200
		Cu	250
		Co, Ni, Li, V, Bi, Mn, Zn	500
		B, Fe, Sr	1,000
	HP1381-C-500	Ag	50
		Sb, P	500
		Mo, Sn, Ti	1,000
		Si	5,000
Inorganic Ventures	Single Element Standard	As	1,000
		Pb	1,000
		Se	1,000
		Tl	1,000

ICP-MS

Vendor	Stock Name	Elements	Conc. (mg/L)
Environmental Express	HP2930-A-500	Al, Ba	2,000
		Ca, Mg, K, Na	10,000
	HP2930-B-500	Se	10
		Pb	20
		As	40
		Tl, Be, Cd	50
		Cr	200
		Cu	250
		Co, Ni, V, Mn, Zn	500
		B, Fe, Sr	1,000
	HP2930-C-500	Ag	50
		Sb	500
		Mo, Sn, Ti	1,000
		Si	5,000
Inorganic Ventures	Single Element Standard	As	1,000
		Pb	1,000
		Se	1,000
		Tl	1,000

TCLP (MS)

Vendor	Stock Name	Elements	Conc. (mg/L)
Inorganic Ventures	STL-TCLP-1A	Hg	25
		Cu	25
		Zn, Ni	50
		Cd, Se, Ag	100
		Cr, As, Pb	500
	Single Element Standard	Ba	10,000

AVS-SEM (LCS)
(10 g/250 mL)

Elements	Conc. (mg/kg)
As	2.5
Cd	1.25
Cr	5.0
Cu	6.25
Pb	2.5
Ni	12.5
Ag	1.25
Zn	12.5

Appendix B.

Example: TALs LIMS Digestion Spreadsheets (014-001 to 014-012)

Metals/Inorganics Analysis Sheet

(To Accompany Samples to Instruments)

Batch Number: 500-106204

Method Code: 500-3005A-500

Analyst: Longtin, Melinda B

Batch Open: 2/24/2011 9:15:00AM

Batch End: 2/24/2011 9:16:00AM

Preparation, Total Recoverable or Dissolved Metals

	Input Sample Lab ID (Analytical Method)	SDG	Matrix	Initial Amount	Final Amount	Due Date	Analytical TAT	Dlv Rank	Comments	Output Sample Lab ID
1	MB~500-106204/1 N/A	N/A		50 mL	50 mL	N/A	N/A	N/A		*MB~500-106204/1-A*
2	LCS~500-106204/2 N/A	N/A		50 mL	50 mL	N/A	N/A	N/A		*LCS~500-106204/2-A*
3	500-31055-A-1 (6010B)	N/A	Water	50 mL	50 mL	3/7/11	8_Days - R	2	Zn	*500-31055-A-1-A*
4	500-31055-A-2 (6010B)	N/A	Water	50 mL	50 mL	3/7/11	8_Days - R	2	Zn	*500-31055-A-2-A*
5	720-33503-D-3 (6020)	N/A	Filtrate	50 mL	50 mL	2/28/11	4_Days - R	2	AS, Cr	*720-33503-D-3-A*
6	500-31062-E-1 (6020)	N/A	Filtrate	50 mL	50 mL	3/7/11	8_Days - R	3	As, Cd, Pb	*500-31062-E-1-B*
7	500-31062-E-2 (6020)	N/A	Filtrate	50 mL	50 mL	3/7/11	8_Days - R	3	As, Cd, Pb	*500-31062-E-2-B*
8	500-31062-E-2~DU (6020)	N/A	Filtrate	50 mL	50 mL	3/7/11	8_Days - R	3	As, Cd, Pb	*500-31062-E-2-C~DU*
9	500-31062-E-2~MS (6020)	N/A	Filtrate	50 mL	50 mL	3/7/11	8_Days - R	3	As, Cd, Pb	*500-31062-E-2-D~MS*
10	500-31062-E-2~MSD (6020)	N/A	Filtrate	50 mL	50 mL	3/7/11	8_Days - R	3	As, Cd, Pb	*500-31062-E-2-E~MSD*

(C-2) C-492

(6014-001)

Metals/Inorganics Analysis Sheet

(To Accompany Samples to Instruments)

Batch Number: 500-106204

Method Code: 500-3005A-500

Analyst: Longtin, Melinda B

Batch Open: 2/24/2011 9:15:00AM

Batch End: 2/24/2011 9:16:00AM

Batch Notes

Digestion Tube/Cup Lot #	1010192
Hot Block ID number	3
Hood ID or number	2
Lot # of Nitric Acid	J11046
Lot # of hydrochloric acid	J32030
Uncorrected Temperature	n/a
Oven, Bath or Block Temperature 1	95
ID number of the thermometer	C24127
Uncorrected Temperature 2	n/a
Oven, Bath or Block Temperature 2	95
Pipette ID	1627
First Start time	0915
First End time	0916
Batch Comment	

(C-2) C-493

(014-002)

Metals/Inorganics Analysis Sheet

(To Accompany Samples to Instruments)

Batch Number: 500-106204
Method Code: 500-3005A-500

Analyst: Longtin, Melinda B

Batch Open: 2/24/2011 9:15:00AM
Batch End: 2/24/2011 9:16:00AM

Comments

Login Comments for Job	31055:	Form 8 required for all report levels.
Login Comments for Job	31062:	BP LaMP specifications apply Form 8s are required for VOCs, SVOCs Internal COC needed

(C-2) C-494

(014-003)

Metals/Inorganics Analysis Sheet

(To Accompany Samples to Instruments)

Batch Number: 500-106204
Method Code: 500-3005A-500

Analyst: Longtin, Melinda B

Batch Open: 2/24/2011 9:15:00AM
Batch End: 2/24/2011 9:16:00AM

Reagent Additions Worksheet

Lab ID	Reagent Code	Amount Added	Final Amount	By	Witness
LCS 500-106204/2	M10KSPKMS_00001	0.5 mL	50 mL		
500-31062-E-2 MS	M10KSPKMS_00001	0.5 mL	50 mL		
500-31062-E-2 MSD	M10KSPKMS_00001	0.5 mL	50 mL		

Other Reagents:

Reagent	Amount/Units	Lot#:

(C-2) C-495

(014004)

Metals/Inorganics Analysis Sheet

(To Accompany Samples to Instruments)

Batch Number: 500-106205
Method Code: 500-3010A-500

Analyst: Longtin, Melinda B

Batch Open: 2/24/2011 9:15:00AM
Batch End: 2/24/2011 9:16:00AM

Preparation, Total Metals

	Input Sample Lab ID (Analytical Method)	SDG	Matrix	Initial Amount	Final Amount	Due Date	Analytical TAT	Dlv Rank	Comments	Output Sample Lab ID
1	MB~500-106205/1 N/A	N/A		50 mL	50 mL	N/A	N/A	N/A		*MB~500-106205/1-A*
2	LCS~500-106205/2 N/A	N/A		50 mL	50 mL	N/A	N/A	N/A		*LCS~500-106205/2-A*
3	500-30797-F-24 (6010B)	N/A	Water	50 mL	50 mL	2/28/11	12_Days - R	2	Ba, Cu	*500-30797-F-24-A*
4	500-30797-E-24 (6010B)	N/A	Filtrate	50 mL	50 mL	2/28/11	12_Days - R	2	As, Ba, B, Cd, Mn, Mo, K, Se, Na, Zn	*500-30797-E-24-A*
5	500-30797-E-24~DU (6010B)	N/A	Filtrate	50 mL	50 mL	2/28/11	12_Days - R	2	As, Ba, B, Cd, Mn, Mo, K, Se, Na, Zn	*500-30797-E-24-B~DU*
6	500-30797-E-24~MS (6010B)	N/A	Filtrate	50 mL	50 mL	2/28/11	12_Days - R	2	As, Ba, B, Cd, Mn, Mo, K, Se, Na, Zn	*500-30797-E-24-C~MS*
7	500-30797-E-24~MSD (6010B)	N/A	Filtrate	50 mL	50 mL	2/28/11	12_Days - R	2	As, Ba, B, Cd, Mn, Mo, K, Se, Na, Zn	*500-30797-E-24-D~MSD*
8	500-30797-F-25 (6010B)	N/A	Water	50 mL	50 mL	2/28/11	12_Days - R	2	Ba, Cu	*500-30797-F-25-A*
9	500-30797-E-25 (6010B)	N/A	Filtrate	50 mL	50 mL	2/28/11	12_Days - R	2	As, Ba, B, Cd, Mn, Mo, K, Se, Na, Zn	*500-30797-E-25-A*
10	500-30797-F-26 (6010B)	N/A	Water	50 mL	50 mL	2/28/11	12_Days - R	2	Ba, Cu	*500-30797-F-26-A*
11	500-30797-E-26 (6010B)	N/A	Filtrate	50 mL	50 mL	2/28/11	12_Days - R	2	As, Ba, B, Cd, Mn, Mo, K, Se, Na, Zn	*500-30797-E-26-A*
12	500-31061-A-1 (6010B)	N/A	Water	50 mL	50 mL	3/7/11	8_Days - R	2	Cu, Na, Zn	*500-31061-A-1-A*
13	500-31061-A-2 (6010B)	N/A	Water	50 mL	50 mL	3/7/11	8_Days - R	2	Cu, Na, Zn	*500-31061-A-2-A*
14	500-31061-A-3 (6010B)	N/A	Water	50 mL	50 mL	3/7/11	8_Days - R	2	Cu, Na, Zn	*500-31061-A-3-A*

(C-2) C-496

(01/4005)

Metals/Inorganics Analysis Sheet

(To Accompany Samples to Instruments)

Batch Number: 500-106205 Analyst: Longtin, Melinda B Batch Open: 2/24/2011 9:15:00AM

Method Code: 500-3010A-500 Batch End: 2/24/2011 9:16:00AM

15	500-31061-A-4 (6010B)	N/A	Water	50 mL	50 mL	3/7/11	8_Days - R	2	Cu, Na, Zn	*500-31061-A-4-A*
16	500-31061-A-5 (6010B)	N/A	Water	50 mL	50 mL	3/7/11	8_Days - R	2	Cu, Na, Zn	*500-31061-A-5-A*

(C-2) C-497

(014-006)

Metals/Inorganics Analysis Sheet

(To Accompany Samples to Instruments)

Batch Number: 500-106205
Method Code: 500-3010A-500

Analyst: Longtin, Melinda B

Batch Open: 2/24/2011 9:15:00AM
Batch End: 2/24/2011 9:16:00AM

Batch Notes

Lot # of hydrochloric acid	J32030
Lot # of Nitric Acid	J11046
Vendor of Reagent used	n/a
Digestion Tube/Cup Lot #	1010192
Hot Block ID number	3
ID number of the thermometer	J59688
Uncorrected Temperature	n/a
Oven, Bath or Block Temperature 1	95
Uncorrected Temperature 2	n/a
Oven, Bath or Block Temperature 2	95
Pipette ID	1627
First Start time	0915
First End time	0916
Batch Comment	

Comments

(C-2) C-498

(614-007)

Metals/Inorganics Analysis Sheet

(To Accompany Samples to Instruments)

Batch Number: 500-106205
Method Code: 500-3010A-500

Analyst: Longtin, Melinda B

Batch Open: 2/24/2011 9:15:00AM
Batch End: 2/24/2011 9:16:00AM

Reagent Additions Worksheet

Lab ID	Reagent Code	Amount Added	Final Amount	By	Witness
LCS 500-106205/2	M11ASPKIC_00001	0.5 mL	50 mL		
500-30797-E-24 MS	M11ASPKIC_00001	0.5 mL	50 mL		
500-30797-E-24 MSD	M11ASPKIC_00001	0.5 mL	50 mL		

Other Reagents:

Reagent	Amount/Units	Lot#:

(C-2) C-499

(014-008)

Metals/Inorganics Analysis Sheet

(To Accompany Samples to Instruments)

Batch Number: 500-106093

Method Code: 500-3050B-500

Analyst: Longtin, Melinda B

Batch Open: 2/23/2011 9:00:00AM

Batch End: 2/23/2011 9:01:00AM

Preparation, Metals

	Input Sample Lab ID (Analytical Method)	SDG	Matrix	Initial Amount	Final Amount	Due Date	Analytical TAT	Dlv Rank	Comments	Output Sample Lab ID
1	MB~500-106093/1 N/A	N/A		1.0 g	100 mL	N/A	N/A	N/A		*MB~500-106093/1-A*
2	LCS~500-106093/2 N/A	N/A		1.0 g	100 mL	N/A	N/A	N/A		*LCS~500-106093/2-A*
3	500-31006-F-1 (6010B)	500-31006-1	Solid	1.1597 g	100 mL	2/25/11	3_Days	2	RCRA	*500-31006-F-1-H*
4	500-31006-G-2 (6010B)	500-31006-1	Solid	1.1180 g	100 mL	2/25/11	3_Days	2	RCRA	*500-31006-G-2-C*
5	500-31006-G-3 (6010B)	500-31006-1	Solid	1.1570 g	100 mL	2/25/11	3_Days	2	RCRA	*500-31006-G-3-C*
6	500-31006-F-4 (6010B)	500-31006-1	Solid	1.1444 g	100 mL	2/25/11	3_Days	2	RCRA	*500-31006-F-4-B*
7	500-31020-F-1 (6010B)	500-31020-1	Solid	1.0292 g	100 mL	2/28/11	4_Days - R	2	HSL -(Mn, Ni)	*500-31020-F-1-D*
8	500-31020-F-1~DU (6010B)	500-31020-1	Solid	1.0389 g	100 mL	2/28/11	4_Days - R	2	HSL -(Mn, Ni)	*500-31020-F-1-E~DU*
9	500-31020-F-1~MS (6010B)	500-31020-1	Solid	1.1916 g	100 mL	2/28/11	4_Days - R	2	HSL -(Mn, Ni)	*500-31020-F-1-F~MS*
10	500-31020-F-1~MSD (6010B)	500-31020-1	Solid	1.1693 g	100 mL	2/28/11	4_Days - R	2	HSL -(Mn, Ni)	*500-31020-F-1-G~MSD*
11	500-31015-A-3 (6010B)	N/A	Solid	1.0011 g	100 mL	3/4/11	8_Days - R	2	Co, CU, Fe, Mn, Ni, Ti	*500-31015-A-3-A*

(C-2) C-500

(600-4109)

Metals/Inorganics Analysis Sheet

(To Accompany Samples to Instruments)

Batch Number: 500-106093

Method Code: 500-3050B-500

Analyst: Longtin, Melinda B

Batch Open: 2/23/2011 9:00:00AM

Batch End: 2/23/2011 9:01:00AM

Batch Notes

Perform Calculation (0=No, 1=Yes)	n/a
Nominal Amount Used	n/a
Digestion Tube/Cup Lot #	1010192
Blank Soil Lot Number	n/a
Balance ID	C1966
Hood ID or number	3
Hot Block ID number	8
Lot # of hydrochloric acid	J32030
Lot # of Nitric Acid	J11046
Logbook ID for diluted Nitric	n/a
Hydrogen peroxide lot number	J16a04
ID number of the thermometer	HB305334
Temperature	95
Acid used for pH adjustment	n/a
Pipette ID	1628
Analyst	MG
First Start time	0900
First End time	0901
Person's name who witnessed reagent drop	MG
Uncorrected Temperature	n/a

(C-2) C-501

(0.4-0.6)

Metals/Inorganics Analysis Sheet

(To Accompany Samples to Instruments)

Batch Number: 500-106093 Analyst: Longtin, Melinda B Batch Open: 2/23/2011 9:00:00AM

Method Code: 500-3050B-500 Batch End: 2/23/2011 9:01:00AM

Oven, Bath or Block Temperature 1	95
Uncorrected Temperature 2	n/a
Oven, Bath or Block Temperature 2	95
Batch Comment	

Comments

Login Comments for Job 31006: SAMPLES IN COOLER 4 AWAITING HOMOGENIZATION.

Partial / prelim results sent 2/24/11 per JK.

(C-2) C-502

(10-4-11)

Metals/Inorganics Analysis Sheet

(To Accompany Samples to Instruments)

Batch Number: 500-106093
Method Code: 500-3050B-500

Analyst: Longtin, Melinda B

Batch Open: 2/23/2011 9:00:00AM
Batch End: 2/23/2011 9:01:00AM

Reagent Additions Worksheet

Lab ID	Reagent Code	Amount Added	Final Amount	By	Witness
LCS 500-106093/2	M11ASPKIC_00001	1.0 mL	100 mL		
500-31020-F-1 MS	M11ASPKIC_00001	1.0 mL	100 mL		
500-31020-F-1 MSD	M11ASPKIC_00001	1.0 mL	100 mL		

Other Reagents:

Reagent	Amount/Units	Lot#:

(C-2) C-503

(014-012)

TITLE: Sample Preparation Semivolatile and Nonvolatile Organic Compounds from a WasteWater or Leachate Matrix using Separatory Funnel Extraction

Approvals (Signature/Date):

09/22/11	9/22/11
Daniel A. Knieriemen Date Supervisor, Organic Extractions	John D. Nagel Date Env. Health & Safety Coordinator
Terese A. Preston 9/22/11	Michael J. Healy 9/22/11
Terese A. Preston Date Quality Assurance Manager	Michael J. Healy Date Laboratory Director

Facility Distribution No: _____ **Distributed To:** Uncontrolled

1.0 SCOPE AND APPLICATION

This Standard Operating Procedure (SOP) outlines the extraction procedure of semivolatile and nonvolatile organic compounds from wastewater or leachate matrices using SW-846, Third Edition, Methods 1311, 3500B and 3500C, and 3510C; and 40CFR, Part 136, Methods 608, 610, and 625 as references.

On occasion, clients request slight modifications to this SOP. These modifications are addressed on a case-by-case basis with the range of accuracy (i.e., MDLs, linearity check or PT sample) verified prior to implementation. Any modifications would be written into a Quality Assurance Plan (QAP), authorized via laboratory signature approval, and mentioned in the data package's case narrative.

1.1 Method Sensitivity

1.1.1 Method Detection Limits

Not Applicable - refer to the analytical SOPs.

1.1.2 Reporting Limits

Not Applicable - refer to the analytical SOPs.

1.1.3 Definitions

Refer to Section 3.0 of the Laboratory's Quality Assurance Manual (UP-QA-QAM).

1.2 Summary of Method

The following procedure is used for the separatory funnel extraction of semivolatile and nonvolatile organics from waste water and leachate matrices for subsequent analysis.

A measured volume of sample (usually 1-liter for waste waters and 100 mLs for leachates) at the appropriate pH is serially extracted with Dichloromethane (DCM, a.k.a., Methylene chloride) using the separatory funnel extraction procedure. The extract is dried, concentrated, exchanged to another solvent if necessary, adjusted to the appropriate final volume, and stored at 4 ± 2°C prior to analysis. Optional cleanup procedures may be performed upon request. The mandatory acid cleanup for PCB samples is described within this SOP. GPC, Florisil, sulfur removal cleanups, and silica gel cleanup are described in UP-SP-003.

2.0 INTERFERENCES

- Solvents, reagents, glassware, and other sample processing hardware may yield artifacts and/or interferences to sample analysis. All these materials must be demonstrated to be free from interferences under the conditions of the analysis by analyzing method blanks.
- Phthalate esters contaminate many types of products commonly found in the laboratory. Plastics, in particular, must be avoided because phthalates are commonly used as plasticizers and are easily extracted from plastic material.
- Soap residue on glassware may cause degradation of certain analytes. This problem is especially pronounced with glassware that may be difficult to rinse. These items should be hand-rinsed very carefully to avoid this problem.
- Matrix interferences may be caused by contaminants that are co-extracted from the sample. These can appear as large, distinct peaks and/or elevated baselines. Occasionally matrix interferences may prevent the proper detection of surrogate and/or analytes, resulting in the reporting of low, or possibly high, surrogate and/or spike recoveries. The extent of matrix interferences will vary considerably from sample to sample, depending upon the nature of the site being sampled. Various cleanup procedures may be performed to remove heavy background or interferences, including GPC cleanup, florisil cleanup, acid cleanup, silica gel cleanup, and sulfur removal procedures.

3.0 SAFETY

Employees must abide by the policies and procedures in the Corporate Safety Manual, Radiation Safety Manual, Lab Specific Addendum to the CSM, and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coat and closed-toe, nonabsorbent shoes are a minimum.

3.1 Specific Safety Concerns or Requirements

- The following analytes have been tentatively classified as known or suspected, human or mammalian carcinogens: benzo(a)anthracene, benzidine, PCBs, 3,3'dichlorobenzindine, benzo(a)pyrene, alpha-BHC, beta-BHC, gamma-BHC, delta-BHC, 4,4'-DDT, dibenz(a,h)anthracene and N-nitrosodimethylamine. Primary standards of these toxic compounds should be prepared in hood.
- The use of separatory funnels to extract aqueous samples with Methylene Chloride creates excessive pressure very rapidly. Initial venting should be done immediately after the sample container has been sealed and inverted. Vent the funnel into the hood away from people and other samples. This is considered a high-risk activity, and a face shield must be worn over safety glasses or goggle when it is performed.
- Nitrile or equivalent gloves are available and must be worn when handling acids, bases, or samples.
- All extractions and handling of solvents should be performed under well-ventilated conditions of a fume hood.
- When pouring large volumes of solvents (100 mLs or greater), using a squeeze bottle, using the Cycletainer toggle, or transferring solvent in any manner such that a splash can occur, safety glasses are inadequate to protect the eyes and face. A face shield must be worn. A hood sash can act as a face shield when working with solvents inside a fume hood.

3.2 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Acetone	Flammable	1000 ppm-TWA	Inhalation of vapors irritates the respiratory tract. May cause coughing, dizziness, dullness, and headache.
Acetonitrile	Flammable Poison	40 ppm-TWA	Early symptoms may include nose and throat irritation, flushing of the face, and chest tightness. Prolonged exposure to high levels of vapors may cause formation of cyanide anions in the body.
Hexane	Flammable Irritant	500 ppm-TWA	Inhalation of vapors irritates the respiratory tract. Overexposure may cause lightheadedness, nausea, headache, and blurred vision. Vapors may cause irritation to the skin and eyes.
Methylene Chloride	Carcinogen Irritant	25 ppm-TWA 125 ppm-STEL	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.
Sodium Hydroxide	Corrosive Poison	2 ppm, 5 mg/m^3	This material will cause burns if comes into contact with the skin or eyes. Inhalation of Sodium Hydroxide dust will cause irritation of the nasal and respiratory system.
Sulfuric Acid	Corrosive Oxidizer Dehydrator	1 mg/m^3	This material will cause burns if comes into contact with the skin or eyes. Inhalation of vapors will cause irritation of the nasal and respiratory system.
1 – Always add acid to water to prevent violent reactions. 2 – Exposure limit refers to the OSHA regulatory exposure limit.			

There are no materials used in this method that have a serious or significant hazard rating. **NOTE: This list does not include all materials used in the method.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

4.0 EQUIPMENT AND SUPPLIES

4.1 Glassware

- 2 Liter separatory funnels
- 250 mL, 500 mL Erlenmeyer flasks
- Long stem glass funnels
- 100 mL, 1,000 mL graduated cylinder
- 500 mL Kuderna-Danish Flask
- 10 mL graduated receivers with attachments, regular and insulated types
- 3-Ball Snyder column
- 1.0 mL, 10.0 mL, class A, volumetric pipet
- 2.0 mL disposable pipet
- 500 uL, 1,000 uL syringe
- 5.0 mL, 10.0 mL pre-marked 16 X 125 test tubes with Teflon lined lids*
- Glass stirring rods
- Shallow Pyrex drying tray
- Teflon squeeze bottles
- Teflon centrifuge bottles, 125 mL

*Volumetrically add the appropriate volume of Hexane to a 16 X 125 screw top test tube and mark the meniscus using a fine-tipped marker. Discard the Hexane into an appropriate waste vessel and let dry. Dilute the sample to volume using this test tube.

4.2 Miscellaneous

- glasswool, soxhlet-extracted using DCM
- transfer pipets
- wide range pH paper 1 to 12
- Centrifuge
- Boiling Chips, Teflon, soxhlet-extracted using DCM
- Muffle furnace
- Desiccator
- Balance, top-loading, capable of weighing to 0.001g
- Waterbath, heated, with concentric ring covers, capable of temperature control - Must be in a hood
- Nitrogen Evaporator - N-Evap by Organomation Associates, Inc.

5.0 REAGENTS AND STANDARDS

A label on any reagent or standard bottle must contain the standard number, concentration of the reagent, name of the reagent, date prepared, expiration date and the analyst who prepared the reagent.

5.1 Reagents

5.1.1 Pesticide Grade Methylene Chloride [Dichloromethane (DCM)]
Pesticide Grade Acetone
Pesticide Grade Hexane
Pesticide Grade Acetonitrile

5.1.2 Heat Purified Granular Sodium Sulfate

Purify by heating to 400°C for 4-hours in a shallow drying tray, cool in a desiccator, and store in a glass bottle.

- Life of Reagent: 1-year
- Storage Requirements: Store in a glass container and keep dry (anhydrous).

5.1.3 Reagent Water

Defined as water in which no interferent is observed at one-half the reporting limit of any target compounds when one liter of the reagent water is extracted and prepared by using the same procedure as for a water sample.

5.1.4 Concentrated Sulfuric Acid

Purchased from a supplier

- Hazardous Properties: Sulfuric Acid (H_2SO_4) is extremely corrosive and toxic to tissues. Vapors are also harmful.
- Life of Reagent: 1-year
- Storage Requirements: Store in glass container. The label on the bottle must contain concentration and name of reagent.

5.1.5 Sulfuric Acid (1:1)

Cautiously add 500 mLs concentrated sulfuric acid to 500 mLs reagent water. Because this is an exothermic reaction, prepare in Pyrex glassware and in a cold water bath.

- Hazardous Properties: Extremely corrosive and toxic to tissues. Vapors are also harmful.
- Life of Reagent: 1-year from preparation date
- Storage Requirements: Store in a glass bottle

5.1.6 10 N Sodium Hydroxide

Cautiously dissolve 400 grams of solid NaOH in reagent water and dilute to 1000 mLs. Always prepare in Pyrex glassware due to the amount of heat given off by the reaction.

- Hazardous Properties: Toxic & Corrosive. Harmful if swallowed, inhaled or absorbed through the skin.
- Life of Reagent: 1-year from preparation date
- Storage Requirements: Store in a glass bottle.

5.2 Quality Control (QC) Solutions

QC solutions must be stored in amber or foil-wrapped, glass containers that have Teflon-lined caps. The label on the container must contain the following information: Name of the solution, the standard number, concentration of the components, date prepared, preparation analyst, and expiration date. All QC solutions are stored in the standards refrigerator in the GC/MS lab, GC Extractables lab, or in the standards refrigerator located in the organic extraction lab. QC solutions are stored at 4 ± 2°C.

Listed below are the various QC solutions and concentrations appropriate for extraction of BNAs, Pesticides, Pesticides/PCBs, PCBs, PAHs, and diesel range organics (DRO) from waste water and TCLP Leachate samples.

5.2.1 Surrogate Spike Solutions

5.2.1.1 BNA Surrogate Spike Working Solution

The BNA Surrogate Spike Stock Solution is purchased directly from a chemical vendor at 10 times the concentration of the levels listed below. See Attachment 3. The BNA Surrogate Spike Working Solution is prepared by pipetting 10.0 mLs of BNA Surrogate Spike Stock Solution to a 100.0 mL flask. Class A glassware must be used. The standard is then brought up to 100.0 mLs with methanol. 500 uLs of BNA Surrogate Spike Solution is added to all samples, method blanks (MBs), matrix spike/matrix spike duplicates (MS/MSDs) and laboratory control samples (LCS).

Compound	Concentration (ug/mL)
p-Terphenyl-d_4	100
Nitrobenzene-d_5	100
2-Fluorobiphenyl	100
1,2-Dichlorobenzene-d_4	100
Phenol-d_5	150
2-Fluorophenol	150
2,4,6-Tribromophenol	150
2-Chlorophenol-d_4	150

- Life of Standard: 6 months from the preparation date (as documented from the vendor).
- Storage Requirements: As stated in Section 5.2

5.2.1.2 BNA Surrogate (SIM) Spike Solution

The BNA Surrogate (SIM) Spike Solution is prepared from the BNA Surrogate Spike Solution. 500 uLs of BNA Surrogate (SIM) Spike Solution is added to all samples, the MB, MS/MSD and LCS.

Compound	Concentration (ug/mL)
p-Terphenyl-d_4	10
Nitrobenzene-d_5	10
2-Fluorobiphenyl	10
1,2-Dichlorobenzene-d_4	10
Phenol-d_5	15
2-Fluorophenol	15
2,4,6-Tribromophenol	15
2-Chlorophenol-d_4	15

- Life of Standard: Same as parent solution.
- Storage Requirements: As stated in Section 5.2

5.2.1.3 BNA Low Level Surrogate Spike Working Solution

The BNA Low Level Surrogate Spike Working Solution is prepared from the BNA Surrogate Spike Solution. 500 uLs of BNA Surrogate Working Solution is added to all samples, the MB, MS/MSD and LCS.

Compound	Concentration (ug/mL)
p-Terphenyl-d_4	50
Nitrobenzene-d_5	50
2-Fluorobiphenyl	50
1,2-Dichlorobenzene-d_4	50
Phenol-d_5	75
2-Fluorophenol	75
2,4,6-Tribromophenol	75
2-Chlorophenol-d_4	75

- Life of Standard: Same as parent solution.
- Storage Requirements: As stated in Section 5.2

5.2.1.4 Pesticide/PCB Surrogate Spike Parent Solution

A certified stock solution is purchased directly from a standards vendor. The GC Extractables lab prepares a surrogate parent solution from these standards and is a solution of decachlorobiphenyl (DCB) and 2,4,5,6-tetrachloro-m-xylene (TCX) in acetone at the concentrations listed below. Refer to the analytical SOP for specific details on the preparation, storage and labeling of these solutions.

Compound	Concentration (ug/mL)
DCB	4.0
TCX	4.0

- Life of Standard: 6 months from date of preparation, not to exceed expiration date of standards
- Storage Requirements: As stated in Section 5.2

5.2.1.5 Pesticide/PCB Surrogate Spike Solution

The surrogate used in the extraction of pesticides/PCBs from waste waters is a solution of DCB and TCX in acetone at the concentrations listed below. The working solution is prepared from the parent solution by performing a 1/10 dilution using Class A volumetric glassware in acetone. 1,000 uLs of Pesticide/PCB Surrogate Spike Solution is added to all samples, the MB, MS/MSD and LCS.

Compound	Concentration (ug/mL)
DCB	0.40
TCX	0.40

- Life of Standard: 6 months from date of preparation, not to exceed expiration date of the parent standard
- Storage Requirements: As stated in Section 5.2

5.2.1.6 PAH Surrogate Spike Solution

The surrogate used in the extraction of PAHs from soil/sediments is a solution of Decafluorobiphenyl and Benzo (e) pyrene in acetone at the concentrations listed below. The HPLC group prepares the working surrogate solution. 500 uLs of PAH Surrogate Spike Solution is added to all samples, the MB, MS/MSD and LCS.

Compound	Concentration (ug/mL)
Decafluorobiphenyl	100
Benzo (e) pyrene	5.0

- Life of Standard: Same as parent solution
- Storage Requirements: As stated in Section 5.2

5.2.1.7 DRO Surrogate Spike Solution

The surrogate used in the extraction of DRO from soil/sediments is a solution of 2-Fluorobiphenyl and o-Terphenyl in acetone at the concentrations listed below. The GC group prepares the working surrogate solution. 500 uLs of DRO Surrogate Spike Solution is added to all samples, the MB, MS/MSD and LCS.

Compound	Concentration (ug/mL)
2-Fluorobiphenyl	200
o-Terphenyl	200

- Life of Standard: Same as parent solution.
- Storage Requirements: As stated in Section 5.2.

5.2.2 Laboratory Control Sample (LCS) & Matrix Spike (MS) Solutions

5.2.2.1 BNA TCL Spike Solution

The BNA TCL Spike Solution is prepared from two stock standards, 8270 LCS Mix and 3,3'-Dimethylbenzidine. See Attachments 4 and 5. 50.0 mLs of 8270 LCS Mix and 5.0 mLs of 3,3'-Dimethylbenzidine are pipetted into a 100.0 mL flask. Class A glassware must be used. The standard is then brought up to 100.0 mLs with methanol. 1000 uLs of BNA TCL Spike Solution is added to the LCS and MS/MSD.

Compound	Concentration (ug/mL)
Acid Compounds	100
Base/Neutral Compounds	100

- Life of Standard: 6 months from the from the preparation date (as documented from the vendor).
- Storage Requirements: As stated in Section 5.2.

5.2.2.2 BNA Matrix Spike (SIM) Solution

The BNA Matrix Spike (SIM) Solution is prepared from the BNA TCL Spike Solution found in section 5.2.2.1. 1000 uLs of BNA Matrix Spike (SIM) Solution is added to the MS/MSD and LCS.

Compound	Concentration (ug/mL)
Acid Compounds	10
Base/Neutral Compounds	10

- Life of Standard: Same as parent solution.
- Storage Requirements: As stated in Section 5.2.

5.2.2.3 BNA Low Level Matrix Spike Solution

The BNA Low Level Matrix Spike Solution is prepared from the BNA TCL Spike Solution found in section 5.2.2.1. 1000 uLs of BNA Low Level Matrix Spike Solution is added to the MS/MSD and LCS.

Compound	Concentration (ug/mL)
Acid Compounds	50
Base/Neutral Compounds	50

- Life of Standard: Same as parent solution.
- Storage Requirements: As stated in Section 5.2.

5.2.2.4 Pesticide (Full List) Spike Solution

The LCS/MS solution used in the extraction of pesticides or pesticides/PCB from soil/sediments is a solution of pesticide compounds in methanol at the following concentrations. 1000 uLs of Pesticide (Full List) Spike Solution is added to MS/MSD and LCS.

Compound	Concentration (ug/mL)
All compounds	0.40

- Life of Standard: Same as parent solution.
- Storage Requirements: As stated in Section 5.2.

5.2.2.5 PCB Spike Solution

The LCS / MS spike solution used in the extraction of PCBs from waste waters is a solution of Arochlor 1016 and Arochlor 1260 in methanol at the following concentrations. 1,000 uLs of PCB Spike Solution is added to the MS/MSD and LCS.

Compound	Concentration (ug/mL)
AR1016	5.0
AR1260	5.0

- Life of Standard: Same as parent solution
- Storage Requirements: As stated in Section 5.2

5.2.2.6 TCLP Pesticide Spike Working Solution

The LCS / MS spike solution used in the extraction of pesticides from TCLP leachates is a solution of pesticide compounds in methanol at the following concentrations. 1,000 uLs of TCLP Pesticide Spike Solution is added to the MS and LCSs that are for Pesticides only

Compound	Concentration (ug/mL)
Lindane	0.1
Heptachlor	0.1
Heptachlor Epoxide	0.1
gamma-Chlordane	0.1
Endrin	0.1
Methoxychlor	1.0

- Life of Standard: Same as parent solution
- Storage Requirements: As stated in Section 5.2

5.2.2.7 Toxaphene Spike Working Solution

The LCS / MS spike solution used in the extraction of pesticides from TCLP leachates is a solution of toxaphene in methanol at the following concentration. 1,000 uLs of Toxaphene Spike Solution is added to the LCS and MSs that are for PCBs only.

Compound	Concentration (ug/mL)
Toxaphene	10

- Life of Standard: Same as parent solution
- Storage Requirements: As stated in Section 5.2

5.2.2.8 PAH Spike Solution

The LCS/MS solution used in the extraction of PAHs from soil/sediments is a solution of analytes in acetonitrile at the following concentrations. The HPLC group prepares the spike solution. 500 uLs of PAH Spike Solution is added to the MS/MSD and LCS.

Compound	Concentration (ug/mL)
Acenaphthene	8
Acenaphthylene	16
Anthracene	0.8
Benzo(a)anthracene	0.8
Benzo(a)pyrene	0.8
Benzo(b)fluoranthene	1.6
Benzo(ghi)perylene	1.6
Benzo(k)fluoranthene	0.8
Chrysene	0.8
Dibenzo(a,h)anthracene	1.6
Fluoranthene	1.6
Fluorene	0.8
Indeno(1,2,3-cd)pyrene	0.8
Naphthalene	8.0
Phenanthrene	0.8
Pyrene	0.8

- Life of Standard: Same as the parent solution.
- Storage Requirements: As stated in Section 5.2.

5.2.2.9 DRO Spike Solution

The LCS/MS solution used in the extraction of DRO from soil/sediments is a solution Diesel Fuel #2 at 1000 ug/mL in methanol. The GC group prepares the spike solution. 500 uLs of DRO Spike Solution is added to the MS/MSD and LCS.

Compound	Concentration (ug/mL)
Diesel Fuel #2	4,000

- Life of Standard: Same as the parent solution.
- Storage Requirements: As stated in Section 5.2.

6.0 CALIBRATION

Not Applicable.

7.0 PROCEDURE

7.1 Quality Control Checks

The following QC is performed with each organic extraction batch. An extraction batch contains samples of similar matrix that are logged in for the same test code. An extraction batch may contain no more than 20 samples.

Quality Control	Frequency
MB	1 in 20 or fewer samples
LCS [1]	1 set of 20 or fewer samples
MS/MSD [2]	At least one set in 20 or fewer samples (may be requested by client)
Surrogates	Every sample / MB / MS / MSD / LCS

[1] LCS Duplicate (LCSD) is performed when insufficient sample is available for the MS/MSD or if requested by the client.
[2] The sample selection for MS/MSD is rotated among client samples so that various matrix problems may be noted and/or addressed.

7.2 Sample Collection, Preservation and Handling

Sample container, preservation technique and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract/client requests. In general, water samples are collected in 1-liter or 80-oz. narrow-mouth, amber, glass jars, with Teflon-lined screw-cap lids. If a Teflon-lined lid is not available, solvent-rinsed aluminum foil can be used as a liner.

NOTE: If the sample matrix is highly acidic or basic, corrosion of the foil may occur and contaminate the sample.

Matrix	Holding Time (VTS) [1]	Preservation
Wastewaters	7 days	Cool, 4 ± 2°C
TCLP Leachates	14 days to TCLP Extract [2] 7 days from the completion of the TCLP extraction for the organic extraction	Cool, 4 ± 2°C

[1] Verified Time of Sampling.
[2] TCLP Extraction performed by Inorganic Prep Group.

Samples, sample extracts and standards are stored separately. Samples are stored at 4 ± 2°C prior to extraction. Any samples received by the preparation group that do not comply must be noted. Deviations in results "could" be explained by the failure of sample collector/client/custodian to comply to sample collection and storage requirements.

7.3 Sample Preparation (Glassware Preparation)

7.3.1 All glassware (except Snyder columns, volumetric flasks, volumetric pipets, and syringes) must be washed thoroughly with warm soapy water, rinsed **5** times with hot tap water and rinsed **3** times with DI water. The stopcocks of the separatory funnels should be cleaned with a small brush. **It is absolutely mandatory that all glassware be scrupulously clean.** Failure to rinse well with water, may leave a soap film that is incompletely removed with solvent rinsing, and with subsequent extraction, "soap" peaks will appear in the chromatograms.

7.3.2 If glassware is very wet it may be towel dried. **All** Glassware should be rinsed with cleaning grade acetone, and the excess should be discarded into the appropriate waste container. This step is performed to ensure that the glassware is dry. **If after rinsing once, the glassware still contains any water, repeat the acetone rinse.** At this point let the glassware dry before continuing to ensure that all of the water is gone. All glassware should then be rinsed with DCM using a Teflon squeeze bottle, and the excess discarded into the appropriate waste container. Repeat this step at least 3-times and allow the glassware to dry. Failure to remove all water prior to DCM rinsing prevents the DCM solvent from coming into contact with all surfaces on the glassware. The solvent rinse is then incomplete, and may leave contaminants on the glass, which are then co-extracted with the sample.

NOTE: DCM is considered a health hazard. It is harmful if swallowed or inhaled. The vapor is irritating and thus should be used in a hood. It is readily absorbed through the skin and contact may be irritating.

NOTE: Pressure builds up quickly in a capped separatory funnel. Vent the pressure immediately and often thereafter.

7.3.3 Procedures for Return of Glassware to Dishroom

Any glassware that has organic residue present, all K-D receivers, and all separatory funnels should be rinsed with cleaning grade acetone, or whatever other solvent which removes the residue, prior to return to the dish room from the lab. Glassware in which the residue can not be removed should be segregated from other glassware being returned to the dish room. Glassware should be carefully placed in a tub and placed on the "Dirty" side of the dish racks. In order to reduce glassware breakage, glassware should not be stacked in the tubs. Severely chipped glassware should be removed from service. Some items are repairable. Check with the supervisor as to what items get repaired.

7.4 Calibration/Standardization

Not Applicable.

7.5 Preventive Maintenance

- Balance should be cleaned and checked for levelness prior to each use. Class "S" weights should be weighed and documented each day the balance is used. Consult your supervisor if the weights are out of acceptable limits. Do not use the balance until the situation is back in control.
- The K-D water bath should contain DI water only. The water level should be checked prior to each use, and should not fall below the heating coils. If the heating coils do become exposed to the air while in use, they could burn-out and would require replacement. Keep the water level approximately one inch from the top of the bath edge.
- The N-Evap water bath should also contain DI water. The water level in the bath should be sufficient to cover 3/4 of the receivers when submerged.
- The thermometer used in the extraction water baths are checked against a reference NIST thermometer on a yearly basis or sooner if necessary.
- The desiccant used in the desiccator should be removed and dried in an oven at 130°C whenever the color indicates that it is saturated with moisture. This is usually indicated by a change of color from blue to purple/pink.
- To avoid the breakage of sample extracts, the analyst needs to take care that the containers are properly balanced on the centrifuge. The container should fit snugly in the holder. Use a paper towel to cushion the centrifuge bottles if necessary.

7.6 Extraction Process

7.6.1 MB and LCS Water Preparation

Add 1.0 liter of Milli-Q water to 2 separatory funnels and label as the MB and LCS.

7.6.2 Sample Preparation

Prior to pouring water samples, sample containers should be inverted a few times to mix the sample. However, if there is a sediment layer present, consult your supervisor or section manager before mixing. Also, if the sample appears to have more than one phase, immediately contact your supervisor, section manager, or project manager. The client will need to be notified.

a. Waste Waters

If the sample container is 1-liter or 1-quart in volume, mark the level on the outside of the bottle. Pour the entire contents of the bottle into the separatory funnel. Rinse the bottle with 60 mls of DCM (the first aliquot of the extraction, and add it to the extraction vessel. This ensures that the sample is qualitatively transferred. Refill the bottle to the mark with water and then measure the volume in the bottle. If a bottle is full with sample, the volume still needs to be measured due to variations from bottle to bottle. Record this amount as the initial sample volume. Occasionally the analyst will encounter samples that contain sediment which has collected at the bottom of the sample bottle. The bottle still must be rinsed with DCM and the DCM/sediment transferred to the separatory funnel. If emulsion occurs, the DCM can be separated by using the centrifuge (see **7.6.9.2 Centrifugation**). If a sample contains an unreasonable amount of sediment (1/4 inch or more) contact the supervisor before continuing. Alternatively, a graduated cylinder for each sample can be used to determine initial sample volume. If a graduated cylinder is used, both it and the sample bottle must be rinsed with the first aliquot of DCM.

Use 1.0 liter aliquots for MS/MSD if sample volume allows. **Note: Splitting of sample volume to perform a MS/MSD is not allowed for DoD QSM, Bp LaMP, and South Carolina DHEC projects.** Label as the sample, MS and MSD.

b. TCLP Leachates

For BNA TCLP samples, measure 900 mLs of Milli-Q water into each sample separatory funnel. Measure 100 mLs of each sample and add it to the labeled separatory funnel containing the water.

For Pesticide TCLP samples, measure 990 mLs of Milli-Q water into each sample separatory funnel. Measure 10 mLs of each sample and add it to the labeled separatory funnel containing the water.

Measure an additional aliquot of sample to those designated to have a MS. LCSD and MSD are not performed.

7.6.3 Check the pH by dipping a transfer pipet into the sample and touching it to wide-range pH paper. Record the pH in the extraction log.

7.6.4 Add the appropriate amount of the surrogate spike solution to every sample including the MB, MS/MSD and LCS. Note: For DoD QSM Projects the surrogate spike must be added to the sample bottle prior to transfer to the separatory funnel. If the sample bottle is completely filled, an aliquot of sample may be transferred to the separatory funnel prior to spiking. Samples which contain sediments that are required to be decanted will be spiked after transfer to the separatory funnel.

7.6.5 Add the appropriate amount of the spike solution to the LCSs and MSs. Note: For DoD QSM Projects the surrogate spike must be added to the sample bottle prior to transfer to the separatory funnel. If the sample bottle is completely filled, an aliquot of sample may be transferred to the separatory funnel prior to spiking. Samples which contain sediments that are required to be decanted will be spiked after transfer to the separatory funnel.

NOTE: All spiking solutions MUST be at room temperature before use. This includes stock solutions when preparing working spike solutions. Actual concentrations will change at colder temperatures. Furthermore, some components may come out of solution in the freezer. Warming to room temperature reverses the process.

7.6.6 Adjust the pH as required (refer to Flowchart in Attachment 2 for the appropriate pH for each parameter). Use sulfuric acid (1:1) to acidify and NaOH (10N) to make basic. Record pH adjustments in the log book.

7.6.7 Plug a long-stem glass funnel with a little soxhlet-extracted glass wool. Fill the funnel half-full with heat-purified granular sodium sulfate and place on a 250 mL collecting flask (500 mLs for BNA). Be sure each separatory funnel and corresponding collecting flask is appropriately labeled with the correct sample number, extraction batch number, and method code.

7.6.8 Seal and shake the separatory funnel vigorously for 2-minutes with periodic venting to release excess pressure.

CAUTION: Pressure builds up quickly in a separatory funnel that has both the stopcock closed and the stopper in place. Initial venting should be done immediately after the separatory funnel has been sealed and inverted. Be sure to vent the separatory funnel frequently by inverting the funnel and opening the stopcock. Do not point the funnel at or near anyone as it is being vented.

7.6.9 Allow the organic layer to separate from the water phase for a minimum of 10 minutes. If the emulsion interface between layers is more than one-third the size of the solvent layer, the analyst must employ one or more of the following techniques to complete the phase separation (document the presence of severe emulsions in the log book). Method 3510C, Section 7.6 specifies that samples that do not meet the 80% recovery of $MeCl_2$ after extraction should be re-extracted via Method 3520. The Project Manager will be contacted if this scenario occurs to determine the best course of action. Projects with samples that historically do not meet this requirement need to be logged with the 3520 preparation method to ensure that sufficient sample volume is available and holding times are met.

7.6.9.1 Stirring Rod

Physically break up the emulsion by stirring.

7.6.9.2 Centrifugation

Separates by density. Draw off the emulsion into a 125 mL, Teflon, centrifuge bottle. Balance the centrifuge. Centrifuge 2-5 minutes. Carefully pour the contents back into the separatory funnel and drain off the DCM layer. Alternatively, after centrifugation, draw out the DCM (bottom layer) directly from the centrifuge bottle using a disposable, 10 mL pipet and deliver it to the collecting flask. Return any aqueous phase back to the appropriate separatory funnel. Add the next DCM aliquot first to the centrifuge bottle as a rinse and then to the appropriate separatory funnel.

7.6.9.3 Salting Out

Addition of salt makes the aqueous phase too polar to support a less polar phase. Add ~100 to 200 g of heat-purified sodium chloride to the aqueous phase and shake the sample again.

7.6.10 Drain the organic layer through the funnel containing sodium sulfate and into the 250 mL collecting flask.

7.6.11 Repeat the extraction 2 more times using 60 mLs of DCM each time, combining all 3 organic layers in the same flask. Rinse down the sodium sulfate with ~20 mLs of DCM after the first and last drain. Unless BNA, the sample is now ready for concentration. Proceed to Section 7.6.12 for K-D concentration instructions

BNA Samples The samples are adjusted to pH>11 and extracted 3-more times as described above, after which, the samples are ready for concentration. Proceed to Section 7.6.12 for K-D concentration instructions.

7.6.12 K-D Sample Concentration

Occasionally, samples extracted from wastewaters for subsequent BNA analysis and/or Pesticide/PCB analysis receive a gel permeation cleanup (GPC). These samples need to be identified prior to concentrating. Refer to the appropriate section of USP-001 or 003 for the concentration of these "pre-GPC" BNA samples. Refer to the appropriate section of USP-003 for the concentration of these "pre-GPC" Pesticide/PCB samples.

7.6.12.1 Assemble a K-D concentrator by attaching a 10 mL concentrator tube (insulated type if BNA or DRO) to a 500 mL K-D flask. Add a couple of boiling chips. Transfer each extract to its own K-D flask (making sure that each flask is appropriately labeled). Rinse the Erlenmeyer flask 3 times with DCM, adding each rinse to the K-D flask. Rinse down the joint with DCM and then attach a 3-ball Snyder column to each and place on the water bath. The temperature control should be set at medium. This setting should correspond to 80-90°C. Concentration to <10 mLs is usually completed in 60-90 minutes. The balls should chatter but the chambers should not flood.

7.6.12.2 Refer to appropriate test for further concentration instructions.

BNA and DRO When the apparent final volume reaches about 2-5 mLs, remove from the bath. Allow to drain and cool for 10 minutes. Remove the Snyder column, rinsing the joint and flask with 1-2 mLs DCM. Allow to drain. Dry off the outside of the joint between the flask and receiver to remove the water. Pull the 2 pieces apart gently, in a twisting motion. Rinse off the joint with DCM into the receiver. Cover the receiver with a piece of aluminum foil. If the extracts are to sit overnight, they are to be stored at 4 ± 2°C.

Proceed to Section 7.6.13 for further concentration using nitrogen evaporation.

PCB and Pesticide/PCB When the apparent volume reaches ~ 5 mLs, a solvent exchange is required. Add 50 mLs of hexane to the K-D flask (through the Snyder column). Concentrate the extract by raising the temperature of the water bath, if necessary, to maintain proper distillation. The Snyder column may be wrapped with aluminum foil to aid in proper evaporation. When the apparent volume reaches 3-5 mLs, remove from the bath. Allow to drain and cool for 10 minutes. Remove the Snyder column, rinsing the joint and flask with 1-2 mLs hexane. Allow to drain. Dry off the outside of the joint between the flask and receiver to remove the water. Pull the 2 pieces apart gently, in a twisting motion. Rinse off the joint with hexane into the receiver.

Transfer the extract into a labeled, 16X125 test tube that has been previously marked at 10.0 mLs. Rinse the receiver 3 times with hexane, transferring the rinsate each time to the 16X125 test tube. Refrigerate the extracts if they are not going to be immediately concentrated.

Proceed to Section 7.6.13 for further concentration using nitrogen evaporation.

PAHs When the apparent volume reaches ~5 mLs, a solvent exchange is required. Add 5 mLs of acetonitrile to the K-D flask (through the Snyder column). Concentrate the extract by raising the temperature of the water bath, if necessary, to maintain proper distillation. The Snyder column may be wrapped with aluminum foil to aid in proper evaporation. When the apparent volume reaches 3-5 mLs, remove from the bath. Allow to drain and cool for 10 minutes. Remove the Snyder column, rinsing the joint and flask with 1-2 mLs acetonitrile. Allow to drain. Dry off the outside of the joint between the flask and receiver to remove the water. Pull the 2 pieces apart gently, in a twisting motion. Rinse off the joint with acetonitrile into the receiver.

Transfer the extract into a labeled, 16X125 test tube that has been previously marked at 5.0 mLs. Rinse the receiver 3 times with acetonitrile, transferring the rinsate each time to the 16X125 test tube. Refrigerate the extracts if they are not going to be immediately concentrated.

Proceed to Section 7.6.14 for further concentration using nitrogen evaporation.

7.6.13 TurboVap Sample Concentration

All samples being concentrated must be recorded in the extraction log.

**Occasionally, samples extracted from soil/sediments for subsequent BNA analysis and/or Pesticide/PCB analysis receive a gel permeation cleanup (GPC). These samples need to be identified prior to concentrating. Refer to UP-SP-003 for this procedure.

Prepare the TurboVap by ensuring that the unit has sufficient nitrogen supply and the water level in the bath is greater or equal to the sample volume in the TurboVap tube. The pressure should be set to between 8 to 15 PSI.

Transfer the extract to an appropriately labeled TurboVap tube. It can only contain 200 mLs of extract. If there is more than 200 mLs of extract to concentrate, concentrate 200 mLs of it down to approximately 20 mLs, then add the remaining extract to the tube. Rinse the Erlenmeyer flask 3 times with DCM, adding each rinse to the tube.

To begin the concentration process, gently place the tube in the water bath. Close the cover. The TurboVap has several endpoint selections. Select the sensor endpoint. The following parameters should be set: bath temperature to 40, endpoint time to 00, and pressure to 14 PSI. Press the START/STOP button for each cell position used. To pause the concentration, raise the cover. To continue operation, lower the cover. When a cell reaches its endpoint, its corresponding light blinks and the beeper sounds briefly every thirty seconds. Raise the cover to silence the beeper. Remove the glassware from the bath.

Refer to the appropriate 'parameter' defined below for further concentration instructions:

BNAs

At the endpoint the extract should be at ~1 mLs. The extract should be removed from the TurboVap before the endpoint sensor is triggered. Low analyte recovery will occur if the extract volume is concentrated to low. Transfer the entire extract to an appropriately labeled 2 mL vial. Rinse the TurboVap tube with a few 100 uLs of DCM 3 times, transferring the rinse to the 2 mL vial each time.

Proceed to Section 7.6.14 for further concentration using nitrogen evaporation.

PCB, Pesticide/PCB

Begin concentrating extract. When an extract volume reaches 10 mLs, perform a hexane exchange. Remove the tube from the TurboVap and add 50 mLs of hexane. This is done to ensure that solvent is not spilled in the water bath. Place the tube back on the TurboVap and concentrate the extract back down to 10 mLs. Remove the glassware from the bath.

Transfer the extract into a labeled, 16 X125 test tube that has been previously marked at a 10.0 mL* volume. Rinse the TurboVap tube 3 times with hexane, transferring the rinsate each time to the 16X125 test tube. Refrigerate the extracts if they are not going to be immediately concentrated.

* Volumetrically pipet 10.0 mL of hexane into a 16 X 125 test tube. Mark the meniscus using a fine-tipped marker. Discard the solvent into an appropriate waste vessel.

Proceed to Section 7.6.14 for further concentration using nitrogen evaporation.

PAHs

Before adding the extract to the TurboVap tube, add 5 mLs of acetonitrile.

Concentrate the extract to 10 mLs. Transfer the extract into a labeled, 16 X125 test tube that has been previously marked at a 5.0 mL* volume. Rinse the TurboVap tube 3 times with acetonitrile, transferring the rinsate each time to the 16X125 test tube. Refrigerate the extracts if they are not going to be immediately concentrated.

* Volumetrically pipet 5.0 mL of DCM into a 16 X 125 test tube. Mark the meniscus using a fine-tipped marker. Discard the solvent into an appropriate waste vessel.

Proceed to Section 7.6.14 for further concentration using nitrogen evaporation.

DRO

At the endpoint the extract should be at approximately 10 mLs. Transfer the extract into a 16 X 125 test tube that has been previously marked at a 5.0 mL* volume. Rinse the TurboVap tube 3 times with DCM, transferring the rinsate each time to the 16 X 125 test tube.

* Volumetrically pipet 5.0 mL of DCM into a 16 X 125 test tube. Mark the meniscus using a fine-tipped marker. Discard the solvent into an appropriate waste vessel.

Proceed to Section 7.6.14 for further concentration using nitrogen evaporation.

7.6.14 Nitrogen Evaporation

7.6.14.1 For further concentration, place each receiver or test tube on the N-Evap tray. The N-Evap outlet needles should be stored in a 600 mL beaker containing DCM:Acetone (1:1) mixture prior to each use. Wipe each outlet needle with a KimWipe and lock into place on the N-Evap. Lower the outlet tube into the receiver being careful not to touch the sides of the receiver. The outlet tube should remain above the surface of the extract. Open the valve of the nitrogen tank and adjust the pressure in order to achieve the appropriate nitrogen stream. The nitrogen stream should make a slight "dimple" in the surface of the extract. If the extract bubbles, the nitrogen stream is **not** gentle enough. Lower the entire N-Evap tray into the warm water bath to aid the evaporation process. Heat is not necessary and if used the temperature should not exceed 35°C.

7.6.14.2 The extracts must never be allowed to go dry. While the extracts are concentrating, occasionally rinse down the sides of the receiver or test tube with appropriate solvent (see corresponding test instructions below).

7.6.14.3 Refer to appropriate test below for further concentration instructions.

BNA and 625

7.6.14.5a Once the extracts reach ~0.5 mLs, remove from the N-Evap. Label a 1.5-2 mL screw capped vial with appropriate sample number and batch number. Transfer the 0.5 mLs to the vial using a transfer pipet. Rinse the receiver well with a little (<1/4 mL) DCM and add to the vial. Compare the sample volume in the vial to a pre-marked, 0.9 mL vial. Rinse the receiver with a little more DCM (<1/4 mL). Transfer to sample vial. Compare the volume to the pre-marked 0.9 mL vial. Dilute the sample (if necessary) to 0.9 mLs with another DCM rinsate. The extraction analyst must rinse the receiver at least 2-times and stay at or below 0.9 mLs. The use of a 500 uL syringe gives the analyst more control when adding the small-volume DCM rinses to the receiver. If the rinsates make the extract more than the 0.9 mL, the analyst may hold the vial under the stream of nitrogen momentarily to reduce the volume back down to 0.9 mL. The GC/MS analyst will bring the sample extract to exactly 1.0 mL prior to analysis. Seal vial with a Teflon-lined screw-cap.

There will be samples that will not concentrate to a 1 mL final volume. In these cases, bring the sample up to the smallest final volume (increments of 1 mL)* that the sample and 3 rinses will transfer into. All changes in final volume must be documented LIMS. Notify your supervisor.

* Volumetrically add the appropriate volume of DCM to a 16 X 125 screw top test tube and mark the meniscus using a fine-tipped marker. Discard the DCM into an appropriate waste vessel. Bring up sample to volume using this test tube.

7.6.14.6a All samples pertaining to one extraction batch are placed in a plastic vial holder and secured. A listing of the entire contents of the batch, including clients, sample numbers, batch number, standard numbers, and date extracted, is to be placed WITH the set. To relinquish samples, the extraction analyst takes the sample extracts to the sample extract refrigerator (located in the GC/MS Lab). The TALS ICOC module is used by the prep analyst to electronically transfer custody of the extracts to the storage location.

7.6.14.7a An analyst from the GC/MS BNA group must be present to receive the samples. Both analysts check for completeness and correctness of the information. All extracts are to be kept at 4 $\pm$ 2°C in the dark, until analysis.

PCBs, Pesticide/PCB and 608

7.6.14.4b Concentrate the sample to 2-3 mLs and dilute to the 10.0 mL mark with hexane.

PCBs Refer to mandatory sulfur acid cleanup (Sec. 7.6.16)

7.6.14.5b All samples pertaining to one extraction batch are placed in a test tube rack. Each test tube should have the extraction batch number, sample number, and test code written on it.

7.6.14.6b To relinquish samples, the extraction analyst takes the sample extracts to the sample cooler area (located in the GC Extractables Lab). The TALS ICOC module is used by the prep analyst to electronically transfer custody of the extracts to the storage location. An analyst from the GC Extractables group must be present to receive the samples. Both analysts check for completeness and correctness of the information. All extracts are to be kept at 4 $\pm$ 2°C in the dark, until analysis.

PAHs

7.6.14.4c Concentrate the sample to ~2 mLs to remove any remaining DCM. Dilute the sample up to 5.0 mLs with acetonitrile.

7.6.14.5c All samples pertaining to one extraction batch are placed in a test tube rack. Each test tube should have the extraction batch number, sample number, and test code written on it.

7.6.14.6c To relinquish samples, the extraction analyst takes the sample extracts to the sample cooler area (located in the GC Extractables Lab). The TALS ICOC module is used by the prep analyst to electronically transfer custody of the extracts to the storage location. An analyst from the HPLC group must be present to receive the samples. Both analysts check for completeness and correctness of the information. All extracts are to be kept at 4 ± 2°C in the dark, until analysis.

Diesel/DRO

7.6.14.4d Concentrate the sample to the 5.0 mL mark. If the solvent goes below the 5.0 mL mark, dilute to 5.0 mLs with DCM.

7.6.14.5d All samples pertaining to one extraction batch are placed in a test tube rack. Each test tube should have the extraction batch number, sample #, and test code written on it.

7.6.14.6d To relinquish samples, the extraction analyst takes the sample extracts to the sample cooler area (located in the GC Extractables lab). The TALS ICOC module is used by the prep analyst to electronically transfer custody of the extracts to the storage location. An analyst from the GC Extractables group must be present to receive the samples. Both analysts check for completeness and correctness of the information. All extracts are to be kept at 4 ± 2°C in the dark, until analysis.

7.6.15 Screening and Cleanup

7.6.15.1 Samples are not pre-screened prior to extraction. Unless previous experience has proven high concentrations and/or background, a 1-liter sample will be extracted.

7.6.15.2 Sludge samples are a mixture of water and suspended solid materials. If the percent moisture of a given sludge is > 90%, a 10 g or 30 g portion may be weighed and added to 1-L of reagent water. It is then extracted like a water matrix as described in this procedure. Despite the extraction method, sludges are generally reported on a dry weight basis and the total solids result must be entered into the spreadsheet.

7.6.15.3 PAH samples do not receive any cleanup procedures.

7.6.16 Extract Cleanup by Gel Permeation Chromatography (GPC)

7.6.16.1 It is not mandatory that BNA or Pesticide/PCB samples be subjected to further cleanup steps prior to analysis. However, if it is requested that these sample sets receive a GPC cleanup, refer to the appropriate section of the UP-SP-003.

7.6.16.2 It is not mandatory that Pesticide/PCB samples be subjected to further cleanup steps prior to analysis. However, if it is requested that the Pesticide/PCB set receives a Florisil cleanup, refer to the appropriate sections of UP-SP-003.

7.6.16.3 It is not mandatory that DRO sample be subjected to further cleanup procedures prior to analysis. However, if it is requested that the DRO set receives a Silica Gel cleanup, refer to the appropriate section in UP-SP-003.

7.6.17 Mandatory Acid Cleanup for PCBs

If the sample is to be analyzed for **PCBs ONLY**, a mandatory sulfuric acid cleanup is performed. Add ~3-4 mLs of concentrated H_2SO_4 to the extract (contained in a screw-top test tube). Shake vigorously for a least 1 minute. Centrifuge the hexane/acid for at least a couple minutes to ensure adequate separation. If the acid layer (the bottom layer) is dark in color, transfer the hexane layer (the top layer) to another screw-top test tube and repeat the acid cleanup. The acid cleanup should be repeated until the acid layer no longer turns dark in color. The hexane extract must always be immediately removed from the acid and transferred into another clean, labeled, screw-top test tube. Document the acid cleanup in LIMS. Relinquish the samples as described in the nitrogen evaporation section.

7.7 Documentation

7.7.1 All extraction information must be carefully documented in 'real time' in the LIMS batch and prep worksheet (Attachment 1). Any additional sample or extraction information is recorded in the comment section of the batch sheet. All problems and/or deviations from normal procedures must be documented in LIMS. The supervisor should be notified and a Non-Conformance Memo (NCM) may need to be initiated.

7.7.2 Different parameters are given different test methods within LIMS. Refer to the batch paperwork for the assigned test methods.

8.0 QUALITY CONTROL

8.1 QC Summary

8.1.1 Method Blank (MB)

The MB is reagent (Milli-Q) water, which has been spiked with the appropriate surrogate, then taken through the entire extraction procedure and is used to monitor the introduction of artifacts into the process. This demonstrates that the materials used are free of interferences.

8.1.2 Laboratory Control Sample (LCS)

The LCS is reagent (Milli-Q) water, which has been spiked with a predetermined quantity of spike solution with the appropriate analytes of interest and the appropriate surrogate.

8.1.3 Matrix Spike/Matrix Spike Duplicate (MS/MSD)

In MS/MSD analysis, predetermined quantities of spike solutions of specified analytes and appropriate surrogate are added to a sample matrix prior to sample extraction and analysis. Samples are split into duplicates, spiked and analyzed. Percent recoveries are calculated for each of the analytes detected by the analysis. The relative percent difference between the samples is calculated and used to assess analytical precision.

8.1.4 Surrogate

A surrogate is an organic compound(s) which is similar to the analytes of interest in chemical composition, extraction and chromatography, but which is not normally found in environmental samples. The surrogate is added to **all** samples, MBs, MS/MSD and LCS prior to extraction and analysis. Percent recoveries are calculated for each surrogate.

8.1.5 Sometimes PCBs are extracted in the same extraction batch as Pesticide samples. When this occurs, the QC includes one MB; one LCS/LCSD for the pesticide compounds; one LCS/LCSD for PCB compounds; one MS/MSD for the pesticide compounds; and one MS/MSD for PCB compounds.

8.1.6 LCDs and MSDs are not performed for BNA TCLP.

8.1.7 LCSD and MSDs are not performed for Pesticide TCLP samples. However, an LCS/LCS along with a MS/MS are extracted and recorded. The TCLP Pesticide Spike and Toxaphene Spike need to remain separate from one another.

8.2 Corrective Action

The extraction analyst must perform the Quality Control described in Section 7.1. The extraction analyst will not know if all QC is in control until the GC/MS, GC/Extractables, or HPLC group analyzes the extracts and determine the results of the MBs, MS/MSDs, LCSs, and surrogate recoveries. It is the responsibility of the analysis group to inform the Organic Extractions Department by documenting in the "BNA Re-Extraction Log" or by initiating a Non-Conformance Memo (NCM) in LIMs when a sample or set of samples requires re-extraction. Re-extractions are to be designated as R1 in the extraction process.

All problems and/or deviations from normal procedures must be documented in LIMS. Your supervisor should be notified and a NCM may need to be initiated.

9.0 DATA ANALYSIS AND CALCULATIONS

Not Applicable.

10.0 POLLUTION CONTROL

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

10.1 Waste Management

Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to UP-WM-001.

The following waste streams are produced when this method is carried out.

- Waste flammable solvents. Solvents will be collected in a waste jar and poured into the Waste Chlorinated Solvent drum using a funnel to reduce splashing.
- Extracted water samples will be poured into the Waste Water drum using a funnel to reduce splashing.
- Remaining solid waste produced by this method shall be disposed of into the Non-Hazardous Waste solids containers.
- Any unused extracts remaining from this method shall be turned over to the EHSC to be collected for disposal.

11.0 METHOD PERFORMANCE CRITERIA

Refer to Section 1, 7 and 8.

12.0 REFERENCES

Refer to Sections 1, 6, 7 and 8.

13.0 ATTACHMENTS

Attachment 1. Example: Extraction Prep Worksheet
Attachment 2. Example: Procedural Flowcharts
Attachment 3: Example: BNA Surrogate Stock Solution
Attachment 4: Example: 8270 LCS Mix
Attachment 5: Example: 3,3'-Dimethylbenzidine

Appendix 1: BNA Halowax
Appendix 2: Alachlor/Atrazine
Appendix 3: Wisconsin DRO
Appendix 4: JP4/JP8
Appendix 5: AR1242/AR1254

14.0 REVISION HISTORY

- Revision 11, was updated on 09/21/11
- Annual Review
- Section 7.6.13 was updated to change TurboVap temperature from 45 to 40 degrees C.
- Attachment 1: Extraction Logbook removed, Prep Worksheet added
- Section 7.6.13, 7.6.17 was updated to remove reference to the Extraction logbook.
- Section 7.6.14.5a was updated to change Extraction log to Extraction worksheet.
- Sections 7.6.14.7a, 6b, 6c, 6d were updated to remove reference to the signing of the Extraction log after extract transfer to reference the electronic transfer of extracts via the TALs ICOC module.
- Section 7.7.1 was updated to change reference from 'Extraction log' to 'LIMS batch and prep worksheet.'

Attachment 1.

Example: Extraction Prep Worksheet (026-001 to 026-002)

Aqueous Extraction Prep Worksheet

Used for Collecting Prep Info)

UP-SP-3510

Batch Number: 500-124272 Analyst: Dillman, Jessica Batch Open: 9/1/2011 8:25:00AM

Method Code: 500-625_Prep-500 Batch End: 9/1/2011 10:00:00AM

Batch Notes

Person's name who did the prep	JD	*Extraction*	
Balance ID	*Leave Blank*		
Prep Solvent Name	DCM	*Extraction*	
Prep Solvent Lot #	K26J01	*Extraction*	
Prep Solvent Volume Used	360	*Extraction*	
Person's name who witnessed reagent drop		*Extraction*	
Pipette ID	*Leave Blank*		
Acid used for pH adjustment	*Leave Blank*		
Acid used for pH adjust Lot #	1450356	*Extraction*	
Time the first extraction started 24 hr	*Leave Blank*		
Time the first extraction ended 24hr	*Leave Blank*		
Base used for pH adjustment	*Leave Blank*		
Base used for pH adjust Lot #	1452161	*Extraction*	
Time the second extration started 24 hr	*Leave Blank*		
Person's name who did the concentration	LK,SS	*Concentration*	
Water Bath ID	*Leave Blank*		
Water Bath Temperature	*Leave Blank*		
Exchange Solvent Name		*Concentration*	
Exchange Solvent Lot #		*Concentration*	
Concentration Start Time	0930	*Concentration*	

(C-2) C-530

(026-001)

CHI-22-16-113/A-9/11

Aqueous Extraction Prep Worksheet

Used for Collecting Prep Info)

Batch Number: 500-124272 Analyst: Dillman, Jessica Batch Open: 9/1/2011 8:25:00AM

Method Code: 500-625_Prep-500 Batch End: 9/1/2011 10:00:00AM

Concentration End Time	1100	*Concentration*
Na2SO4 Lot Number	K10624	*Extraction*
Sufficient volume for MS/MSD?	N	*Extraction*
N-evap #	C-0655	*Concentration*
N-evap temperature	29.9	*Concentration*
Uncorrected N-evap Temperature	30	*Concentration*
SOP Number	*Leave Blank*	
Syringe Lot #	A9/B10	*Extraction*
Batch Comment		

(C-2) C-531

Comments

500-38494-M-1

Method Comments:

Login Comments for Job 38543: Do not dilute 625 Custom list more than 10x. See project notes for limits.

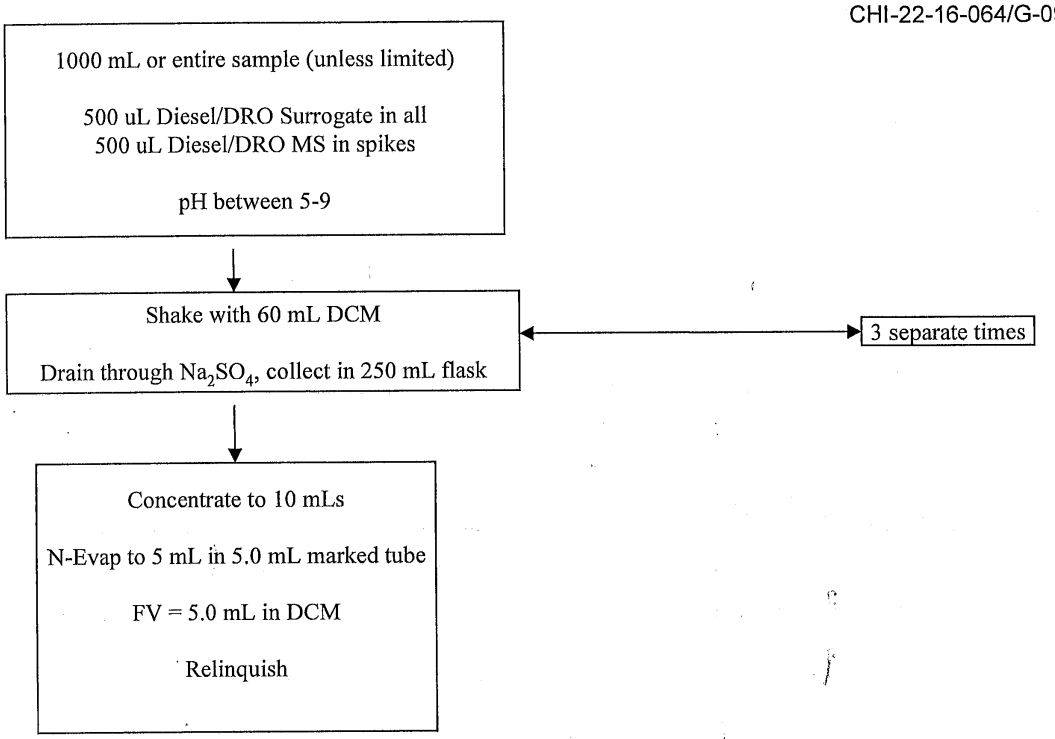
(026-002)

Attachment 2.

Example: Procedural Flowcharts (027-001 to 027-012)

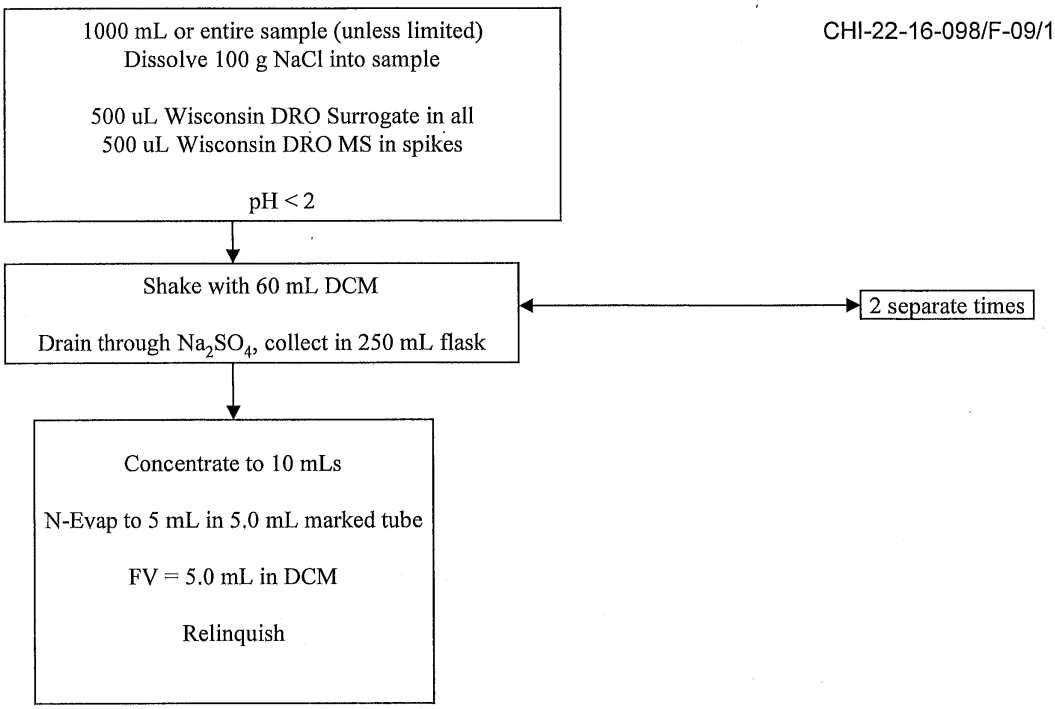
Flowchart for DRO Water (8015)
by 3510

CHI-22-16-064/G-09/11



(027-001)

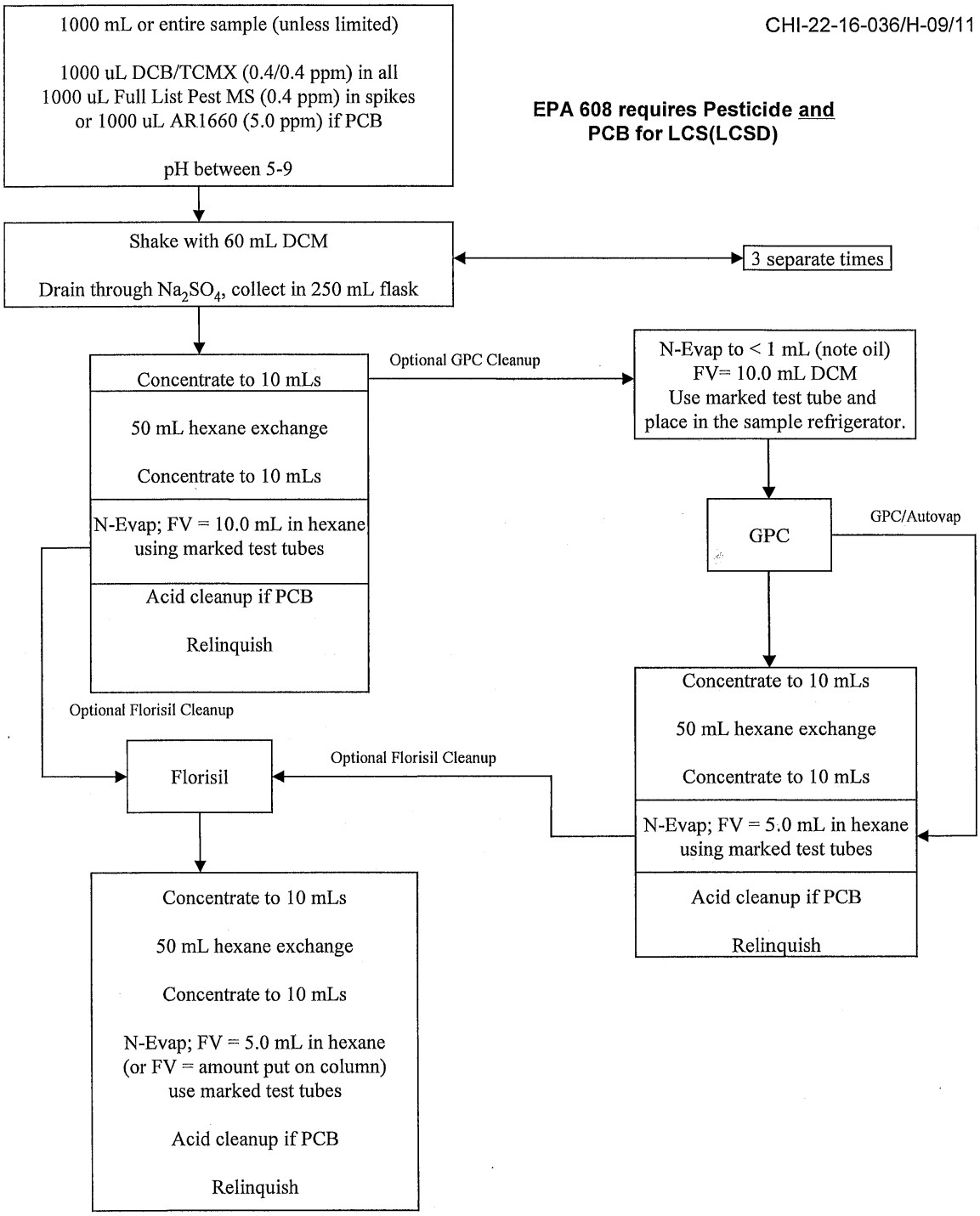
Flowchart for Wisconsin DRO Water by 3510



CHI-22-16-098/F-09/11

(027-002)

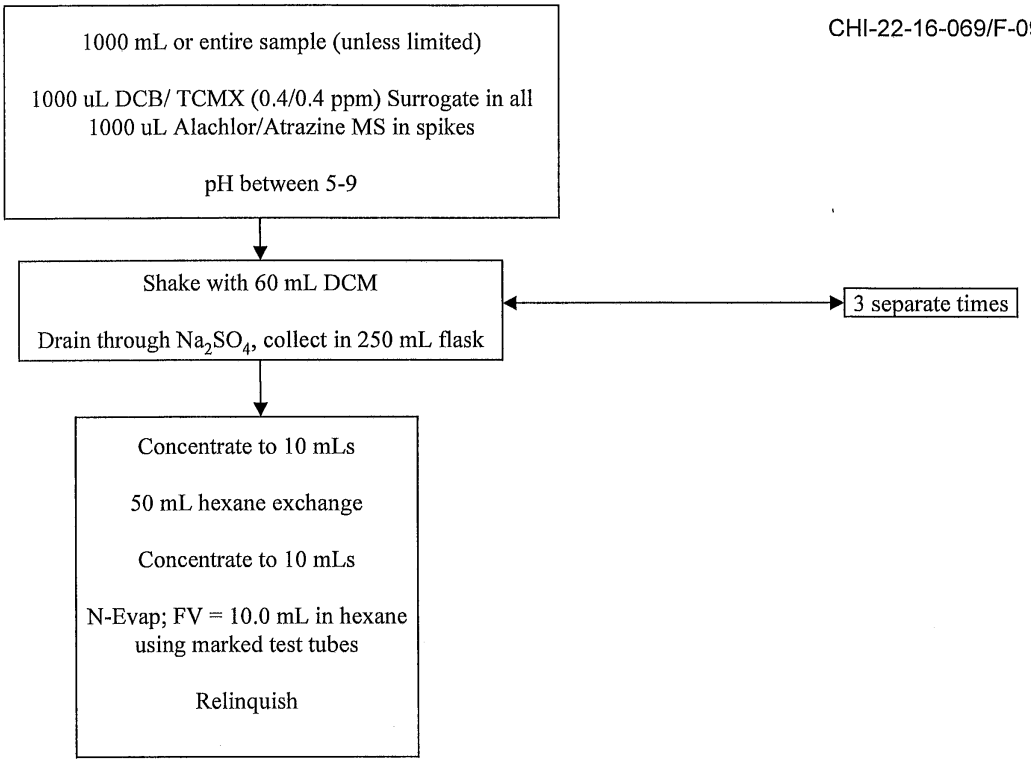
Flowchart for Pest/PCB Water (8081, 8082, 608) by 3510



(027-004)

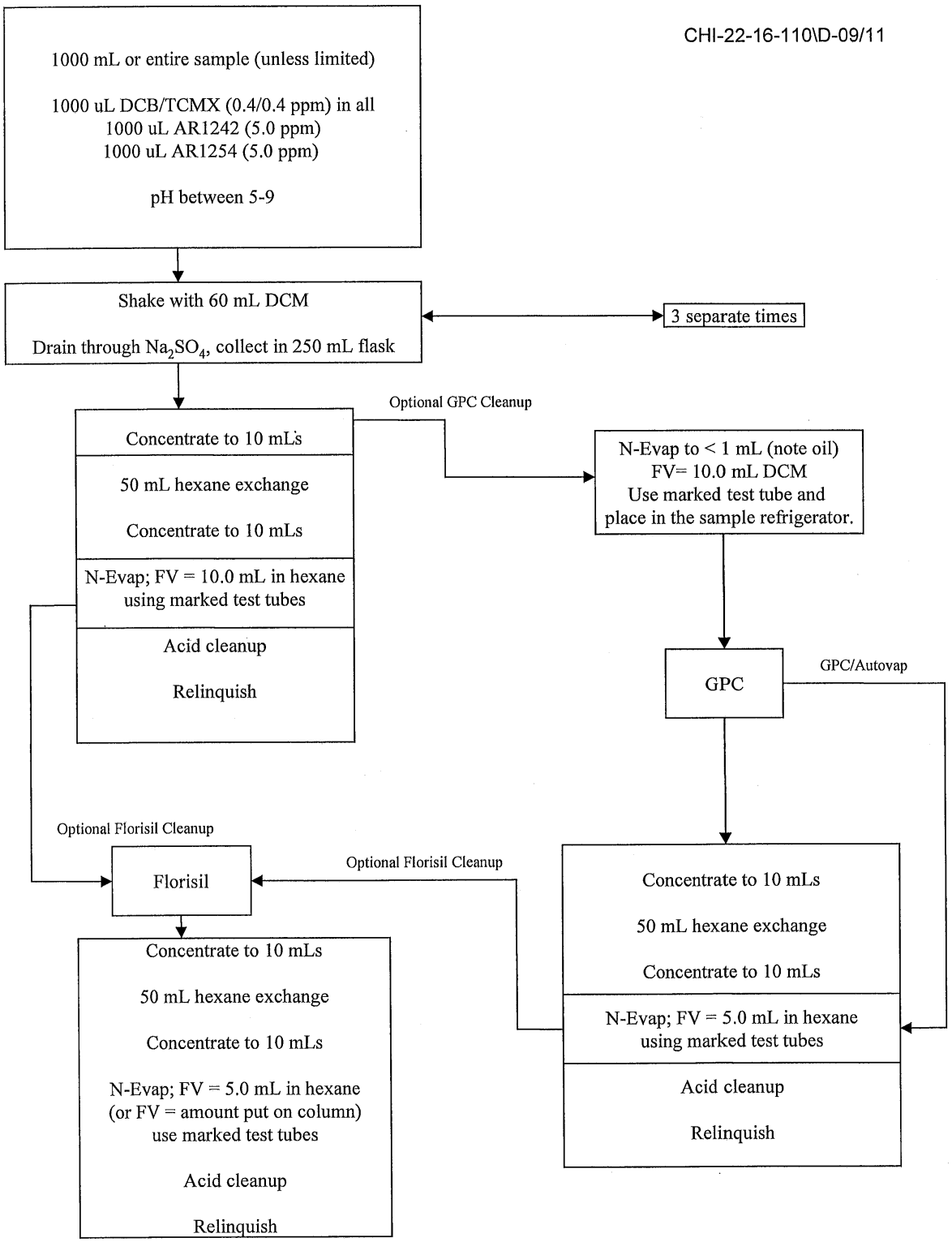
Flowchart for Alachlor/Atrazine Water (8081A) by 3510C

CHI-22-16-069/F-09/11



(027-005)

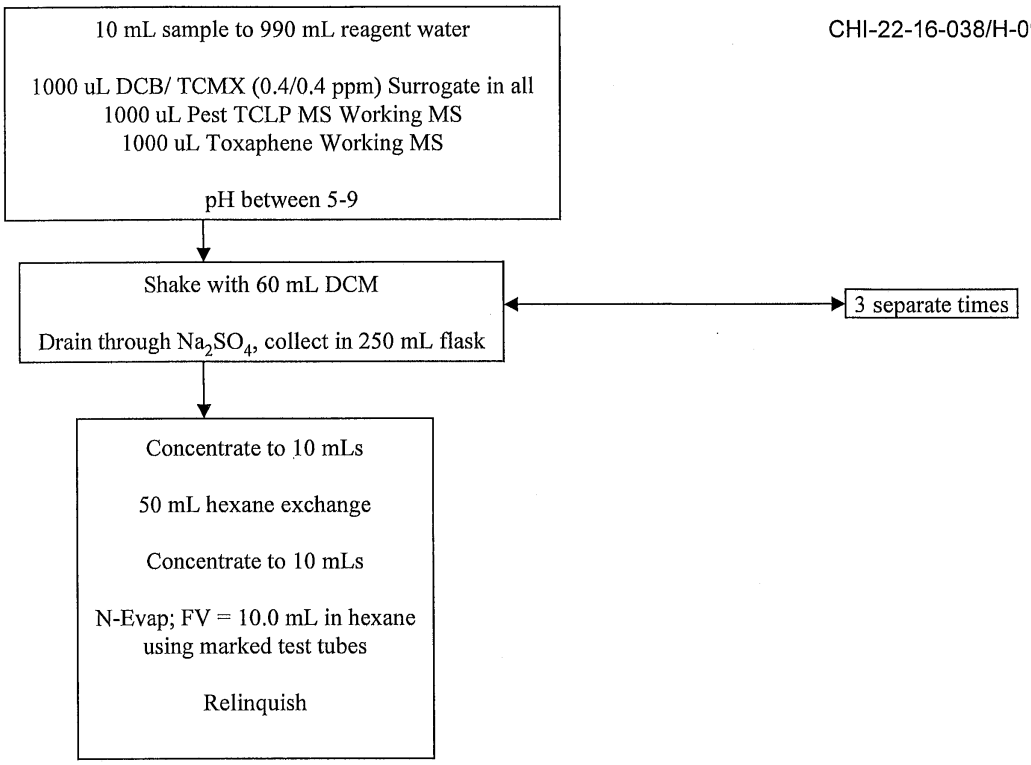
Flowchart for AR1242, AR1254 Water (8082) by 3510



(027-006)

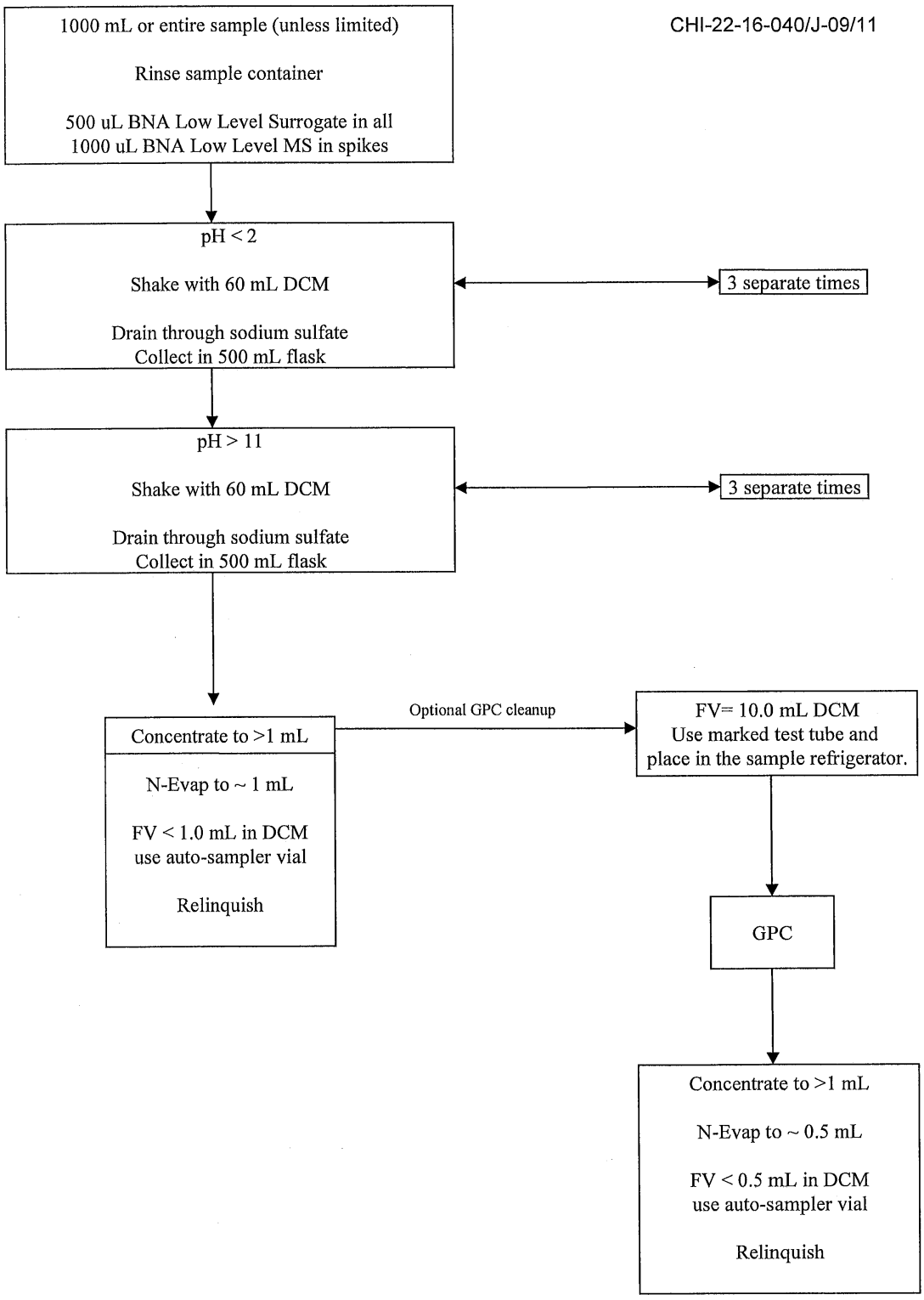
Flowchart for Pest/PCB TCLP by 3510C

CHI-22-16-038/H-09/11



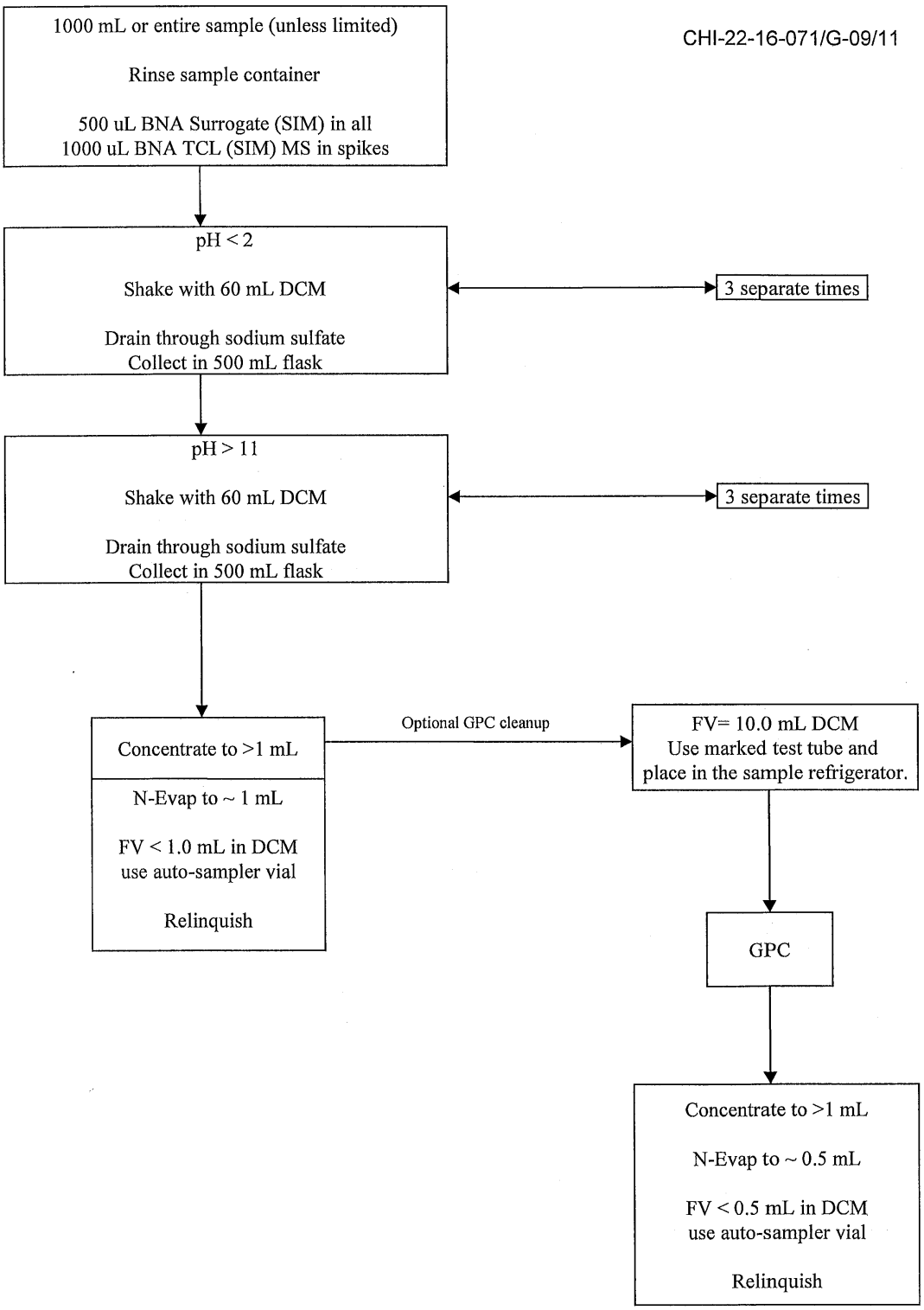
(027-007)

Flowchart for BNA Water (8270C, 625) by 3510C



(027-008)

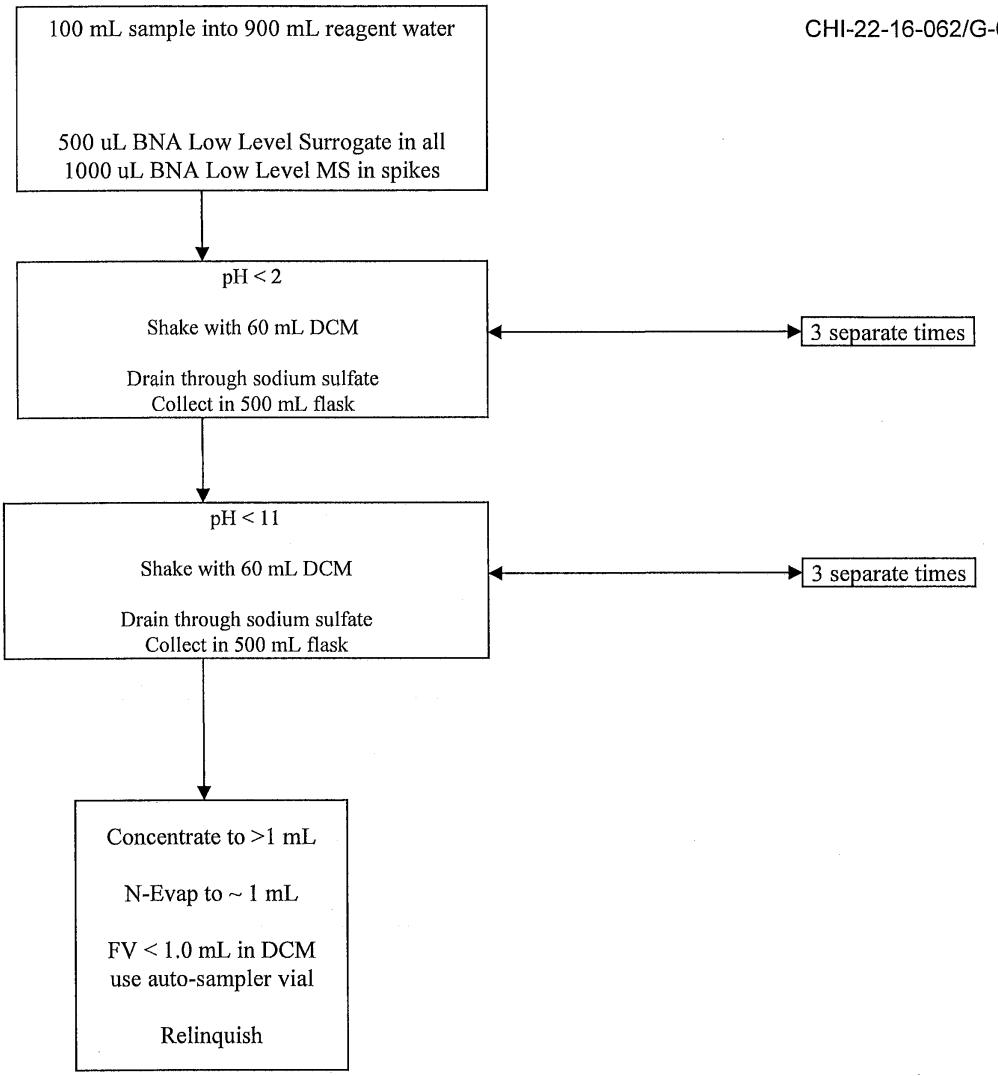
Flowchart for BNA SIM Water by 3510C



(027-009)

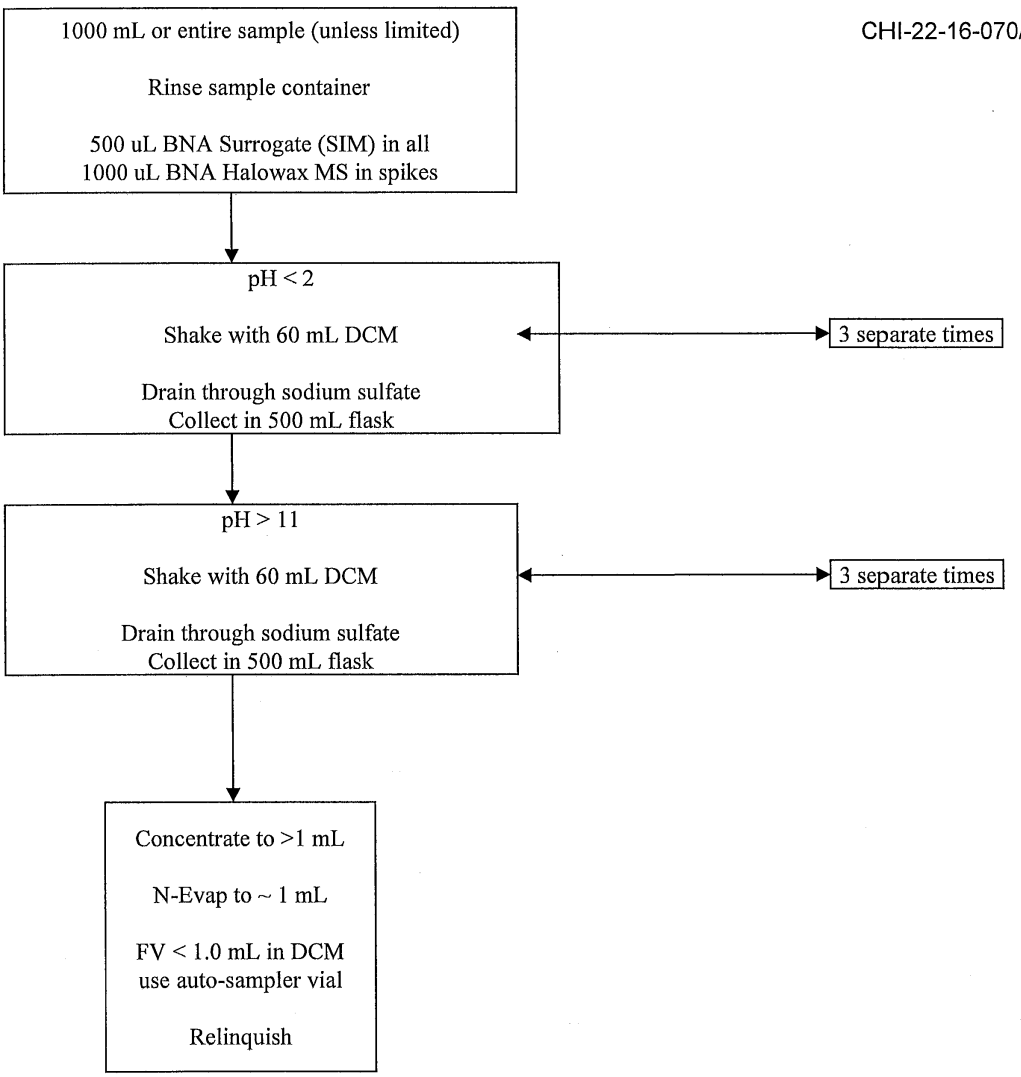
Flowchart for BNA TCLP
by 3510C

CHI-22-16-062/G-09/11



(027-010)

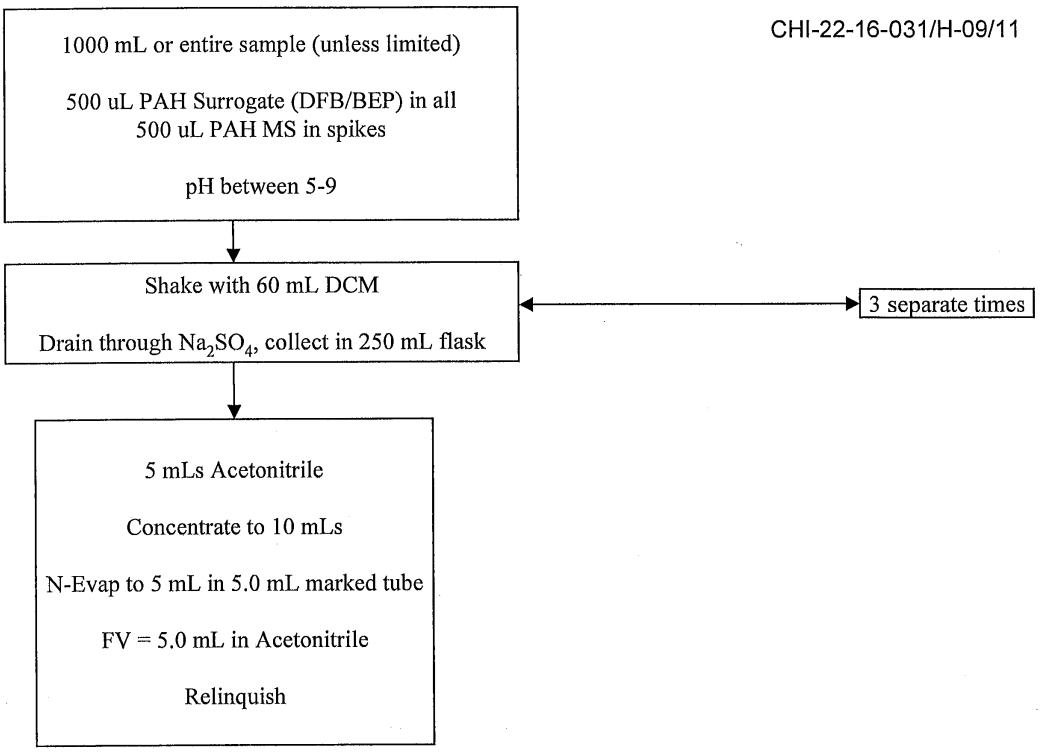
Flowchart for BNA Halowax Water
by 3510C



(027-011)

Flowchart for PAH Water (8310, 610) by 3510

CHI-22-16-031/H-09/11



(027-012)

Attachment 3:

Example: BNA Surrogate Stock Solution (028-001)

Certificate of Composition

1002553
ID: EXBNASUP_00019
Exp: 04/30/13 Prpd: DAK
BNA Surrogate Parent

DESCRIPTION: Semi-Volatile Acid/Base Surrogate Spike (High)

CATALOG NO.: 861142 MFG DATE: Apr-2010

LOT NO.: LB75131 EXPIRATION DATE: Apr-2013

Rec: 05/14/10

SOLVENT: METHYLENE CHLORIDE

ANALYTE (1)	CAS NUMBER	PERCENT PURITY (2)	WEIGHT CONCENTRATION (3)	SUPELCO LOT NO
NITROBENZENE-D5	4165-60-0	99.9	1000	LB47918
P-TERPHENYL-D14	1718-51-0	99.9	1000	LB65558
PHENOL-D6	13127-88-3	99.9	1500	LB72515
1,2-DICHLOROBENZENE-D4	2199-69-1	99.9	1000	LB74053
2-CHLOROPHENOL D4	93951-73-6	99.4	1500	LB74323
2-FLUOROBIPHENYL	321-60-8	99.9	1000	LB68246
2-FLUOROPHENOL	367-12-4	99.9	1500	LB72440
2,4,6-TRIBROMOPHENOL	118-79-6	99.9	1500	LB59603

(1) Listed in alphabetical order.
(2) Determined by capillary GC-FID, unless otherwise noted.
(3) NIST traceable weights are used to verify balance calibration with the preparation of each lot. Concentration of analyte in solution is ug/ml +/- 0.5%, uncertainty based upon balance and Class A volumetric glassware. Weights are corrected for analytes less than 98% pure.

Elwood Doughty
QA Manager

SUPELCO Analytical
595 North Harrison Road • Bellefonte, PA
16823-0048 USA • Phone (814) 359-3441

(028-001)

Attachment 4:

Example: 8270 LCS Mix
(029-001 to 029-004)

Cerilliant
Analytical Reference Standards

ERS-077
ER112008-02
Revision 1

Certificate of Analysis

8270 LCS Mix

1002554
ID: EXBNASPP_00053
Exp: 11/30/13 Prpd: DAK
8270 LCS Mix Parent

Rec: 05/14/10

Catalog Number:	ERS-077
Solution Lot:	ER112008-02
Expiration Date:	November 2013
Solvent:	Methanol::Methylene chloride::Benzene (70::25::5)
Amount per Ampule:	5 mL
Storage:	Protect from light, store in freezer.
Handling:	Sonicate for 5 minutes before use. We advise laboratories to use measured volumes of this standard solution before diluting to the desired concentration.
Intended Use:	For laboratory use only. Not suitable for human or animal consumption.

* *All analyte data can be found on the following pages.*

Cerilliant certifies that this standard meets or exceeds the specifications stated in this data sheet. Accuracy is ensured by purity determinations and gravimetric preparation using balances calibrated with NIST traceable weights. Precision is guaranteed by triplicate analysis and comparison to previous lots (when available). Homogeneity is demonstrated by random analysis of the ampuled standard.

Authorized Signature: Lara Sparks

Lara Sparks, Quality Assurance Director

April 15, 2009

Date

Cerilliant Corporation 811 Paloma Drive, Suite A, Round Rock, TX 78665 800-848-7837 / 512-238-9974

(029-001)

Component	Lot Number	Chromatographic Purity	Concentration [1]
Acenaphthene	29697-41	99%	200.0 µg/mL
Acenaphthylene	ER030707-01	99%	200.0 µg/mL
Acetophenone	EB-1807	99%	200.0 µg/mL
Aniline	EB-1265	99%	200.0 µg/mL
Anthracene	ER061407-04	99%	200.0 µg/mL
Azobenzene	P004554	99%	200.0 µg/mL
Benz(a)anthracene	ER121707-01	99%	200.0 µg/mL
Benzidine [3]	PR081108-03	99%	200.0 µg/mL
Benzo(b)fluoranthene	ER022008-02	99%	200.0 µg/mL
Benzo(k)fluoranthene	ER061608-02	99%	200.0 µg/mL
Benzo(g,h,i)perylene	ER020708-08	99%	200.0 µg/mL
Benzo(a)pyrene	ER050707-01	99%	200.0 µg/mL
Benzoic acid	EB-1766	99%	200.0 µg/mL
Benzyl alcohol	EB-1963	99%	200.0 µg/mL
Biphenyl	PER_053_97109	99%	200.0 µg/mL
Bis(2-chloroethoxy)methane	P004590	98%	200.0 µg/mL
Bis(2-chloroethyl)ether	PER_456_102708	99%	200.0 µg/mL
Bis(2-chloroisopropyl)ether	P004719	99%	200.0 µg/mL
Bis(2-ethylhexyl) phthalate	P004582	99%	200.0 µg/mL
4-Bromophenyl phenyl ether	P004550	99%	200.0 µg/mL
Butyl benzyl phthalate	EB-2108	99%	200.0 µg/mL
Carbazole	P004638	95%	200.0 µg/mL
p-Chloroaniline	EB-1801	99%	200.0 µg/mL
4-Chloro-3-methylphenol	PR060807-01	99%	200.0 µg/mL
2-Chloronaphthalene	P004640	99%	200.0 µg/mL
2-Chlorophenol	PR060807-02	99%	200.0 µg/mL
4-Chlorophenyl phenyl ether	P004659	98%	200.0 µg/mL
Chrysene	ER081006-02	99%	200.0 µg/mL
Dibenz(a,h)anthracene	ER091206-01	98%	200.0 µg/mL
Dibenzofuran	P004552	99%	200.0 µg/mL
Di-n-butyl Phthalate	P004541	99%	200.0 µg/mL
1,2-Dichlorobenzene	EB-2097	99%	200.0 µg/mL
1,3-Dichlorobenzene	EB-1908	99%	200.0 µg/mL
1,4-Dichlorobenzene	PER_226_104287	99%	200.0 µg/mL
3,3'-Dichlorobenzidine	ER083107-03	99%	200.0 µg/mL
2,4-Dichlorophenol	P004712	99%	200.0 µg/mL
2,6-Dichlorophenol	P004533	100%	200.0 µg/mL
Diethyl Phthalate	PER_096_102706	99%	200.0 µg/mL
2,4-Dimethylphenol	P004557	99%	200.0 µg/mL
Dimethyl Phthalate	EB-1774	100%	200.0 µg/mL
2,4-Dinitrophenol	PR062607-03	99%	200.0 µg/mL
2,4-Dinitrotoluene	EB-1379	99%	200.0 µg/mL
2,6-Dinitrotoluene	PER_083_93401	99%	200.0 µg/mL
Dinoseb	ER042903-01	99%	200.0 µg/mL
Di-n-octyl Phthalate	P004648	99%	200.0 µg/mL
Diphenyl ether	P004715	99%	200.0 µg/mL
1,4-Dioxane	PR102607-09	99%	200.0 µg/mL
2-Ethoxyethanol	PER_052_102546	99%	200.0 µg/mL
Fluoranthene	ER060506-01	99%	200.0 µg/mL

Component	Lot Number	Chromatographic Purity	Concentration [1]
Fluorene	35001-29	99%	200.0 µg/mL
Hexachlorobenzene	P004546	99%	200.0 µg/mL
Hexachloro-(1,3)-butadiene	P141819	97%	200.0 µg/mL
Hexachlorocyclopentadiene	EB-2076	96%	200.0 µg/mL
Hexachloroethane	EB-2003	99%	200.0 µg/mL
Hexachlorophene [3]	P004584	95%	200.0 µg/mL
Hexachloropropene	EB-1977	97%	200.0 µg/mL
Indeno(1,2,3,-c,d)pyrene	ER082107-02	99%	200.0 µg/mL
Isophorone	EB-1778	98%	200.0 µg/mL
2-Methyl-4,6-dinitrophenol	P004549	99%	200.0 µg/mL
2-Methylnaphthalene	EB-1779	97%	200.0 µg/mL
2-Methylphenol	PR061107-01	99%	200.0 µg/mL
4-Methylphenol	PER_016_99798	99%	200.0 µg/mL
Naphthalene	22599-76	99%	200.0 µg/mL
2-Nitroaniline	EB-1781	99%	200.0 µg/mL
3-Nitroaniline	P004543	99%	200.0 µg/mL
4-Nitroaniline	EB-1796	99%	200.0 µg/mL
Nitrobenzene	PER_117_93604	99%	200.0 µg/mL
2-Nitrophenol	PR060807-03	99%	200.0 µg/mL
4-Nitrophenol	P004713	99%	200.0 µg/mL
N-Nitrosodi-n-butylamine	P004651	99%	200.0 µg/mL
N-Nitrosodiethylamine	P004558	99%	200.0 µg/mL
N-Nitrosodimethylamine	P004569	99%	200.0 µg/mL
N-Nitrosodi-n-propylamine	P004632	99%	200.0 µg/mL
N-Nitrosodiphenylamine	PR102606-01	97%	200.0 µg/mL
N-Nitrosomethylethylamine	P004718	99%	200.0 µg/mL
N-Nitrosopiperidine	P004545	99%	200.0 µg/mL
N-Nitrosopyrrolidine	P004551	99%	200.0 µg/mL
Pentachlorobenzene	P004714	99%	200.0 µg/mL
Pentachloroethane	EB-1954	99%	200.0 µg/mL
Pentachlorophenol	P004716	99%	200.0 µg/mL
Phenanthrene	ER042805-02	99%	200.0 µg/mL
Phenol	PR061107-02	99%	200.0 µg/mL
Pyrene	ER050305-03	98%	200.0 µg/mL
Pyridine	PER_039_102530	99%	200.0 µg/mL
1,2,4,5-Tetrachlorobenzene	EB-1955	99%	200.0 µg/mL
2,3,4,6-Tetrachlorophenol	ER100206-01	99%	200.0 µg/mL
1,2,4-Trichlorobenzene	EB-1677	99%	200.0 µg/mL
2,4,5-Trichlorophenol	PR040307-02	98%	200.0 µg/mL
2,4,6-Trichlorophenol	P004555	99%	200.0 µg/mL

[1] The range of the prepared concentration is determined by statistical process control of our production and analysis systems with a 95% confidence.

[2] Concentration values are determined by comparison to an independent calibration curve. We suggest using the prepared concentration value for dilutions. The concentration range is calculated from the distribution of multiple analyses of the new standard with a 95% degree of confidence.

[3] Due to stability problems, it is recommended not to use this analyte for quantitation.

Standard Solution Assay Parameters

Analysis Method:	GC/MS	**Calibration Curve:**	Linear Regression
Column:	DB-5ms, 30 m x 0.25 mm ID, 0.25 µm film thickness	**Number of Points:**	1
Temp Program:	40°C (hold 4 mins) to 270°C at 10°C/min (hold 11 mins)		
Injector Temp:	Cool on-column		
Detector Temp:	280°C	*Each point analyzed in triplicate*	

Standard Solution Comparability and Homogeneity

Compound	*Mean Concentration (µg/mL)* [2]	*E. M. L % RSD*	*% Difference from Target*	*% Difference from Existing*
Acenaphthene	202.1	4.2	1.1	0.7
Benzo(a)pyrene	203.8	4.6	1.9	2.4
Benzyl alcohol	199.2	2.7	-0.4	-0.5
Bis(2-ethylhexyl)phthalate	202.9	4.7	1.5	0.7
Carbazole	202.6	4.8	1.3	0.1
4-Chloroaniline	204.2	3.9	2.1	6.8
4-Chloro-3-methylphenol	204.5	4.5	2.2	-1.3
Dibenzofuran	203.2	4.3	1.6	-0.4
1,4-Dichlorobenzene	202.1	3.4	1.0	-1.1
3,3'-Dichlorobenzidine	199.3	3.0	-0.3	1.6
2,4-Dichlorophenol	203.5	4.5	1.7	-2.2
2,4-Dinitrotoluene	201.0	4.4	0.5	1.4
Di-n-octyl-phthalate	204.9	4.6	2.5	0.0
Fluoranthene	202.4	4.4	1.2	2.8
Hexachloro-(1,3)-butadiene	204.3	4.4	2.2	0.3
Isophorone	204.4	4.7	2.2	9.9
2-Methylnaphthalene	203.8	3.8	1.9	-1.4
3-Nitroaniline	203.3	4.2	1.7	0.3
2-Nitrophenol	203.7	4.7	1.8	-1.0
Nitrobenzene	200.6	2.9	0.3	-0.9
N-Nitrosodi-n-butylamine	204.7	4.3	2.4	-0.2
N-Nitrosodiphenylamine	202.8	4.4	1.4	3.4
Pentachlorobenzene	203.3	4.5	1.6	-0.7
Pentachlorophenol	199.3	5.0	-0.4	-4.4
Phenol	201.9	2.2	0.9	-5.9
Pyridine	198.9	3.0	-0.5	-0.7

This standard was prepared from six stocks that have been verified for accuracy and homogeneity. Verification of this standard was performed by monitoring 26 components listed in the table above, including the Method 8270 Calibration Check Compounds (CCC) and at least one analyte from each stock standard.

Attachment 5:

Example: 3,3'-Dimethylbenzidine
(030-001 to 030-003)

Cerilliant
Analytical Reference Standards

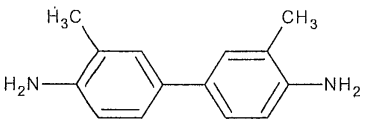
ERD-132S
ER061108-02
Revision 0

1002565
ID: EXDMBSPP_00047
Exp: 06/30/11 Prpd: DAK
BNA LCS 3,3'-Dimethylbenz

Rec: 05/14/10

Certificate of Analysis

3,3'-Dimethylbenzidine



Catalog Number:	ERD-132S
Solution Lot:	ER061108-02
Expiration Date:	June 2011
Solvent:	Methylene chloride
Amount per Ampule:	1.2 mL
Storage:	Protect from light, store in freezer.
Handling:	We advise laboratories to use measured volumes of this standard solution before diluting to the desired concentration.
Intended Use:	For laboratory use only. Not suitable for human or animal consumption.

Component	Chromatographic Purity[1]	Prepared Concentration[2]	Analyzed Concentration[3]
3,3'-Dimethylbenzidine	99%	2000 ± 62 µg/mL	1932 ± 10 µg/mL

Standard Solution Comparability

Standard Solution	*Lot Number*	*Concentration[3] (µg/mL)*	*% Difference from Target*
New Lot	ER061108-02	1932	-3.4
Previous Lot	ER042006-01	1887	-5.7

Standard Solution Homogeneity

Ampuling Position	*Concentration[3] (µg/mL)*	*Mean*	*% RSD*
Early	1924		
Middle	1943		
Late	1928	1932	0.5

[1] See following pages for more information.

[2] The prepared concentration is corrected for chromatographic purity, residual water, and residual solvents (see data on following pages). The range of the prepared concentration is determined by statistical process control of our production and analysis systems with a 95% confidence.

[3] Concentration values are determined by comparison to an independent calibration curve. We suggest using the prepared concentration value for dilutions. The concentration range is calculated from the distribution of multiple analyses of the new standard with a 95% degree of confidence.

Cerilliant certifies that this standard meets or exceeds the specifications stated in this data sheet. Accuracy is ensured by purity determinations and gravimetric preparation using balances calibrated with NIST traceable weights. Precision is guaranteed by triplicate analysis and comparison to previous lots (when available). Homogeneity is demonstrated by random analysis of the ampuled standard.

Authorized Signature: Lara Sparks

Lara Sparks, Quality Assurance Director

July 15, 2008

Date

Cerilliant Corporation 811 Paloma Drive, Suite A, Round Rock, TX 78665 800-848-7837 / 512-238-9974

(030-001)

Standard Solution Assay Parameters

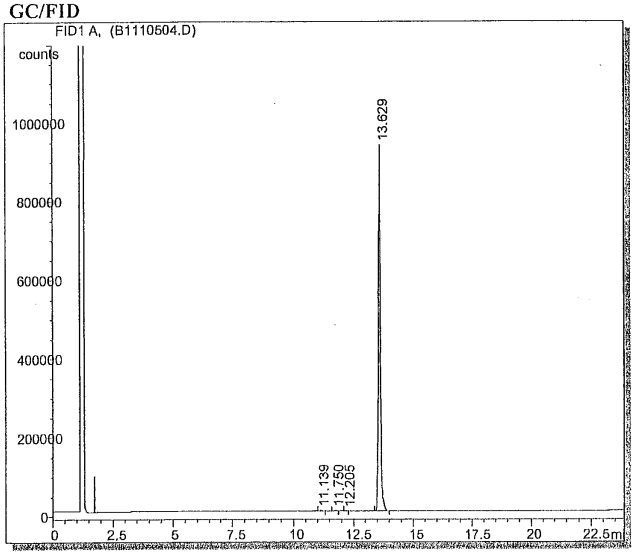
Analysis Method:	GC/FID	**Calibration Curve:**	Linear Regression
Column:	DB-5ms 30 m x 0.53 mm ID, 1.5 µm film thickness	**Number of Points:**	4
Temp Program:	60°C to 280°C at 40°C/min hold 7 min	**Linearity (r):**	0.999
Injector Temp:	Cool-on-Column		
Detector Temp:	325°C		

Neat Material Data

Compound Name:	3,3'-Dimethylbenzidine	**Chemical Formula:**	$C_{14}H_{16}N_2$
Compound Lot:	PER_015_93442	**CAS Number:**	119-93-7
Chromatographic Purity:	99%	**Molecular Weight:**	212.29

Spectral and Physical Data

GC/FID



Column:	DB-5ms, 30 m x 0.53 mm ID, 1.5 µm film thickness
Temp Program:	40°C to 200°C at 40°C/min
	200°C to 245°C at 5°C/min
	245°C to 280°C at 2°C/min hold 10 min
Injector Temp:	Cool-on-Column
Detector Temp:	325°C
Data File Name:	C:\HPCHEM\1\DATA\B1110504.D
Operator:	RPC
Instrument:	GC #2
Sample Name:	PER_015_93442
Method File:	PER064.M
Acquired:	November 10, 2005 10:59 AM

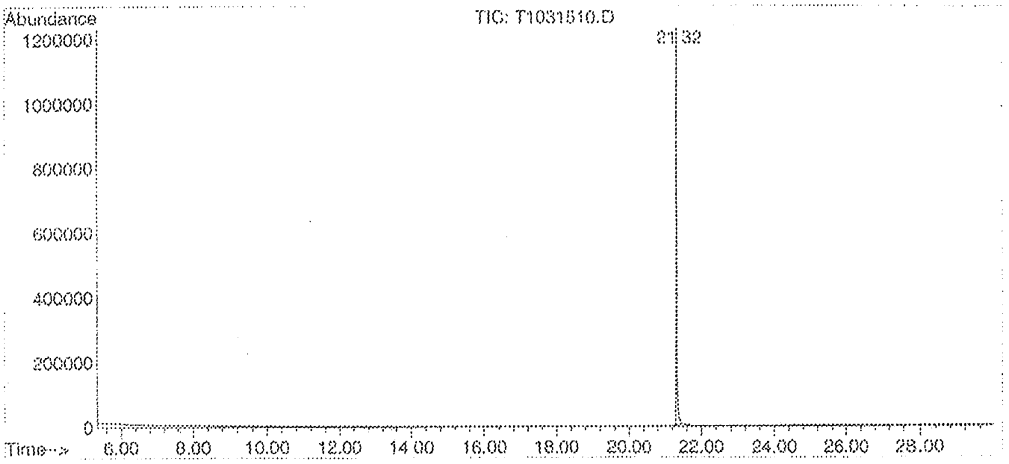
Peak #	Ret Time	Area	Height	Area %
1	11.14	9242	2023	0.17
2	11.75	5806	1325	0.11
3	12.21	805	218	0.02
4	13.63	5332500	930653	99.70

(C 30-002)

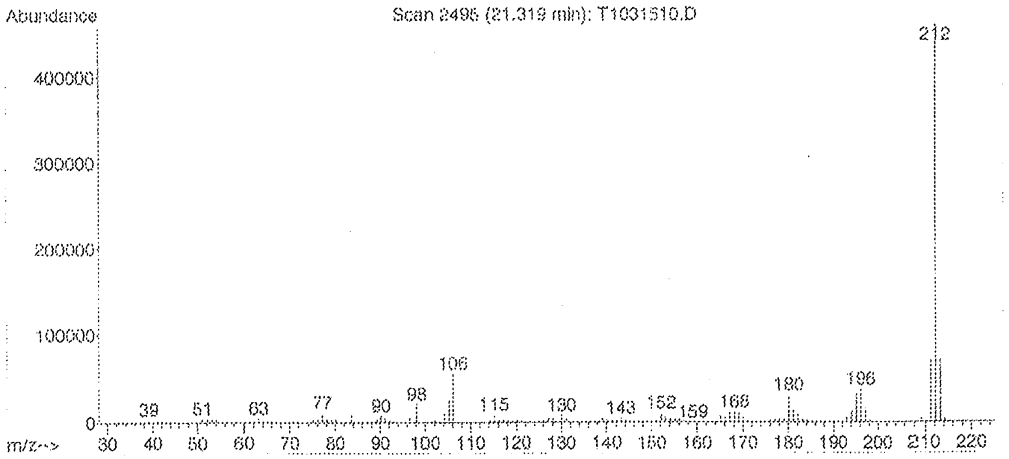
Compound Name : 3,3'-Dimethylbenzidine
Lot Number : PER_015_93442
Instrument : Agilent 5973N MSD/6890N GC
Operator-Inst ID : DMH - C10106
Date Reported : Wed Jul 19 16:42:39 2006
Column Type : DB-5ms, 30m x 0.25mm ID, 0.25um film thickness
Temp. Program : 50°C to 300°C @ 10°C/min, 5min hold
Injector Temp. : Cool on-column
Carrier Gas : Helium
Flow Rate (mL\min) : 0.80 mL/min
Transfer Line Temp. : 280°C

Scan Range : 35-600

Total Ion Chromatogram



Mass Spectrum



(030-003)

Appendix 1

BNA Halowax

Other than having different standards, the BNA Halowax extraction is identical to the BNA separatory funnel procedure.

1.0 Surrogate and Matrix Spike Solutions

1.1 BNA Surrogate Spike (SIM) Solution

The surrogate used for the halowax extraction is the same solution used for the BNA SIM extraction. The BNA Surrogate Spike (SIM) Solution is prepared from the BNA Surrogate Spike at the levels listed below. 500 uLs of BNA Surrogate (SIM) Spike Solution is added to all samples, MBs, LCS and MS/MSDs.

Compound	Concentration (ug/mL)
p-Terphenyl-d_4	10
Nitrobenzene-d_5	10
2-Fluorobiphenyl	10
1,2-Dichlorobenzene-d_4	10
Phenol-d_5	15
2-Fluorophenol	15
2,4,6-Tribromophenol	15
2-Chlorophenol-d_4	15

- Life of Standard: 6-months from the preparation date (as documented from the vendor).
- Storage Requirements: As stated in Section 5.2

1.2 BNA Halowax Matrix Spike Solution

The BNA Halowax Matrix Spike Solution is prepared by the BNA analytical group. The composition and concentration of this standard is beyond the scope of this SOP. See the analytical SOP for specifics about this standard. 1000 uLs of the matrix spike solution is added to the LCS and MS/MSD.

- Life of Standard: 6-months from the preparation date (as documented from the vendor).
- Storage Requirements: As stated in Section 5.2

Appendix 2

Alachlor/Atrazine

Other than having different standards, the Alachlor/Atrazine extraction is identical to the pesticide separatory funnel procedure.

1.0 Surrogate and Matrix Spike Solutions

1.1 Pesticide/PCB Surrogate Spike Solution

The surrogate used for the Alachlor and Atrazine extraction is the same solution used for the pesticides/PCBs extraction. The surrogate used in the extraction of pesticides/PCBs from waters is a solution of Decachlorobiphenyl (DCB) and 2,4,5,6-Tetrachloro-m-xylene (TCX) in acetone at the concentrations listed below. 1,000 uLs of Pesticide/PCB Surrogate Spike Solution is added to all samples, MBs, LCS and MS/MSD.

Compound	Concentration (ug/mL)
DCB	0.4
TCX	0.4

- Life of Standard: Same as parent solution.
- Storage Requirements: As stated in Section 5.2.

1.2 Alachlor/Atrazine Matrix Spike Solution

The LCS/MS solution used in the extraction Alachlor and Atrazine from waters is a solution in methanol. The composition and concentration of this standard is beyond the scope of this SOP. See the analytical SOP for specifics about this standard. 1,000 uLs of Alachlor/Atrazine Spike Solution is added to LCS and MS/MSD.

- Life of Standard: Same as parent solution.
- Storage Requirements: As stated in Section 5.2.

Appendix 3

Wisconsin DRO

In addition using specific surrogate and spike solutions, the Wisconsin DRO extraction differs from the SW-846 DRO extraction. The changes are noted in this appendix.

1.0 Reagents

1.1 Heat-Purified Sodium Chloride

Sodium chloride purchased directly from vendor. Purify by heating at 400°C for four hours in a shallow drying tray, cool in a desiccator, and store in a glass bottle.

- Life of Reagent: 1-year.
- Storage Requirements: Store in a glass container and keep dry (anhydrous).

2.0 Surrogate and Matrix Spike Solutions

2.1 Wisconsin DRO Surrogate Solution

The Wisconsin DRO surrogate solution is prepared by the DRO analyst at the levels listed below. 500 uLs of the surrogate is added to all samples, MBs, LCS/LCD and MS/MSDs.

Compound	Concentration (ug/mL)
C9-Nonane	100

- Life of Standard: 6-months from the preparation date (as documented from the vendor).
- Storage Requirements: As stated in Section 5.2

2.2 Wisconsin DRO Matrix Spike Solution

The Wisconsin DRO Matrix Spike Solution is prepared by the DRO analyst. The composition and concentration of this standard is beyond the scope of this SOP. See the analytical SOP for specifics about this standard. 500 uLs of the matrix spike solution is added to the LCS/LCD and MS/MSD.

- Life of Standard: 6-months from the preparation date (as documented from the vendor).
- Storage Requirements: As stated in Section 5.2

3.0 Wisconsin DRO Extraction

3.1 Clarification of section MB and LCS water preparation in **7.6.1**. Add 1.0 liter of Milli-Q water to 3 separatory funnels and label as the MB, LCS and LCD. A LCD must be extracted even if there is a MS/MSD.

3.2 Add to section **7.6.3**. Samples should be preserved in the field. If sample pH is greater than two, sample results must be flagged.

3.3 Add to section **7.6.4**. Add 100 g NaCl to separtory funnel, then shake. Ensure that the salt is dissolved into sample before adding surrogate and spike.

3.4 In section **7.6.11**, repeat the extraction only once more for a total of two, 60 mL extractions.

3.5 Make the following changes in section **7.6.12.1**. Use the 10 mL insulated concentrator tubes. The K-D bath should be at 70° C.

3.6 In section **7.6.12**, add rinse a small amount of DCM through the Snyder column before removing it from the K-D flask.

3.7 Refer to Diesel/DRO section in **7.6.14**.

Appendix 4

JP4 and JP8

Other than having different standards, the JP4 or JP8 extraction is identical to the DRO separatory funnel procedure.

1.0 Surrogate and Matrix Spike Solutions

1.1 DRO Surrogate Spike Solution

The surrogate used for the JP4 or JP8 extraction is the same solution used for the DRO extraction. The surrogate used in the extraction of DRO from waters is a solution of 2-Fluorobiphenyl and o-Terphenyl in acetone at the concentrations listed below. The GC group prepares the working surrogate solution. 500 uLs of DRO Surrogate Spike Solution is added to all samples, MBs, LCS and MS/MSD.

Compound	Concentration (ug/mL)
2-Fluorobiphenyl	100
o-Terphenyl	100

- Life of Standard: Same as parent solution.
- Storage Requirements: As stated in Section 5.2.

1.2 JP4 Matrix Spike Solution

The LCS/MS solution used in the extraction of JP4 from waters is a solution in methanol. The composition and concentration of this standard is beyond the scope of this SOP. See the analytical SOP for specifics about this standard. 500 uLs of JP4 Spike Solution is added to LCS and MS/MSD.

- Life of Standard: Same as parent solution.
- Storage Requirements: As stated in Section 5.2.

1.2 JP8 Matrix Spike Solution

The LCS/MS solution used in the extraction of JP8 from waters is a solution in methanol. The composition and concentration of this standard is beyond the scope of this SOP. See the analytical SOP for specifics about this standard. 500 uLs of JP8 Spike Solution is added to LCS and MS/MSD.

- Life of Standard: Same as parent solution.
- Storage Requirements: As stated in Section 5.2.

Appendix 5

AR1242 and AR1254 PCBs

Other than having different matrix spike standards, the AR1242 and AR1254 extraction is identical to the PCB water extraction.

1.0 Matrix Spike Solutions

These two matrix spike solutions need to be used separately. Samples batches will contain an LCS/MS/MSD for AR1242 and an LCS/MS/MSD for AR1254.

1.1 AR1242 Spike Solution

The LCS/MS solution used in the extraction of PCBs from soil/sediments is a solution of Arochlor 1242 in methanol at the following concentrations. 1000 uLs of PCB Spike Solution is added to the LCS and MS/MSD.

Compound	Concentration (ug/mL)
AR 1242	5.0

- Life of Standard: Same as parent solution.
- Storage Requirements: As stated in Section 5.2.

1.1 AR1254 Spike Solution

The LCS/MS solution used in the extraction of PCBs from soil/sediments is a solution of Arochlor 1254 in methanol at the following concentrations. 1000 uLs of PCB Spike Solution is added to the LCS and MS/MSD.

Compound	Concentration (ug/mL)
AR 1242	5.0

- Life of Standard: Same as parent solution.
- Storage Requirements: As stated in Section 5.2.

TITLE: Wet Chemistry
Inorganic Ions by Ion Chromatography

Approvals (Signature/Date):

Carla Bonner 11/30/10
Carla Bonner Date
Supervisor, Wet Chemistry

Diane L. Harper 11/30/10
Diane L. Harper Date
Inorganics Manager

Terese A. Preston 11/30/10
Terese A. Preston Date
Quality Assurance Manager

11/30/10
John D. Nagel Date
Env. Health & Safety Coor.

Michael J. Healy 11/30/10
Michael J. Healy Date
Laboratory Director

Facility Distribution No: _____ **Distributed To:** Uncontrolled

1.0 SCOPE / APPLICATION

This Standard Operating Procedure (SOP) defines the procedure for determining the following inorganic anions by Ion Chromatography: Fluoride, Chloride, Nitrite-N, Bromide, Nitrate-N, Ortho-Phosphate-P, and Sulfate. This SOP was written using EPA Method 300.0, revision 2.1; SW-846, 3rd Edition, Method 9056; and SW 846, Update IV, Method 9056A as references.

On occasion, clients request slight modifications to this SOP. These modifications are addressed on a case-by-case basis with the range of accuracy (i.e., MDLs, linearity check or PT sample) verified prior to implementation. Any modifications would be written into a Quality Assurance Plan (QAP), authorized via laboratory signature approval, and mentioned in the data package's case narrative.

Specific requirements pertaining to the DoD QSM Version 4.2 are located in Attachment 7. These requirements are additionally applicable to all NFESC projects. Any deviations from these procedures and/or variances from must be addressed appropriately in accordance with standard operating protocol and pre-approved on a project-by-project basis.

1.1 Method Sensitivity

NOTE: Results of linear range and MDL studies indicate the need for changes in calibration standard concentrations; ICV/LCS concentrations; and reporting limits.

1.1.1 Method Detection Limits

The method detection limit (MDL), referred to as the detection limit (DL) in NELAC and DOD QSM version 4.1 documents, is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to Appendix B of 40 CFR 136, "Guidelines Establishing Test Procedures for the Analysis of Pollutants" with additional details are provided in the TestAmerica Corporate SOP, *CA-QS-006, Detection Limits* and the TestAmerica Chicago SOP, *UP-QA-017, Method Detection Limit Studies.* MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; the MDL will be verified on a quarterly basis to meet the requirements of the DoD QSM version 4.2.

Attachment 1 (Standards/QC Summary Table) defines the preparation and concentrations of the IDL / MDL standards.

1.1.2 Demonstration of Capability

Before the laboratory may institute a new method and begin reporting results, the laboratory shall confirm that it can properly operate the method. A demonstration of capability is performed whenever there is a change in instrument type, method or personnel. An Initial Demonstration of Capability (IDOC) must be thoroughly documented and approved by the Department Manager/Supervisor and QA Manager prior to independently analyzing client samples. All associated documentation must be retained in the QA Department and in the Analyst Training files. For additional details on the demonstration of capability procedures followed, refer to the laboratory SOP, *UP-QA-QAM, Quality Assurance Manual, Sections 20.4.2 and 20.4.3.*

1.1.3 Linear Range Verification

The upper end of the calibration curve must be verified every 6 months by running an alternate source standard at the concentration of the highest standard. This is called the Linear Range Standard (LRS) and must be within 5% of the known concentration.

1.1.4 Instrument Detection Limits

Instrument Detection Limits (IDLs) are generated every 6 months. The MDLs are performed at the same frequency and same concentration. As current IDL's do not require true sample preparation, the two are equivalent and used interchangeably.

1.1.5 Reporting Limits

Reporting Limits are defined as the lowest concentration of an analyte determined by a given method in a given matrix that the laboratory feels can be reported with acceptable quantitative error; values specified by the EPA methods; or other project and client requirements. Reporting limits are maintained at values ~3-5x the respective MDL to ensure confidence in the value reported. Refer to Attachment 1 for the laboratory's reporting limits.

NOTES: Reporting limits will vary depending on sample size; dilutions associated with matrix interference or exceedence of the linear concentration range; and dry weight reporting.

The DoD QSM, version 4.2 and NELAC use the term Limit of Quantitation (LOQ) for the RL.

1.2 Definitions

Refer to Section 3.0 of the Laboratory's Quality Assurance Manual (UP-QA_QAM).

1.3 Summary of Method

A small volume of sample, typically 2-3 mLs, is introduced into an ion chromatograph (IC). The anions of interest are separated and measured, using a system comprised of a guard column, separator column, suppressor device, and a conductivity detector. For solids, the analysis is preceded by an extraction procedure.

2.0 INTERFERENCES

- Interferences can be caused by substances with retention times that are similar to and overlap those of the anion of interest. Large amounts of an anion can interfere with the peak resolution of an adjacent anion. Sample dilution and/or fortification can be used to solve most interference problems.
- Possible interference from the water dip or negative peak that elutes near the fluoride peak can usually be eliminated by the addition of the equivalent of 1 mL of concentrated eluent to 100 mLs of each standard and sample.
- Method interferences may be caused by contaminants in the reagent water, reagents, glassware, and other sample processing apparatus that lead to discrete artifacts or elevated baseline in ion chromatograms.
- The quantitation of un-retained peaks should be avoided. Low molecular weight organic acids (formate, acetate, propionate, etc.) which are conductive and co-elute with or near the fluoride peak would bias the fluoride quantitation in some drinking and most waste waters. Ion chromatography is not recommended for analysis of TCLP extracts in which acetic acid is used.

3.0 SAFETY

Employees must abide by the policies and procedures in the Corporate Safety Manual, Radiation Safety Manual, Lab Specific Addendum to the CSM, and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coat and closed-toe, nonabsorbent shoes are a minimum.

3.1 Specific Safety Concerns or Requirements

- Potassium Nitrate and Sodium Nitrate are strong oxidizers; therefore, do not store them near any combustible materials. These chemical are stable under prescribed conditions of use and storage (i.e., store protected from air). Note: These chemicals are not routinely use at TestAmerica Chicago for IC analysis. They may be used in the event a purchased stock standard becomes unusable and the analyst must prepare a stock locally. They are kept in inventory.
- Sodium Fluoride is highly toxic. Note: It is not routinely used at TestAmerica Chicago, but may be, in the event a fluoride stock must be prepared locally. It is kept in inventory.
- Exercise caution when using syringes with attached filter assemblies. Application of excessive force has, upon occasion, caused a filter disc to burst during the process.

3.2 Primary Materials Used

Because TestAmerica Chicago purchases 1000 mg/L stock standards for IC analysis, the hazardous salts in the table below are not used except in the event that local preparation of a stock becomes necessary.

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Potassium Nitrate	Oxidizer	None	Causes irritation to the respiratory tract, skin and eyes. Symptoms may include coughing, shortness of breath. Symptoms include redness, itching, and pain.
Sodium Fluoride	Poison	2.5 Mg/M3-TWA as F	**Highly Toxic.** Causes severe irritation to the respiratory tract, symptoms may include coughing, sore throat, and labored breathing. Causes irritation, with redness and pain. Solutions are corrosive. Eye irritant! May cause irritation and serious eye damage. Effects may not appear immediately.
Sulfuric Acid	Corrosive Oxidizer Dehydrator Poison Carcinogen	1 Mg/M3-TWA	Inhalation produces damaging effects on the mucous membranes and upper respiratory tract. Symptoms may include irritation of the nose and throat, and labored breathing. Symptoms of redness, pain, and severe burn can occur. Contact can cause blurred vision, redness, pain and severe tissue burns. Can cause blindness.
1 – Always add acid to water to prevent violent reactions. 2 – Exposure limit refers to the OSHA regulatory exposure limit.			

4.0 EQUIPMENT AND SUPPLIES

- Dionex DX-120 Ion Chromatograph (IC4)
- Ionpac AG14A-5 um (3 X 30 mm) Guard Column
- Ionpac AS14A-5 um (3 X 150 mm) Analytical Column
- ASRS Ultra-4 mm Suppressor
- DX LAN Card for DX-120
- Dionex Advanced Computer Interface
- PC equipped with PeakNet software, Release 5.1
- Dionex Automated Sampler
- Vials with or without filter caps for Automated Sampler
- 100 mL volumetric flasks
- High Purity Helium Gas or Nitrogen Gas set to deliver 90-120 psi.
- Eluent reservoirs
- OnGuard II H_{TM} pre-treatment cartridges
- 5 or 10 mL syringes
- 0.45 um syringe filters

5.0 REAGENTS AND STANDARDS

All reagents and blank solutions prepared daily must include the following information on the container labels: date of preparation, method identification, reagent or blank identification, relevant SOP section.

Use only reagent grade chemicals that meet ACS criteria.

5.1 Eluent, 100x Concentrate

On a balance capable of reading to 0.1 g, weigh out 84.8 grams of Sodium Carbonate and 8.4 grams Sodium Bicarbonate. Dissolve both in a 1-L class A volumetric flask filled with ~700 mLs Milli-Q water. Dilute to volume.

- Life of Reagent: 6-Months
- Storage Requirements: None

5.2 Eluent Working Solution (8.0 mM Na_2CO_3 / 1.0 mM $NaHCO_3$)

Pipet 10 mLs of the Eluent Stock Solution into a 1-L volumetric flask filled with ~700 mLs of Milli-Q water. Dilute to volume.

- Life of Reagent: 6-Months
- Storage Requirements: None

5.3 Anion Stock Standards

Stock standards are purchased from a vendor (received with the certified values and applying the manufacturer's expiration date). The calibration curve is prepared from Stock Standard I; the ICV/CCV, LCS and MSs are prepared from Stock Standard II.

5.3.1 Purchased Stock Standards (1,000 mg/L)

Stock	Suggested TALS ID
IC Fluoride Stock I	**W S T I C F 1**
IC Chloride Stock I	**W S T I C CL 1**
IC Nitrate Stock I	**W S T I C N O 3 1**
IC Bromide Stock I	**W S T I C B R1**
IC Nitrite Stock I	**W S T I CN I 1**
IC O-Phos Stock I	**W S T I C P 1**
IC Sulfate Stock I	**W S T I C S 1**
IC Chloride Stock II	**W S T I C C L2**
IC Fluoride Stock II	**W S T I C F 2**
IC Sulfate Stock II	**W S T I C S 2**
IC Nitrate Stock II	**W S T I C N O 3 2**
IC Bromide Stock II	**W S T I C B R 2**
IC Nitrite Stock II	**W S T I C N I 2**
IC O-Phos Stock II	**W S T I C P 2**

*The Stock II solutions are from an alternate manufacturer source than the Stock I solutions.

- Life of Standard: 1-Year or manufacturer's recommendation, whichever is sooner
- Storage Requirements: Refrigerate at 4 ± 2°C

5.3.2 Calibration Curve

Refer to Attachment 1 for concentrations and preparation from Stock I.

5.3.3 Initial Calibration Verification (ICV)
Laboratory Control Sample (LCS)
Matrix Spike (MS) / Matrix Spike Duplicate (MSD)

Refer to Attachment 1 for concentrations and preparation from Stock II.

MS/MSDs will most likely need to be done on multiple sample dilutions. Calculate and report spikes on the same dilution from which the original sample is reported.

5.3.4 Continuing Calibration Verification (CCV)

Refer to Attachment 1 for concentrations and preparation from Stock I.

6.0 CALIBRATION (NON-DAILY)

The calibration curve is prepared and run monthly or whenever a significant change in instrument response is observed. Some examples of the need for recalibration may include low recovery or concentration of the ICV standard, significant changes in retention times of the anions, or instrument maintenance, which may affect the chromatography. Some sample matrices will affect the retention times of subsequent injections. The calibration is checked immediately after the curve is run and daily with the initial run of an ICV with the requirement of ±10% acceptance criteria. (Refer to Section 7.4) (Refer to the TestAmerica Corporate Policy, CA-T-P-002, *Selection of Calibration Points* for further guidance.)

No blank is run as part of the curve, but each curve consists of 6 mixed standards, each of which contains a given concentration of each of the seven anions.

Running a curve over-writes the previous curve, and all subsequent analyses will be processed against the new curve. The Dionex ICs integrate peak areas. Refer to the PeakNet software manual for specifics.

6.1 Retention Times (RTs)

RTs may vary according to ionic size, ionic charge, ion concentration, column type, and ionic composition of the mobile phase. The RT window will be routinely determined using ±5% of the RT for each analyte contained in the Initial Calibration Verification (ICV) standard. Attachment 5 is an example of the table used for the calculation of the RT windows and included with the raw data.

DoD requires that the retention time be established as 3X the standard deviation for each analyte over a 24-hour period. TestAmerica Chicago does not have sufficient points in a 24-hour period, but determines the standard deviation of standards over a period of several days annually and calculates the retention time window. If the +/-5% window is narrower, TestAmerica Chicago will continue to use that method, but will keep the other data on file.

Analyst discretion may be used for RT shifts resulting from complex sample matrices. The instrument must be recalibrated or obtain section manager or QA approval if the RT drifts outside this window. RTs may vary with the use of different columns.

7.0 PROCEDURE

7.1 Quality Control Checks, non-calibration

Quality Controls	Frequency [1]	Default Control Limits
Method Blank (MB)	1 / set of < 20 samples	< Reporting Limit (<1/2 RL or LOD for DoD QSM)
LCS [3]	1 / set of < 20 samples	Statistical, QAPP, or method limits; whichever is applicable[2]
MS / MSD [4]	[5]	75 - 125% (9056) 80-120% (9056A and 300.0)
Duplicate (DU or MSD) [6]	See above	≤ 20 RPD (≤ 15 RPD for 9056A)

[1] Client-specific QAPPs may include QC limits and frequency requirements that supersede those given above. Certain client QAPPs may require additional QC types, such as an MRL or DLCK to be run in sequence.
[2] Statistical control limits are available for those clients or projects that require the use of statistical limits. Method 9056 requires 80-120% limits; EPA 300.0 requires 90-110%.
[3] LCS Duplicate (LCSD) is performed only when insufficient sample is available for the MS/MSD or when requested by the client/project/contract.
[4] The sample selection for the MS/MSD is rotated among client samples so that various matrix problems may be noted and/or addressed.
[5] EPA Method 300.0 requires MS's at a 10% frequency (1 in 10 samples); SW-846 Method 9056 requires MS's at a 5% frequency.
[6] 9056 requires an unspiked DU at a frequency of 1 in 10 samples (MS at 1 in 20). For analysis of non-drinking water samples by 9056A and 300.0, MS and MSDs are the default matrix QC, and a DU is only done by client or program request.

Note: Drinking water samples are analyzed in sets of 10 with a MS and DU performed on the drinking water matrix. Control limits are ≤ 10 RPD for duplicates and 85 - 115% for matrix spikes. LCS recoveries must be 90 - 110%.

Note: The Method Blank must be treated exactly like the samples in order to make a valid assessment of the contamination level. This includes filtration and any other sample handling techniques.

7.2 Sample Preservation and Storage

Samples should be collected in scrupulously clean glass or polyethylene bottles.

Anion	Preservation	Hold Time
Fluoride	Cool 4 ± 2°C	28 Days
Chloride	Cool 4 ± 2°C	28 Days
Nitrite-N	Cool 4 ± 2°C	48 Hours
Bromide	Cool 4 ± 2°C	28 Days
Nitrate-N	Cool 4 ± 2°C	48 Hours
Ortho-Phosphate-P	Cool 4 ± 2°C	48 Hours
Sulfate	Cool 4 ± 2°C	28 Days

7.3 Sample Preparation

7.3.1 Waters

Water samples should be filtered prior to analysis using a 0.45 um membrane type filter if the sample appears to have particulates in it. If transition metals are known or suspected to be present in a sample, it is good practice to put the sample through a Dionex OnGuard II H_{TM} column prior to analysis, since transition metals will corrupt the columns. Use these columns with care and appropriate rinsing.

7.3.2 Soils

Weigh ~10 grams weighed to the nearest 0.1 gram of the solid sample into a 125 mL Erlenmeyer flask and add 100 mLs of reagent grade water. Spike at this point if applicable. Mix the solution well using a wrist action shaker for 10 minutes, and then filter the slurry solution using a 0.45 u membrane type filter. (It is best to let the slurry solution settle for few hours prior to filtering to allow the particulates to settle. This allows for an easier filtration process. **Never compromise the representative composition of the sample in the original container!** Centrifuging is another pre-filtration option.) Soil extracts may also be treated through OnGuard II H_{TM} columns.

7.4 Calibration /Calibration Verification[1]

Calibration Controls	Sequence	Control Limit
Standard Curve	Monthly or as needed	r ≥ 0.995; Stds. ± 10% of known; y-intercept < abs. of RL
ICV (Initial Calibration Verification)	Immediately after curve and at start of each run	± 10%
ICB (Initial Calibration Blank)	After ICV	< Reporting Limit (<1/2 RL or LOD for DoD QSM)
CCV (Cont. Calibration Verification)	After every 10 readings and after last sample reading	± 10%
CCB (Cont. Calibration Blank)	After every CCV	< Reporting Limit (<1/2 RL or LOD for DoD QSM)

[1] See Attachment 1 for details of standard preparation.
[2] The lowest standard in each curve is at the TestAmerica Chicago lower limit of quantitation (RL, referred to as LOQ in DoD QSM version 4.1 and NELAC) and must be within 50% of the true value for 9056A. The remaining standards should be within 10% of the true value.
[3] If the recovery of the CCV has changed more than 5% within a run, recalibrate for that analyte to comply with SW-846 Method 9056.

7.5 Preventive Maintenance

Instrument maintenance is recorded in the instrument maintenance log (Attachment 2). The following checks are completed as necessary to ensure efficient operation of the instrument.

- Periodically check for leaks or spills within the valve compartments. Isolate and repair any leaks.
- Clean up any spills.
- Rinse any dried eluents or reagents off the system components with DI water.
- Check all air and liquid lines for discoloration or crimping. Relocate pinched lines and replace damaged lines.
- Change eluent reservoir filter and in-line filters as needed.
- Some sample matrices will degrade the IC components over time. Placing one to three vials containing concentrated eluent in the schedule after the final CCB may help to prevent the degradation.
- Clean the columns periodically as needed following the Dionex manual. Be sure to place the guard column after the separator column for this procedure.

7.6 Sample Analysis

Enter the sequence of analysis of samples and standards in the instrument's "Schedule File" that is located at C:\PeakNet\Schedule*Month date year.sch.*

Pour approximately 5 mL of each calibration standard, method blank, LCS, and prepared sample into a labeled autosampler vial. Place the vials in sequence into the 6-place vial holders, then place the holders into the autosampler. Take care to limit the number of holders to 10 if the autosampler will be left unattended. More holders can be added when an equal number of spent holders have been removed. Fill the eluent reservoir with working eluent solution (reagent 5.2). Eluent will need to flow through the system until it is equilibrated or for an hour before starting. Start the run when the instrument's general operating conditions are met (see 7.6.1). Refer to the instrument's operational manual for complete details.

7.6.1 General Operating Conditions

Item	Description
Sample Loop Volume	25 uLs
Analytical Column	Ionpac AS14A 5 um (3 X 150 mm)
Eluent	8.0 mM Na_2CO_3 / 1.0 mM $NaHCO_3$
Eluent Flow Rate	0.5 mL / min
Suppressor	ASRS Ultra 4mm Suppressor
Background Conductivity	~ 22 uS (stable)
System Pressure	1600 – 2000 psi (stable)

7.7 Data Evaluation and Documentation

7.7.1 Data File Processing

At the completion of the run, print the Schedule File and the individual Data Files (chromatograms). Additionally, create a .csv file for the run. Export the .csv file to TALS, by following the keyboard sequence found in Attachment 6.

7.7.2 Raw Data Evaluation and Documentation

Go through the hard copies of each chromatogram while checking the imported data in the TALs LIMS sample result screen. Circle the peak number for each anion being reported on each chromatogram. When multiple dilutions have been made, report each ion from a peak that falls within the upper half of the curve when clear resolution has been obtained. To the right of each anion name write the appropriate 2-letter data qualifier explaining why the other anions are not being reported (see Attachment 3). Sign and date each chromatogram as it's evaluated.

TestAmerica Chicago logs in all ion chromatography samples under a method code with only 48 hours applied as a holding time. The analyst must remove the "H" flags from the 28-day analytes during review of the ADII batch.

If manual integration is advised, be sure to print the chromatogram before and after the corrections, preferably with an expanded scale so that the need for the correction is apparent, and be sure to document on the hard copy why it was done. Manual integrations must be co-signed by the trained reviewer, supervisor, Inorganics Manager, or QA Manager. See the corporate SOP on Manual Integration (CA-Q-S-002) and Section 9.4 of this SOP for approved manual integration practices.

Complete a Cover Page for each analysis run (CHI-22-12-084). Documentation on this page includes the TALs LIMS Batch, File ID, Instrument, Calibration Date, Standard Traceability, Calibration Range and Data Qualifiers used in the evaluation of the data. The analyst and reviewer signatures are required on this page. Refer to Attachment 3 for examples of above mentioned forms and an example of the TALs LIMS Forms that serve to report the data and its associated QC in the TALs LIMS system.

The complete raw data package for an IC run includes the Cover Page (Attachment 3), the Schedule File, the Retention Time Table (Attachment 5), and the Sample Analysis Reports from the IC. For Level 4 reports, the calibration curve data must also be included.

7.7.3 Traceability of Standards

Upon receipt or preparation, each chemical salt, solvent, acid, standard, or other reagent is entered into LIMS and is issued a unique ID# based upon the type and sequential order in which the item was prepared or received. Further information entered into the TALs LIMS includes the manufacturer, lot # (if applicable), the date received or prepared, the expiration date, volume/weight received; concentration (if applicable); preparation details (if applicable), initials of the recording analyst, and the description of the item (i.e., IC Nitrate Stock Solution – LCS/MS). Once the record is created, a unique label is printed and affixed to the appropriate standard/reagent container. Non-standard reagents, such as eluent, and such items as filter lot #s, are recorded in the batch information screens of the prep batch (if applicable) and the analytical batch, either by scanning or manual entry. The standards are entered in the Reagent tab in ADII, along with the volume added for final concentration calculation.

7.7.4 Data Review

Analytical data goes through a 200% review cycle. As results are generated, analysts review their work to ensure that the results generated meet QC requirements and relevant EPA methodologies. The analyst transfers the data into TALs in the Analyst Desktop module. Where non-compliance is observed, the analyst creates Non-Conformance Memos (NCMs) in TALs. Flags and data qualifiers can be method, project, program or QAPP specific. The analyst documents the initial review on a data review checklist (Attachment 4) and sets the batch status in

LIMs to 1st level review. The second level or peer review of the data is conducted by another individual who has been trained on the review process. This secondary review is documented on the same checklist, making any necessary corrections to the data or additions to the NCMs as necessary. The batch is then set to 2nd level review. Any Spectra and all manual integrations are reviewed. The raw data, including the checklist, instrument print-outs, and manual entries, and electronic files are retained for easy retrieval in accordance with the laboratory's record and retention policy outlined in the SOP, *UP-QA-QAM, Section 15.*

Examples of items included in the above reviews are as follows:

- QC data are outside the specified control limits for accuracy and precision
- Unusual detection limit changes are observed
- Samples having unusually high results
- Samples exceeding a known regulatory limit
- Raw data indicating some type of contamination or poor technique
- Inconsistent peak integration (if applicable)
- Transcription errors
- Results outside of calibration range

8.0 QUALITY CONTROL

8.1 QC Summary

8.1.1 One MB and one LCS will be included in each batch of samples (refer to Section 7.1. The MB will be examined to determine if contamination is being introduced in the laboratory. The LCS will be examined to determine accuracy of the method and cumulative LCS data will provide precision data.

8.1.2 Accuracy will be measured by the percent recovery (%) of the LCS. The recovery must be in range, as determined by in-house control limits or statistical analysis, in order to be considered acceptable. And, precision will be measured by the cumulative reproducibility of the LCSs.

8.1.3 One MS/MSD (or MS/DU) is required per matrix per sample batch (refer to Section 7.1), unless otherwise requested. Results must agree within the limits defined in Section 7.1 or within internally-derived statistical limits in order to be considered acceptable.

8.2 Corrective Actions

When an out-of-control situation occurs, the analysts must use their best analytical judgment and available resources to determine the corrective action to be taken. The out-of-control situation may be caused by more than one variable. The analysts should seek the assistance of their immediate supervisor or manager, QA personnel, or other experienced staff if they are uncertain of the cause of the out-of-control situation. The analysis must not be resumed until the source of the problem and an in-control status is attained. All samples associated with the out of control situation should be reanalyzed. Out-of-control data must never be released without approval of the supervisor, inorganics manager, project manager, QA personnel or the lab manager.

Listed below are steps that MUST be taken when an out of control situation occurs:

- demonstrate that all the problems creating the out of control situation were addressed;
- document the problem and the action which was taken to correct the problem on the data review checklist;
- document on the checklist that an in-control has been achieved; and write the appropriate Non-Conformance Memo (NCM) in the batch;
- receive approval (signature) of the Supervisor, Inorganics Manager, QA personnel, Project Manager, or the Laboratory Manager prior to the release of any analytical data associated with the problem.

It may be necessary to forward an NCM to the PM/Client for further action/approval prior to reporting of the data.

The following are suggested actions to specific out of control situations:

QC Standard	Suggested Corrective Actions
Calibration Curve	• reanalyze the standard curve; • prepare a new stock and/or working standards; • check the reagents/solutions and prepare fresh if necessary. • 6-month LRS outside 95-105% recovery requires re-calibration.
ICV	• repeat the ICV to verify proper preparation; • prepare a new ICV from the original stock; • check for instrument base-line drift; • restandardize the instrument with existing standards, reanalyze; • check the reagents/solutions and prepare fresh if necessary; • prepare a new stock and/or working standards and recalibrate.
ICB	• prepare a new ICB to verify proper preparation; • verify that the instrument base-line is stable and/or perform necessary maintenance, cleaning, etc... to achieve stability; • determine the source of contamination by the process of elimination, correct the problem and reanalyze. (Carry over from a previous analysis or reagent contamination are two common sources).
LCS	• reanalyze the LCS to verify that an out-of-control situation exists; • determine the source of error within the preparation procedure, correct the problem and repeat the sample set. (Sources of contamination could be either the reagents, the LCS stock solution, or the preparation area.) • Note any out-of-control LCSs on the data review checklist and in an NCM.
If an LCS Duplicate (LCSD) is required, it must meet the control limits of ≤ 20 RPD. If this criteria is not met, and both LCS's meet the % Recovery control limits, then see your supervisor for proper corrective action.	

QC Standard	Suggested Corrective Actions
MB	• Reanalyze the MB to verify contamination at a level > Reporting Limit; • Determine the source of contamination and correct the problem; • All samples whose concentration is <10 times the contaminated MB level must be reprocessed and reanalyzed; any sample which is >10 times the MB level need not be reanalyzed. However, note the out-of-control MB on the data review checklist and write an NCM. • If the sample is a non-detect for the parameter in question, the MB is appropriately flagged in TALS and the samples are reported without further corrective action, according to the client/project/QAPP requirements and may contain an NCM noting the presence of contamination in the blank. • If the sample concentration is above the RL, the MB and sample are appropriately flagged in TALS. Further corrective action (re-analysis of samples) is dependent upon client/project/QAPP requirements. At least, the samples will be flagged and an NCM will note the presence of the analyte in the MB. The report format will be consistent with client/project/QAPP requirements. • If the sample concentration is between the MDL and the RL, the sample data are reported as estimated and flagged for the presence of the analyte in the MB. The report format and additional corrective action is dependent upon client/project/QAPP requirements. • In all cases, MB data is used to assess contamination in the laboratory environment. Samples associated with MBs above the MDL will be evaluated, and at a minimum will be flagged in both TALS and in the final deliverable, and an NCM/narrative will note the problem. Additional corrective action is client/project/QAPP dependent. • MB data will be monitored to ensure that a systematic cause or source of contamination is not being introduced into the system. Upon evidence to that effect, the lab will take immediate corrective action to identify the source and correct the problem. Any associated samples analyzed during that time will be evaluated and suspect samples will be reanalyzed if appropriate.
DU (If Required)	• the sample must be reprocessed and reanalyzed unless the sample concentration is <5 times the Reporting Limit, then the ± Reporting Limit rule applies; • if the reanalysis is within the control limits, the second value is reported; • if the reanalysis is still outside of the control limits, the data must be flagged and noted in an NCM.
MS	• If a single spike is performed and it is outside the acceptance limits, it must be repeated to verify the matrix effect. Generally, an MS and MSD are performed together in the same batch as the original sample analysis. • Report all spikes, whether or not they are in control. • Note out-of-control spikes on the data review checklist and NCMs.
CCV	• repeat the CCV to verify proper preparation; • report non-detect sample results if the CCV bias is high; write and NCM • prepare a new CCV from the original stock; • check for instrument base-line drift; • check the reagents/solutions and prepare fresh if necessary; • recalibrate with a new standard curve and repeat all detected if CCV is biased high)samples since the previous in-control CCV; • never dispose of any samples until you are sure that all QC are within their designated control limits.

QC Standard	Suggested Corrective Actions
CCB	• prepare a new CCB to verify proper preparation; • verify that the instrument base-line is stable and/or perform necessary maintenance, cleaning, etc... to achieve stability; • determine the source of contamination by the process of elimination, correct the problem and reanalyze all the samples since the previous in-control CCB. (Carry over from a previous analysis or reagent contamination are two common sources); • never dispose of any samples until you are sure that all QC are within their designated control limits. • Report only non-detects and samples >10X the contamination level. Write an NCM

9.0 Data Analysis and Calculations

9.1 Sample Concentrations

9.1.1 Waters mg/L = instrument reading x dilution factor

9.1.2 Soils $\text{mg/kg} = \frac{\text{instr. reading (mg/L) x final vol. (mLs)}}{\text{sample wt. (g)}} \times \text{dilution factor}$

9.2 Accuracy

9.2.1 ICV/CCV and LCS % Recoveries $= \frac{\text{observed concentration}}{\text{actual concentration}} \times 100$

9.2.2 MS % Recovery $= \frac{\text{(spiked sample conc. - original sample conc.)}}{\text{spike concentration}} \times 100$

9.3 Precision

9.3.1 Matrix Dup. and LCD Relative Percent Difference (RPD)

$$\text{RPD} = \frac{|\text{orig. sample value - dup. sample value}|}{[(\text{orig. sample value + dup. sample value})/2]} \times 100$$

9.4 Manual Integration Policy

In each case where the file has been edited or manual integrations have been performed, the following guidelines apply:

- Manual integrations should be consistent among all files integrated.
- Manual integrations should not be performed solely to meet QC criteria.
- Excessive manual integrations may reflect an instrumental or methodological problem that should be addressed.
- Manual integrations shall follow the TestAmerica Corporate SOP for manual integrations (CA-Q-S-002).

Manual integrations are most often performed for the following reasons:

- Assignment of correct peak that was mis-identified by the data system.
- Incomplete auto-integration due to high level of target compound detected.
- Incomplete auto-integration due to background interference.
- Incorrect auto-integration due to co-elution or near co-elution of compounds.
- Missed peaks.

All integrations are reviewed by the analyst. All chromatograms and reports are printed after any integration takes place and are routinely included in the data packages.

10.0 POLLUTION CONTROL

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

10.1 Waste Management

Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to UP-WM-001.

The following waste streams are produced when this method is carried out.

- Waste from this procedure will enter the "Wastewater" wastestream.
- Single component standards will be turned over to the EHSC or Waste Technician.

11.0 METHOD PERFORMANCE CRITERIA

Refer to Sections 1, 6, 7 and 8.

12.0 REFERENCES

Refer to Section 1.0

13.0 ATTACHMENTS

Attachment 1: Example: IC Standards / QC Summary Table
Attachment 2: Example: Daily Maintenance Log
Attachment 3: Example: IC Cover Page/Schedule File
Attachment 4: Example: Data Review Checklist
Attachment 5: Example: Retention Time Window Summary Table
Attachment 6: Example: Data Export to LIMS: Keystroke Sequence
Attachment 7: DoD QSM Version 4.2: Appendix F QC Requirements Summary (Table F-1 and Table F-11)
Attachment 8: Example: Retention time study (3X standard deviation)

14.0 REVISION HISTORY

- Revision 12 updated on 11/30/10
- Annual Review
- Changed CCV preparation from Stock II to Stock I
- Added text to section 7.7.2 about removing the H flags from 28-day anions when appropriate while reviewing ADII
- Changed 7.1 and 7.4 to include DoD LOD limits for blanks
- Incorporated SOP change form in section 7.1, footnote 6.
- Added non-standard reagent traceability note to section 7.7.3

Attachment 1.

Example: IC Standards / QC Summary Table (017-001)

TestAmerica Chicago

CHI-22-12-070\N-11/10

IC - Standards / QC Summary Table

Parameter		Fluoride	Chloride	NO2-N	Bromide	NO3-N	O-PO4 as P	Sulfate	High Range Chloride	High Range Sulfate
Method		EPA 300.0 SW-846 9056/A	EPA 300.0 SW-846 9056/A	EPA 300.0 SW-846 9056/A	EPA 300.0 SW-846 9056/A	EPA 300.0 SW-846 9056/A	EPA 300.0 SW-846 9056/A	EPA 300.0 SW-846 9056/A	EPA 300.0 SW-846 9056/A	EPA 300.0 SW-846 9056/A
Report Limit	mg/L	0.2	0.2	0.1	0.2	0.1	0.2	0.2	5	5
Report Limit	mg/Kg	2.0	2.0	1.0	2.0	1.0	1.0	2.0	50	50

Standard	Stock Std.	Curve (mg/L) / (ul volume of Stock Standard added per 100 mLs of DI water in a volumetric flask)								
1	I	0.2 (20uL)	0.2 (20uL)	0.1 (10uL)	0.2 (20uL)	0.1 (10uL)	0.20 (20uL)	0.2 (20uL)	5 (500uL)	5 (500uL)
2	I	0.4 (40uL)	0.5 (50uL)	0.5 (50uL)	0.5 (50uL)	0.5 (50uL)	0.50 (50uL)	0.5 (50uL)	20 (2mL)	20 (2mL)
3	I	0.6 (60uL)	1.0 (100uL)	1.0 (100uL)	1.0 (100uL)	1.0 (100uL)	1.0 (100uL)	2.5 (250uL)	50 (5mL)	50 (5mL)
4	I	1.0 (100uL)	3.0 (300uL)	2.0 (200uL)	2.0 (200uL)	2.0 (200uL)	2.0 (200uL)	5.0 (500uL)	100 (10mL)	100 (10mL)
5	I	1.5 (150uL)	5.0 (500uL)	3.0 (300uL)	3.0 (300uL)	3.0 (300uL)	3.0 (300uL)	7.5 (750uL)	150 (15mL)	150 (15mL)
6	I	2.0 (200uL)	7.5 (750uL)	5.0 (500uL)	5.0 (500uL)	4.0 (400uL)	4.0 (400uL)	10 (1000ul)		

QC Solution	Stock Std.	Concentration (mg/L) / (ul volume of Stock Standard added per 100 mLs of DI water in a volumetric flask)								
ICV/LCS (+/- 10%)	II	1 (100uL)	3 (300uL)	2 (200uL)	2 (200uL)	2 (200uL)	2 (200uL)	5 (500uL)	50 (5mL)	50 (5mL)
CCV (+/- 10%)	I	0.6 (60uL)	1.0 (100uL)	1.0 (100uL)	1.0 (100uL)	1.0 (100uL)	1.0 (100uL)	2.5 (250uL)	20 (2mL)	20 (2mL)
MDL/ IDL/ LOQ	II	0.2 (20uL)	0.2 (20uL)	0.1(10uL)	0.2(20uL)	0.1(10uL)	0.2 (20uL)	0.2 (20uL)	NA	NA
MS (75-125%)	II	1 (100uL)	3 (300uL)	2 (200uL)	2 (200uL)	2 (200uL)	2 (200uL)	5 (500uL)	50 (5mL)	50 (5mL)

Notes:

1. Reporting Limits will vary depending on sample size/volume, dilution factors and dry weight reporting for soils.
2. All Standards and QC Solutions are prepared fresh daily.
3. LOD Verification Standard =1/2 dilution of LOQ Standard.

Revision: 11/30/10

(C-2) C-578

(DI 7-001)

Attachment 2.

Example: Daily Maintenance Log (018-001)

TestAmerica Chicago
IC-4 Dionex Series DX-120
Maintenance Log

Page No.:_____

Action	Initial/Date	Initial/Date	Initial/Date	Initial/Date	Initial/ Date	Initial/Date	Initial/Date
Date analytical column changed							
Analytical column serial number (record when changed)							
Date guard column changed							
Guard column serial number (record when changed)							
Date Nitrogen Tank changed							
Eluent flow rate (~2 mL/min.)							
Background conductivity (~12-20 uS and stable)							
System pressure (~1600-2000 psi and stable)							

Non-Routine Maintenance/Comments:

Reviewed by:_____ Date:_____

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(018-001)

Attachment 3.

IC Cover Page / Schedule File
(019-001 to 019-002)

TestAmerica Chicago

Cover Page - Ion Chromatograph

LIMS Batch: _____ **FILE ID:**_____**.csv**

Calibration Date: _____

Instrument: (Circle)	
a. IC4: Dionex Series DX-120	DX120.met
b. IC3: Dionex Series DX-100	DX100.met

Standard Traceability

Note: Working Standards are prepared daily from the noted Stock Solutions.

Test	Calibration Curve CCV1 / CCV2 Stock ID	LCS ICV MS Stock ID	LCS mg/L	(ICV-CCV1)/CCV2 mg/L	MS mg/L
Fluoride	WSTICF1-	WSTICF2-	1.0	1.0 / 0.6	1.0
Chloride	WSTICCL1-	WSTICCL2-	3.0	3.0 / 1.0	3.0
Nitrite-N	WSTICNI1-	WSTICNI2-	2.0	2.0 / 1.0	2.0
Bromide	WSTICBR1-	WSTICBR2-	2.0	2.0 / 1.0	2.0
Nitrate-N	WSTICNO31-	WSTICNO32-	2.0	2.0 / 1.0	2.0
O-PO4	WSTICP1-	WSTICP2-	2.0	2.0 / 1.0	2.0
Sulfate	WSTICS1-	WSTICS2-	5.0	5.0 / 2.5	5.0

Test	Range of Curve (mg/L)
Fluoride	0.20 - 2.0
Chloride	0.20 - 7.5
Nitrite-N	0.10 - 5.0
Bromide	0.20 - 5.0
Nitrate-N	0.10 - 4.0
O-PO4	0.20 - 4.0
Sulfate	0.20 - 10.0

IC Data Qualifiers used by the analyst in the raw data evaluation.

CE = **C**o-**E**lution or masking of peak(s) has been identified

OR = **O**ver **R**ange - Peak exceeds the highest standard of the calibration curve

OD = **O**ver **D**ilution - The ion is reported from a less dilute injection

NR = **N**ot **R**equired - analysis was not required for this ion

AD = **A**nalyst **D**iscretion was used in the evaluation and the reporting of the ion

RE = **R**eported **E**lsewhere

RT = **R**etention **T**ime Shift

Analyst Signature: _____ Date: _____

Reviewer Signature: _____ Date: _____

(019-001)

Line	Sample	Sample Type	Level	Method	Data File	Volume	Dilution	Weight	Int. Std.
1	ICV	Sample		dx-120.met	20101129\d5997_001.dxd	1	1	1	1
2	ICB	Sample		dx-120.met	20101129\d5997_002.dxd	1	1	1	1
3	MB	Sample		dx-120.met	20101129\d5997_003.dxd	1	1	1	1
4	LCS	Sample		dx-120.met	20101129\d5997_004.dxd	1	1	1	1
5	500-29483-D-2	Sample		dx-120.met	20101129\d5997_005.dxd	1	25	1	1
6	500-29483-D-2	Sample		dx-120.met	20101129\d5997_006.dxd	1	250	1	1
7	500-29483-D-3	Sample		dx-120.met	20101129\d5997_007.dxd	1	25	1	1
8	500-29483-D-3	Sample		dx-120.met	20101129\d5997_008.dxd	1	250	1	1
9	500-29483-E-6	Sample		dx-120.met	20101129\d5997_009.dxd	1	25	1	1
10	500-29483-E-6	Sample		dx-120.met	20101129\d5997_010.dxd	1	250	1	1
11	500-29483-D-7	Sample		dx-120.met	20101129\d5997_011.dxd	1	25	1	1
12	500-29483-D-7	Sample		dx-120.met	20101129\d5997_012.dxd	1	250	1	1
13	CCV	Sample		dx-120.met	20101129\d5997_013.dxd	1	1	1	1
14	CCB	Sample		dx-120.met	20101129\d5997_014.dxd	1	1	1	1
15	500-29483-E-8	Sample		dx-120.met	20101129\d5997_015.dxd	1	25	1	1
16	500-29483-E-8	Sample		dx-120.met	20101129\d5997_016.dxd	1	250	1	1
17	500-29483-C-1	Sample		dx-120.met	20101129\d5997_017.dxd	1	25	1	1
18	500-29483-E-2	Sample		dx-120.met	20101129\d5997_018.dxd	1	25	1	1
19	500-29483-E-2	Sample		dx-120.met	20101129\d5997_019.dxd	1	250	1	1
20	500-29483-E-3	Sample		dx-120.met	20101129\d5997_020.dxd	1	25	1	1
21	500-29483-E-3	Sample		dx-120.met	20101129\d5997_021.dxd	1	250	1	1
22	500-29483-C-4	Sample		dx-120.met	20101129\d5997_022.dxd	1	25	1	1
23	500-29483-C-5	Sample		dx-120.met	20101129\d5997_023.dxd	1	25	1	1
24	500-29483-D-6	Sample		dx-120.met	20101129\d5997_024.dxd	1	25	1	1
25	CCV	Sample		dx-120.met	20101129\d5997_025.dxd	1	1	1	1
26	CCB	Sample		dx-120.met	20101129\d5997_026.dxd	1	1	1	1
27	500-29483-D-6	Sample		dx-120.met	20101129\d5997_027.dxd	1	250	1	1
28	500-29483-E-7	Sample		dx-120.met	20101129\d5997_028.dxd	1	250	1	1
29	500-29483-D-8	Sample		dx-120.met	20101129\d5997_029.dxd	1	250	1	1
30	500-29483-C-9	Sample		dx-120.met	20101129\d5997_030.dxd	1	25	1	1
31	500-29483-C-10	Sample		dx-120.met	20101129\d5997_031.dxd	1	25	1	1
32	500-29483-C-10 MS	Sample		dx-120.met	20101129\d5997_032.dxd	1	25	1	1
33	500-29483-C-10 MSD	Sample		dx-120.met	20101129\d5997_033.dxd	1	25	1	1
34	CCV	Sample		dx-120.met	20101129\d5997_034.dxd	1	1	1	1
35	CCB	Sample		dx-120.met	20101129\d5997_035.dxd	1	1	1	1
36	SHUTDOWN	Sample		shutdown120.met	d5997	1	1	1	1

Attachment 4.

Example: Data Review Checklist
(020-001)

TestAmerica Chicago
INORGANICS – LIMS DATA REVIEW CHECKLIST **Test** _____ **Analytical Batch#** _____

Prep Batch # _____
(File by analytical batch #)

Batch Entry Date: _____ Analysis Date: _____ No. of Jobs in Batch: _____

Analyst / Primary Reviewer: _____ 1st Level Review Date: _____

Secondary Reviewer: _____ 2nd Level Review Date: _____

	PRI REV	SEC REV	COMMENTS
1) Analyst correct			
2) Instrument Code present			
3) Was Data Imported _____ Manually entered _____ Balance Interface Used _____			
4) Samples & all QC in order as analyzed?			
5) Sample Date/Time analyzed correct			
6) Reagent Codes present and Amount Spiked correct?			
7) Dilution factors all present and correct?			
8) Are correct Sample ID's used? Are all samples designated with a Blue P?			
9) Are correct QC ID's used? Are all QC designated with a Blue P?			
10) Are all QC correctly related to the samples?			
11) Do all entries match raw data?			
12) Is all QC calculated and are correct flags applied?			
13) Is an NCM needed? ICV, MB, LCS, LCSD, DU, MS, MSD, RPD out; holding time missed or Manual Integration Required			NCM # _____ Approved By: _____ hitials
Raw Data: 1) Is AD Batch # is clearly noted?			
2) Are manual calculations and final results clearly shown?			
3) Are all errors crossed out with single line & initialed and dated?			
4) Is unreported data clearly identified with reason & initialed and dated?			
5) All unused portions of the page(s) Z'd out?			
6) Is data signed & dated by analyst & reviewer?			

CHI-22-12-109/E-08/08

(020-001)

Attachment 5.

Example: Retention Time Window Summary Table (021-001)

TestAmerica Chicago

Retention Time Window Summary Table

Instrument:	IC4: DX-120	Method: DX120.met
Analytical Batch:	72193	
Date:	9/22/2009	

Analyst:	Pete Ficarello

Parameter	ICV RT	RT Window	
	Minutes	+/- 5 %	
Fluoride	2.58	2.45	2.71
Chloride	3.60	3.42	3.78
Nitrite	4.22	4.01	4.43
Bromide	5.27	5.01	5.53
Nitrate	5.88	5.59	6.17
Orthophosphate	7.27	6.91	7.63
Sulfate	8.62	8.19	9.05

Analyst Signature: _____ Date: _____

CHI-22-12-130/A-11/09

(021-001)

Attachment 6.

Example: Data Export to LIMS: Keystroke Sequence (022-001)

TestAmerica Chicago

Ion Chromatography .CSV Export to TAL LIMS

1. From "PeakNet MainMenu" (Click the TAB under the specific Instrument "DXLAN or ACI"
2. [Batch]
3. [File]
4. Select C\Peaknet\Schedule\tallims.bch. It should be recently opened item 1.
5. [Processing]
6. [Export]
7. Select Process Method "from data files"
8. [Processing]
9. [Export]
10. [Browse]
11. Navigate to and open Peaknet\CSV
12. Type the schedule date in the filename box with the extension .csv
13. [Save]
14. For report type indicate "Summary" the [OK]
15. [Processing]
16. [Start]
17. Close batch window after processing complete. Do not save changes.
18. Open Peaknet CSV files folder on desktop.
19. Find the .csv file you just created.
20. Right click on the file and Send to "IC4 Raw data for TAL LIMS".

(022-001)

Attachment 7:

DoD QSM Version 4.2: Appendix F QC Requirements Summary
Table F-1 and Table F-11
(023-001 to 023-004)

TestAmerica Chicago

DoD QSM Version 4.2: Appendix F - Quality Control Requirements Summary

Table F-1 Summary of QC Check Definitions, Purpose, and Evaluation – Inorganics (WC)

QC Check	Definition	Purpose	Evaluation
Calibration Blank	Reagent water containing no analytes of interest.	To determine the zero point of the calibration curve for all initial and continuing calibrations.	This is a required QC procedure. Continuing calibration blank responses above the LOD require corrective action
Continuing calibration verification (CCV)	This verification of the ICAL that is required during the course of analysis at periodic intervals. Continuing calibration verification applies to both external standard and internal standard calibration techniques, as well as to linear and non-linear calibration models	To verify that instrument response is reliable, and has not changed significantly from the current ICAL curve.	If the values for the analytes are outside the acceptance criteria, the ICAL may not be stable. Results associated with out-of-control CCV results require reanalysis or flagging
Demonstrate Acceptable Analytical Capability	QC samples are analyzed in series to verify ability to produce data of acceptable precision and bias.	To verify the ability to produce data of acceptable precision and bias for a specific instrument type, matrix, method, and analyst.	The average recovery of the spikes and standard deviation of the replicates must be within designated acceptance criteria. Analysis of field samples may not be conducted until this check is successful.
Duplicate Sample (replicate)	Two identical portions of material collected for chemical analysis, and identified by unique alphanumeric codes. The duplicate may be portioned from the same sample, or may be two identical samples taken from the same site. The two portions are prepared and analyzed identically. (modified QSM)	To provide information on the heterogeneity of the sample matrix or to determine the precision of the intralaboratory analytical process for a specific sample matrix	A duplicate sample will provide information on the heterogeneity of the sample matrix. The greater the heterogeneity of the matrix, the greater the relative percent difference between the sample and the sample duplicate. If the sample matrix is homogeneous (such as with drinking water) and the relative pecent difference is high, this could indicate a problem in the analytical system.
Initial calibration for all analytes (ICAL)	Analysis of analytical standards at different concentrations that are used to determine and calibrate the quantitation range of the response of the analytical detector or method	To establish a calibration curve for the quantification of the analytes of interest	Statistical procedures are used to determine the relationship between the signal response and the known concentration of analytes of interest. The ICAL must be successful before any samples or other QC check samples can be analyzed.
Laboratory control sample (LCS) containing all analytes to be reported	A sample matrix, free from the analytes of interest, spiked with known amounts of analytes or a material containing known and verified amounts of analytes.	Used to evaluate the performance of the total analytical system, including all preparation and analysis steps. Assesses the ability of the laboratory/analyst to successfully recover the target analytes from a control (clean) matrix. Control limits for LCS recovery, typically expressed as percent recovery, are used for the development of statistical control limits and serve as acceptance criteria for determining whether an analytical run is in control (batch acceptance).	This is a required QC Check. The inability to achieve acceptable recoveries in the LCS indicate problems with the precision and bias of the measurement system. Failure to achieve acceptable recoveries in a "clean" matrix is an indicator of possible problems achieving acceptable recoveries in field samples.
MS	A sample prepared by adding a know mass of target analyte to a specified amount of matrix sample for which an independent estimate of target analyte concentration is available.	To assess the performance of the method as applied to a particular matrix. Matrix spikes are used, for example, to determine the effect of the matrix on a method's recovery efficiency. The recovery of target analytes from the matrix spike sample is used to determine the bias of the method in the specific sample matrix.	The lack of acceptable recoveries in the MS often points to problems with the sample matrix. One test of this is a comparison to the LCS recoveries. If the corresponding LCS recoveries are within acceptable limits, a matrix effect is likely. The lab should not correct for recovery; only report the results of the analyses and the associated MS results and indicate that the results from these analyses have increased uncertainty
MSD	A second replicate matrix spike prepared in the laboratory and analyzed to obtain a measure of the precision of recovery for each analyte	To assess the performance of the method as applied to a particular matrix and provide information on the homogeneity of the matrix. Also used to determine the precision of the intralaboratory analytical process for a specific sample matrix.	When compared to the MS, the MSD will provide information on the heterogeneity of the sample matrix. The greater the heterogeneity of the matrix, the greater the RPD between the matrix spike and the matrix spike duplicate. If the sample matrix is homogeneous, such as with drinking water, and the RPD is high, this could indicate a problem in the analytical system.

(C-2) C-591

(023-001)

F-1 (cont.)			
QC Check	**Definition**	**Purpose**	**Evaluation**
Matrix Verification sample (CR+6 only)	A pH-adjusted filtrate that has been spiked with CR+6 to ensure that the sample matrix does not have a reducing condition or other interferents that could affect color development. (Modified Method)	To ensure that the sample matrix does not have a reducing condition or other interferents that affect color development.	To verify the absence of an interference, the spike recovery must be between 85% and 115%. If the result of verification indicates a suppressive interference, the sample should be diluted and reanalyzed. If the interference persists after sample dilution, an alternative method (Method 7195, Coprecipitation, or Method 7197, Chelation/Extraction) should be used.
MB	A sample of a matrix similar to the batch of associated samples (when available) that is free from the analytes of interest and is processed simultaneously with an under the same conditions as samples through all steps of the analytical procedures, and in which no target analytes or interferences are present at concentrations that impact the analytical results for sample analyses.	To assess background interferences or contamination in the analytical system that might lead to high bias or false positive data. Results of method blanks provide an estimate of the within-batch variability of the blank response and an indication of bias introduced by the preparation and analytical procedure.	This is one of the QC samples used to measure lab accuracy/bias. The sample could indicate whether contamination is occurring during sample prep and analysis. If analytes are detected > ½ RL, reanalyze or qualify (B-flag) all results for the specific analytes(s) in all samples in the associated prep batch as appropriate. For common lab contaminants, no analytes detected > the RL. See Section D.1.1.1 and Box D-1
RT window position establishment for each analyte (and surrogate) (all chromatographic methods only)	Determination of the placement of the RT window (i.e. start/stop time) of each analyte or group of analytes as it elutes through the chromatographic column so that analyte identification can be made during sample analysis. This is done during the ICAL.	To idendify analytes of interest	Incorrect window position may result in false negatives, require additional manual integrations, or cause unnecessary reanalysis of samples when surrogates or spiked compounds are erroneously not identified.
RT window width calculated for each analyte (and surrogate) (non-MS chromatographic methods only)	Determination of the length of time between sample injection and the appearance of a peak at the detector. The total length of time (window) is established for each analyte or group of analytes and is set for complete elution of analyte peaks. It is based upon a series of analyses and statistical calculations that establish the measured band on the chromatogram that can be associated with a specific analyte or group of analytes.	To ensure that the chromatographic system is operating reliably and that the system conditions have been optimized for the target anaytes and surrogates in the standards and sample matrix to be analyzed. It is done to minimize the occurrence of both false positive and false negative results.	Used to evaluate continued system performance. Tight RT windows may result in false negatives or may cuase unnecessary reanalysis of samples when surrogates or spiked compounds are erroneously not identified. Overly wide RT windows may result in false positive results that cannot be confirmed upon further analysis.
Second source calibration verification (ICV)	A standard obtained or prepared from a source independent of the source of standards for the ICAL. Its concentration should be at or near the middle of the calibration range. It is done after the ICAL.	To verify the accuracy of the ICAL.	The concentration of the 2nd source calibration verification, determined from the analysis, is compared with the known value of the standard to determine the accuracy of the ICAL. This independent verification of the ICAL must be acceptable before sample analysis can begin.

Notes:
1. Project-specific requirements identified by the client supersede any requirements listed. The requirements are meant to be default, to be used when project-specific direction based on DQOs is not available.
2. If there is a contradiction between the method and the DoD tables, the requirements specified in the tables shall be followed.

(023-002)

TestAmerica Chicago

DoD QSM Version 4.2 Appendix F - Quality Control Requirements Summary

Table F-11: Inorganic Analysis by Common Anions: Method 9056

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Demonstrate acceptable analytical capability	Prior to using any test method and at any time there is a significant change in instrument type, personnel, test method, or sample matrix.	QC acceptance criteria published by DoD, if available; otherwise method- specified criteria.	Recalculate results; locate and fix problem, then rerun demonstration for those analytes that did not meet criteria (see Section C.1.f).	NA	This is a demonstration of analytical ability to generate acceptable precision and bias per the procedure in Appendix C. No analysis shall be allowed by analyst until successful demonstration of capability is complete.
LOD Determination and verification (See Box D-13)					
LOQ Establishment and verification (See Box D-14)					
RT window width calculated for each analyte	After method set-up and after major maintenance (e.g., column change).	RT width $\pm$ 3 times standard deviation for each analyte RT over a 24-hour period.	NA	NA	
Initial Calibration (ICAL) for all analytes min 3 standards and one calibration blank	ICAL prior to sample analysis	$r \geq 0.995$.	Correct problem, then repeat ICAL.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until ICAL has passed.
2nd Source calibration verification (ICV)	Once after each ICAL, prior to beginning a sample run.	All analytes within $\pm$ 10% of true value and RTs within appropriate windows.	Correct problem and verify 2nd source standard. Rerun ICV. If that fails, correct problem and repeat ICAL.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until calibration has been verified.
RT window position establishment for each analyte	Once per ICAL	Position shall be set using the midpoint standard of the ICAL curve when ICAL is performed. On days when ICAL is not performed, the initial CCV is used.	NA	NA	
Midrange continuing calibration verification (CCV)	After every 10 field samples and at the end of the analysis sequence.	All project analytes within established RT windows. Within $\pm$ 10% of true value.	Correct problem, then rerun calibration verification. If that fails, then repeat ICAL. Reanalyze all samples since the last successful calibration verification.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to all results for the specific analyte(s) in all samples since the last acceptable calibration verification.	Problem must be corrected. Results may not be reported without a valid CCV. Flagging is only appropriate in cases where the samples cannot be reanalyzed. RT windows are updated per the method.

F-11 (cont.)					
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action**	**Flagging Criteria**	**Comments**
Method Blank (MB)	One per prep batch	No analytes detected > ½ RL and greater than 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater). Blank results must not otherwise affect sample results (see Box D-1).	Correct problem, then see criteria in box D-1; If required, reprep and reanalyze MB and all samples processed with the contaminated blank.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply B-flag to all results for the specific analyte(s) in all samples in the associated prep batch.	Problem must be corrected. Results may not be reported without a valid method blank. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
LCS containing all analytes to be reported	One per prep batch	Laboratory in-house limits not to exceed ± 20%. Control limits may be not greater than ± 3 times the standard deviation of the mean LCS recovery. See Box D-3.	Correct problem, then reprep and reanalyze the LCS and all samples in the associated prep batch for failed analytes, if sufficient sample material is available (see full explanation in Appendix G).	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to specific analyte(s) in all samples in the associated prep batch.	Problem must be corrected. Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Matrix Spike (MS)	One per prep batch per matrix (see Box D-7)	For matrix evaluation, use laboratory in-house LCS limits (not to exceed ± 20%).	Examine the project-specific DQOs. Contact client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met.	For matrix evaluation only. If MS results are outside the LCS limits, the data shall be evaluated to determine the source of difference and to determine if there is a matrix effect or analytical error.
Matrix Spike Duplicate (MSD)	One per prep batch per matrix (see Box D-7)	For matrix evaluation use laboratory in-house LCS limits (not to exceed ± 20%). RPD ≤ 15% (between MS and MSD).	Examine the project-specific DQOs. Contact client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met.	The data shall be evaluated to determine the source of difference.
Sample duplicate (replicate)	One per every 10 samples.	% D ≤ 10% (between sample and sample duplicate).	Correct problem and reanalyze sample and duplicate.	Apply J-flag if sample cannot be rerun or reanalysis does not correct problem.	The data shall be evaluated to determine the source of difference.
Results reported between DL and LOQ	NA	NA	NA	Apply J-flag to all results between DL and LOQ	

Notes:
1. Project-specific requirements identified by the client supersede any requirements listed. The requirements are meant to be default, to be used when project-specific direction based on DQOs is not available.
2. If there is a contradiction between the method and the DoD tables, the requirements specified in the tables shall be followed.

(400-22004) (023-004)

Attachment 8:

Example: Retention time study (3X standard deviation)
(024-001)

TestAmerica Chicago
Instrument: Dionex D-120 Ion Chromatograph
Retention time window establishment - 4th Quarter, 2010
ICV, CCV, LCS data points used for study

Batch #	Date	F	Cl	NO2-N	Br	NO3-N	PO4-P	SO4
97802	10/20/10	1.0320	3.2369	2.0883	2.0829	2.2002		5.3632
		1.0357	3.2823	2.1439	2.1670	2.2446		5.3544
		1.0574	3.1973	2.0791	2.1081	2.1757		5.2601
97924a	10/22/10	1.0659	3.1687	2.0789	2.1003	2.1725		5.3948
97924b	10/23/10	1.0387	3.0550	2.0325	2.0023	2.0874		5.0396
		1.0471	3.0354	2.0105	1.9912	2.0919		5.2067
98127	10/26/10	1.0018	3.0063	1.9880	1.9181	2.0781		4.9757
		1.0193	3.0468	1.9675	1.9154	2.0931		5.0553
98331	10/27/10	1.0918	3.1159	2.0476	2.0828	2.2020		5.4156
		1.0757	3.2047	2.0981	2.1103	2.2109		5.3977
98472	10/28/10	1.0722	3.0483	1.8896	1.9628	1.9847	2.0382	5.1952
		1.0934	3.1302	1.9251	2.0055	2.0375	2.0575	5.3545
98566	10/29/10	1.1012	3.2213	1.9674	1.9323	2.0629	1.8219	5.4232
98626	10/30/10	1.0491	3.1415	1.9828	2.1161	2.0643	2.0088	5.2821
		1.0900	3.1473	1.9740	2.0177	2.0443	1.9077	5.2650
98736	11/01/10	1.0306	3.0167	1.8395	1.9033	1.9577	1.8248	5.0485
		0.8865	3.0946	1.8917	1.9288	1.9973	1.7396	5.1916
99425	11/09/10	1.0051	2.9793	1.8353	1.9628	1.9971	2.1068	5.3609
		1.0397	3.0321	1.9159	2.1406	2.0594	2.1697	5.4716
99717	11/11/10	1.0677	3.0242	1.8620	2.0744	2.0723	2.1500	5.2405
100199	11/17/10	1.0134	2.8268	1.7167	1.8520	1.9989	2.0515	4.9336
		0.9804	2.8549	1.7554	2.0056	2.0295	2.0769	4.9915
100314	11/18/10	0.9941	3.0741	1.9262	1.8502	1.9335	1.9613	5.1116
		1.0355	3.1051	1.9497	1.9295	1.9554	1.9486	5.0211
100423	11/19/10	1.042	3.1421	1.9184	1.8301	1.9618	1.9617	5.0886
		1.0041	3.0155	1.8553	1.8255	1.8519	1.7851	4.7052
100790	11/24/10	1.0401	3.1452	1.871	1.806	1.9653	1.9016	5.0356
		1.0253	3.1568	1.9237	1.931	2.0282	1.9435	5.052
	Average	1.0370	3.0895	1.9476	1.9840	2.0557	1.9697	5.1870
	Std. Dev.	0.043226	0.104673	0.103133	0.104400	0.095231	0.124388	0.186518
	3X Std Dev	0.1297	0.3140	0.3094	0.3132	0.2857	0.3732	0.5596
	5%	0.0518	0.1545	0.0974	0.0992	0.1028	0.0985	0.2593

(024-001)

TITLE: WET CHEMISTRY
Alkalinity

Approvals (Signature/Date):

Carla Bonner 2/25/11
Carla Bonner Date
Supervisor, Wet Chemistry

Diane L. Harper 2/25/11
Diane L. Harper Date
Inorganics Manager

Terese A. Preston 2/25/11
Terese A. Preston Date
Quality Assurance Manager

2/25/2011
John D. Nagel Date
Env. Health & Safety Coor.

Michael J. Healy 3/1/11
Michael J. Healy Date
Laboratory Director

Facility Distribution No: _____ **Distributed To:** Uncontrolled

1.0 SCOPE / APPLICATION

This Standard Operating Procedure (SOP) is used to determine the alkalinity in drinking, surface, and ground waters, and solid matrices. This SOP was written using Standard Methods, 20th Edition, Method 2320B as the primary reference.

There may be several references to the equivalent method EPA 600/4-79-020 Method 310.1 throughout this SOP. As of March 12, 2007, this method has been withdrawn from the Federal Register. There will be a transition period in which the method is removed from laboratory, agency and permit documentation. Regardless, TestAmerica Chicago will remain in compliance with approved methods and regulations.

On occasion, clients request slight modifications to this SOP. These modifications are addressed on a case-by-case basis with the range of accuracy (i.e., MDLs or PT sample) verified prior to implementation. Any modifications would be written into a Quality Assurance Plan (QAP), authorized via laboratory signature approval, and mentioned in the data package's case narrative.

1.1 Method Sensitivity

1.1.1 Method Detection Limits

The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to Appendix B of 40 CFR 136, "Guidelines Establishing Test Procedures for the Analysis of Pollutants" with additional details are provided in the TestAmerica Corporate SOP, *CA-QS-006, Detection Limits* and the TestAmerica Chicago SOP, *UP-QA-017, Method Detection Limit Studies.* MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; these are verified at least annually.

1.1.2 Demonstration of Capability

Before the laboratory may institute a new method and begin reporting results, the laboratory shall confirm that it can properly operate the method. A demonstration of capability is performed whenever there is a change in instrument type, method or personnel. An Initial Demonstration of Capability (IDOC) must be thoroughly documented and approved by the Department Manager/Supervisor and QA Manager prior to independently analyzing client samples. All associated documentation must be retained in the QA Department and in the Analyst Training files. For additional details on the demonstration of capability procedures followed, refer to the laboratory SOP, *UP-QA-QAM, Quality Assurance Manual, Sections 20.4.2 and 20.4.3.*

1.1.3 Reporting Limits

Reporting Limits are defined as the lowest concentration of an analyte determined by a given method in a given matrix that the laboratory feels can be reported with acceptable quantitative error or client requirements, values specified by the EPA methods or other project and client requirements. The laboratory maintains the reporting limits to values ~3-5x the respective MDL to ensure confidence in the value reported.

Matrix	Reporting Limit
Waters	5 mg/L as $CaCO_3$
Wastes/Soils	0.05% (500 mg/kg) as $CaCO_3$

1.1.4 Definitions

Refer to Section 3.0 of the Laboratory's Quality Assurance Manual (UP-QA_QAM).

1.2 Summary of Method

Alkalinity is the quantitative capacity of an aqueous medium to react with hydrogen ions. Hydroxide (OH^-), carbonate ($CO_3^{=}$) and bicarbonate (HCO_3^-) anions expressed as calcium carbonate are responsible for the measure of alkalinity.

Hydroxyl ions present in solutions, as a result of dissociation or hydrolysis of solutes, react with additions of standard acid. In titration of a single ionic species, as in the standardization of reagents, the most accurate endpoint is obtained at the inflection point of the titration curve. The inflection point is the point at which a pH change per milliliter of added acid versus volume of acid added is the maximum. Because accurate identification of inflection points may be difficult or impossible in buffered or complex mixtures, the titration is carried to arbitrary endpoints.

The endpoint is monitored potentiometrically at TestAmerica Chicago. Total alkalinity, with the endpoint pH 4.5, is reported most commonly, but regardless of the species requested, the PC Titrate is programmed to record and calculate total, phenolphthalein, carbonate, bicarbonate, and hydroxide alkalinity for all samples.

All samples are initially titrated with 0.02 N titrant, and samples that are over-range are then re-analyzed in a separate run with 0.1 N titrant.

2.0 INTERFERENCES

- Soaps, oily matter or suspended solids may coat the glass electrode and cause a sluggish response.
- The interfering materials may not be removed to increase precision because some may contribute an important part of the acid-consuming property.
- Similarly, the development of a precipitate during titration may make the glass electrode sluggish and cause high results.

3.0 SAFETY

Employees must abide by the policies and procedures in the Corporate Safety Manual, Radiation Safety Manual, Lab Specific Addendum to the CSM, and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coat and closed-toe, nonabsorbent shoes are a minimum.

3.1 Specific Safety Concerns or Requirements

- Although titrant solutions are relatively weak, caution must be exercised during the preparation and handling of these solutions.

3.2 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **NOTE:** This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table. A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Sulfuric Acid	Corrosive Oxidizer Dehydra-dator	1 mg/m^3	This material will cause burns if comes into contact with the skin or eyes. Inhalation of vapors will cause irritation of the nasal and respiratory system.
1 – Always add acid to water to prevent violent reactions. 2 – Exposure limit refers to the OSHA regulatory exposure limit.			

4.0 EQUIPMENT AND SUPPLIES

- ManTech PC Titrate configured for alkalinity with pH probe and ATC and including the following component model numbers: PC-1104-00, PC-1300-475, PC-1000-550, PC-1000-102/4
- Compatible autosampler
- 50 mL centrifuge tubes compatible with autosampler
- Class A glassware
- Eppendorf pipets

5.0 REAGENTS AND STANDARDS

All reagents are prepared from ACS Reagent grade or better chemicals and with Type II Deionized (DI) Water.

5.1 pH 4 Buffer
pH 7 Buffer
pH 10 Buffer
pH 7 Buffer - Alternate Source

Purchased through a chemical vendor.

- Life of Reagent: 1 year or manufacturer recommendation
- Storage Requirements: None

Note: The following reagents (5.2, 5.3, 5.5, and 5.6) are necessary only if reagent 5.4, the ~0.02 N sulfuric acid titrant is prepared and standardized in-house.

5.2 Sodium Carbonate (Na_2CO_3) Solution, 0.05 N

On a balance capable of reading 0.1 mg, weigh out 2.50 $\pm$ 0.2 grams of analytical reagent grade Sodium Carbonate (Na_2CO_3) (dried at 250°C for 4-hours and cooled in a desiccator). Quantitatively transfer to a 1.0-L volumetric flask and dilute to volume with DI water. Record the exact weight of sodium carbonate used.

- Life of Reagent: 1 week
- Storage Requirements: None

5.3 ~0.1 N Sulfuric Acid (H_2SO_4)

Dilute 3.0 mLs of concentrated Sulfuric Acid to 1.0-L with DI water. Standardize versus 40.0 mLs of 0.05 N Sodium Carbonate Solution (Rgt. 5.2) with about 60 mL DI water by titrating potentiometrically to pH 5. Lift the electrode and rinse it off into the beaker. Boil the solution gently for 3-5 minutes under a watch glass cover. Cool to room temperature. Rinse the watch glass into the beaker. Continue titrating to the pH inflection point. Standardize weekly if prepared in house. This solution is used as the titrant for high samples.

- Life of Reagent: 1 year
- Storage Requirements: None

Alternatively, this titrant may be purchased from a chemical vendor, at a certified normality. JT Baker #5641-3 or equivalent is appropriate for this test.

5.4 ~0.02 N H_2SO_4

Dilute 200 mLs of 0.1 N H_2SO_4 to 1.0-L with DI water. Standardize by potentiometric titration of 15.0 mLs of ~0.05 N Na_2CO_3 Solution (Rgt. 5.2) as above. Standardize weekly if prepared in-house.

- Life of Reagent: 1-year
- Storage Requirements: None

Alternatively, this titrant may be purchased from a chemical vendor, at a certified normality. JT Baker #5693-07 or equivalent is appropriate for this test.

5.5 Potassium Hydrogen Phthalate (KHP) Solution, ~0.05 N

Crush 15 - 20 grams of primary standard KHP to about 100 mesh and dry at 120°C for 2- hours. Cool in a desiccator. Weigh 1.00 ± 0.01 grams of the crushed KHP and transfer it to a 100 mL volumetric flask. Dilute with DI water. This solution is used to standardize the 0.1 N sodium hydroxide solution (Rgt. 5.6).

- Life of Reagent: 1-year
- Storage Requirements: none

5.6 0.1 N Sodium Hydroxide (NaOH)

Dissolve 4.00 grams of dry NaOH pellets in carbon dioxide-free DI water in a 1.0 L volumetric flask. Dilute to volume. This solution is the Stock Standard for Alkalinity, which is equivalent to 100 mg/L as $CaCO_2$ at a 1/50 dilution as used in this SOP. Since this is used as a standard and NaOH itself can't be used as a primary standard, it must be standardized when prepared.

To Standardize:

Transfer 20.0 mLs of Reagent 5.5 into a small beaker on a stir plate, using a 20.0 volumetric pipet. Add sufficient DI water to allow gentle stirring with a small stir bar. Add a few drops of phenolphthalein indicator. The solution will be colorless.

Rinse and then fill a 25.0 mL buret with the 0.1 N NaOH. Titrate, slowing as you near the pink endpoint. Record the mL of NaOH required to reach the endpoint and determine the normality using the following equation:

N x vol = N x vol

Or specifically:

N of NaOH x mL of NaOH = 0.05 x 20.0mL

Adjust the N to 0.10 +/- 0.005 if necessary by adding water or NaOH.

The alkalinity in this standard is hydroxide alkalinity, and is reported as total alkalinity only. The other species will be calculated as non-detects or nearly non-detects and must be rejected in the analytical batches.

- Life of Reagent: 1-year
- Storage Requirements: Secure cap tightly to protect from atmospheric CO_2.

5.7 Standards Preparation

QC Solution	Preparation
Initial Calibration	4.0, 7.0, and 10.0 pH Buffers
Initial and Cont. Calibration Verification (PHC), after calibration and every 10 readings	Alt. 7.00 pH Buffer
Lab Control Sample (LCS)[1] (100 mg/L, total alkalinity only), one per 20 or fewer samples	1 mL Stock Sol. (Rgt. 5.6) to 50 mLs DI Water

[1] An LCS duplicate (LCSD) is sometimes required by client-specific QAPPs.

6.0 CALIBRATION (NON-DAILY)

Not applicable.

7.0 PROCEDURE

7.1 Quality Control Checks

Calibration/QC Standards	Frequency	Control Limit
Method Blank (MB)	1 in 20 samples	< Reporting Limit
Laboratory Control Standard (LCS)	1 in 20 samples	80 – 120% Recovery
Matrix Duplicate (DU) [1]	1 in 20 samples	≤ 20 RPD

[1] The sample selection for the DU, if not defined by the client on the chain-of-custody, is rotated among client samples so that various matrix problems may be noted and/or addressed.

7.2 Sample Preservation and Storage

Holding time, preservation techniques and sample container may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client request. Listed below are the holding times and the references which include container and preservation requirements for compliance with the Clean Water Act (CWA) and Safe Drinking Water Act (SDWA).

Regulation	Holding Time	Preservation	Reference
CWA	14 days	Cool 4 ± 2°C	40 CFR Part 136.3
SDWA	14 days	Cool 4 ± 2°C	EPA 600/4-79-020

7.3 Sample Preparation

7.3.1 Sample Size

This method is for all concentration ranges of alkalinity.

Matrix	Sample Size
Waters	~50 mLs (Fills the autosampler tube. 20 mL is titrated by PC Titrate)
Wastes	>/=1.0 g/100 mLs DI Water[1]. Use ~50 mL of the extract in autosampler.

[1] Extract for 10 minutes on a wrist-action shaker, allow to settle, then filter.

7.4 Calibration / Standardization

Calibration/QC Standards	Frequency	Control Limit
4, 7, and 10 buffers	Initial Calibration	
pH 7.0 (as ICV,CCV)	Initially, every 10 readings, and at the end of the run	± 0.2 pH unit

7.5 Preventive Maintenance

Occasionally, the pH probe should be thoroughly cleaned according to manufacturer's recommendations. Generally, the PC Titrate should be kept clean and free of corrosive residues. The PC should be backed up on a regular basis to preserve data integrity. The tubing on the peristaltic pump will need to be changed periodically for proper functioning. Consult the manual for specific maintenance instructions. All maintenance must be recorded in the log.

7.6 Analysis

7.6.1 Verify that the water reservoir is filled and the drain lines are properly positioned.

7.6.2 Place ~50 mLs of each standard, sample, buffer, and blank in labeled centrifuge tubes and place the tubes in order from back to front, left to right, in the autosampler.

Note: After the final pH 7.0 buffer (last PHC), place a tube containing red pH 4.0 buffer, spiked with fluoride, since the fluoride probe remains in the titration vessel and a fluoride solution extends the life of the probe. The instrument automatically draws from this position to fill the reaction vessel at the end of the run.

7.6.3 From the PC desktop, click on the PC Titrate icon, and log on. Select the syringe icon and purge the syringe. Then select alkalinity by clicking on the green beaker icon at the bottom of the screen.

7.6.4 The run template that appears will have the run sequence already in order under the "sample name" column. Continuing under that column, enter all the run-specific standards and sample IDs in order.

7.6.5 Click on "save template". Enter today's date under "enter search text", and then click on "OK".

7.6.6 Click on "check time table" and then "start".

7.6.7 The instrument will first calibrate the pH meter, then it will continue to the end of the run. The instrument will draw each sample or standard from the autosampler into the reaction vessel, where the ~0.02 N H_2SO_4 and the 20-mL volume of TISAB are added. The PC Titrate records the alkalinity result, in mg/L as $CaCO_3$, when it is electronically stable at each of the endpoints, when applicable. The time required may vary from sample to sample.

Documentation

When the run is complete, select items to print from across the top of the screen and print them one by one. Make sure all the raw data is printed before you exit the program. Attach these pages to the data review checklist to be reviewed against the LIMS entries.
The run is saved automatically. Transfer the run directly into TALs by double-clicking on the instrument PC desktop ICON labeled "ManTech File Transfer Utility for STL LIMS". In the pop-up screen, click on the [...] button to the right of the space for the "Raw Data File", then identify your file by date (usually it will be the last file in the list). Open the file and highlight the alkalinity parameters from the list on the left side of the screen: pH, alk, palk, carb, bicarb, hydrox.. Click on "transfer file". You will get confirmation that the transfer was successful.

7.7.1 Raw Data

The analysis of samples and standards is documented on the PC Titrate print-outs which must be accompanied by an Alkalinity Calculation page (Attachment 1).

7.7.2 Traceability of Standards

Upon preparation, the standard NaOH is entered into TALs LIMS and is issued a unique ID#. Further information entered into the database includes the manufacturer, lot # (if applicable), the date prepared, the expiration date, volume/weight received; concentration; preparation details (if applicable), initials of the analyst, and the description of the item. Once the record is created, a unique label is printed and affixed to the standard container.

7.7.3 Data Review

Analytical data goes through a 200% review cycle. As results are generated, analysts review their work to ensure that the results generated meet QC requirements and relevant EPA methodologies. The analyst transfers the data into TALs in the Analyst Desktop module. Where non-compliance is observed, the analyst creates Non-Conformance Memos (NCMs) in TALs. Flags and data qualifiers can be method, project, program or QAPP specific. The analyst documents the initial review on a data review checklist (Attachment 2) and sets the batch status in

LIMs to 1st level review. The second level or peer review of the data is conducted by another individual who has been trained on the review process. This secondary review is documented on the same checklist, making any necessary corrections to the data or additions to the NCMs as necessary. The batch is then set to 2nd level review. The raw data, including the checklist, instrument print-outs, and manual entries, and electronic files are retained for easy retrieval in accordance with the laboratory's record and retention policy outlined in the SOP, *UP-QA-QAM, Section 15.*

Examples of items included in the above reviews are as follows:

- QC data are outside the specified control limits for accuracy and precision
- Unusual detection limit changes are observed
- Samples having unusually high results
- Samples exceeding a known regulatory limit
- Raw data indicating some type of contamination or poor technique
- Transcription errors
- Results outside of calibration range

Notes:

Very high alkalinities may appear incorrectly on the instrument pages as non-detects. Review the results carefully. Any zero result for a sample with pH >4.5 must be diluted and repeated. Dilutions must be manually entered on the instrument printouts.

Because pH is a logarithmic scale, the acceptance limits are +/- 0.2 SU (Standard Units). TALS is unable to evaluate or report +/- limits; therefore, the ICV/CCVs are not reported from TALS.

8.0 QUALITY CONTROL

8.1 QC Summary

8.1.1 One MB and one LCS will be included in each laboratory lot of 20 or fewer samples. The MB will be examined to determine if contamination is being introduced in the laboratory. The LCS will be examined to determine accuracy of the method and cumulative LCS data will provide precision data.

8.1.2 Accuracy will be measured by the percent recovery (%) of the LCS. The recovery must be in range, as determined by in-house control limits or statistical analysis, in order to be considered acceptable. And, precision will be measured by the cumulative reproducibility of the LCSs.

8.1.3 The Method Duplicate, identified in TALS as DU, is performed per matrix per 20-sample analytical set, unless otherwise requested. Results must agree within the in-house precision limits or statistical control limits in order to be considered acceptable.

8.2 Corrective Actions

When an out-of-control situation occurs, the analysts must use his/her best analytical judgment and available resources to determine the corrective action to be taken. The out-of-control situation may be caused by more than one variable. The analyst should seek the assistance of his/her immediate supervisor, QA personnel, or other experienced staff if he/she is uncertain of the cause of the out-of-control situation. The test must not be resumed until the source of the problem and an in-control status is attained. All samples associated with the out of control situation should be reanalyzed. Out-of-control data must never be released without approval of the Supervisor, Inorganics Manager, Project Manager, QA personnel or the Lab Director.

Listed below are steps that MUST be taken when an out-of-control situation occurs:

- demonstrate that all the problems creating the out of control situation were addressed;
- document the problem and the action which was taken to correct the problem on the data review checklist and in a Non-Conformance Memo (NCM) in TALS;
- receive approval (signature) of the supervisor, Project Manager, QA personnel, or the Laboratory Director prior to the release of any analytical data associated with the problem.

Suggested Actions to specific out of control situations:

QC Indicator	Suggested Corrective Actions
ICV	• repeat the ICV to verify proper preparation; • pour fresh buffer; • check for instrument base-line drift; • re-calibrate with fresh buffers, reanalyze.
LCS	• reanalyze LCS to verify that an out of control situation exists; • determine the source of error within the preparation procedure, correct the problem and repeat the sample set. (Sources of contamination could be the reagents, the LCS stock solution, or the preparation area.) • Non-detect samples may be reported if the LCS bias is high, but note out-of-control LCSs on the data review checklist and in an NCM.
MB	• Reanalyze the MB to verify that the contamination is at a level > Reporting Limit; • Determine the source of contamination and correct the problem; • all samples whose concentration is <10 times the MB level must be reprocessed and reanalyzed; any sample which is >10 times the MB level need not be reanalyzed. However, note out-of-control MBs on the data review checklist and in an NCM.
Matrix Duplicate (DU)	• the sample must be reprocessed and reanalyzed unless the sample concentration is <5 times the Reporting Limit, then the ± Reporting Limit rule applies; • if the reanalysis is within the control limits, the second value is reported; • if the reanalysis is still outside of the control limits, a CAR must be written and then approved by your Section Manager. • Note out-of-control DUs on the data review checklist and in an NCM.
CCV	• Repeat the CCV to verify a proper reading; • Pour a fresh CCV buffer; • Check for instrument base-line drift; • Recalibrate with a fresh buffers and repeat all samples since the previous in-control CCV; • Never dispose of any samples until you are sure that all QC are within their designated control limits.

9.0 DATA ANALYSIS AND CALCULATIONS

9.1 Normality of Titrant, N = $\frac{A \times B}{53 \times C}$

Where:
A = g Na_2CO_3 weighed into 1.0 L
B = mL Na_2CO_3 solution taken from titration
C = mL acid used to inflection point

This is only required when preparing titrant at the laboratory. Purchased titrant is certified by the manufacturer at a certain normality, given on the label.

9.2 Alkalinity: >20 mg/L as $CaCO_3$ mg $CaCO_3$/L (kg) = $\frac{A \times N \times 50{,}000}{\text{mL or g sample}}$

Where:
A = mL standard acid used
N = Normality of standard acid (titrant)

The sample results are calculated by the PC Titrate software, based on 20 mL of sample volume, the volume of titrant added by the auto-titrator, and the normality of the titrant as entered by the analyst.

9.3 Alkalinity: <20 mg/L as $CaCO_3$ mg $CaCO_3$/L (kg) = $\frac{(2B - C) \times N \times 50{,}000}{\text{mg or g sample}}$

Where:
B = mL titrant to 1st recorded pH
C = B + mL titrant to lower pH the additional 0.30 units
N = Normality of standard acid (titrant)

The additional low-level titration and this corrective calculation are done automatically by the PC Titrate software.

9.4 Converting Soil/Waste Results to Percent Alkalinity = $\frac{\text{mg/kg}}{10{,}000}$

This calculation is done in TALS.

9.5 Accuracy

9.5.1 LCS % Recovery = $\frac{\text{observed concentration}}{\text{actual concentration}} \times 100$

9.6 Precision

9.6.1 DU Relative Percent Difference (RPD)

$$RPD = \frac{|\text{orig. sample value} - \text{dup. sample value}|}{[(\text{orig. sample value} + \text{dup. sample value})/2]} \times 100$$

9.7 Alkalinity Relationships

The following discussion is included for explanatory purposes only. The PC Titrate provides the results for all alkalinity forms for all samples.

In addition to total alkalinity, calculation of alkalinity relationships is also possible. The results obtained for a phenolphthalein end point and the total alkalinity determination offers a stoichiometric classification of the three principle forms of alkalinity present in many waters. The classification ascribes the entire alkalinity to bicarbonate, carbonate and hydroxide, and assumes the absence of other (weak) inorganic or organic acids such as phosphoric and boric acids.

When alkalinity relationships are requested, the sample aliquot must first be titrated to an endpoint of pH 8.3. Record the titrant used. Then the titration is continued to pH 4.5. From the phenolphthalein alkalinity (to pH 8.3) and the total alkalinity, the various forms are calculated using the following table. Again, this is done for every sample by the PC Titrate. The analyst need only report what the client requests.

Relationship	Hydroxide Alkalinity	Carbonate Concentration	Bicarbonate Concentration
P = 0	< 5	< 5	T
P < 1/2 T	< 5	2P	T – 2P
P = 1/2 T	< 5	2P	< 5
P > 1/2 T	2P – T	2(T-P)	< 5
P = T	T	< 5	< 5

P = Phenolphthalein alkalinity (as $CaCO_3$ titrated to pH 8.3)
T = Total alkalinity (as $CaCO_3$ titrated to pH 4.5)

10.0 POLLUTION CONTROL

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

10.1 Waste Management

Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to UP-WM-001.

There are no special waste streams associated with this method.

- Waste from this procedure will enter the "Corrosive wastewater" wastestream.
- Single component standards will be turned over to the EHSC or Waste Technician.

11.0 METHOD PERFORMANCE CRITERIA

Refer to Sections 1, 6, 7, and 8.

12.0 REFERENCES

Refer to Section 1.0

13.0 ATTACHMENTS

Attachment 1. Example: Alkalinity Calculations page
Attachment 2. Example: Data Review Checklist
Attachment 3. Example: PC Titrate alkalinity print-outs

14.0 REVISION HISTORY

- Revision 12 updated on 02/24/11
- Annual Review
- Added PC Tritrate model numbers to section 4.0
- Added standardization instructions to section 5.6
- Added the option of purchasing Reagent 5.3

Attachment 1.

Example: Alkalinity Calculations page (014-001)

TestAmerica Chicago

Alkalinity

Analytical Batch # _____

NCM # _____

Method: SM 2320B

Instrument: PC Titrate

Alkalinity Calculations :

> 20 mg/L alk = tcon x 50,000 x ep1 or ep2a / svol

< 20 mg/L alk = (2 X ep2a - ep2b) X tcon X 50,000 / svol

where:

ep1 = mLs titrant to pH 8.3 (endpoint for Phenolphthalein Alkalinity)
ep2a = mLs Titrant to pH 4.5 (endpoint for Total Alkalinity)
ep2b = total mLs Titrant to lower pH an additional 0.30 units
tcon = Titrant normality
svol = mL sample volume

Alkalinity Relationships :

Result (as CaCO3)	OH Alk.	Carb. Alk.	Bicarb. Alk.
P = 0	0	0	T
P < 1/2 T	0	2P	T - 2P
P = 1/2 T	0	2P	0
P > 1/2 T	2P - T	2(T - P)	0
P = T	T	0	0

Key

P = Phenophthalein Alkalinity (as CaCO3 titr. To pH 8.3)
T = Total Alkalinity (as CaCO3 titr. To pH 4.5)
OH = Hydroxide Alkalinity

Note: All calculations are performed by the PC Titrate Software. The PC Titrate autosampler draws 20 ml of each buffer, standard, and sample for titration.

Standard Traceability:

ICAL	pH Buffer 4.0	_____
ICAL	pH Buffer 7.0	_____
CCAL	pH Buffer 7.0	_____
	LCS	_____

Spiking Levels:

LCS _____ mg/L

Analyst: _____ Analysis Date: ____

Reviewer: _____ Date: _____

CHI-22-12-114/C-11/09

(014-001)

Attachment 2.

Example: Data Review Checklist (015-001)

TestAmerica Chicago
INORGANICS – LIMS DATA REVIEW CHECKLIST **Test** _____ **Analytical Batch#** _____

Prep Batch # _____
(File by analytical batch #)

Batch Entry Date: _____ Analysis Date: _____ No. of Jobs in Batch: _____

Analyst / Primary Reviewer: _____ 1st Level Review Date: _____

Secondary Reviewer: _____ 2nd Level Review Date: _____

	PRI REV	SEC REV	COMMENTS
1) Analyst correct			
2) Instrument Code present			
3) Was Data Imported _____ Manually entered _____ Balance Interface Used _____			
4) Samples & all QC in order as analyzed?			
5) Sample Date/Time analyzed correct			
6) Reagent Codes present and Amount Spiked correct?			
7) Dilution factors all present and correct?			
8) Are correct Sample ID's used? Are all samples designated with a Blue P?			
9) Are correct QC ID's used? Are all QC designated with a Blue P?			
10) Are all QC correctly related to the samples?			
11) Do all entries match raw data?			
12) Is all QC calculated and are correct flags applied?			
13) Is an NCM needed? ICV, MB, LCS, LCSD, DU, MS, MSD, RPD out; holding time missed or Manual Integration Required			NCM # _____ Approved By: _____Initials
Raw Data: 1) Is AD Batch # is clearly noted?			
2) Are manual calculations and final results clearly shown?			
3) Are all errors crossed out with single line & initialed and dated?			
4) Is unreported data clearly identified with reason & initialed and dated?			
5) All unused portions of the page(s) Z'd out?			
6) Is data signed & dated by analyst & reviewer?			

CHI-22-12-109/E-08/08

(015-001)

Attachment 3.

Example: PC Titrate alkalinity print-outs
(016-001 to 016-005)

105925

CM 2-18-11

TAL Chicago

Wet Lab

Report Date: 02/18/2011 **Run Number** **Order Number**

Report Time: 15:19 **3171** **20110218-1**

SampleID	RunDate	RunTime	pH	palk-ppm	Total Alk (ppm)	Bi-Carbonate	Carbonate	Hydroxide	F-ppm
phc	02/18/2011	12:43	6.93	.00	2,152.58	2,152.58	.00	.00	-1.00
mb	02/18/2011	12:47	4.97	.00	.97	.97	.00	.00	-1.00
lcs	02/18/2011	12:55	10.89	88.59	96.79	.00	16.40	80.39	-1.00
500-30936-A-1	02/18/2011	13:04	8.16	.00	510.42	510.42	.00	.00	-1.00
500-30972-I-1	02/18/2011	13:11	7.86	.00	127.40	127.40	.00	.00	-1.00
680-65669-A-1	02/18/2011	13:18	6.05	.00	4.10	4.10	.00	.00	-1.00
680-65669-A-2	02/18/2011	13:24	7.27	.00	39.38	39.38	.00	.00	-1.00
680-65705-A-1	02/18/2011	13:31	7.26	.00	32.05	32.05	.00	.00	-1.00
680-65705-A-2	02/18/2011	13:38	7.30	.00	32.46	32.46	.00	.00	-1.00
680-65705-A-3	02/18/2011	13:43	4.88	.00	-.68	-.68	.00	.00	-1.00
680-65705-A-3 DU	02/18/2011	13:48	4.76	.00	-.58	-.58	.00	.00	-1.00
phc-1	02/18/2011	14:08	7.00	.00	2,286.13	2,286.13	.00	.00	-1.00
680-65762-O-1	02/18/2011	14:13	6.02	.00	2.17	2.17	.00	.00	-1.00
680-65762-O-2	02/18/2011	14:20	7.29	.00	25.11	25.11	.00	.00	-1.00
680-65762-O-3	02/18/2011	14:27	6.56	.00	4.52	4.52	.00	.00	-1.00
680-65762-O-4	02/18/2011	14:32	4.33	.00	-1.37	-1.37	.00	.00	-1.00
680-65762-O-5	02/18/2011	14:36	4.03	.00	.00	.00	.00	.00	-1.00
680-65762-O-6	02/18/2011	14:40	4.08	.00	.00	.00	.00	.00	-1.00
680-65762-O-7	02/18/2011	14:45	4.60	.00	-1.00	-1.00	.00	.00	-1.00
680-65762-O-8	02/18/2011	14:51	5.34	.00	.53	.53	.00	.00	-1.00
680-65762-S-9	02/18/2011	14:57	6.03	.00	2.03	2.03	.00	.00	-1.00
phc-2	02/18/2011	15:17	7.00	.00	2,220.32	2,220.32	.00	.00	-1.00

PC-TitratION PLUS by Man-Tech Associates, Inc.

(016-001)

105925

CM 2-18-11

PC-Titrate For Windows

Exported Equation Results

Sample ID	pH	pHmV	stbT	Temp
phc	6.932	-18.180	91.350	22.187
mb	4.974	97.290	92.144	21.946
lcs	10.888	-253.660	91.845	22.339
500-30936-A-1	8.157	-88.250	26.085	19.641
500-30972-I-1	7.860	-72.020	92.314	19.945
680-65669-A-1	6.054	33.560	92.219	19.918
680-65669-A-2	7.273	-37.590	91.694	19.852
680-65705-A-1	7.258	-36.740	92.085	20.094
680-65705-A-2	7.298	-38.940	92.164	19.931
680-65705-A-3	4.884	102.780	92.386	20.867
680-65705-A-3 DU	4.760	110.350	91.564	21.237
phc-1	7.000	-21.850	91.840	20.895
680-65762-O-1	6.022	36.010	91.965	20.936
680-65762-O-2	7.291	-38.690	91.450	20.991
680-65762-O-3	6.560	4.390	91.509	21.078
680-65762-O-4	4.330	135.490	71.330	21.107
680-65762-O-5	4.027	153.560	81.060	21.498
680-65762-O-6	4.083	150.630	79.931	22.202
680-65762-O-7	4.599	120.360	91.542	21.599
680-65762-O-8	5.338	76.290	92.450	21.512
680-65762-S-9	6.030	35.400	91.590	21.903
phc-2	7.000	-21.600	91.646	21.715

Date: 02/18/2011

(016-002)

105925

CM 2-18-11

PC-Titrate For Windows

Exported Equation Results

Sample ID	palk	talk	ep1	ep2	svol	tcon
phc	0.000	2152.575	0.000	43.052	20.000	0.020
mb	0.000	1.769	0.000	0.035	20.000	0.020
lcs	88.587	96.786	1.772	1.936	20.000	0.020
500-30936-A-1	0.000	510.421	0.000	10.208	20.000	0.020
500-30972-I-1	0.000	127.405	0.000	2.548	20.000	0.020
680-65669-A-1	0.000	5.680	0.000	0.114	20.000	0.020
680-65669-A-2	0.000	39.381	0.000	0.788	20.000	0.020
680-65705-A-1	0.000	32.050	0.000	0.641	20.000	0.020
680-65705-A-2	0.000	32.458	0.000	0.649	20.000	0.020
680-65705-A-3	0.000	1.424	0.000	0.028	20.000	0.020
680-65705-A-3 DU	0.000	1.089	0.000	0.022	20.000	0.020
phc-1	0.000	2286.131	0.000	45.723	20.000	0.020
680-65762-O-1	0.000	3.382	0.000	0.068	20.000	0.020
680-65762-O-2	0.000	25.108	0.000	0.502	20.000	0.020
680-65762-O-3	0.000	5.916	0.000	0.118	20.000	0.020
680-65762-O-4	0.000	0.000	0.000	0.000	20.000	0.020
680-65762-O-5	0.000	0.000	0.000	0.000	20.000	0.020
680-65762-O-6	0.000	0.000	0.000	0.000	20.000	0.020
680-65762-O-7	0.000	0.599	0.000	0.012	20.000	0.020
680-65762-O-8	0.000	1.925	0.000	0.039	20.000	0.020
680-65762-S-9	0.000	3.510	0.000	0.070	20.000	0.020
phc-2	0.000	2220.316	0.000	44.406	20.000	0.020

Date: 02/18/2011

(016-003)

105925

CM 2-18-11

PC-Titrate For Windows

Exported Equation Results

Sample ID	cond	pH	palk	talk	bcarb	carb	hydrx	flrd	Cl	NH3
phc	-1.000	6.932	0.000	2152.575	2152.575	0.000	0.000	-1.000	-1.000	-1.000
mb	-1.000	4.974	0.000	0.967	0.967	0.000	0.000	-1.000	-1.000	-1.000
lcs	-1.000	10.888	88.587	96.786	0.000	16.398	80.388	-1.000	-1.000	-1.000
500-30936-A-1	-1.000	8.157	0.000	510.421	510.421	0.000	0.000	-1.000	-1.000	-1.000
500-30972-I-1	-1.000	7.860	0.000	127.405	127.405	0.000	0.000	-1.000	-1.000	-1.000
680-65669-A-1	-1.000	6.054	0.000	4.102	4.102	0.000	0.000	-1.000	-1.000	-1.000
680-65669-A-2	-1.000	7.273	0.000	39.381	39.381	0.000	0.000	-1.000	-1.000	-1.000
680-65705-A-1	-1.000	7.258	0.000	32.050	32.050	0.000	0.000	-1.000	-1.000	-1.000
680-65705-A-2	-1.000	7.298	0.000	32.458	32.458	0.000	0.000	-1.000	-1.000	-1.000
680-65705-A-3	-1.000	4.884	0.000	-0.680	-0.680	0.000	0.000	-1.000	-1.000	-1.000
680-65705-A-3 DU	-1.000	4.760	0.000	-0.582	-0.582	0.000	0.000	-1.000	-1.000	-1.000
phc-1	-1.000	7.000	0.000	2286.131	2286.131	0.000	0.000	-1.000	-1.000	-1.000
680-65762-O-1	-1.000	6.022	0.000	2.171	2.171	0.000	0.000	-1.000	-1.000	-1.000
680-65762-O-2	-1.000	7.291	0.000	25.108	25.108	0.000	0.000	-1.000	-1.000	-1.000
680-65762-O-3	-1.000	6.560	0.000	4.519	4.519	0.000	0.000	-1.000	-1.000	-1.000
680-65762-O-4	-1.000	4.330	0.000	-1.370	-1.370	0.000	0.000	-1.000	-1.000	-1.000
680-65762-O-5	-1.000	4.027	0.000	0.000	0.000	0.000	0.000	-1.000	-1.000	-1.000
680-65762-O-6	-1.000	4.083	0.000	0.000	0.000	0.000	0.000	-1.000	-1.000	-1.000
680-65762-O-7	-1.000	4.599	0.000	-0.998	-0.998	0.000	0.000	-1.000	-1.000	-1.000
680-65762-O-8	-1.000	5.338	0.000	0.526	0.526	0.000	0.000	-1.000	-1.000	-1.000
680-65762-S-9	-1.000	6.030	0.000	2.030	2.030	0.000	0.000	-1.000	-1.000	-1.000
phc-2	-1.000	7.000	0.000	2220.316	2220.316	0.000	0.000	-1.000	-1.000	-1.000

Date: 02/18/2011

(016-004)

105925

CM 2-18-11

PC-Titrate For Windows

Exported Equation Results

Sample ID	palk	talk	ep1	ep2a	ep2b	svol	tcon
mb	0.000	0.967	0.000	0.035	0.051	20.000	0.020
680-65669-A-1	0.000	4.102	0.000	0.114	0.145	20.000	0.020
680-65705-A-3	0.000	-0.680	0.000	0.028	0.071	20.000	0.020
680-65705-A-3 DU	0.000	-0.582	0.000	0.022	0.055	20.000	0.020
680-65762-O-1	0.000	2.171	0.000	0.068	0.092	20.000	0.020
680-65762-O-3	0.000	4.519	0.000	0.118	0.146	20.000	0.020
680-65762-O-4	0.000	-1.370	0.000	0.000	0.027	20.000	0.020
680-65762-O-5	0.000	0.000	0.000	0.000	0.000	20.000	0.020
680-65762-O-6	0.000	0.000	0.000	0.000	0.000	20.000	0.020
680-65762-O-7	0.000	-0.998	0.000	0.012	0.044	20.000	0.020
680-65762-O-8	0.000	0.526	0.000	0.039	0.066	20.000	0.020
680-65762-S-9	0.000	2.030	0.000	0.070	0.100	20.000	0.020

Date: 02/18/2011

(016-005)

TITLE: WET CHEMISTRY
Chemical Oxygen Demand (COD)

Approvals (Signature/Date):

Carla Bonner 9/30/11
Carla Bonner Date
Supervisor, Wet Chemistry

Diane L. Harper 9/30/11
Diane L. Harper Date
Inorganics Manager

Terese A. Preston 9/30/11
Terese A. Preston Date
Quality Assurance Manager

10/3/11
John D. Nagel Date
Env. Health & Safety Coor.

Michael J Healy 9/30/11
Michael J. Healy Date
Laboratory Director

Facility Distribution No: _____ **Distributed To:** Uncontrolled

1.0 SCOPE / APPLICATION

This Standard Operating Procedure (SOP) is used to determine the Chemical Oxygen Demand (COD) content in water samples at concentrations of 0 - 1500 mg/L. Occasional requests are made to analyze soils or sludge and instructions are included for that matrix.

- Low Range: 0 -150 mg/L
- High Range: 50 -1500 mg/L

This SOP is based on Standard Methods, 20th Ed., Method 5220C.

On occasion, clients request slight modifications to this SOP. These modifications are addressed on a case-by-case basis with the range of accuracy (i.e., MDLs or PT sample) verified prior to implementation. Any modifications would be written into a Quality Assurance Plan (QAP) and authorized via laboratory signature approval; and amended to the data packages case narrative.

Specific requirements pertaining to the DOD QSM version 4.2 are located in Attachment 3. These requirements are additionally applicable to all NFESC projects. Any deviations from these procedures and/or variances from must be addressed appropriately and pre-approved on a project-by-project basis.

1.1 Method Sensitivity

1.1.1 Method Detection Limits

The method detection limit (MDL), referred to as the detection limit (DL) in NELAC and DOD QSM documents, is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to Appendix B of 40 CFR 136, "Guidelines Establishing Test Procedures for the Analysis of Pollutants" with additional details are provided in the TestAmerica Corporate SOP, *CA-QS-006, Detection Limits* and the TestAmerica Chicago SOP, *UP-QA-017, Method Detection Limit Studies.* MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; the MDL will be verified on a quarterly basis to meet the requirements of the DoD QSM version 4.2.

1.1.2 Demonstration of Capability

Before the laboratory may institute a new method and begin reporting results, the laboratory shall confirm that it can properly operate the method. A demonstration of capability is performed whenever there is a change in instrument type, method or personnel. An Initial Demonstration of Capability (IDOC) must be thoroughly documented and approved by the Department Manager/Supervisor and QA Manager prior to independently analyzing client samples. All associated documentation must be retained in the QA Department and in the Analyst Training files. For additional details on the demonstration of capability procedures followed, refer to the laboratory SOP, *UP-QA-QAM, Quality Assurance Manual, Sections 20.4.2 and 20.4.3.*

1.1.3 Reporting Limits

Reporting Limits are defined as the lowest concentration of an analyte determined by a given method in a given matrix that the laboratory feels can be reported with acceptable quantitative error or client requirements, values specified by the EPA methods or other project and client requirements. Because of the high level of quantitative error associated with determinations at the level of the MDL, the laboratory maintains reporting limits higher than the MDL. Wherever

possible, reporting is limited to values ~3-5x the respective MDL to ensure confidence in the value reported.

Range	Reporting Limits[1]
Low-Range Waters	10 mg/L
High-Range Waters	100 mg/L
Solid/Sludge	400 mg/Kg[2]

[1] Reporting limits will vary depending on sample size/volume, dilution factors and annual MDL determinations.
[2] This limit is based on a maximum sample size of 0.5 grams per 2 mL, and is a wet weight limit which will vary with actual size and % solids correction. It also assumes the high range vials are used.

1.1.3 Definitions

Refer to Section 3.0 of the Laboratory's Quality Assurance Manual (UP-QA_QAM).

1.2 Summary of Method

The COD is used as a measure of the oxygen equivalent of the organic matter content of a sample that is susceptible to oxidation by a strong chemical oxidant. Most types of organic material are oxidized by potassium dichromate ($K_2Cr_2O_7$) in the presence of strong sulfuric acid and high temperature (150°C). Chromium is reduced in the process.

$$Cr^{+6} \text{ (yellow brown)} \quad \rightarrow \quad Cr^{+3} \text{ (blue)}$$

The method titrimetrically measures the amount of unreacted potassium dichromate available after the oxidation has taken place to determine the COD of a sample.

2.0 mLs of thoroughly mixed sample, or a small portion of well-mixed solid sample diluted to ~2 mL with DI water, is added to a HACH COD digestion vial, tightly capped, shaken vigorously, and allowed to reflux in a block digester at 150°C for 2 hours. After the vials have cooled, the unreacted Cr^{+6} in the vials is titrated with ferrous ammonium sulfate (FAS) to determine the amount of Cr^{+6} that was not reduced by the sample. The difference between the initial and final amounts of Cr^{+6} is used to calculate the COD.

2.0 INTERFERENCES

- Chlorides are quantitatively oxidized by dichromate and represent a positive interference. The COD vials contain enough mercuric sulfate to complex more than 1,000 mg/L chloride.
- Silver sulfate has been added to the vial as a catalyst to oxidize straight-chain aliphatic compounds. Silver sulfate, however, does react with chloride, bromide, and iodide to produce precipitates that are oxidized only partially.
- Reduced inorganic species are oxidized under test conditions. If significant levels of species such as ferrous iron, sulfide, or manganous manganese are present, stoichiometric oxidation can be assumed and corrections can be made to the COD value obtained, although this is not done at TestAmerica Chicago.

3.0 SAFETY

Employees must abide by the policies and procedures in the Corporate Safety Manual, Radiation Safety Manual, Lab Specific Addendum to the CSM, and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coat and closed-toe, nonabsorbent shoes are a minimum.

3.1 Specific Safety Concerns or Requirements

The disposable glass COD digestion tubes used in this analysis contain Mercuric Sulfate, a compound composed of Mercuric Oxide Red, which may affect the central nervous system, chromium, and Sulfuric acid.

The vials will get hot when mixed, and there is the possibility of pressure explosion, which would distribute the toxins listed above and hot sulfuric acid. Always wear protective gloves, a lab coat, and a face shield when shaking the vials prior to digestion.

3.2 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **NOTE: This list does not include all materials used in the method.** The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table. A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Mercuric Oxide, Red	Oxidizer Corrosive Poison	0.1 Mg/M3 Ceiling (Mercury Compounds)	Extremely toxic. Causes irritation to the respiratory tract. Causes irritation. Symptoms include redness and pain. May cause burns. May cause sensitization. Can be absorbed through the skin with symptoms to parallel ingestion. May affect the central nervous system. Causes irritation and burns to eyes. Symptoms include redness, pain, and blurred vision; may cause serious and permanent eye damage.
Potassium Dichromate	Oxidizer Corrosive Carcinogen	0.1 Mg/M3 TWA as CrO3	Extremely destructive to tissues of the mucous membranes and upper respiratory tract. May cause ulceration and perforation of the nasal septum. Symptoms of redness, pain, and severe burn can occur. Dusts and strong solutions may cause severe irritation. Contact can cause blurred vision, redness, pain and severe tissue burns. May cause corneal injury or blindness.
Sulfuric Acid	Corrosive Oxidizer Dehydrator Poison Carcinogen	1 Mg/M3-TWA	Inhalation produces damaging effects on the mucous membranes and upper respiratory tract. Symptoms may include irritation of the nose and throat, and labored breathing. Symptoms of redness, pain, and severe burn can occur. Contact can cause blurred vision, redness, pain and severe tissue burns. Can cause blindness.
1 – Always add acid to water to prevent violent reactions. 2 – Exposure limit refers to the OSHA regulatory exposure limit.			

4.0 EQUIPMENT AND SUPPLIES

- COD vial titration stand
- Block digester
- 10 mL Class A buret
- Micro stir bar
- Stir plate
- PPE including a face shield

5.0 REAGENTS AND STANDARDS

All standards, reagents, and QC solutions are prepared with Milli-Q Water, unless otherwise stated, in class A volumetric flasks. Purchased chemicals must be reagent grade.

5.1 Reagents

5.1.1 Ferroin Indicator

Dissolve 1.485 g 1,10-Phenanthroline monohydrate and 695 mg Ferrous Sulfate·7 H_2O in DI water and dilute to 100 mLs. Alternatively, this may be purchased.

- Life of Reagent: 1 Year
- Storage Requirements: None

5.1.2 Sulfuric Acid, Concentrated

Purchased.

- Life of Reagent: See vendor requirements
- Storage Requirements: Store in acid cabinet

5.2 Standards

5.2.1 Low Range

5.2.1.1 COD Stock I; 10,000 mg/L

On a balance capable of reading to 0.1 mg, weigh out 4.2517 g dried Potassium Hydrogen Phthalate (KHP). Dissolve in ~400 mL Milli-Q water in a 500 m L class A volumetric flask and bring to volume.

- Life of Standard: 2 Months
- Storage Requirements: Refrigerate

5.2.1.2 ~0.0125 N FAS Titrant

On a balance capable of reading to 0.1 mg, weigh out 4.9012 g Ferrous Ammonium Sulfate (FAS) [$Fe(NH_4)_3(SO_4)_2 \cdot 6H_2O$]. Dissolve in ~800 mL Milli-Q water in a 1.0 L volumetric flask. Add 20 mL concentrated sulfuric acid and dilute to volume. Standardize daily using potassium dichromate (0.025 N).

- Life of Reagent: 1 Year, monitor daily standardization
- Storage Requirements: Amber bottle, store in the dark

5.2.1.3 0.025 N Potassium Dichromate

On a balance capable of reading to 0.1 mg, weigh out 1.2258 g potassium dichromate (dried for 2 hours at 103-105°C) and dissolve in ~800 mL Milli-Q water in a 1.0 L volumetric flask.

- Life of Reagent: 1 Year
- Storage Requirements: None

5.2.1.4 QC Samples (Low Range)

Quality Control	Preparation
Method Blank (MB)	2 mLs of Milli-Q water added to a low-range vial.
Lab Control Sample (LCS) (50 mg/L)	Dilute 500 uLs of Rgt. 5.2.1.1 to 100 mLs of Milli-Q water in a 100 mL volumetric flask.
Matrix Spike (MS) / MS Duplicate (MSD) (50 mg/L)	Add 500 uLs of Rgt. 5.2.1.1 to ~90 mLs of sample in a 100 mL volumetric flask. Dilute to mark with sample.

5.2.2 High Range

5.2.2.1 COD Stock II, 50,000 mg/L

On a balance capable of reading to 0.1 mg, weigh out 4.2517 g dried KHP. Dissolve in ~90 mLs of warm Milli-Q water in a 100 mL volumetric flask. Allow to cool to room temperature and dilute to mark.

- Life of Standard: 2 Months
- Storage Requirements: Refrigerate

5.2.2.2 ~0.125 N FAS Titrant

On a balance capable of reading to 0.1 mg, weigh out 49.012 g Ferrous Ammonium Sulfate (FAS) [$Fe(NH_4)_3(SO_4)_2 \cdot 6H_2O$]. Dissolve in ~800 mL Milli-Q water in a 1.0 L volumetric flask. Add 20 mL of concentrated sulfuric acid and dilute to volume. Standardize daily.

- Life of Reagent: 1 Year, monitor daily standardization
- Storage Requirements: Amber bottle, store away from light

5.2.2.3 0.25 N Potassium Dichromate

On a balance capable of reading to 0.1 mg, weigh out 12.259 g dried potassium dichromate [$K_2Cr_2O_7$]. Dissolve in ~800 mL Milli-Q water in a 1.0 L volumetric flask.

- Life of Reagent: 1 Year
- Storage Requirements: None

5.2.2.4 QC Samples (High Range)

Quality Control	Preparation
Method Blank (MB)	2 mLs of Milli-Q water added to a high-range vial.
Lab Control Sample (LCS) (500 mg/L)	Dilute 1000 uLs of Rgt. 5.2.2.1 to 100 mLs of Milli-Q in a 100 mL volumetric flask.
Matrix Spike (MS) / MS Duplicate (MSD) (500 mg/L)	Add 1000 uLs of Rgt. 5.2.2.1 to 100 mLs of sample in a 100 mL volumetric flask.

6.0 CALIBRATION (NON-DAILY)

Not Applicable.

7.0 PROCEDURES

7.1 Quality Control Checks

QC Sample	Frequency	Control Limit
MB	1 in 20 samples	< Reporting Limit (<1/2 RL DoD)
LCS [1]	1 in 20 samples	80 - 120% Recovery
DU [2]	1 in 20 samples	≤ 20RPD
MS / MSD [2]	1 in 20 samples	75 – 125% Recovery

[1] LCS Duplicate (LCSD) is performed only when insufficient sample is available for the MS/MSD or when requested by the client/project/contract.
[2] The sample selection for MS/MSD is rotated among client samples so that various matrix problems may be noted and/or addressed. A Matrix Duplicate (DU) is performed only when requested by the client/project/contract. The MS/MSD are the routinely performed matrix QC indicators.

7.2 Sample Preservation and Storage

Sample container, preservation technique and holding time may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client request. Listed below are the holding times and references that include container and preservation requirements for compliance with the Clean Water Act (CWA).

Reference	Holding Time	Preservation
40 CFR Part 136	28 days	pH < 2, H_2SO_4, Cool </=6°C

7.3 Sample Preparation

- Preheat the block digester to 150°C.
- Only solids that have been homogenized, identifiable by a green H on the bottle, are to be used for analysis.

7.3.1 Sample Size

Matrix	Sample Size
Waters	2 mLs
Soils	Variable

7.3.2 Vial Preparation

The chemistry used in the high range and low range COD tests are exactly the same. The standard and reagent concentrations have simply been adjusted to provide optimal accuracy in the analytical range of interest.

7.3.2.1 Inspect the sample to be analyzed to estimate whether the COD of the sample will fall within the high or low range. Samples that are clear and odorless should most likely be analyzed in the low range. Samples that are dirty, contain suspended particles, or have an odor should be analyzed in the high range. Historical data is very useful in determining the range. If a wrong estimation is made, the affected sample is simply reanalyzed in the appropriate range.

NOTE:

- Samples are never diluted to be analyzed by low range. They are reanalyzed in the high range.
- All high range COD results <200 mg/L will be reanalyzed by the low range method for verification. If the low range result is <150 mg/L, it will be reported.
- Soil samples are always analyzed in the high range.

7.3.2.2 Shake the sample or mix it in a blender to ensure homogeneity. Deliver 2 mLs of sample into the appropriate range HACH vial with an eppendorf pipet. Carefully replace the cap onto the vial and then manually shake the vial. **CAUTION: The vial will get hot!! See safety, Section 3.** If a blue color develops quickly in a low-range vial, the sample must be reanalyzed using a high-range vial. If a green-to-black color develops quickly in a high-range vial, the sample must be diluted and reanalyzed.

7.3.2.3 If the sample is a soil, mix the sample thoroughly and place a small aliquot in a weigh boat. Weigh the sample and boat. Carefully place a portion of the aliquot in a high range COD vial. Weigh the remaining sample and the boat and calculate by difference the amount of sample added to the vial. Add 2 mLs of DI water to the vial, carefully replace the cap, and shake the vial. **CAUTION: The vial will get hot!!** If a green-to-black color develops quickly in the vial, re-prepare another vial using less sample. If no color change occurs, consider preparing additional vials using more sample.

7.3.2.4 Place the vials in the block digester and allow them to reflux for 2 hours. Remove the samples from the digester and allow them to cool to room temperature. If there will be a delay until the vials are titrated, it is best to place the cooled vials in a dark place such as a cabinet.

7.4 Calibration / Standardization

7.4.1 Daily Standardization of FAS Titrants

- ~0.0125 N FAS titrant for low range COD must be titrated with 0.025 N potassium dichromate.
- ~0.125 N FAS titrant for high range COD must be titrated with 0.25 N potassium dichromate.

Prepare 3-empty vials by adding 2 mLs of the appropriate potassium dichromate standard and 3 mLs of concentrated sulfuric acid to each. Shake to mix and allow to cool. In addition, add 2 mLs of Milli-Q water to a prepared HACH vial. Mix and allow to cool. Titrate all 4-vials and enter the results in the logbook. The average titrant volume of the 3 potassium dichromate vials is "C" in the final equation used to calculate the sample results (see section 9.1). It corrects for the normality of the titrant. The titrant volume used for the 4th vial containing only milli-Q water provides the "A" in the results equation.

7.5 Preventive Maintenance

- Clean the exterior of the digester block.
- Cover the burette to keep dust and dirt out and rinse with DI water for storage. Rinse with titrant before filling.

7.6 Sample Analysis

- Low range COD samples are titrated using ~0.0125 N FAS titrant.
- High range COD samples are titrated using ~0.125 N FAS titrant.

To titrate the samples, carefully remove the vial cap and rinse the inside walls with <1 mL of Milli-Q water. Add 1 drop of ferroin indicator and a micro stir bar to the vial. Position the vial in the vial holder and mix the sample. Record the mLs of titrant required to just change the color to orange-brown. If the color of the sample changes to brown when the ferroin indicator is added, the COD is >150 mg/L in the low range or >1500 mg/L in the high range. The digestion step must be repeated in the high range or the sample must be diluted and reanalyzed in the high range.

7.7 Documentation

7.7.1 Analysis Logbook

The analysis of samples and standards is documented within the logbook (Attachment 1), which must be completed for each day's analysis.

7.7.2 TALS

This test requires a preparation batch and an analytical batch in TALS. It is important to enter the true initial weight/volume in the preparation batch, because entering it in both will apply the factor twice and result in incorrect reported values. TALS does not have a calculation for this test, and TestAmerica Chicago uses an Excel spreadsheet, available on the W drive, to calculate the uncorrected initial results that then are entered manually into ADII. Spike and LCS recoveries that are generated in Excel are very close to the actual recoveries, but may be slightly off since TALS subtracts initial results using different criteria.

All filter paper lot numbers, reagent IDs, etc., must be entered into the Batch Information screen in the appropriate batch (prep or analytical).

7.7.3 Traceability of Standards

Upon receipt or preparation, each standard, or other reagent is entered into TALs LIMS and is issued a unique ID#. Further information entered into the database includes the manufacturer, lot # (if applicable), the date received or prepared, the expiration date, volume/weight received; concentration (if applicable); preparation details (if applicable), initials of the recording analyst, and the description of the item (i.e., COD Stock Solution – LCS). Once the record is created, a unique label is printed and affixed to the appropriate standard/reagent container.

7.7.4 Data Review

Analytical data goes through a 200% review cycle. As results are generated, analysts review their work to ensure that the results generated meet QC requirements and relevant EPA methodologies. The analyst transfers the data into TALs in the Analyst Desktop module. Where non-compliance is observed, the analyst creates Non-Conformance Memos (NCMs) in TALs. Flags and data qualifiers can be method, project, program or QAPP specific. The analyst documents the initial review on a data review checklist (Attachment 2) and sets the batch status in LIMs to 1st level review. The second level or peer review of the data is conducted by another individual who has been trained on the review process. This secondary review is documented on the same checklist, making any necessary corrections to the data or additions to the NCMs as necessary. The batch is then set to 2nd level review. Any Spectra and all manual integrations are reviewed. The raw data, including the checklist, instrument print-outs, and manual entries, and electronic files are retained for easy retrieval in accordance with the laboratory's record and retention policy outlined in the SOP, *UP-QA-QAM, Section 15.*

Examples of items included in the above reviews are as follows:

- QC data are outside the specified control limits for accuracy and precision
- Unusual detection limit changes are observed
- Samples having unusually high results
- Samples exceeding a known regulatory limit
- Raw data indicating some type of contamination or poor technique
- Transcription errors
- Results outside of calibration range

8.0 QUALITY CONTROL

8.1 QC Summary

8.1.1 One MB and one LCS will be included in each laboratory lot of 20 or fewer samples. Regardless of the matrix being processed, the LCS and method blanks will be in an aqueous media. The MB will be examined to determine if contamination is being introduced in the laboratory.

8.1.2 Accuracy will be measured by the percent recovery (%R) of the LCS. The recovery must be in range, as determined by in-house control limits or statistical analysis, in order to be considered acceptable. Additionally, %R will be plotted on control charts to monitor method accuracy.

8.1.3 One MS/MSD is routinely performed per matrix per 20 sample analytical set. Results must agree within the in-house precision/accuracy limits or statistical control limits in order to be considered acceptable.

8.2 Corrective Actions

When an out-of-control situation occurs, the analysts must use their best analytical judgment and available resources to determine the corrective action to be taken. The out- of-control situation may be caused by more than one variable. The analyst should seek the assistance of his/her immediate supervisor, QA personnel, or other experienced staff if he/she are uncertain of the cause of the out of control situation. The test must not be resumed until the source of the problem and an in-control status is attained. All samples associated with the out-of-control situation should be reanalyzed. Out-of-control data must never be released without approval of the supervisor, project manager, QA personnel or the lab manager.

Listed below are steps that must be taken when an out of control situation occurs:

- demonstrate that all the problems creating the out of control situation were addressed;
- document the problem and the action which was taken to correct the problem on the data review checklist; and
- receive approval (signature) of the Wet chemistry Supervisor, Inorganics Manager, Project Manager, QA personnel, or the Laboratory Manager prior to the release of any analytical data associated with the problem.

Suggested Actions to specific out of control situations:

8.2.1 Laboratory Control Sample (LCS)

If the LCS exceeds acceptance limits:

- reanalyze to verify that an out of control situation exists;
- determine the source of error within the preparation procedure, correct the problem and repeat the sample set. (Sources of contamination could be either the reagents, the LCS stock solution, or the preparation area.)

8.2.2 Method Blank (MB)

- reanalyze to verify contamination at a level > Reporting Limit;
- determine the source of contamination and correct the problem;
- all samples whose concentration is >RL or <10 times the MB level must be reprocessed and reanalyzed; any sample which is <RL or >10 times the MB level need not be reanalyzed. However, note the out-of-control MB in an NCM.

8.2.3 Matrix Duplicate (DU)

- TestAmerica Chicago does not control data on the basis of matrix QC. If the MB and LCS are in control, an out of control DU is considered a matrix effect and is reported, flagged, and narrated by means of an NCM.
- If the results are <5 times the Reporting Limit, then the ± Reporting Limit rule applies;

8.2.4 Matrix Spike (MS)

- TestAmerica Chicago does not control data on the basis of matrix QC. If the MB and LCS are in control, an out of control MS or MSD is considered a matrix effect and is reported, flagged, and narrated by means of an NCM.
- Report all spikes, whether or not they are in control.

9.0 DATA ANALYSIS AND CALCULATIONS

9.1 Sample Concentration; COD mg/L = (A - B) x (2,000/C) x M

Where:
A = mLs used in titration of HACH vial standardization blank
B = mLs used in titration of sample
C = average mLs of titrant for standardization
M = 0.1 for low range vials 1.0 for high range vials

Note: This equation is from the Hach Co. manual and is equivalent to the equation found in Standard Methods 5220C, with the M and C factors replacing the molarity, and the 2000 factor replacing the 8000 factor. Since Hach vials are used in the preparation, this modified equation is simpler and more appropriate to use. A mathematical equivalency page is available in the Inorganics Manager's office COD file.

9.2 Accuracy

9.2.1 LCS % Recoveries = $\frac{\text{observed concentration}}{\text{actual concentration}} \times 100$

9.2.2 Matrix Spike % Recovery = $\frac{\text{(spiked sample) - (unspiked sample)}}{\text{spike concentration}} \times 100$

9.3 Precision

9.3.1 Matrix Duplicate Relative Percent Difference (RPD):

$$RPD = \frac{|\text{orig. sample value - dup. sample value}|}{[(\text{orig. sample value + dup. sample value})/2]} \times 100$$

10.0 POLLUTION CONTROL

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

10.1 Waste Management

Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to UP-WM-001.

- Waste from this procedure contains mercury and chromium and must be disposed of in the waste stream drum labeled 'Heavy Metal Corrosive Waste Water'.
- Single component standards will be turned over to the EHSC or waste technician for disposal.

11.0 METHOD PERFORMANCE CRITERIA

Refer to sections 1, 6, 7 and 8.

12.0 REFERENCES

Refer to Section 1.0

13.0 ATTACHMENTS

Attachment 1: Example: Analysis Logbook
Attachment 2: Example: Data Review Checklist
Attachment 3. DoD QSM Version 4.2: Appendix F QC Requirements Summary (Tables F-1)

14.0 REVISION HISTORY

- Revision 14 updated on 09/30/11
- Annual Review
- Added section 7.7.2
- Added text concerning calculation verification to section 9.1

Attachment 1.

Example: Analysis Logbook
(014-001)

TestAmerica Chicago

Method: a. SM5220C (High) b. SM5220C (Low)
Matrix: a. Water b. Waste c. Soil

Chemical Oxygen Demand - Titrimetric

Analytical Batch:_____
Prep. Batch:_____
Labnet:_____

Page #: _____
Book # _____
NCM # _____
Block Digester Temp.: _____ C

F.A.S. Titrant Standardization
a. High: 0.125 N
b. Low: 0.0125 N

(mLs used)
1 _____
2 _____
3 _____
C _____ (Average mLs)

Calculations:
mg/L = (A-B) x (2000/C) x M x Dil. Factor
mg/kg = mg/L Result x 2 mL* / g sample
mg/kg "d" = mg/kg Result / % Solids (as decimal)

Eppendorf Volume: _____uL
Eppendorf ID: _____

Key:
A = mLs used in titration of a blank
B = mLs used in titration of sample
C = Avg. mLs of titrant for standardization
M = 0.1 for Low Range COD vials
= 1.0 for High Range COD vials
= 10.0 for High Range Plus COD vials
* 2 mL final volume in vial
A = _____

	Job #	Analytical Dilution or Sample Size	mLs Titrant B	Result	Units	% Rec RPD	Comments
MB							
LCS		S					
LCS		T					
1							
2							
3							
4							
5							
6							
7							
8							
9							
10							
11							
12							
13							
14							
15							
16							
17							
18							
19							
20							
—							
—							
—							

Standard Traceability: **Note:** Working Standards are prepared daily from the noted Stock Solns.

KHP Standard ID #: _____

Spiking Levels (mg/L): LCS = _____ MS (Water) = _____ MS (Soil) = _____

Preparation Signature:_____ Date:_____ Start Time:_____ End Time:_____

Analyst Signature:_____ Date:_____ Start Time:_____ End Time:_____

Reviewer Signature:_____ Date:_____

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(014-001)

Attachment 2.

Example: Data Review Checklist (015-001)

TestAmerica Chicago
INORGANICS – LIMS DATA REVIEW CHECKLIST

Test _____ **Analytical Batch#** _____

Prep Batch # _____
(File by analytical batch #)

Analyst / Primary Reviewer: _____ 1st Level Review Date: _____

Secondary Reviewer: _____ 2nd Level Review Date: _____

	PRI REV	SEC REV	COMMENTS
1) Is the Batch Information screen complete? Analyst correct? ____ Start/End times correct? ____ Instrument Code entered? ____ Are Batch Notes complete? (i.e. reagents, filters, temperatures, etc….) ____			
2) Was Data Imported ____ Manually entered ____ Balance Interface Used ____			
3) Samples & all QC in order as analyzed? (All readings must be present in sequence.)			
4) Sample List Screen Date(s)/Time(s) correct			
5) Standard Codes present and Amount Spiked correct?			
6) Dilution factors all present and correct?			
7) Are correct Sample ID's used? ____ Data entries match raw data? ____ Are all samples designated with a Blue P?			
8) Are correct QC ID's used? ____ Data entries match raw data? ____ Are all QC designated with a Blue P? ____			
9) Is QC correctly linked to each sample?			
10) Is all QC calculated and are correct flags applied?			
11) Is an NCM needed? ICV, MB, LCS, LCSD, DU, MS, MSD, RPD out; ____ Holding Time missed or Manual Integration required? ____			NCM # _____ Approved By: _____ Initials
Raw Data: 1) Are calibration criteria met? $r \geq 0.995$; $\lvert$y-intercept$\rvert$ < RL; (<1/2 RL DoD QSM)			
2) Is AD Batch # clearly identified?			
3) Are manual calculations and final results clearly shown?			
4) Are all errors crossed out with single line & initialed and dated? Are all unused portions of the pg. Z'd out?			
5) Is unreported data clearly identified with reason & initialed and dated?			
6) Is data signed & dated by analyst & reviewer?			

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(015001)

Attachment 3.

DoD QSM Version 4.2: Appendix F QC Requirements Summary, Table F-1 (016-001 to 016-002)

TestAmerica Chicago

DoD QSM Version 4.2: Appendix F - Quality Control Requirements Summary

Table F-1 Summary of QC Check Definitions, Purpose, and Evaluation – Inorganics (WC)

QC Check	Definition	Purpose	Evaluation
Calibration Blank	Reagent water containing no analytes of interest.	To determine the zero point of the calibration curve for all initial and continuing calibrations.	This is a required QC procedure. Continuing calibration blank responses above the LOD require corrective action
Continuing calibration verification (CCV)	This verification of the ICAL that is required during the course of analysis at periodic intervals. Continuing calibration verification applies to both external standard and internal standard calibration techniques, as well as to linear and non-linear calibration models	To verify that instrument response is reliable, and has not changed significantly from the current ICAL curve.	If the values for the analytes are outside the acceptance criteria, the ICAL may not be stable. Results associated with out-of-control CCV results require reanalysis or flagging
Demonstrate Acceptable Analytical Capability	QC samples are analyzed in series to verify ability to produce data of acceptable precision and bias.	To verify the ability to produce data of acceptable precision and bias for a specific instrument type, matrix, method, and analyst.	The average recovery of the spikes and standard deviation of the replicates must be within designated acceptance criteria. Analysis of field samples may not be conducted until this check is successful.
Duplicate Sample (replicate)	Two identical portions of material collected for chemical analysis, and identified by unique alphanumeric codes. The duplicate may be portioned from the same sample, or may be two identical samples taken from the same site. The two portions are prepared and analyzed identically. (modified QSM)	To provide information on the heterogeneity of the sample matrix or to determine the precision of the intralaboratory analytical process for a specific sample matrix	A duplicate sample will provide information on the heterogeneity of the sample matrix. The greater the heterogeneity of the matrix, the greater the relative percent difference between the sample and the sample duplicate. If the sample matrix is homogeneous (such as with drinking water) and the relative pecent difference is high, this could indicate a problem in the analytical system.
Initial calibration for all analytes (ICAL)	Analysis of analytical standards at different concentrations that are used to determine and calibrate the quantitation range of the response of the analytical detector or method	To establish a calibration curve for the quantification of the analytes of interest	Statistical procedures are used to determine the relationship between the signal response and the known concentration of analytes of interest. The ICAL must be successful before any samples or other QC check samples can be analyzed.
Laboratory control sample (LCS) containing all analytes to be reported	A sample matrix, free from the analytes of interest, spiked with known amounts of analytes or a material containing known and verified amounts of analytes.	Used to evaluate the performance of the total analytical system, including all preparation and analysis steps. Assesses the ability of the laboratory/analyst to successfully recover the target analytes from a control (clean) matrix. Control limits for LCS recovery, typically expressed as percent recovery, are used for the development of statistical control limits and serve as acceptance criteria for determining whether an analytical run is in control (batch acceptance).	This is a required QC Check. The inability to achieve acceptable recoveries in the LCS indicate problems with the precision and bias of the measurement system. Failure to achieve acceptable recoveries in a "clean" matrix is an indicator of possible problems achieving acceptable recoveries in field samples.
MS	A sample prepared by adding a know mass of target analyte to a specified amount of matrix sample for which an independent estimate of target analyte concentration is available.	To assess the performance of the method as applied to a particular matrix. Matrix spikes are used, for example, to determine the effect of the matrix on a method's recovery efficiency. The recovery of target analytes from the matrix spike sample is used to determine the bias of the method in the specific sample matrix.	The lack of acceptable recoveries in the MS often points to problems with the sample matrix. One test of this is a comparison to the LCS recoveries. If the corresponding LCS recoveries are within acceptable limits, a matrix effect is likely. The lab should not correct for recovery; only report the results of the analyses and the associated MS results and indicate that the results from these analyses have increased uncertainty
MSD	A second replicate matrix spike prepared in the laboratory and analyzed to obtain a measure of the precision of recovery for each analyte	To assess the performance of the method as applied to a particular matrix and provide information on the homogeneity of the matrix. Also used to determine the precision of the intralaboratory analytical process for a specific sample matrix.	When compared to the MS, the MSD will provide information on the heterogeneity of the sample matrix. The greater the heterogeneity of the matrix, the greater the RPD between the matrix spike and the matrix spike duplicate. If the sample matrix is homogeneous, such as with drinking water, and the RPD is high, this could indicate a problem in the analytical system.

F-1 (cont.)			
QC Check	**Definition**	**Purpose**	**Evaluation**
Matrix Verification sample (CR+6 only)	A pH-adjusted filtrate that has been spiked with CR+6 to ensure that the sample matrix does not have a reducing condition or other interferents that could affect color development. (Modified Method)	To ensure that the sample matrix does not have a reducing condition or other interferents that affect color development.	To verify the absence of an interference, the spike recovery must be between 85% and 115%. If the result of verification indicates a suppressive interference, the sample should be diluted and reanalyzed. If the interference persists after sample dilution, an alternative method (Method 7195, Coprecipitation, or Method 7197, Chelation/Extraction) should be used.
MB	A sample of a matrix similar to the batch of associated samples (when available) that is free from the analytes of interest and is processed simultaneously with an under the same conditions as samples through all steps of the analytical procedures, and in which no target analytes or interferences are present at concentrations that impact the analytical results for sample analyses.	To assess background interferences or contamination in the analytical system that might lead to high bias or false positive data. Results of method blanks provide an estimate of the within-batch variability of the blank response and an indication of bias introduced by the preparation and analytical procedure.	This is one of the QC samples used to measure lab accuracy/bias. The sample could indicate whether contamination is occurring during sample prep and analysis. If analytes are detected > ½ RL, reanalyze or qualify (B-flag) all results for the specific analytes(s) in all samples in the associated prep batch as appropriate. For common lab contaminants, no analytes detected > the RL. See Section D.1.1.1 and Box D-1
RT window position establishment for each analyte (and surrogate) (all chromatographic methods only)	Determination of the placement of the RT window (i.e. start/stop time) of each analyte or group of analytes as it elutes through the chromatographic column so that analyte identification can be made during sample analysis. This is done during the ICAL.	To idendify analytes of interest	Incorrect window position may result in false negatives, require additional manual integrations, or cause unnecessary reanalysis of samples when surrogates or spiked compounds are erroneously not identified.
RT window width calculated for each analyte (and surrogate) (non-MS chromatographic methods only)	Determination of the length of time between sample injection and the appearance of a peak at the detector. The total length of time (window) is established for each analyte or group of analytes and is set for complete elution of analyte peaks. It is based upon a series of analyses and statistical calculations that establish the measured band on the chromatogram that can be associated with a specific analyte or group of analytes.	To ensure that the chromatographic system is operating reliably and that the system conditions have been optimized for the target anaytes and surrogates in the standards and sample matrix to be analyzed. It is done to minimize the occurrence of both false positive and false negative results.	Used to evaluate continued system performance. Tight RT windows may result in false negatives or may cuase unnecessary reanalysis of samples when surrogates or spiked compounds are erroneously not identified. Overly wide RT windows may result in false positive results that cannot be confirmed upon further analysis.
Second source calibration verification (ICV)	A standard obtained or prepared from a source independent of the source of standards for the ICAL. Its concentration should be at or near the middle of the calibration range. It is done after the ICAL.	To verify the accuracy of the ICAL.	The concentration of the 2nd source calibration verification, determined from the analysis, is compared with the known value of the standard to determine the accuracy of the ICAL. This independent verification of the ICAL must be acceptable before sample analysis can begin.

Notes:
1. Project-specific requirements identified by the client supersede any requirements listed. The requirements are meant to be default, to be used when project-specific direction based on DQOs is not available.
2. If there is a contradiction between the method and the DoD tables, the requirements specified in the tables shall be followed.

(2009-016)

TITLE: Wet Chemistry
Total Organic Carbon / Total Inorganic (Dissolved) Carbon

Approvals (Signature/Date):

Carla Bonner 11/24/10
Carla Bonner Date
Supervisor, Wet Chemistry

Diane L. Harper 11/24/10
Diane L. Harper Date
Inorganics Manager

Terese A. Preston 11/29/10
Terese A. Preston Date
Quality Assurance Manager

11/29/10
John D. Nagel Date
Env. Health & Safety Coor.

Michael J. Healy 12/29/10
Michael J. Healy Date
Laboratory Director

Facility Distribution No: _____ **Distributed To:** Uncontrolled

1.0 SCOPE / APPLICATION

This Standard Operating Procedure is used to quantitatively determine the Total Organic Carbon (TOC) and Total Inorganic (Dissolved) Carbon (TIC/DIC) content in water matrices using UV/persulfate oxidation and non-dispersive IR detection. Total Carbon and Inorganic Carbon may also be measured by setting up the instrument differently. This SOP was written using Standard Methods 5310C and SW-846 Method 9060 and 9060A as references. The analyst also must reference the individual instrument Operations Manual.

There may be several references to the equivalent method EPA 600/04-79-020 415.1 throughout this SOP. As of March 12, 2007, this method has been withdrawn from the Federal Register. There will be a transition period in which the method is removed from laboratory, agency and permit documentation. Regardless, TestAmerica Chicago will remain in compliance with approved methods and regulations.

On occasion, clients request slight modifications to this SOP. These modifications are addressed on a case-by-case basis with the range of accuracy (i.e., MDLs, linearity check or PT sample) verified prior to implementation. Any modifications would be written into a Quality Assurance Plan (QAP) and authorized via laboratory signature approval; and amended to the data packages case narrative.

Specific requirements pertaining to the DOD QSM Version 4.2 are located in Attachment 4. These requirements are additionally applicable to all NFESC projects. Any deviations from these procedures and/or variances from must be addressed appropriately in accordance with standard operating protocol and pre-approved on a project by project basis.

1.1 Method Sensitivity

1.1.1 Method Detection Limits

The method detection limit (MDL), referred to as the detection limit (DL) in DoD QSM version 4.2 and in NELAC documents, is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to Appendix B of 40 CFR 136, "Guidelines Establishing Test Procedures for the Analysis of Pollutants" with additional details are provided in the TestAmerica Corporate SOP, *CA-QS-006, Detection Limits* and the TestAmerica Chicago SOP, *UP-QA-017, Method Detection Limit Studies.* MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; these are verified at least annually.

1.1.2 Demonstration of Capability

Before the laboratory may institute a new method and begin reporting results, the laboratory shall confirm that it can properly operate the method. A demonstration of capability is performed whenever there is a change in instrument type, method or personnel. An Initial Demonstration of Capability (IDOC) must be thoroughly documented and approved by the Department Manager/Supervisor and QA Manager prior to independently analyzing client samples. All associated documentation must be retained in the QA Department and in the Analyst Training files. For additional details on the demonstration of capability procedures followed, refer to the laboratory SOP, *UP-QA-QAM, Quality Assurance Manual, Sections 20.4.2 and 20.4.3.*

1.1.3 Reporting Limits

Matrix	Reporting Limit
Waters	1.0 mg/L

Reporting Limits (RL) are defined as the lowest concentration of an analyte determined by a given method in a given matrix that the laboratory feels can be reported with acceptable quantitative error or client requirements, values specified by the EPA methods or other project and client requirements. Because of the high level of quantitative error associated with determinations at the level of the MDL, the laboratory keeps the reporting limit higher than the MDL. Wherever possible, reporting is limited to values approximately 3-5x the respective MDL to ensure confidence in the value reported. Client specific requests for reporting to the MDL are special circumstances not to be confused with the previous statement.

The RLs are referred to as the Limit of Quantitation (LOQ) in DoD QSM version 4.2 and NELAC documents. These documents also refer to the Level of Detection (LOD), which at TestAmerica Chicago is set at ½ the LOQ.

1.1.4 Definitions

Refer to Section 3.0 of the Laboratory's Quality Assurance Manual (UP-QA_QAM).

1.2 Summary of Method

The measurement of TOC indicates the presence of organically bound carbon. Organic chemicals are of a primary environmental concern because of their wide spread industrial and agricultural uses. TOC analysis provides the basic screening measurement to warrant subsequent specific analyses if high TOC levels are found. DOC is determined by the same process, but on a filtered sample.

Inorganic carbon, including carbonates, and bicarbonates, is removed by acidifying the sample to convert to CO_2 and sparging with oxygen. This also removes any dissolved CO_2 initially present in the sample. The remaining organic carbon is oxidized to carbon dioxide by acidified persulfate in the presence of ultraviolet light. This carbon dioxide is carried to an infrared analyzer. A microprocessor calculates the area of the peaks produced by the analyzer, compares them to the peak area of the calibration standards stored in the memory, and prints out a calibrated organic carbon value in mg/L. TIC may be determined by setting the instrument in TIC mode. In TIC mode the instrument bypasses the UV-persulfate oxidation and quantifies from the sparging step, against a previously run inorganic carbon curve. DIC values are determined the same way, but on filtered samples.

1.2.1 Theory of Operation

Liquid samples are introduced through a sipper probe on an automatic analyzer. TIC and TOC may be determined on the same sample. The inorganic carbonates are converted to CO_2 and measured by a Non-Dispersive Infrared Analyzer (NDIR) as TIC. The ultra-violet source is then activated and sodium persulfate is added to the elevated temperature reactor module to convert the organic carbon to carbon dioxide which is measured by the NDIR and is directly proportional to the TOC in the sample. Spent sample and reagents are purged from the unit and the next sample is then analyzed after an automatic system wash.

2.0 INTERFERENCES

- Persulfate oxidation is slowed in samples containing significant concentrations of chloride by the preferential oxidation of chloride. At a concentration of 0.1% chloride, oxidation of organic matter may be inhibited completely. This is why TestAmerica Chicago prefers to analyze sulfuric acid-preserved samples.
- To obtain TOC results, carbonates and bicarbonates, positive interferences, must be completely removed by acidification and sparging of a clean gas (O_2 or N_2) to remove the CO_2.
- Excessive acidification of sample, producing a reduction in pH of the persulfate solution to 1 or less, can result in sluggish and incomplete oxidation of organic carbon.
- The intensity of the ultraviolet light reading the sample matrix may be reduced by highly turbid samples, resulting in sluggish or incomplete oxidation.
- Large organic particles or very large or complex organic molecules may be oxidized slowly because persulfate oxidation is rate limited.
- Improper sample handling and treatment is a likely source of contamination.

3.0 SAFETY

Employees must abide by the policies and procedures in the Corporate Safety Manual, Radiation Safety Manual, Lab Specific Addendum to the CSM, and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coat and closed-toe, nonabsorbent shoes are a minimum.

3.1 Specific Safety Concerns or Requirements

- The auto sampler has a probe that is sharp; use caution not to stick yourself.
- The UV reactor is hot and can cause burns if touched.
- The Sodium Persulfate is a strong oxidizer. Avoid contact with combustible materials, organic materials, strong reducing agents, and excess heat.

3.2 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **NOTE: This list does not include all materials used in the method.** The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table. A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Sulfuric Acid	Corrosive Oxidizer Dehydrator	1 mg/m^3	This material will cause burns if comes into contact with the skin or eyes. Inhalation of vapors will cause irritation of the nasal and respiratory system.
Phosphoric Acid	Corrosive	1 Mg/M3 TWA	Inhalation is not an expected hazard unless misted or heated to high temperatures. May cause redness, pain, and severe skin burns. May cause redness, pain, blurred vision, eye burns, and permanent eye damage.
Sodium Persulfate	Oxidizer Corrosive	0.1 Mg/M3-TWA as Persulfates	Causes irritation to the respiratory tract. Symptoms may include sore throat, shortness of breath, inflammation of nasal passages, coughing, and wheezing. Causes severe irritation or burns to the skin and eyes. Symptoms include redness, itching, pain and burns. May cause allergic skin reactions. Can cause eye damage.
1 – Always add acid to water to prevent violent reactions. 2 – Exposure limit refers to the OSHA regulatory exposure limit.			

4.0 EQUIPMENT AND SUPPLIES

- Tekmar-Dohrmann Phoenix 8000 TOC Analyzer (known as TOC 3) with XYZ Autosampler and TOCTalk version 3.2 software on the accompanying PC
- Teledyne-Tekmar Fusion TOC analyzer (known as TOC 5) with built-in carrousel autosampler and TekLink version 1.1.0.189 software on the accompanying PC
- VOA vials, with septa for the Fusion or without septa for the Phoenix sampler.
- Class A volumetric flasks: 100 mL, 200 mL, 1000 mL
- Eppendorf pipettes

5.0 REAGENTS AND STANDARDS

All standards and reagents are prepared with Type II Deionized (DI) Water, unless otherwise specified. All standards and reagents are prepared in Class A volumetric glassware and stored at 4 ± 2°C when required. Only reagent grade chemicals meeting ACS criteria are to be used.

5.1 Reagents

5.1.1 Acids

- Phosphoric acid, concentrated
- Sulfuric acid, concentrated

5.1.2 Sodium Persulfate

Dissolve 25 grams of sodium persulfate in 213 mLs of Milli-Q water and add 9 mLs of concentrated phosphoric acid. Double or triple the preparation as required. It is best to prepare this reagent at least one day in advance of its use.

- Life of Reagent: 1 – year
- Storage Requirements: None

5.1.3 21% Phosphoric Acid

Add 37 mLs of concentrated phosphoric acid to 188 mLs of Milli-Q water.

- Life of Reagent: 1 – year
- Storage Requirements: None

5.2 Standards

5.2.1 TOC Stock I, 2000 mg/L

5.2.2 TOC Stock II, 2000 mg/L

On a balance capable of reading 0.1 mg, weigh out 425 mg of reagent grade potassium hydrogen phthalate dried to a constant weight and desiccated, transfer quantitatively with DI water to a 100 mL Class A volumetric flask, dissolve, and add 0.1 mL of concentrated sulfuric acid. Dilute to volume with DI water.

- Life of Standard: 1 – month
- Storage Requirements: Glass amber bottle

*Stock II is prepared as Stock I **EXCEPT** an alternate stock source is used.

5.2.3 TIC Stock I, 2000 mg/L

In a 1.0 L volumetric flask, dissolve 17.6667 g sodium carbonate in freshly boiled and cooled Milli-Q water.

- Life of Standard: 1 – month
- Storage Requirements: Glass amber bottle

5.2.4 TIC Stock II, 1000 mg/L

In a 100 mL volumetric flask, dissolve 0.35 g sodium bicarbonate and 0.4418 g sodium carbonate in freshly boiled and cooled Milli-Q water.

- Life of Standard: 1 – month
- Storage Requirements: Glass amber bottle

5.2.5 Calibration Curve

Using Eppendorf pipettes, prepare the calibration curve on a monthly basis.

Concentration	Volume (uLs) Stock I (Rgt. 5.2.1)	Volume of DI Water
0	0	200 mLs
1	100	|
5	500	|
10	1000	Dilute to
15	1500	200 mLs w/
20	2000	DI Water
30	3000	|
40	4000	|

NOTE: For TIC/DIC, prepare the curve using the same volume of standard as TOC but use the TIC Stock I (Rgt. 5.2.3).

5.2.6 Matrix Spike (MS) / MS Duplicate (MSD) Sample, 10 mg/L

Prepare fresh prior to each use.

TOC: Dilute 0.5 mLs of Stock Organic Carbon Standard II (Std. 5.2.2) to 100 mLs with sample. Alternatively, add 200 uL of TOC Standard II to 40 mLs of sample.

TIC/DIC: Dilute 1.0 mL TIC Standard II (Rgt. 5.2.4) to 100 mLs with sample. Alternatively, add 400 uL of TIC Standard II to 40 mL of sample.

5.2.7 Initial and Continuing Calibration Verification (ICV/CCV); 20 mg/L

TOC: Dilute 10 mLs of TOC Stock II, Rgt. 5.2.2, for the ICV, or TOC Stock I, Rgt. 5.2.1, for the CCV into a 1000 mL volumetric flask. Dilute to volume with Milli-Q water acidified with 1.0 mL sulfuric acid. Prepare weekly.

TIC/DIC: Dilute 4 mLs of TIC Stock II, Rgt. 5.2.4, for the ICV, or TIC Stock I, Rgt. 5.2.1, for the CCV, into a 200 mL volumetric flask. Dilute to volume with Milli-Q water. Do not preserve. Prepare daily.

5.2.7.1 Alternate CCV, 10 mg/L

NOTE: The CCV concentration will be alternated between the 20 mg/L and 10 mg/L standards throughout the analytical sequence.

TOC: Dilute 5 mL of TOC Stock I (Rgt. 5.2.1) into a 1000 mL volumetric flask. Dilute to volume with Milli-Q water acidified with 1.0 mL sulfuric acid. Prepare weekly.

TIC/DIC: Dilute 2 mLs of TIC Stock I (Rgt. 5.2.3) into a 200 mL volumetric flask. Dilute to volume with Milli-Q water. Prepare fresh prior to each use.

5.2.8 Initial / Continuing Calibration Blank (ICB / CCB)

TOC / TIC / DIC: Milli-Q water acidified with sulfuric acid at 0.1 mL per 100 mL (40 uL per 40-mL vial.)

5.2.9 Laboratory Control Sample (LCS), 10 mg/L

TOC: Dilute 1.0 mL of Stock Organic Carbon Standard II (Std. 5.2.2) into a 200 mL volumetric flask. Dilute to volume with Milli-Q water, acidified with 0.2 mL sulfuric acid.

TIC/DIC: Dilute 2.0 mL of TIC Standard II (Rgt. 5.2.4) into a 200 mL volumetric flask. Dilute to volume with Milli-Q water. Do not acidify.

5.2.10 TIC standard for TOC/DOC batches, 10 mg/L

Dilute 2.0 mL of TIC Standard II (Rgt. 5.2.4) into a 200 mL volumetric flask. Dilute to volume with Milli-Q water. Do not acidify. Prepare daily.

Note: This standard is analyzed near the beginning of each TOC/DOC batch to verify that the analyzer is in the correct mode and that the inorganic sparger is working properly.

6.0 CALIBRATION (NON-DAILY)

Although the Dohrmann analyzers have the option of a single concentration calibration standard or a multiple point curve calibration, the latter is used. The linearity must be confirmed at least monthly by running a linear range curve with a blank and concentrations which cover the anticipated range of measurement (refer to Section 5.2.5). One of the calibration standards will be at the desired reporting limit. (Refer to the TestAmerica Corporate Policy, P-T-001, *Selection of Calibration Points* for further guidance.)

If a correlation coefficient of ≥0.995 is not met, the instrument will be recalibrated prior to analysis of samples. Calibration data, to include the correlation coefficient, will be entered into the TOC bench cover sheet kept with the sample data to maintain a permanent record of instrument calibrations.

The ICVs and CCVs are analyzed daily to confirm linearity.

7.0 PROCEDURE

7.1 Quality Control Checks

Quality Controls	Frequency	Control Limit
Method Blank (MB)	1 in 20 samples	< Reporting Limit, <LOD for DoD
LCS	1 in 20 samples	*80 – 120% [1]
LCS Duplicate (LCSD) [2]	1 in 20 samples	*80 – 120%[1]; ≤ 20 RPD
Matrix Duplicate (DU)[3,5]	1 in 20 samples	≤20 RPD
Matrix Spike (MS/MSD)[4,5]	1 in 20 samples	*75-125%[1], 80-120% for DoD

All TOC water samples are analyzed in duplicate (E415.1) or in quadruplicate (SW9060).

Note: For samples received from the state of South Carolina, quadruplicate analysis is required to be performed for each sample for method 9060. Some clients with samples from alternate states may request 9060 analysis to be performed in duplicate to meet their project objectives.

Blanks and quality control samples in the above table are analyzed in duplicate.

[1] In-house statistical limits or individual QAPP limits may be required rather than those in the table. Refer to the paper work accompanying a project's in-house chain-of-custody for appropriate limits to use for each sample.
[2] The reading of a second LCS, in duplicate or quadruplicate, is optional and specifically not needed for some clients. Refer to the paper work accompanying a project's in-house chain-of-custody for appropriate QC requirements.
[3] DUs are performed as requested by the client/project.
[4] When analyzing quads according to SW-846 9060, a MS/MSD must be analyzed on 1 in 10 samples.
[5] The sample selection for MS/MSD or DU is rotated among client samples so that various matrix problems may be noted and/or addressed.

7.2 Sample Preservation and Storage

Holding time, preservation techniques and sample container may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client request. Listed below are the holding times, and the references which include container and preservation requirements for compliance with the Clean Water Act (CWA).

Regulation	Holding Time	Preservation	Reference
CWA	28 days	pH<2 (sulfuric acid); Cool 4 ± 2°C (HCl is approved, but not preferred at TestAmerica Chicago – See Sec. 2.0)	40CFR, Part 136.3

7.3 Sample Preparation

The pH of every sample is checked with pH indicator paper prior to analysis to ensure proper preservation. If the pH of a sample is not <2, three drops of sulfuric acid are added to the sample after it has been poured into an auto sampler tube for analysis.

Care is taken not to mix or shake the TOC sample container in order to prevent loss of volatile organic carbon compounds. If a sample contains sediment, the liquid portion is decanted into a clean beaker. The beaker contents are mixed thoroughly, pH adjusted if necessary, poured into sample vials or tubes, and arranged on the auto sampler.

If the sample appears dirty or has a strong odor, the sample usually contains a high amount of TOC and is to be diluted prior to its initial analysis to avoid overloading the infrared analyzer or clogging instrument tubing. All dilutions must be preserved with concentrated sulfuric acid. Should a diluted sample display a TOC result below the reporting limit, the sample is reanalyzed at a lesser dilution or at full strength. All analysis must be retained as raw data, and all dilutions must be justified.

Samples that contain gross solids or insoluble matter, homogenize until satisfactory replication is obtained. It may be necessary to decant some groundwater samples that contain unwanted sediments. Mix well before decanting.

If dissolved Organic Carbon is to be determined and the sample was not field filtered, filter the sample and a reagent blank through glass fiber filters under vacuum. Pre-treat filters by soaking overnight in a 1:1 solution of HNO3 and reagent water. Collect in new VOA vials (2 per sample).

7.4 Calibration / Standardization

Instrument calibration consists of two types: Initial Calibration and Continuing Calibration. The ICV and CCV following standards are used to verify stable calibration throughout the sequence, and/or to demonstrate that instrument response did not drift during a period of non-use of the instrument.

Calibration Controls	Frequency	Control Limit
Calibration Curve	Monthly or as needed; prior to samples	y-int. < Reporting Limit
Corr. Coeff.	Prior to samples	≥ 0.995
ICV / CCV	every 10 samples; end of sequence	85 - 115%
ICB / CCB	every 10 samples; end of sequence	< Reporting Limit, < LOD for DoD
TIC standard	One in every run	< Reporting Limit

*Calibration check standards and blanks are analyzed with a single analysis.

7.5 Preventive Maintenance

Follow the individual instrument manual recommendations for preventive maintenance. Make sure that the laboratory maintenance manual is maintained for each instrument on each day of operation. (Attachment 3)

7.6 Sample Analysis

Sample Size.....10 mLs per injection (4 mL is actually analyzed)

Follow the individual analyzer manual for instructions for setting up the computer and operating the instrument. Analysis of each injection requires approximately 6-10 minutes, depending on the concentration of TOC in the sample.

7.7 Documentation

7.7.1 Instrument Print-out

The sequence of analysis of the samples and standards is contained in the instrument summary print-out, which serves as the analysis log. The analyst must note recoveries on the print-out, and carefully cross out unused data, being careful to document the reason and data is not reported.

7.7.2 Traceability of Standards

Upon receipt or preparation, each chemical salt, solvent, acid, standard, or other reagent is entered into TALs LIMS and is issued a unique ID#. Further information entered into the database includes the manufacturer, lot # (if applicable), the date received or prepared, the expiration date, volume/weight received; concentration (if applicable); preparation details (if applicable), initials of the recording analyst, and the description of the item (i.e., TOC Stock Solution – LCS). Once the record is created, a unique label is printed and affixed to the appropriate standard/reagent container.

7.7.3 Data Review

Analytical data goes through a 200% review cycle. As results are generated, analysts review their work to ensure that the results generated meet QC requirements and relevant EPA methodologies. The analyst transfers the data into TALs in the Analyst Desktop module. Where non-compliance is observed, the analyst creates Non-Conformance Memos (NCMs) in TALs. Flags and data qualifiers can be method, project, program or QAPP specific. The analyst documents the initial review on a data review checklist (Attachment 1) and sets the batch status in LIMs to 1st level review. The second level or peer review of the data is conducted by another individual who has been trained on the review process. This secondary review is documented on the same checklist, making any necessary corrections to the data or additions to the NCMs as necessary. The batch is then set to 2nd level review. Any Spectra and all manual integrations are reviewed. The raw data, including the checklist, instrument print-outs, and manual entries, and electronic files are retained for easy retrieval in accordance with the laboratory's record and retention policy outlined in the SOP, *UP-QA-QAM, Section 15.*

Examples of items included in the above reviews are as follows:

- QC data are outside the specified control limits for accuracy and precision
- Unusual detection limit changes are observed
- Samples having unusually high results
- Samples exceeding a known regulatory limit
- Raw data indicating some type of contamination or poor technique
- Inconsistent peak integration (if applicable)
- Transcription errors
- Results outside of calibration range

8.0 QUALITY CONTROL

8.1 QC Summary

1. One MB and one LCS will be included in each laboratory lot of 20 or fewer samples. The MB will be examined to determine if contamination is being introduced in the laboratory. The LCS, when prepared in duplicate (LCSD) as required by the client and/or QAPP, will be examined to determine both accuracy and precision.

2. Accuracy will be measured by the percent recovery (%R) of the LCS. The recovery must be in range, as determined by in-house control limits or statistical analysis, in order to be considered acceptable. Additionally, %R will be plotted on control charts to monitor method accuracy.

3. Precision will be measured by the reproducibility of both LCS injections for each analysis, but will only be statistically generated for LCS data. Results must agree within in-house control limits or statistical control limits (if required) in order to be considered acceptable.

4. As a default, one MS, read in duplicate is analyzed per matrix per 20 samples for EPA 415.1 and per 10 sample analytical set for SW 9060. Results must agree within the in-house precision/accuracy limits or statistical control limits (if required) in order to be considered acceptable.

8.2 Corrective Action

When an out-of-control situation occurs, the analysts must use his/her best analytical judgment and available resources to determine the corrective action to be taken. The out-of-control situation may be caused by more than one variable. The analyst should seek the assistance of his/her immediate supervisor, QA personnel, or other experienced staff if he/she is uncertain of the cause of the out-of-control situation. The test must not be resumed until the source of the problem and an in-control status is attained. All samples associated with the out of control situation should be reanalyzed. Out-of-control data must never be released without approval of the Wet Chemistry Supervisor, Inorganics Manager, Project Manager, QA personnel or the Laboratory Director.

Listed below are steps that MUST be taken when an out-of-control situation occurs:

- demonstrate that all the problems creating the out-of-control situation were addressed;
- document the problem and the action which was taken to correct the problem on the data review checklist;
- document on the checklist that an in control status has been achieved; and
- receive approval (signature) of the Wet Chemistry Supervisor, Inorganics Manager, Project Manager, QA personnel or the Laboratory Director prior to the release of any analytical data associated with the problem.

Suggested Actions to specific out of control situations (all reported data associated with the following should have an NCM written in AD):

QC Standard	Suggested Corrective Actions
Calibration Curve	• reanalyze the standard curve; • prepare new stock and/or working standards; • check reagents/solutions and prepare fresh if necessary.
Initial Calibration Verification (ICV)	• repeat to verify proper preparation; • prepare new ICV from original stock; • check for instrument base-line drift; • restandardize with existing standards, reanalyze; • check reagents/solutions and prepare fresh if necessary; • prepare new stock and/or working standards and recalibrate;
TIC Standard in TOC/DOC batches	• check instrument mode and reagent levels
Initial Calibration Blank (ICB)	• prepare a new ICB to verify proper preparation; • verify that the instrument base-line is stable and/or perform necessary maintenance, cleaning, etc... to achieve stability; • determine the source of contamination by the process of elimination, correct the problem and reanalyze. (Carry over from a previous analysis or reagent contamination are two common sources).
Laboratory Control Sample (LCS)	• reanalyze to verify that an out of control situation exists; • determine the source of error within the preparation procedure, correct the problem and repeat the sample set. Sources of contamination could be the reagents, the LCS stock solution, or the preparation area. **NOTE:** When LCS(s) is high, any samples that are < reporting limit may be reported. Precision: LCS and LCD must meet the control limits of ≤ 20 RPD. If this criteria is not met, and both LCS's meet the % Recovery control limits, then see your supervisor for proper corrective action.
Method Blank (MB)	• reanalyze to verify contamination at a level > reporting limit; • determine the source of contamination and correct the problem; • all samples whose concentration is <10x the MB level must be reprocessed and reanalyzed; any sample which is >10x the MB level need not be reanalyzed. However, the situation must be documented on the data review checklist.
Matrix Duplicate (DU)	• the sample must be reprocessed and reanalyzed unless the sample concentration is <5x reporting Limit, then the ± Reporting Limit rule applies; • if the reanalysis is within the control limits, the second value is reported; • The situation must be documented on the data review checklist and approved by your supervisor.

QC Standard	Suggested Corrective Actions
Matrix Spike (MS)	• If a single spike is done and it is outside the acceptance limits, it must be repeated to verify the matrix effect. • Report all spikes, whether or not they are in control. • Note out-of-control spikes on the data review checklist.
Continuing Calibration Verification (CCV)	• reanalyze to verify proper preparation; • prepare new CCV from original stock; • check for instrument base-line drift; • check reagents/solutions and prepare fresh if necessary; • recalibrate with a new standard curve and repeat all samples since the previous in control CCV; • never dispose of any samples until you are sure that all QC are within designated control limits.
Continuing Calibration Blank (CCB)	• prepare a new CCB to verify proper preparation; • verify that the instrument base-line is stable and/or perform necessary maintenance, cleaning, etc... to achieve stability; • determine the source of contamination by the process of elimination, correct the problem and reanalyze all the samples since the previous in control CCB. (Carry over from a previous analysis or reagent contamination are two common sources). • never dispose of any samples until you are sure that all QC are within their designated control limits.

9.0 DATA ANALYSIS AND CALCULATIONS

9.1 Sample Results

mg/L values (TOC, TIC, DOC, and DIC) are read directly from the instrument printer and entered into TALs LIMS.

*Either the individual result from each duplicate or quadruplicate analysis of samples is reported and/or the mean of the readings is reported. The mean is not given directly in the Phoenix print-out, but is calculated in the laboratory's LIMS. The replicate results must meet the laboratory's precision requirements. The high and low results obtained from quadruplicate analysis must meet the laboratory's precision requirements.

9.2 Accuracy

9.2.1 ICV/CCV, LCS % Recoveries = $\frac{\text{avg. observed concentration}}{\text{actual concentration}} \times 100$

9.2.2 MS % Recovery = $\frac{\text{(spike sample result) - (unspiked sample result)}}{\text{spike concentration}}$

9.3 Precision

Matrix Duplicate and LCD (when required) Relative Percent Difference (RPD):

$$RPD = \frac{|\text{orig. sample value - dup. sample value}|}{[(\text{orig. sample value + dup. sample value})/2]} \times 100$$

*For calculating duplicates and MSs, the original sample value is the average of the replicate/quadruplicate results of the sample.

10.0 POLLUTION CONTROL

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

10.1 Waste Management

Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to UP-WM-001.

- Waste from this procedure will enter the 'Corrosive Wastestream'.

11.0 METHOD PERFORMANCE CRITERIA

Refer to Sections 1, 6, 7 and 8.

12.0 REFERENCES

Refer to Section 1.

13.0 ATTACHMENTS

Attachment 1: Data Review Checklist
Attachment 2: Analytical Data Cover Page
Attachment 3: TOC Maintenance Logs
Attachment 4: DoD QSM Version 4.2: Appendix F QC Requirements Summary (Table F-1)

14.0 REVISION HISTORY

- Revision 12 updated on 11/24/10
- Annual Review
- Change CCV prep from Stock II to Stock I
- Add instrument designation
- Add Fusion instrument
- Add software version
- Add Fusion maintenance manual page to Attachment 3

Attachment 1.

Example: Data Review Checklist (015-001)

TestAmerica Chicago
INORGANICS – LIMS DATA REVIEW CHECKLIST **Test** _____ **Analytical Batch#** _____

Prep Batch # _____
(File by analytical batch #)

Batch Entry Date: _____ Analysis Date: _____ No. of Jobs in Batch: _____

Analyst / Primary Reviewer: _____ 1st Level Review Date: _____

Secondary Reviewer: _____ 2nd Level Review Date: _____

	PRI REV	SEC REV	COMMENTS
1) Analyst correct			
2) Instrument Code present			
3) Was Data Imported _____ Manually entered _____ Balance Interface Used _____			
4) Samples & all QC in order as analyzed?			
5) Sample Date/Time analyzed correct			
6) Reagent Codes present and Amount Spiked correct?			
7) Dilution factors all present and correct?			
8) Are correct Sample ID's used? Are all samples designated with a Blue P?			
9) Are correct QC ID's used? Are all QC designated with a Blue P?			
10) Are all QC correctly related to the samples?			
11) Do all entries match raw data?			
12) Is all QC calculated and are correct flags applied?			
13) Is an NCM needed? ICV, MB, LCS, LCSD, DU, MS, MSD, RPD out; holding time missed or Manual Integration Required			NCM # _____ Approved By: _____ Initials
Raw Data: 1) Is AD Batch # is clearly noted?			
2) Are manual calculations and final results clearly shown?			
3) Are all errors crossed out with single line & initialed and dated?			
4) Is unreported data clearly identified with reason & initialed and dated?			
5) All unused portions of the page(s) Z'd out?			
6) Is data signed & dated by analyst & reviewer?			

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(015-001)

Attachment 2.

Analytical Data Cover Page
(016-001)

TestAmerica Chicago

Total Organic/Inorganic Carbon

Method:

EPA 415.1 _____
EPA 415.1, mod. for IC _____
SW846 9060 _____

Analytical Batch #: _____
Analytical Batch #: _____
Prep Batch #: _____

NCM #: _____

Instrument: Phoenix 8000 TOC 3
Matrix: Water

Calibration Range: 1.0 – 40.0 mg/L
Reporting Limit: 1.0 mg/L

Standard Curve: Date of Curve _____

Calculations:

Waters: mg/L = mg/L X dilution

Standards Traceability:

Stock I: Calibration and Curve: _____

Stock II: LCS, ICV, CCV, Matrix spikes _____

Stock II: TIC sparger check _____

Spiking Levels:

LCS	_____ mg/L
ICV	_____ mg/L
CCV	_____ mg/L
CCV (alt.)	_____ mg/L
MS/MSD	_____ mg/L
TIC check	_____ mg/L

Comments:

_____
_____
_____

Analyst:_____Date of analysis:_____

Reviewer: _____ Date: _____

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(016-001)

Attachment 3.

Example: TOC Maintenance Logs
(017-001 to 017-002)

TestAmerica Chicago
TOC3 Maintenance Log - Phoenix 8000 Page No.:_____

Schedule	Action	Date	Date	Date	Date	Date	Date	Date
DAILY	Carrier Gas, 500+ psi from tank to run scheduled load							
	Ample persulfate supply for load							
	Ample acid supply for load							
	Replaced and ample DI water supply for load							
	Check chlorine scrubber to ensure ample life for load							
	Carrier Gas Flow Rate (200 cc/min ± 10%)							
	Gas/Liquid Separator water level filled to waste outlet							
	Empty Mist Trap							
	Make sure 8-port valve thumbscrews are hand-tightened							
WEEKLY	Clean UV Reactor and IC Sparger with soap and water as needed							
	Change reagents, if needed							
	Replace water in Gas/Liquid Separator							
MONTHLY	Change Chlorine Scrubber							
	Inspect Permeation Dryer for damage, water accumulation							
	ANALYST INITIALS:							

Non-Routine Maintenance:_____

Reviewed by:_____ Date:_____

CHI-22-12-091/C-06/10

(017-001)

TESTAMERICA CHICAGO Page: _____

TOC5 – TELEDYNE – TEKMAR TOC ANALYZER: FUSION

DAILY MAINTENANCE LOG

General Information:

Date:							
Analyst Initials:							

Daily:

N_2 Levels – Tank >500 PSI Reg. ~70PSI							
Reagent levels OK							
Water bottle filled							
Copper/Tin OK							

Weekly:

Run leak check							
Perm. Dryer OK							

Monthly:

Prepare New Standards							
Flush sample transfer line							
Clean reactor and sparger if needed							
Add a few acid drops to mist trap							

Comments:

_____

_____

_____

_____

_____

_____

Return to control:

_____

_____

Reviewer Signature:_____ Date:_____

CHI-22-12-133/A-08/10 (017-002)

Attachment 4.

DoD QSM Version 4.2: Appendix F QC Requirements Summary Table F-1 (018-001 to 018-002)

TestAmerica Chicago

DoD QSM Version 4.2: Appendix F - Quality Control Requirements Summary

Table F-1 Summary of QC Check Definitions, Purpose, and Evaluation – Inorganics (WC)

QC Check	Definition	Purpose	Evaluation
Calibration Blank	Reagent water containing no analytes of interest.	To determine the zero point of the calibration curve for all initial and continuing calibrations.	This is a required QC procedure. Continuing calibration blank responses above the LOD require corrective action
Continuing calibration verification (CCV)	This verification of the ICAL that is required during the course of analysis at periodic intervals. Continuing calibration verification applies to both external standard and internal standard calibration techniques, as well as to linear and non-linear calibration models	To verify that instrument response is reliable, and has not changed significantly from the current ICAL curve.	If the values for the analytes are outside the acceptance criteria, the ICAL may not be stable. Results associated with out-of-control CCV results require reanalysis or flagging
Demonstrate Acceptable Analytical Capability	QC samples are analyzed in series to verify ability to produce data of acceptable precision and bias.	To verify the ability to produce data of acceptable precision and bias for a specific instrument type, matrix, method, and analyst.	The average recovery of the spikes and standard deviation of the replicates must be within designated acceptance criteria. Analysis of field samples may not be conducted until this check is successful.
Duplicate Sample (replicate)	Two identical portions of material collected for chemical analysis, and identified by unique alphanumeric codes. The duplicate may be portioned from the same sample, or may be two identical samples taken from the same site. The two portions are prepared and analyzed identically. (modified QSM)	To provide information on the heterogeneity of the sample matrix or to determine the precision of the intralaboratory analytical process for a specific sample matrix	A duplicate sample will provide information on the heterogeneity of the sample matrix. The greater the heterogeneity of the matrix, the greater the relative percent difference between the sample and the sample duplicate. If the sample matrix is homogeneous (such as with drinking water) and the relative pecent difference is high, this could indicate a problem in the analytical system.
Initial calibration for all analytes (ICAL)	Analysis of analytical standards at different concentrations that are used to determine and calibrate the quantitation range of the response of the analytical detector or method	To establish a calibration curve for the quantification of the analytes of interest	Statistical procedures are used to determine the relationship between the signal response and the known concentration of analytes of interest. The ICAL must be successful before any samples or other QC check samples can be analyzed.
Laboratory control sample (LCS) containing all analytes to be reported	A sample matrix, free from the analytes of interest, spiked with known amounts of analytes or a material containing known and verified amounts of analytes.	Used to evaluate the performance of the total analytical system, including all preparation and analysis steps. Assesses the ability of the laboratory/analyst to successfully recover the target analytes from a control (clean) matrix. Control limits for LCS recovery, typically expressed as percent recovery, are used for the development of statistical control limits and serve as acceptance criteria for determining whether an analytical run is in control (batch acceptance).	This is a required QC Check. The inability to achieve acceptable recoveries in the LCS indicate problems with the precision and bias of the measurement system. Failure to achieve acceptable recoveries in a "clean" matrix is an indicator of possible problems achieving acceptable recoveries in field samples.
MS	A sample prepared by adding a know mass of target analyte to a specified amount of matrix sample for which an independent estimate of target analyte concentration is available.	To assess the performance of the method as applied to a particular matrix. Matrix spikes are used, for example, to determine the effect of the matrix on a method's recovery efficiency. The recovery of target analytes from the matrix spike sample is used to determine the bias of the method in the specific sample matrix.	The lack of acceptable recoveries in the MS often points to problems with the sample matrix. One test of this is a comparison to the LCS recoveries. If the corresponding LCS recoveries are within acceptable limits, a matrix effect is likely. The lab should not correct for recovery; only report the results of the analyses and the associated MS results and indicate that the results from these analyses have increased uncertainty
MSD	A second replicate matrix spike prepared in the laboratory and analyzed to obtain a measure of the precision of recovery for each analyte	To assess the performance of the method as applied to a particular matrix and provide information on the homogeneity of the matrix. Also used to determine the precision of the intralaboratory analytical process for a specific sample matrix.	When compared to the MS, the MSD will provide information on the heterogeneity of the sample matrix. The greater the heterogeneity of the matrix, the greater the RPD between the matrix spike and the matrix spike duplicate. If the sample matrix is homogeneous, such as with drinking water, and the RPD is high, this could indicate a problem in the analytical system.

(C-2) C-662

(C18-0d)

F-1 (cont.)			
QC Check	**Definition**	**Purpose**	**Evaluation**
Matrix Verification sample (CR+6 only)	A pH-adjusted filtrate that has been spiked with CR+6 to ensure that the sample matrix does not have a reducing condition or other interferents that could affect color development. (Modified Method)	To ensure that the sample matrix does not have a reducing condition or other interferents that affect color development.	To verify the absence of an interference, the spike recovery must be between 85% and 115%. If the result of verification indicates a suppressive interference, the sample should be diluted and reanalyzed. If the interference persists after sample dilution, an alternative method (Method 7195, Coprecipitation, or Method 7197, Chelation/Extraction) should be used.
MB	A sample of a matrix similar to the batch of associated samples (when available) that is free from the analytes of interest and is processed simultaneously with an under the same conditions as samples through all steps of the analytical procedures, and in which no target analytes or interferences are present at concentrations that impact the analytical results for sample analyses.	To assess background interferences or contamination in the analytical system that might lead to high bias or false positive data. Results of method blanks provide an estimate of the within-batch variability of the blank response and an indication of bias introduced by the preparation and analytical procedure.	This is one of the QC samples used to measure lab accuracy/bias. The sample could indicate whether contamination is occurring during sample prep and analysis. If analytes are detected > ½ RL, reanalyze or qualify (B-flag) all results for the specific analytes(s) in all samples in the associated prep batch as appropriate. For common lab contaminants, no analytes detected > the RL. See Section D.1.1.1 and Box D-1
RT window position establishment for each analyte (and surrogate) (all chromatographic methods only)	Determination of the placement of the RT window (i.e. start/stop time) of each analyte or group of analytes as it elutes through the chromatographic column so that analyte identification can be made during sample analysis. This is done during the ICAL.	To idendify analytes of interest	Incorrect window position may result in false negatives, require additional manual integrations, or cause unnecessary reanalysis of samples when surrogates or spiked compounds are erroneously not identified.
RT window width calculated for each analyte (and surrogate) (non-MS chromatographic methods only)	Determination of the length of time between sample injection and the appearance of a peak at the detector. The total length of time (window) is established for each analyte or group of analytes and is set for complete elution of analyte peaks. It is based upon a series of analyses and statistical calculations that establish the measured band on the chromatogram that can be associated with a specific analyte or group of analytes.	To ensure that the chromatographic system is operating reliably and that the system conditions have been optimized for the target anaytes and surrogates in the standards and sample matrix to be analyzed. It is done to minimize the occurrence of both false positive and false negative results.	Used to evaluate continued system performance. Tight RT windows may result in false negatives or may cuase unnecessary reanalysis of samples when surrogates or spiked compounds are erroneously not identified. Overly wide RT windows may result in false positive results that cannot be confirmed upon further analysis.
Second source calibration verification (ICV)	A standard obtained or prepared from a source independent of the source of standards for the ICAL. Its concentration should be at or near the middle of the calibration range. It is done after the ICAL.	To verify the accuracy of the ICAL.	The concentration of the 2nd source calibration verification, determined from the analysis, is compared with the known value of the standard to determine the accuracy of the ICAL. This independent verification of the ICAL must be acceptable before sample analysis can begin.

Notes:
1. Project-specific requirements identified by the client supersede any requirements listed. The requirements are meant to be default, to be used when project-specific direction based on DQOs is not available.
2. If there is a contradiction between the method and the DoD tables, the requirements specified in the tables shall be followed.

(C-2) C-663

(018-002)

COMMONWEALTH OF PENNSYLVANIA
DEPARTMENT OF ENVIRONMENTAL PROTECTION

OFFICE OF FIELD OPERATIONS

BUREAU OF LABORATORIES

pennsylvania
DEPARTMENT OF ENVIRONMENTAL PROTECTION

Certifies that

02-00538

MICROSEEPS INCORPORATED
220 WILLIAM PITT WAY
PITTSBURGH, PA 15238-1328

Having duly met the requirement of
The Act of June 29, 2002 (P.L. 596, No. 90)
dealing with Environmental Laboratory Accreditation
(27 Pa. C.S. §§4101-4113) and the
National Environmental Laboratory Accreditation Conference Standard

is hereby approved as an

Accredited Laboratory

As more fully described in the attached Scope of Accreditation

Expiration Date: **11/30/2011**
Certificate Number: **008**

Continued accreditation status depends on successful ongoing participation in the Program
Certificate not transferable Surrender upon revocation
To be conspicuously displayed at the Laboratory
Not valid unless accompanied by a valid Scope of Accreditation
Shall not be used to imply endorsement by the Commonwealth of Pennsylvania
Customers are urged to verify the laboratory's current accreditation status
PA DEP is a NELAP recognized accreditation body

Aaren S. Alger, Chief
Laboratory Accreditation Program
Bureau of Laboratories

1500-FM-LAB0016 Rev. 8/2009

(C-2) C-664

Attachment to Certificate of Accreditation 008, expiration date November 30, 2011. This listing of accredited analytes should be used only when associated with a valid certificate of accreditation.

State Laboratory ID: 2-00538 **EPA Lab Code: PA00076** **(412) 826-5245**

Microseeps Inc
220 William Pitt Way
Pittsburgh, PA 15238

Program Non-Potable Water

Method	Analyte	Accreditation Type	Primary	Effective Date
EPA 5030B	Aqueous-phase purge-and-trap	NELAP	PA	1/6/2006
EPA 624	1,1,1-Trichloroethane	NELAP	PA	1/6/2006
EPA 624	1,1,2,2-Tetrachloroethane	NELAP	PA	1/6/2006
EPA 624	1,1,2-Trichloroethane	NELAP	PA	1/6/2006
EPA 624	1,1-Dichloroethane	NELAP	PA	1/6/2006
EPA 624	1,1-Dichloroethene (1,1-Dichloroethylene)	NELAP	PA	1/6/2006
EPA 624	1,2-Dichlorobenzene (o-Dichlorobenzene)	NELAP	PA	1/6/2006
EPA 624	1,2-Dichloroethane	NELAP	PA	1/6/2006
EPA 624	1,2-Dichloropropane	NELAP	PA	1/6/2006
EPA 624	1,3-Dichlorobenzene (m-Dichlorobenzene)	NELAP	PA	1/6/2006
EPA 624	1,4-Dichlorobenzene (p-Dichlorobenzene)	NELAP	PA	1/6/2006
EPA 624	2-Chloroethyl vinyl ether	NELAP	PA	1/6/2006
EPA 624	Acrolein (Propenal)	NELAP	PA	1/6/2006
EPA 624	Acrylonitrile	NELAP	PA	1/6/2006
EPA 624	Benzene	NELAP	PA	1/6/2006
EPA 624	Bromodichloromethane	NELAP	PA	1/6/2006
EPA 624	Bromoform	NELAP	PA	1/6/2006
EPA 624	Carbon tetrachloride	NELAP	PA	1/6/2006
EPA 624	Chlorobenzene	NELAP	PA	1/6/2006
EPA 624	Chloroethane	NELAP	PA	1/6/2006
EPA 624	Chloroform	NELAP	PA	1/6/2006
EPA 624	Dibromochloromethane	NELAP	PA	1/6/2006
EPA 624	Ethylbenzene	NELAP	PA	1/6/2006
EPA 624	Methyl bromide (Bromomethane)	NELAP	PA	1/6/2006
EPA 624	Methyl chloride (Chloromethane)	NELAP	PA	1/6/2006
EPA 624	Methylene chloride (Dichloromethane)	NELAP	PA	1/6/2006
EPA 624	Tetrachloroethene (PCE, Perchloroethylene)	NELAP	PA	9/24/2007
EPA 624	Toluene	NELAP	PA	1/6/2006
EPA 624	Trichloroethene (TCE, Trichloroethylene)	NELAP	PA	2/8/2008
EPA 624	Trichlorofluoromethane (Freon 11)	NELAP	PA	1/6/2006
EPA 624	Vinyl chloride (Chloroethene)	NELAP	PA	1/6/2006
EPA 624	Xylenes, total	NELAP	PA	1/6/2006
EPA 624	cis-1,3-Dichloropropene	NELAP	PA	1/6/2006
EPA 624	trans-1,2-Dichloroethene	NELAP	PA	1/6/2006
EPA 624	trans-1,3-Dichloropropene	NELAP	PA	1/6/2006
EPA 7199 Modified	Divalent manganese	NELAP	PA	11/15/2010

The Pennsylvania Department of Environmental Protection Laboratory Accreditation Program is a NELAP recognized accrediting authority. Customers are urged to verify the laboratory's current accreditation standing.
www.dep.state.pa.us **Issue Date: 11/15/2010**

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State Laboratory ID: 2-00538 **EPA Lab Code: PA00076** **(412) 826-5245**

Microseeps Inc
220 William Pitt Way
Pittsburgh, PA 15238

Program Non-Potable Water

Method	Analyte	Accreditation Type	Primary	Effective Date
EPA 7199 Modified	Ferric iron	NELAP	PA	11/15/2010
EPA 7199 Modified	Ferrous iron	NELAP	PA	11/15/2010
EPA 8260	1,1,1,2-Tetrachloroethane	NELAP	PA	1/6/2006
EPA 8260	1,1,1-Trichloroethane	NELAP	PA	1/6/2006
EPA 8260	1,1,2,2-Tetrachloroethane	NELAP	PA	1/6/2006
EPA 8260	1,1,2-Trichloroethane	NELAP	PA	1/6/2006
EPA 8260	1,1-Dichloroethane	NELAP	PA	1/6/2006
EPA 8260	1,1-Dichloroethene (1,1-Dichloroethylene)	NELAP	PA	1/6/2006
EPA 8260	1,1-Dichloropropene	NELAP	PA	1/6/2006
EPA 8260	1,2,3-Trichlorobenzene	NELAP	PA	1/6/2006
EPA 8260	1,2,3-Trichloropropane (1,2,3-TCP)	NELAP	PA	1/6/2006
EPA 8260	1,2,4-Trichlorobenzene	NELAP	PA	1/6/2006
EPA 8260	1,2,4-Trimethylbenzene	NELAP	PA	1/6/2006
EPA 8260	1,2-Dibromo-3-chloropropane (DBCP, Dibromochloropropane)	NELAP	PA	2/8/2008
EPA 8260	1,2-Dibromoethane (EDB, Ethylene dibromide)	NELAP	PA	9/26/2007
EPA 8260	1,2-Dichlorobenzene (o-Dichlorobenzene)	NELAP	PA	9/26/2007
EPA 8260	1,2-Dichloroethane	NELAP	PA	1/6/2006
EPA 8260	1,2-Dichloropropane	NELAP	PA	1/6/2006
EPA 8260	1,3,5-Trimethylbenzene	NELAP	PA	1/6/2006
EPA 8260	1,3-Dichlorobenzene (m-Dichlorobenzene)	NELAP	PA	1/6/2006
EPA 8260	1,3-Dichloropropane	NELAP	PA	1/6/2006
EPA 8260	1,4-Dichlorobenzene (p-Dichlorobenzene)	NELAP	PA	1/6/2006
EPA 8260	2,2-Dichloropropane	NELAP	PA	1/6/2006
EPA 8260	2-Butanone (Methyl ethyl ketone, MEK)	NELAP	PA	9/26/2007
EPA 8260	2-Chlorotoluene	NELAP	PA	1/6/2006
EPA 8260	2-Hexanone	NELAP	PA	9/26/2007
EPA 8260	4-Chlorotoluene	NELAP	PA	1/6/2006
EPA 8260	4-Methyl-2-pentanone (MIBK)	NELAP	PA	1/6/2006
EPA 8260	Acetone	NELAP	PA	1/6/2006
EPA 8260	Benzene	NELAP	PA	1/6/2006
EPA 8260	Bromobenzene	NELAP	PA	1/6/2006
EPA 8260	Bromochloromethane	NELAP	PA	1/6/2006
EPA 8260	Bromodichloromethane	NELAP	PA	1/9/2009
EPA 8260	Bromoform	NELAP	PA	1/6/2006
EPA 8260	Carbon disulfide	NELAP	PA	1/6/2006
EPA 8260	Carbon tetrachloride	NELAP	PA	1/6/2006

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Microseeps Inc
220 William Pitt Way
Pittsburgh, PA 15238

Program Non-Potable Water

Method	Analyte	Accreditation Type	Primary	Effective Date
EPA 8260	Chlorobenzene	NELAP	PA	1/6/2006
EPA 8260	Chloroethane	NELAP	PA	1/6/2006
EPA 8260	Chloroform	NELAP	PA	1/6/2006
EPA 8260	Dibromochloromethane	NELAP	PA	1/6/2006
EPA 8260	Dibromochloropropane (1,2-Dibromo-3-chloropropane, DBCP)	NELAP	PA	8/16/2006
EPA 8260	Dibromomethane	NELAP	PA	1/6/2006
EPA 8260	Dichlorodifluoromethane (Freon 12)	NELAP	PA	1/6/2006
EPA 8260	Ethanol	NELAP	PA	9/26/2007
EPA 8260	Ethylbenzene	NELAP	PA	1/6/2006
EPA 8260	Hexachlorobutadiene (1,3-Hexachlorobutadiene)	NELAP	PA	1/6/2006
EPA 8260	Isopropylbenzene	NELAP	PA	1/6/2006
EPA 8260	Methyl bromide (Bromomethane)	NELAP	PA	1/6/2006
EPA 8260	Methyl chloride (Chloromethane)	NELAP	PA	1/6/2006
EPA 8260	Methyl tert-butyl ether (MTBE)	NELAP	PA	1/6/2006
EPA 8260	Methylene chloride (Dichloromethane)	NELAP	PA	1/6/2006
EPA 8260	Naphthalene	NELAP	PA	1/6/2006
EPA 8260	Styrene	NELAP	PA	1/6/2006
EPA 8260	Tetrachloroethene (PCE, Perchloroethylene)	NELAP	PA	9/24/2007
EPA 8260	Toluene	NELAP	PA	1/6/2006
EPA 8260	Trichloroethene (TCE, Trichloroethylene)	NELAP	PA	1/6/2006
EPA 8260	Trichlorofluoromethane (Freon 11)	NELAP	PA	1/6/2006
EPA 8260	Vinyl chloride (Chloroethene)	NELAP	PA	1/6/2006
EPA 8260	Xylenes, total	NELAP	PA	1/6/2006
EPA 8260	cis-1,2-Dichloroethene	NELAP	PA	1/6/2006
EPA 8260	cis-1,3-Dichloropropene	NELAP	PA	1/6/2006
EPA 8260	n-Butylbenzene	NELAP	PA	1/6/2006
EPA 8260	n-Propylbenzene	NELAP	PA	1/6/2006
EPA 8260	sec-Butylbenzene	NELAP	PA	1/6/2006
EPA 8260	tert-Butyl alcohol (2-Methyl-2-propanol)	NELAP	PA	9/26/2007
EPA 8260	tert-Butylbenzene	NELAP	PA	1/6/2006
EPA 8260	trans-1,2-Dichloroethene	NELAP	PA	1/6/2006
EPA 8260	trans-1,3-Dichloropropene	NELAP	PA	1/6/2006
EPA 8260-Extended	Methyl isobutyl ketone (MIBK, 4-Methyl-2-pentanone)	NELAP	PA	11/7/2005
EPA 9040B	pH	NELAP	PA	3/20/2009
EPA 9056A	Chloride	NELAP	PA	4/21/2009

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Microseeps Inc
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Pittsburgh, PA 15238

Program Non-Potable Water

Method	Analyte	Accreditation Type	Primary	Effective Date
EPA 9056A	Nitrate	NELAP	PA	1/6/2006
EPA 9056A	Nitrite	NELAP	PA	1/6/2006
EPA 9056A	Sulfate	NELAP	PA	1/6/2006
EPA 9060	Total organic carbon (TOC)	NELAP	PA	11/15/2010
Microseeps SOP- AM20Gax	Carbon dioxide	NELAP	PA	1/6/2006
Microseeps SOP- AM20Gax	Carbon monoxide	NELAP	PA	1/6/2006
Microseeps SOP- AM20Gax	Ethane	NELAP	PA	1/6/2006
Microseeps SOP- AM20Gax	Ethene	NELAP	PA	1/6/2006
Microseeps SOP- AM20Gax	Hydrogen	NELAP	PA	1/6/2006
Microseeps SOP- AM20Gax	Isobutane (2-Methylpropane)	NELAP	PA	1/6/2006
Microseeps SOP- AM20Gax	Methane	NELAP	PA	1/6/2006
Microseeps SOP- AM20Gax	Oxygen	NELAP	PA	1/6/2006
Microseeps SOP- AM20Gax	Propane	NELAP	PA	1/6/2006
Microseeps SOP- AM20Gax	Propene	NELAP	PA	1/6/2006
Microseeps SOP- AM20Gax	n-Butane	NELAP	PA	1/6/2006
Microseeps SOP- AM20Gax	Inorganic carbon, total	NELAP	PA	1/6/2006
Microseeps SOP- AM20Gax	Acetylene (Ethyne)	NELAP	PA	1/6/2006
Microseeps SOP- AM21G	Acetic acid (Ethanoic acid)	NELAP	PA	3/20/2009
Microseeps SOP- AM21G	Lactic acid	NELAP	PA	3/20/2009
Microseeps SOP- AM21G	Butyric acid	NELAP	PA	3/20/2009
Microseeps SOP- AM21G	Propionic acid	NELAP	PA	3/20/2009
Microseeps SOP- AM21G	Pyruvic acid	NELAP	PA	3/20/2009
Microseeps SOP-AM 4 02	Total petroleum hydrocarbons (TPH)	NELAP	PA	1/6/2006
Microseeps SOP-AM 4 02	n-Decane	NELAP	PA	1/6/2006
Microseeps SOP-AM 4 02	n-Dodecane	NELAP	PA	1/6/2006
Microseeps SOP-AM 4 02	n-Hexadecane	NELAP	PA	1/6/2006
Microseeps SOP-AM 4 02	1,1,1-Trichloroethane	NELAP	PA	1/6/2006
Microseeps SOP-AM 4 02	1,1,2,2-Tetrachloroethane	NELAP	PA	1/6/2006
Microseeps SOP-AM 4 02	1,1,2-Trichloroethane	NELAP	PA	1/6/2006
Microseeps SOP-AM 4 02	1,1-Dichloroethane	NELAP	PA	1/6/2006
Microseeps SOP-AM 4 02	1,1-Dichloroethene (1,1-Dichloroethylene)	NELAP	PA	1/6/2006
Microseeps SOP-AM 4 02	1,2,3-Trichlorobenzene	NELAP	PA	1/6/2006
Microseeps SOP-AM 4 02	1,2,4-Trichlorobenzene	NELAP	PA	1/6/2006
Microseeps SOP-AM 4 02	1,2,4-Trimethylbenzene	NELAP	PA	1/6/2006
Microseeps SOP-AM 4 02	1,2-Dichlorobenzene (o-Dichlorobenzene)	NELAP	PA	1/6/2006
Microseeps SOP-AM 4 02	1,2-Dichloroethane	NELAP	PA	1/6/2006

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State Laboratory ID: 2-00538 **EPA Lab Code: PA00076** **(412) 826-5245**

Microseeps Inc
220 William Pitt Way
Pittsburgh, PA 15238

Program Non-Potable Water

Method	Analyte	Accreditation Type	Primary	Effective Date
Microseeps SOP-AM 4 02	1,2-Dichloropropane	NELAP	PA	1/6/2006
Microseeps SOP-AM 4 02	1,3,5-Trimethylbenzene	NELAP	PA	1/6/2006
Microseeps SOP-AM 4 02	1,3-Dichlorobenzene (m-Dichlorobenzene)	NELAP	PA	1/6/2006
Microseeps SOP-AM 4 02	1,4-Dichlorobenzene (p-Dichlorobenzene)	NELAP	PA	1/6/2006
Microseeps SOP-AM 4 02	2-Butanone (Methyl ethyl ketone, MEK)	NELAP	PA	1/6/2006
Microseeps SOP-AM 4 02	2-Chloroethyl vinyl ether	NELAP	PA	1/6/2006
Microseeps SOP-AM 4 02	2-Hexanone	NELAP	PA	1/6/2006
Microseeps SOP-AM 4 02	2-Propanol (Isopropyl alcohol)	NELAP	PA	1/6/2006
Microseeps SOP-AM 4 02	4-Methyl-2-pentanone (MIBK)	NELAP	PA	1/6/2006
Microseeps SOP-AM 4 02	Acetone	NELAP	PA	1/6/2006
Microseeps SOP-AM 4 02	Benzene	NELAP	PA	1/6/2006
Microseeps SOP-AM 4 02	Bromodichloromethane	NELAP	PA	1/6/2006
Microseeps SOP-AM 4 02	Bromoform	NELAP	PA	1/6/2006
Microseeps SOP-AM 4 02	Carbon tetrachloride	NELAP	PA	1/6/2006
Microseeps SOP-AM 4 02	Chlorobenzene	NELAP	PA	1/6/2006
Microseeps SOP-AM 4 02	Chloroethane	NELAP	PA	1/6/2006
Microseeps SOP-AM 4 02	Chloroform	NELAP	PA	1/6/2006
Microseeps SOP-AM 4 02	Dibromochloromethane	NELAP	PA	1/6/2006
Microseeps SOP-AM 4 02	Dichlorodifluoromethane (Freon 12)	NELAP	PA	1/6/2006
Microseeps SOP-AM 4 02	Ethylbenzene	NELAP	PA	1/6/2006
Microseeps SOP-AM 4 02	Isopropylbenzene	NELAP	PA	1/6/2006
Microseeps SOP-AM 4 02	Methyl bromide (Bromomethane)	NELAP	PA	1/6/2006
Microseeps SOP-AM 4 02	Methyl chloride (Chloromethane)	NELAP	PA	1/6/2006
Microseeps SOP-AM 4 02	Methyl tert-butyl ether (MTBE)	NELAP	PA	1/6/2006
Microseeps SOP-AM 4 02	Methylene chloride (Dichloromethane)	NELAP	PA	1/6/2006
Microseeps SOP-AM 4 02	Naphthalene	NELAP	PA	1/6/2006
Microseeps SOP-AM 4 02	Styrene	NELAP	PA	1/6/2006
Microseeps SOP-AM 4 02	Tetrachloroethene (PCE, Perchloroethylene)	NELAP	PA	1/6/2006
Microseeps SOP-AM 4 02	Toluene	NELAP	PA	1/6/2006
Microseeps SOP-AM 4 02	Trichloroethene (TCE, Trichloroethylene)	NELAP	PA	1/6/2006
Microseeps SOP-AM 4 02	Trichlorofluoromethane (Freon 11)	NELAP	PA	1/6/2006
Microseeps SOP-AM 4 02	Vinyl chloride (Chloroethene)	NELAP	PA	1/6/2006
Microseeps SOP-AM 4 02	Xylenes, total	NELAP	PA	1/6/2006
Microseeps SOP-AM 4 02	cis-1,2-Dichloroethene	NELAP	PA	1/6/2006
Microseeps SOP-AM 4 02	cis-1,3-Dichloropropene	NELAP	PA	1/6/2006
Microseeps SOP-AM 4 02	n-Heptane	NELAP	PA	1/6/2006

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www.dep.state.pa.us **Issue Date: 11/15/2010**

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State Laboratory ID: 2-00538 **EPA Lab Code: PA00076** **(412) 826-5245**

Microseeps Inc
220 William Pitt Way
Pittsburgh, PA 15238

Program Non-Potable Water

Method	Analyte	Accreditation Type	Primary	Effective Date
Microseeps SOP-AM 4 02	n-Hexane	NELAP	PA	1/6/2006
Microseeps SOP-AM 4 02	trans-1,2-Dichloroethene	NELAP	PA	1/6/2006
Microseeps SOP-AM 4 02	trans-1,3-Dichloropropene	NELAP	PA	1/6/2006
Microseeps SOP-AM23G	Acetic acid (Ethanoic acid)	NELAP	PA	1/6/2006
Microseeps SOP-AM23G	Hydroxy-isobutyric acid (HIBA)	NELAP	PA	1/6/2006
Microseeps SOP-AM23G	Isohexanoic acid	NELAP	PA	1/6/2006
Microseeps SOP-AM23G	Lactic acid	NELAP	PA	1/6/2006
Microseeps SOP-AM23G	n-Hexanoic acid	NELAP	PA	1/6/2006
Microseeps SOP-AM23G	Butyric acid	NELAP	PA	1/6/2006
Microseeps SOP-AM23G	Isopentanoic acid	NELAP	PA	1/6/2006
Microseeps SOP-AM23G	Propionic acid	NELAP	PA	1/6/2006
Microseeps SOP-AM23G	Pyruvic acid	NELAP	PA	1/6/2006
Microseeps SOP-AM23G	n-Pentanoic acid	NELAP	PA	1/6/2006
Microseeps SOP-GCMS3	BTEX (Benzene, toluene, ethylbenzene, and xylenes)	NELAP	PA	11/27/2007
Microseeps SOP-GCMS3	Ethanol	NELAP	PA	11/27/2007
Microseeps SOP-GCMS3	Ethyl tert-butyl ether (ETBE)	NELAP	PA	11/27/2007
Microseeps SOP-GCMS3	Isopropylbenzene	NELAP	PA	11/27/2007
Microseeps SOP-GCMS3	Methyl tert-butyl ether (MTBE)	NELAP	PA	11/27/2007
Microseeps SOP-GCMS3	Naphthalene	NELAP	PA	11/27/2007
Microseeps SOP-GCMS3	tert-Amyl alcohol (2-Methyl-2-butanol)	NELAP	PA	11/27/2007
Microseeps SOP-GCMS3	tert-Amyl ethyl ether (TAEE)	NELAP	PA	11/27/2007
Microseeps SOP-GCMS3	tert-Amyl methyl ether (TAME)	NELAP	PA	11/27/2007
Microseeps SOP-GCMS3	tert-Butyl alcohol (2-Methyl-2-propanol)	NELAP	PA	11/27/2007
Microseeps SOP-PM 01C	Carbon dioxide	NELAP	PA	1/6/2006
Microseeps SOP-PM 01C	Ethane	NELAP	PA	1/6/2006
Microseeps SOP-PM 01C	Ethene	NELAP	PA	1/6/2006
Microseeps SOP-PM 01C	Isobutane (2-Methylpropane)	NELAP	PA	1/6/2006
Microseeps SOP-PM 01C	Methane	NELAP	PA	1/6/2006
Microseeps SOP-PM 01C	Nitrogen	NELAP	PA	1/6/2006
Microseeps SOP-PM 01C	Oxygen	NELAP	PA	1/6/2006
Microseeps SOP-PM 01C	Propane	NELAP	PA	1/6/2006
Microseeps SOP-PM 01C	Propene	NELAP	PA	1/6/2006
Microseeps SOP-PM 01C	n-Butane	NELAP	PA	1/6/2006
Microseeps SOP-PM 01C	Inorganic carbon, total	NELAP	PA	1/6/2006
Microseeps SOP-PM 01C	Acetylene (Ethyne)	NELAP	PA	1/6/2006
Microseeps SOP-SM 9	Carbon dioxide	NELAP	PA	1/6/2006

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Microseeps Inc
220 William Pitt Way
Pittsburgh, PA 15238

Program Non-Potable Water

Method	Analyte	Accreditation Type	Primary	Effective Date
Microseeps SOP-SM 9	Ethane	NELAP	PA	1/6/2006
Microseeps SOP-SM 9	Ethene	NELAP	PA	1/6/2006
Microseeps SOP-SM 9	Hydrogen	NELAP	PA	1/6/2006
Microseeps SOP-SM 9	Isobutane (2-Methylpropane)	NELAP	PA	1/6/2006
Microseeps SOP-SM 9	Nitrogen	NELAP	PA	1/6/2006
Microseeps SOP-SM 9	Oxygen	NELAP	PA	1/6/2006
Microseeps SOP-SM 9	Propane	NELAP	PA	1/6/2006
Microseeps SOP-SM 9	Propene	NELAP	PA	1/6/2006
Microseeps SOP-SM 9	n-Butane	NELAP	PA	1/6/2006
RSK-175	Ethane	NELAP	PA	3/20/2009
RSK-175	Ethene	NELAP	PA	3/20/2009
RSK-175	Methane	NELAP	PA	3/20/2009
RSK-175	Propane	NELAP	PA	3/20/2009
SM 2320 B	Alkalinity as CaCO3	NELAP	PA	12/7/2004
SM 4500-H+ B	pH	NELAP	PA	3/20/2009
SM 4500-S F	Sulfide	NELAP	PA	5/10/2007
SM 5310 C	Total organic carbon (TOC)	NELAP	PA	11/15/2010

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State Laboratory ID: 2-00538 **EPA Lab Code:** PA00076 **(412) 826-5245**

Microseeps Inc
220 William Pitt Way
Pittsburgh, PA 15238

Program Solid and Chemical Materials

Method	Analyte	Accreditation Type	Primary	Effective Date
EPA 5035	Closed-system purge-and-trap (bisulfate option)	NELAP	PA	1/6/2006
EPA 5035	Closed-system purge-and-trap (methanol option)	NELAP	PA	1/6/2006
EPA 8260	1,1,1,2-Tetrachloroethane	NELAP	PA	11/7/2005
EPA 8260	1,1,1-Trichloroethane	NELAP	PA	12/7/2004
EPA 8260	1,1,2,2-Tetrachloroethane	NELAP	PA	12/7/2004
EPA 8260	1,1,2-Trichloroethane	NELAP	PA	12/7/2004
EPA 8260	1,1-Dichloroethane	NELAP	PA	12/7/2004
EPA 8260	1,1-Dichloroethene (1,1-Dichloroethylene)	NELAP	PA	12/7/2004
EPA 8260	1,2,3-Trichlorobenzene	NELAP	PA	11/7/2005
EPA 8260	1,2,3-Trichloropropane (1,2,3-TCP)	NELAP	PA	11/7/2005
EPA 8260	1,2,4-Trichlorobenzene	NELAP	PA	11/7/2005
EPA 8260	1,2,4-Trimethylbenzene	NELAP	PA	11/7/2005
EPA 8260	1,2-Dibromo-3-chloropropane (DBCP, Dibromochloropropane)	NELAP	PA	2/8/2008
EPA 8260	1,2-Dibromoethane (EDB, Ethylene dibromide)	NELAP	PA	12/7/2004
EPA 8260	1,2-Dichlorobenzene (o-Dichlorobenzene)	NELAP	PA	11/7/2005
EPA 8260	1,2-Dichloroethane	NELAP	PA	12/7/2004
EPA 8260	1,2-Dichloropropane	NELAP	PA	12/7/2004
EPA 8260	1,3-Dichlorobenzene (m-Dichlorobenzene)	NELAP	PA	11/7/2005
EPA 8260	1,3-Dichloropropane	NELAP	PA	11/7/2005
EPA 8260	1,4-Dichlorobenzene (p-Dichlorobenzene)	NELAP	PA	11/7/2005
EPA 8260	2,2-Dichloropropane	NELAP	PA	11/7/2005
EPA 8260	2-Chlorotoluene	NELAP	PA	11/7/2005
EPA 8260	2-Hexanone	NELAP	PA	11/7/2005
EPA 8260	4-Chlorotoluene	NELAP	PA	11/7/2005
EPA 8260	4-Methyl-2-pentanone (MIBK)	NELAP	PA	12/7/2004
EPA 8260	Acetone	NELAP	PA	12/7/2004
EPA 8260	Benzene	NELAP	PA	12/7/2004
EPA 8260	Bromobenzene	NELAP	PA	11/7/2005
EPA 8260	Bromochloromethane	NELAP	PA	11/7/2005
EPA 8260	Bromodichloromethane	NELAP	PA	12/7/2004
EPA 8260	Bromoform	NELAP	PA	12/7/2004
EPA 8260	Carbon disulfide	NELAP	PA	12/7/2004
EPA 8260	Carbon tetrachloride	NELAP	PA	12/7/2004
EPA 8260	Chlorobenzene	NELAP	PA	12/7/2004
EPA 8260	Chloroethane	NELAP	PA	12/7/2004
EPA 8260	Chloroform	NELAP	PA	12/7/2004

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Microseeps Inc
220 William Pitt Way
Pittsburgh, PA 15238

Program Solid and Chemical Materials

Method	Analyte	Accreditation Type	Primary	Effective Date
EPA 8260	Dibromochloromethane	NELAP	PA	12/7/2004
EPA 8260	Dibromomethane	NELAP	PA	11/7/2005
EPA 8260	Dichlorodifluoromethane (Freon 12)	NELAP	PA	11/7/2005
EPA 8260	Ethylbenzene	NELAP	PA	12/7/2004
EPA 8260	Isopropylbenzene	NELAP	PA	12/7/2004
EPA 8260	Methyl bromide (Bromomethane)	NELAP	PA	12/7/2004
EPA 8260	Methyl chloride (Chloromethane)	NELAP	PA	12/7/2004
EPA 8260	Methyl tert-butyl ether (MTBE)	NELAP	PA	12/7/2004
EPA 8260	Methylene chloride (Dichloromethane)	NELAP	PA	12/7/2004
EPA 8260	Naphthalene	NELAP	PA	12/7/2004
EPA 8260	Styrene	NELAP	PA	12/7/2004
EPA 8260	Tetrachloroethene (PCE, Perchloroethylene)	NELAP	PA	12/7/2004
EPA 8260	Toluene	NELAP	PA	12/7/2004
EPA 8260	Trichloroethene (TCE, Trichloroethylene)	NELAP	PA	12/7/2004
EPA 8260	Trichlorofluoromethane (Freon 11)	NELAP	PA	11/7/2005
EPA 8260	Vinyl chloride (Chloroethene)	NELAP	PA	12/7/2004
EPA 8260	Xylenes, total	NELAP	PA	12/7/2004
EPA 8260	cis-1,2-Dichloroethene	NELAP	PA	12/7/2004
EPA 8260	cis-1,3-Dichloropropene	NELAP	PA	12/7/2004
EPA 8260	n-Butylbenzene	NELAP	PA	11/7/2005
EPA 8260	n-Propylbenzene	NELAP	PA	1/6/2006
EPA 8260	sec-Butylbenzene	NELAP	PA	1/6/2006
EPA 8260	tert-Butyl alcohol (2-Methyl-2-propanol)	NELAP	PA	2/8/2008
EPA 8260	tert-Butylbenzene	NELAP	PA	11/7/2005
EPA 8260	trans-1,2-Dichloroethene	NELAP	PA	12/7/2004
EPA 8260	trans-1,3-Dichloropropene	NELAP	PA	12/7/2004
EPA 8260-Extended	Methyl isobutyl ketone (MIBK, 4-Methyl-2-pentanone)	NELAP	PA	11/7/2005
EPA 9040B	pH	NELAP	PA	3/20/2009
EPA 9045	pH	NELAP	PA	3/20/2009
Microseeps SOP-AM 4 02	Total petroleum hydrocarbons (TPH)	NELAP	PA	3/20/2009
Microseeps SOP-AM 4 02	n-Decane	NELAP	PA	3/20/2009
Microseeps SOP-AM 4 02	n-Dodecane	NELAP	PA	3/20/2009
Microseeps SOP-AM 4 02	n-Hexadecane	NELAP	PA	3/20/2009
Microseeps SOP-AM 4 02	1,1,1-Trichloroethane	NELAP	PA	3/20/2009
Microseeps SOP-AM 4 02	1,1,2,2-Tetrachloroethane	NELAP	PA	3/20/2009
Microseeps SOP-AM 4 02	1,1,2-Trichloroethane	NELAP	PA	3/20/2009

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Pittsburgh, PA 15238

Program Solid and Chemical Materials

Method	Analyte	Accreditation Type	Primary	Effective Date
Microseeps SOP-AM 4 02	1,1-Dichloroethane	NELAP	PA	3/20/2009
Microseeps SOP-AM 4 02	1,1-Dichloroethene (1,1-Dichloroethylene)	NELAP	PA	3/20/2009
Microseeps SOP-AM 4 02	1,2,3-Trichlorobenzene	NELAP	PA	3/20/2009
Microseeps SOP-AM 4 02	1,2,4-Trichlorobenzene	NELAP	PA	3/20/2009
Microseeps SOP-AM 4 02	1,2,4-Trimethylbenzene	NELAP	PA	3/20/2009
Microseeps SOP-AM 4 02	1,2-Dichlorobenzene (o-Dichlorobenzene)	NELAP	PA	3/20/2009
Microseeps SOP-AM 4 02	1,2-Dichloroethane	NELAP	PA	3/20/2009
Microseeps SOP-AM 4 02	1,2-Dichloropropane	NELAP	PA	3/20/2009
Microseeps SOP-AM 4 02	1,3,5-Trimethylbenzene	NELAP	PA	3/20/2009
Microseeps SOP-AM 4 02	1,3-Dichlorobenzene (m-Dichlorobenzene)	NELAP	PA	3/20/2009
Microseeps SOP-AM 4 02	1,4-Dichlorobenzene (p-Dichlorobenzene)	NELAP	PA	3/20/2009
Microseeps SOP-AM 4 02	2-Butanone (Methyl ethyl ketone, MEK)	NELAP	PA	3/20/2009
Microseeps SOP-AM 4 02	2-Chloroethyl vinyl ether	NELAP	PA	3/20/2009
Microseeps SOP-AM 4 02	2-Hexanone	NELAP	PA	3/20/2009
Microseeps SOP-AM 4 02	2-Propanol (Isopropyl alcohol)	NELAP	PA	3/20/2009
Microseeps SOP-AM 4 02	4-Methyl-2-pentanone (MIBK)	NELAP	PA	3/20/2009
Microseeps SOP-AM 4 02	Acetone	NELAP	PA	3/20/2009
Microseeps SOP-AM 4 02	Benzene	NELAP	PA	3/20/2009
Microseeps SOP-AM 4 02	Bromodichloromethane	NELAP	PA	3/20/2009
Microseeps SOP-AM 4 02	Bromoform	NELAP	PA	3/20/2009
Microseeps SOP-AM 4 02	Carbon tetrachloride	NELAP	PA	3/20/2009
Microseeps SOP-AM 4 02	Chlorobenzene	NELAP	PA	3/20/2009
Microseeps SOP-AM 4 02	Chloroethane	NELAP	PA	3/20/2009
Microseeps SOP-AM 4 02	Chloroform	NELAP	PA	3/20/2009
Microseeps SOP-AM 4 02	Dibromochloromethane	NELAP	PA	3/20/2009
Microseeps SOP-AM 4 02	Dichlorodifluoromethane (Freon 12)	NELAP	PA	3/20/2009
Microseeps SOP-AM 4 02	Ethylbenzene	NELAP	PA	3/20/2009
Microseeps SOP-AM 4 02	Isopropylbenzene	NELAP	PA	3/20/2009
Microseeps SOP-AM 4 02	Methyl bromide (Bromomethane)	NELAP	PA	3/20/2009
Microseeps SOP-AM 4 02	Methyl chloride (Chloromethane)	NELAP	PA	3/20/2009
Microseeps SOP-AM 4 02	Methyl tert-butyl ether (MTBE)	NELAP	PA	3/20/2009
Microseeps SOP-AM 4 02	Methylene chloride (Dichloromethane)	NELAP	PA	3/20/2009
Microseeps SOP-AM 4 02	Naphthalene	NELAP	PA	3/20/2009
Microseeps SOP-AM 4 02	Styrene	NELAP	PA	3/20/2009
Microseeps SOP-AM 4 02	Tetrachloroethene (PCE, Perchloroethylene)	NELAP	PA	3/20/2009
Microseeps SOP-AM 4 02	Toluene	NELAP	PA	3/20/2009

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Microseeps Inc
220 William Pitt Way
Pittsburgh, PA 15238

Program Solid and Chemical Materials

Method	Analyte	Accreditation Type	Primary	Effective Date
Microseeps SOP-AM 4 02	Trichloroethene (TCE, Trichloroethylene)	NELAP	PA	3/20/2009
Microseeps SOP-AM 4 02	Trichlorofluoromethane (Freon 11)	NELAP	PA	3/20/2009
Microseeps SOP-AM 4 02	Vinyl chloride (Chloroethene)	NELAP	PA	3/20/2009
Microseeps SOP-AM 4 02	Xylenes, total	NELAP	PA	3/20/2009
Microseeps SOP-AM 4 02	cis-1,2-Dichloroethene	NELAP	PA	3/20/2009
Microseeps SOP-AM 4 02	cis-1,3-Dichloropropene	NELAP	PA	3/20/2009
Microseeps SOP-AM 4 02	n-Heptane	NELAP	PA	3/20/2009
Microseeps SOP-AM 4 02	n-Hexane	NELAP	PA	3/20/2009
Microseeps SOP-AM 4 02	trans-1,2-Dichloroethene	NELAP	PA	3/20/2009
Microseeps SOP-AM 4 02	trans-1,3-Dichloropropene	NELAP	PA	3/20/2009
Microseeps SOP-AM23G	Acetic acid (Ethanoic acid)	NELAP	PA	1/6/2006
Microseeps SOP-AM23G	Hydroxy-isobutyric acid (HIBA)	NELAP	PA	1/6/2006
Microseeps SOP-AM23G	Isohexanoic acid	NELAP	PA	1/6/2006
Microseeps SOP-AM23G	Lactic acid	NELAP	PA	1/6/2006
Microseeps SOP-AM23G	n-Hexanoic acid	NELAP	PA	1/6/2006
Microseeps SOP-AM23G	Butyric acid	NELAP	PA	1/6/2006
Microseeps SOP-AM23G	Isopentanoic acid	NELAP	PA	1/6/2006
Microseeps SOP-AM23G	Propionic acid	NELAP	PA	1/6/2006
Microseeps SOP-AM23G	Pyruvic acid	NELAP	PA	1/6/2006
Microseeps SOP-AM23G	n-Pentanoic acid	NELAP	PA	1/6/2006

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www.dep.state.pa.us **Issue Date: 11/15/2010**

MICROSEEPS, INC.
220 William Pitt Way
Pittsburgh, PA 15238
412-826-5245

QUALITY SYSTEMS MANUAL

Controlled Copy No. _____

Signatures of Final Approval:

Ruth Welsh — 7-29-10
Ruth Welsh — Date
Laboratory Manager

Patrick McLoughlin — 7-29-10
Patrick McLoughlin Ph.D. — Effective Date
Quality Systems Manager

Organizational Units

Customer Service

Risk

Wet Chemistry

Volatiles

CSIA

Semi-Volatiles

Sales & Marketing

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1.0 Introduction, Purpose, and Scope

1.1 Introduction

Microseeps, Inc. (Microseeps) recognizes its crucial role in providing reliability and excellence in the environmental analytical industry. The laboratory provides information necessary for engineering, industrial, and regulatory clients to make informed judgments and applicable policy decisions. The laboratory's analytical services also assist clients in complying with major environmental regulations. Microseeps' management acknowledges that uncompromising dedication to quality is fundamental to remaining a competitive force in the analytical services market.

1.2 Purpose

The purpose of this Quality Systems Manual is to outline a program of policies, procedures, and documentation, which assures that our analytical services meet a defined standard of quality on an ongoing basis. This document defines the standards under which all laboratory operations will be performed. As supplemented by Standard Operating Procedures (SOPs), the Quality Systems Manual describes the laboratory's organization, objectives, and operating philosophy.

Microseeps Quality Systems Manual contains references to the laboratory's policies and operational procedures that have been established in order to meet the quality requirements of the NELAC Standards and the following Quality Policy Statement.

1.2.1 Microseeps' Quality Policy Statement

Microseeps is an organization that provides information and environmental services to a very diverse client base. In all processes from initial customer contact to project completion, Microseeps aim is to offer clients a reliable product of the best quality that is delivered in a timely manner at a reasonable price. This Policy Statement represents a top level management commitment and shall be achieved using the following goals:

1. Production of accurate information.
2. Adherence to an ethical standard that demands continuous honesty and integrity.
3. Achievement of customer satisfaction.
4. Establishment of a documented trail to support the results.
5. Maintenance of a pleasant working environment where employees are treated equitably and fairly.

These goals are fundamental to all of Microseeps' actions.

The Standard Operating Procedures and Microseeps Quality Systems Manual detail the procedures for achieving these goals. Management personnel are charged with ensuring all applicable procedures are completed in the spirit of the Quality Policy. They are available to all employees to assist in applying these principles and to advise appropriate action. It is the responsibility of each Microseeps employee to consistently act as directed by the five goals of quality. Further, it is their responsibility to consult their immediate superior for direction if an issue arises for which the response is unclear.

If Microseeps' Director Level Management realizes that these goals are not achievable but the validity and appropriateness of client results is not jeopardized, we will inform affected clients of the situation and allow them to alter their employ of Microseeps' services accordingly. If the client's data are to be negatively impacted where the validity of the results is in question, or a clients' project is operationally or legally jeopardized, the following procedures shall be followed:

1. Concerned operations will be suspended.

2. Affected clients will be notified.

3. Alternative procedures for securing the requested services will be employed until the corrective actions are completed to a level that is consistent with Microseeps' documented procedures and is satisfactory to management.

To achieve these quality goals, all laboratory data must be properly documented, legally defensible, and supported by statistically defined and verifiable confidence limits. Falsification of data under any circumstance is unacceptable and is grounds for termination. Microseeps has an Employee Handbook which provides policies and procedures to help employees avoid involvement in any activities that would diminish confidence in Microseeps competence, impartiality, judgment and/or operational integrity.

Microseeps uses EPA-approved methodologies such as those found in Standard Methods and SW-846, whenever methods are available. If an EPA-approved method has not been specified, Microseeps will select an industry recognized and validated method for use or will develop an internal method based upon thorough research and good scientific methods. In all instances of scientific innovation, Microseeps recognizes the value of a firm commitment to quality and integrity.

1.3 Scope of Quality Systems Manual

This document serves as both the Microseeps, Inc. Quality Systems Manual and the Microseeps Laboratory Quality Assurance Plan. It contains both quality assurance policies and quality control procedures that are followed to ensure and document the quality of analytical data. This manual provides detail concerning quality management requirements employed at Microseeps

for the documented acquisition of samples, analysis of those samples via specific tests, the reporting of that data to the client, and the ultimate disposal of samples.

1.4 Scope of Services

Microseeps offers a comprehensive scope of laboratory analytical services to environmental consultants, industries, governmental agencies, and municipalities. The scope of services include:

Wastewater, storm water, solid waste, and hazardous waste analyses

- Volatile analyses via GC/MS
- Ion analysis via Ion Chromatography
- Wet Chemistry analyses for pH, alkalinity, TOC, and sulfide.

Intrinsic Bioremediation Analysis

Training Seminars

Soil Vapor Extraction Analysis

1.5 Certifications

Microseeps holds the following certifications:

- National Environmental Laboratory Accreditation Program (NELAP): Pennsylvania
- Florida
- Connecticut
- West Virginia
- South Carolina
- Louisiana
- Texas
- New York
- New Jersey
- Kentucky

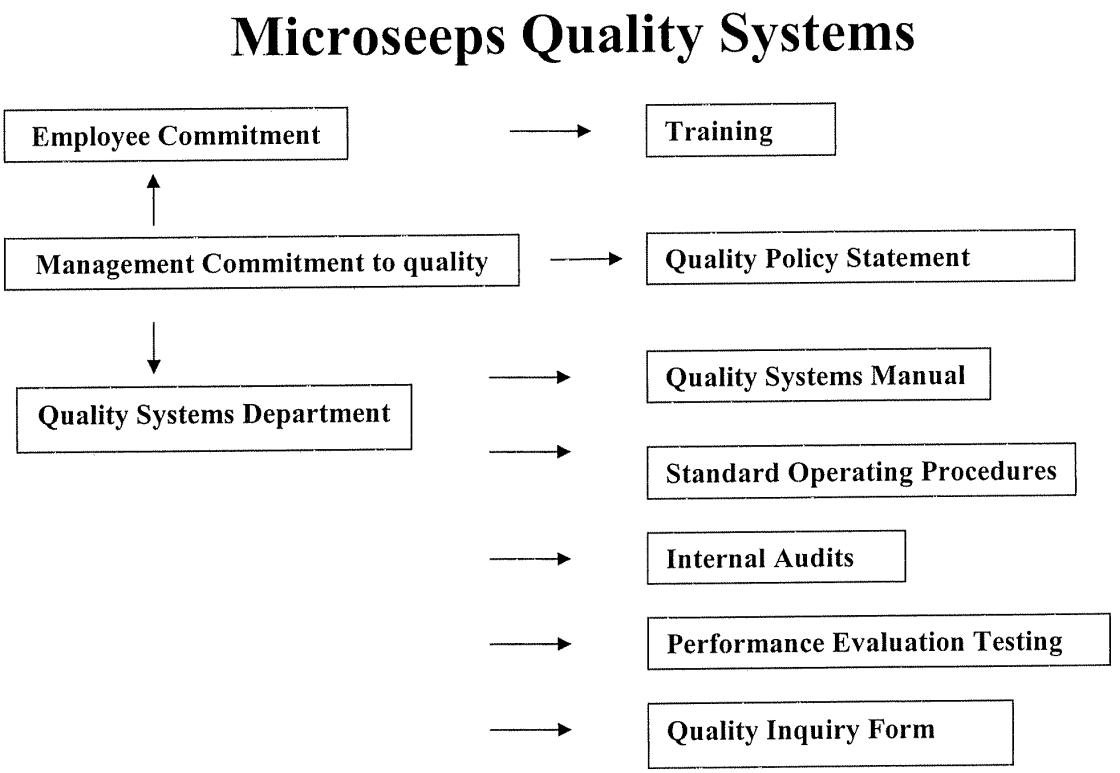
Specific parameter lists for the various certifications are available from the Customer Service Department upon request.

2.0 Quality Systems – Establishment and Audits

2.1 Establishment of a Quality System

Microseeps has established a quality system based upon the fundamentals of good laboratory practices and the requirements outlined in Chapter Five of the National Environmental Laboratory Accreditation Program. Although Microseeps provides a variety of environmental services, Microseeps' Quality System has been designed according to the type, range, and volume of analytical testing activities undertaken in the laboratories. This section describes Microseeps' Quality System and outlines the policy and procedures for implementing corrective action when non-conforming work or departures from policies and procedures occur.

2.2 Elements of Microseeps Quality System



This flowchart shows the general elements of Microseeps Quality Systems. Each area of this flowchart is addressed or referenced in this Quality Systems Manual.

2.2.1 Quality Manual Review

The Microseeps Quality Systems Manual is reviewed annually for accuracy and applicability. These records are maintained in the Quality Systems Coordinator's Office. All revisions will be conducted in accordance with Microseeps Standard Operating Procedure for Document Control.

2.2.1.1 Document Control

Document control procedures are specified in Microseeps Standard Operating Procedure for Document Control SOP-ADM 5. The Standard Operating Procedure outlines document control procedures for generating, formatting, revising, approving, tracking, distributing, indexing, archiving, and destroying controlled documents, including the Quality Systems Manual.

2.2.2 Internal Audits

Technical audits serve to verify compliance with method-specific procedures including operations related to test methods. Any audit that is conducted on an operation that is involved with data generation and the assurance of its quality is a technical audit. System audits function to verify compliance with the laboratory's quality system. Types of procedures that would be reviewed as a part of a systems audit could include: (1) response to complaints; (2) sample tracking methodologies; and (3) sample acceptance policies.

The Quality Systems Department will conduct all technical and system audits. Audits shall be conducted by individuals who are independent of the activity to be audited. All internal auditors shall be trained and qualified in the areas in which they will be conducting the audit.

2.2.2.1 Audit Frequency

Internal Audits are scheduled and conducted by the Quality Systems Department and may either be scheduled or unannounced. Microseeps conducts internal technical audits and internal system audits at least monthly. The audits are conducted to insure that Microseeps' operations continue to comply with the Quality Systems requirements specified in the Quality Systems Manual and Standard Operating Procedures. The Quality Systems Coordinator maintains an audit schedule to ensure that all laboratory groups and programs are audited.

2.2.2.2 Procedures for Internal Audits

Audits will be conducted on analytical groups or systems according to the annual audit schedule posted in the Quality Systems Coordinator's Office. Immediately prior to starting the audit Operations will be notified. Microseeps' Standard Operating Procedure ADM-4 for Internal Laboratory Audits outlines the specific procedures for conducting an internal audit.

According to the SOP, the audit is performed using the appropriate audit check sheet as a guide. The audit check sheet will be selected and used as the auditing guide for the area that is to be

audited. The audit check sheets delineate the activities and records that will be reviewed. The sheet is completed and additional notes are made by the auditor based upon observations, interviews, and record reviews.

2.2.2.3 Internal Audit Findings and Corrective Action

Using the audit check sheets and supplementary notes, an Internal Audit Report (Figure 2-1) is prepared by the Quality Systems Coordinator. The Internal Audit Report contains an overall summary of the audit, including both items of a positive nature, as well as deficiencies. The cause of each deficiency is determined using Root Cause Analysis. Root Cause Analysis is the foundation upon which all Corrective Action is based. This process and an example are outlined in Figure 2-2 in this Quality Systems Manual.

Corrective Action based upon the root cause analysis is indicated on a Corrective Action Report (see Figure 2-3). A Corrective Action Report is prepared for each deficiency listed on the Internal Audit Report. The Internal Audit Report and Corrective Action Report are forwarded to the appropriate Department Head for corrective action. A follow-up audit is conducted.

If the audit was of a technical nature, the Audit Report will be forwarded to the Laboratory Director. The Laboratory Director will meet with the specific Department Head the first business day following the audit. The audit will be discussed along with the recommendations for corrective action. Corrective action is expected to take place immediately or as soon as possible following the audit. A follow-up audit of any deficient area(s) will be conducted within one week of audit completion, or as soon as corrective action is completed in order to monitor the effectiveness of corrective action.

Where any audit findings or defective measuring or test equipment may cast doubt on the correctness or validity of the laboratory's calibrations or test results, Microseeps shall take immediate corrective action as specified in Microseeps' Quality Policy Statement. If the subsequent investigation shows that laboratory results have been affected, the affected client shall be notified in writing by the Customer Service Office.

2.2.2.4 Work Stoppage

The Quality Systems Department has the authority to stop any work that is found to be unsatisfactory or to prevent reporting of unjustifiable results. The work will not be ordered to commence until corrective action is taken that insures satisfactory work and reliable results according to the Quality Systems Department's discretion.

2.2.3 Performance Evaluation Audits

Performance evaluation audits enable Microseeps to measure the precision, accuracy, and comparability of laboratory-generated data through the use of blind reference materials. Microseeps participates in performance evaluation studies required by the EPA and several state

agencies. Performance evaluation studies are completed as required to maintain necessary certifications, and as required as an integral part of an internal audit at the Quality Systems Department's discretion.

Performance evaluation studies for all accredited parameters are planned for testing at least twice each year. The Quality Systems office maintains EXCEL spreadsheets of results so that trends are easily noticeable.

In situations where the analyst is to know that a performance evaluation is being conducted, the analyst prepares the samples according to the instructions provided. If the PE is to be a blind audit, the Quality Systems Department prepares the samples. The samples are analyzed as soon as possible after opening the vials to avoid sample deterioration. Prior to reporting the results to the appropriate agency, the Laboratory Director and the Quality Systems Manager evaluate results of the performance evaluation samples.

2.2.3.1 Performance Evaluation Findings and Corrective Action

Once the evaluation report from the Performance Evaluation Provider or appropriate agency is received, the Quality Systems Coordinator forwards the findings to the Laboratory Director. Unacceptable results are investigated to determine the root cause of the failures. The following points are addressed during the investigation:

- Potential for reporting/calculation errors
- Preparation of calibration standards
- Evaluation of quality control data associated with the analysis
- Evaluation of analytical technique and instrument performance

Additional Performance Evaluation samples may be submitted for analysis if the Quality Systems Department determines it is necessary to ensure that an analytical method, technique, or instrument performance problem is corrected.

2.2.4 External Audits

External Audits are conducted as necessary to retain laboratory certifications or at a client's request. The Quality Systems Department is the liaison between Microseeps and an external auditor. The Quality Systems Department is responsible for notifying the laboratory staff of upcoming audits. This notification will include the agency that will perform the audit, the reason for the audit, the dates involved, and the areas of concern.

During the audit, laboratory personnel will be available to the auditor as requested, as will any documentation necessary for the auditor to obtain sufficient information to effectively evaluate the laboratory. If laboratory information of a proprietary nature is necessary to complete an audit, the auditor will be required to complete and sign Microseeps Confidentiality Agreement.

2.2.4.1 External Audit Findings and Corrective Action

The Quality Systems Department will prepare a report summarizing the findings of the post audit meeting. Following review of this report, any deficiencies noted by the auditor that affect the validity of data in any area will be addressed immediately. Corrective action will be taken to eliminate the cause of a deficiency and to prevent recurrence. The Corrective Action process shall identify and implement corrective actions to eliminate the root cause of the deficiency. The development and implementation of a corrective action plan shall not be contingent upon the receipt of the external auditor's report.

When the External Audit Report is received at Microseeps, deficiencies will be addressed in the order of their priority as determined by the auditor. The Quality Systems Department will prepare a response plan for correcting the reported deficiencies and will submit a Corrective Action Plan to the auditing agency. Corrective action will be taken according to the timetable outlined by the plan.

2.2.5 Managerial Review

Laboratory Management shall conduct a review of the Quality System and its testing and calibration activities. This review shall be conducted to ensure the Quality System's suitability and effectiveness and to introduce any necessary changes or improvements in the quality system and laboratory operations. This review shall be conducted annually by department level management or higher.

The review will be conducted in the same manner as an Internal Systems Audit, but the auditors will be made up of department level management. Microseeps' Standard Operating Procedure ADM-4 includes an Audit Check Sheet for the Managerial Review Process, which outlines the review procedure.

2.2.5.1 Managerial Review Findings and Corrective Action

The review findings shall be documented on an Audit Report Form and submitted to the Quality Systems Department for review. Corrective Action Reports will be generated for the audit deficiencies and will be resolved as necessary. A timeline for resolution is specified on the Corrective Action Report and the review findings become an item for discussion at the Board of Directors meeting where management ensures the corrective actions are completed.

2.2.5.2 Board of Directors Meetings

Regular discussions of quality assurance issues are necessary to provide a forum in which upper management are informed of problems and changes that affect the laboratory operations. The Quality Systems Department regularly provides a report to the President, Senior Vice President,

Director of Operations, and Director of Sales to discuss quality issues. These meetings serve as a general review of factors affecting quality and will include the following topics at a minimum:

- Personnel changes
- Instrument changes
- Internal and External Audit findings
- Certification changes
- Testing and calibration activities
- Quality System implementation activities and progress

2.2.5.3 Documentation

At the end of the meeting, the Technical Director will document the results of the meeting, prepare a summary of the discussion, and set an agenda for additional follow-up, if required. This report shall serve as documentation of management commitment to the Quality System. A report of each meeting will be kept on file in the Quality System Coordinator's Office.

2.3 Additional Quality Control Checks

In addition to periodic audits, Microseeps ensures the quality of results provided to clients by implementing additional checks to monitor the quality of the laboratory's analytical activities. Some of these checks are as follows:

- Use of certified standards in many of our quality control samples
- Replicate testing using the same and different test methods
- Correlation of results for different but related analysis of a sample
- Participation in Proficiency Testing

2.4 Audit and Review Documentation

All audit records shall be kept on file in the Quality Systems Coordinator's Office. These records shall be available for external auditors, as well as, individuals involved in a Managerial Review of the Microseeps Quality System. This documentation will include:

- Audit Check Sheets
- Audit Report Forms
- Corrective Action Reports

2.5 Quality Inquiry Forms

Microseeps has instituted a procedure for reporting when departures from documented policies, procedures, and quality control have occurred. The Quality Inquiry Form QIF (see Figure 2-4) is a means for anyone in the company to communicate quality concerns to the Technical

Department. These forms are a unique part of Microseeps' Quality System, which provide an avenue for all employees to reflect a genuine concern for quality throughout the company. The QIF is also used to document Customer Complaints and initiate the process of resolving those complaints. All items pertaining to the Customer complaint shall be recorded on the QIF including investigation results and resolution to the satisfaction of the client. All QIFs that deal with Customer Complaints shall be kept on file in the Customer Service Office.

Note: If the quality concern can be corrected by the person who discovered the quality problem, a CAR should be completed in lieu of a QIF.

2.6 Corrective Action

An integral part of Microseeps' Quality Systems Program is the system for identifying, reporting, and correcting deficiencies in the laboratory operation. There are several areas in the laboratory that may require corrective action. It is the responsibility of every employee to be aware of potential problems and to notify the appropriate personnel of situations requiring corrective action.

2.6.1 Problem Isolation and Identification

Identification and isolation of problems in the laboratory are not always easy tasks. The need to perform corrective action may become apparent at any point of the analytical process. Corrective action should be initiated and documented as soon as a problem becomes evident. The analyst at the bench detects some situations such as malfunctioning equipment. Corrective action for these situations takes the form of repairing the instrument, either internally or through the use of a service call. The corrective action is documented in the instrument maintenance log and the data obtained just prior to the failure is closely scrutinized for acceptability.

Other situations may not be easily identifiable. For example, systematic drift or sensitivity fluctuations may not be identified until the time that data is validated. Other occurrences that may trigger the need for corrective action include the following:

- Recoveries for surrogates, matrix spike, matrix spike duplicates, and laboratory control standards outside of acceptance limits.
- Percent differences for duplicate analyses outside acceptance limits.
- Trends noted in quality control data.

Out of control events that concern sample analysis and data generation must be documented in a Case Narrative, which becomes a permanent part of the client's project file and final data report.

2.6.2 Sample Handling Problems

Problems involving sample handling may include missing or broken containers, discrepancies between the chain of custody and actual shipment, improperly preserved bottles, insufficient volume, and missed holding times. When one of these problems is identified, a Non-Conformance

Form is completed and acted upon in accordance with the Standard Operating Procedure for Sample Receiving (SOP-S2). If necessary, the client is contacted to discuss possible resolutions to the problem. The Non-Conformance Form becomes a part of the client's permanent file.

2.6.3 Sample Analysis Problems

Problems incurred during sample analysis may bring procedures and data into question. These problems may include the following:

- Unacceptable calibration
- Improper procedures
- Unacceptable blank, LCS, and/or surrogate recovery
- Quantitation error
- Required QC not performed
- Retention time shifts

Resolving these problems may include preparing and analyzing the samples a second time, re-calibrating the instrument, or making new standard solutions and reagents. Standard Operating procedures detail corrective action steps that are specific to an analytical procedure. The documentation becomes part of the client's permanent file.

The person identifying the problem documents the situation and identifies possible sources of the problem. If this individual can immediately correct the situation, for example, by re-calibrating the instrument, they will do so and document the action that was taken in the case narrative. If the problem cannot be corrected immediately, the person documents the situation and notifies the Laboratory Director. The Laboratory Director is responsible for ensuring that the appropriate corrective actions are followed.

2.6.4 Corrective Action Reports

A Corrective Action Report (CAR) will be completed for each audit deficiency, legitimate issue reported on a Quality Inquiry Form, and any systemic out of control event that occurs. The Quality Systems Coordinator is responsible for maintaining a supply of forms. To track the corrective action, each CAR will be given a unique identifier in the form of YY-XXXX where:

- YY = the year in which the CAR was initiated and
- XXXX = is the sequential number of the record starting with 0001

2.6.4.1 Corrective Action Report Initiation and Procedures

A Corrective Action Report may be generated by anyone in the company who discovers and can correct a systemic out of control event. There are two types of situations in which an employee

will generate a CAR. First, when the root cause is obvious to the person who discovered the out of control event, and secondly when the root cause is not obvious.

In the first situation, the person who discovers an out of control event shall institute and document corrective action by completing the top portion of the CAR (above the dotted line). They will enter their name, the current date, a description of the event that needs corrective action, and the specific corrective action that was taken. The Lead Analyst or Department supervisor shall be notified of the event and the corrective action necessary to solve the non-conformance. The person who completes the corrective action shall sign the form and forward it to their immediate supervisor for a signature. The form will then be forwarded to the Quality Systems Department for cataloging. If additional action is required, the form shall be returned to the department concerned and returned to the Quality Department when corrective action is complete.

If the root cause is not immediately obvious, the Quality Systems Department shall be notified immediately to assist in Root Cause Analysis and determining the appropriate corrective action.

The Quality Systems Department will forward a copy of the CAR to the Laboratory Director. If it is determined that the client needs to be notified of the incident, the Quality Systems Department shall forward a copy of the CAR to the Customer Service Department for further action.

2.6.4.2 External Audits and Performance Evaluation Studies

The Quality Systems Department will initiate CAR(s) in response to third party audits and deficiencies on performance evaluation studies. In the case of third party audits, if the resolution to the finding is easily identifiable, the Quality Systems Department will discuss the situation with the Laboratory Director and the corrective action will be implemented. If the finding concerns a procedural change or is interdisciplinary, the Quality Systems Department will form a task team to investigate the problem and develop an appropriate course of action. The task team's findings will be presented to the Quality Systems Department for discussion and possible implementation. Once a corrective action plan is implemented, the situation will be monitored for a reasonable period of time to ensure that the action has been effective.

The Quality Systems Department will prepare a report for management that summarizes all corrective actions (See Figure 2-5). The report will provide a brief description of the problem, the steps that are being taken to correct the situation, and the status of the item.

2.7 Preventive Actions

The preferred course of laboratory quality and improvement is to identify opportunities for improvement rather than react to the occurrence of problems or complaints. Microseeps is continually seeking ways to improve its performance and product. When these areas are identified, a plan is developed by the staff, usually including the Quality Systems Department, Laboratory Manager, or Manager of Technical Systems. Preventative actions are implemented according to the time table specified in the plan and monitored at weekly Operations Meetings. Preventive

action procedures include follow-up actions and applications of controls in order to ensure effectiveness.

2.8 Management Arrangements for Permitting Departures from Documented Procedures or Standard Specifications

It is Microseeps management's intent to ensure that documented procedures are followed. Rarely, a situation may occur that requires a departure from documented quality procedures. When this type of situation occurs, the Quality Systems Department and Laboratory Director, and any other manager whose department may be affected, will discuss and unanimously agree upon the action to be taken. The departure will be documented in memo form and kept on file in the Quality System Coordinator's Office. Corrective action will be taken as soon as possible to prevent the necessity of the departure from reoccurring.

Figure 2-1

Internal Audit Report

Audit Date: _____ **Department:** _____

Auditors: _____

Lead Analyst/Supervisor: _____

Employees Present: _____

Audit Summary:

Areas of Excellence:

Deficiencies:

Suggestions:

Technical Director Signature: _____ **Date:** _____

Figure 2-2

Root Cause Analysis

Deficiencies are always analyzed from the non-conformance back to the root cause as follows:

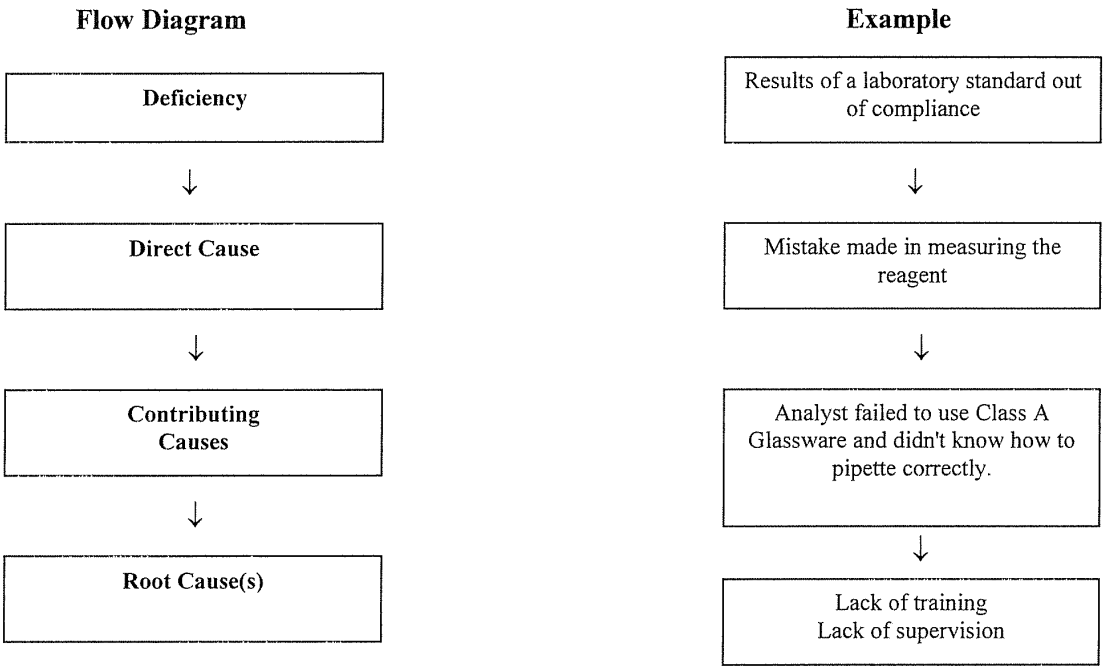


Figure 2-3

Corrective Action Report

This form is to be initiated by the person who discovers an event that needs corrective action. Please complete only the portion of the form above the dotted line. When complete, forward to the Quality Systems Department.

Initiated by: _____ **Date:** _____
Print Name

Description of the event needing corrective action and corrective action required.

Root Cause: _____

This Corrective Action must be acted upon in the time frame indicated below:

Immediately ___ **One Week** ___ **Two Weeks** ___ **Other** _____
Est. Date

Corrective Action Taken: **Due Date:**

Action Completed by: _____ **Date:** _____

Supervisor Review: _____ **Date:** _____

Project #: _____ **Sample Numbers:** _____

Additional Action: is___ is not ___ necessary. Customer Contact: is___ is not ___ necessary.

Action Closed: _____ **Date:** _____

cc: Laboratory Director, Technical Director **CAR #** _____

Figure 2-4

Quality Inquiry Form

This form is to be used as a means to communicate quality issues within the company and as a means of documenting customer complaints. The first resource to resolving any internal concern is direct communication. This form is provided as a means to pass inquiries to the Technical Department when direct communication is inappropriate or ineffective. Please fill out as completely as possible and forward to the Technical Director.

Date of inquiry _____

Concerned Department:	Customer Service	Organics	Bottle Preparation	Receiving
	Wet Chemistry	Risk Waters	Risk Vapors	Quality Systems
	General	Management		

Please categorize the issue (Please circle one of the following numbered items):

Procedure related

1) Failure to follow SOP
2) Improper SOP
3) No SOP known

Record keeping related

4) Items not or improperly recorded
5) Records improperly stored
6) Records improperly handled (lost, insecure, removed from building)

Client Communication related

7) Client was knowingly promised the unachievable
8) Client's request was disregarded
9) Information was inappropriately kept from client
10) Report was incomplete or inaccurate
11) Other client complaint (describe below)

Internal Communication related

12) Internal chain of command (as per organizational chart) not followed
13) Client's request not passed to appropriate personnel
14) Supervisor was unresponsive
15) Redundant efforts were caused by poor communication among analysts

Please describe the issue and means taken to resolve (use other side if necessary):

_____

_____

_____

_____

Figure 2-5

Quality Systems Update and
Corrective Action Summary Report
To Management

1. **Brief Description of audit deficiencies and quality inquiry forms:**

CAR #	Date	Deficiency	Resolution	Update

2. **Steps being taken to remedy the deficiency:** (Obtain information from other Management Team members who have unresolved CAR's and update them on status of Technical Directors'.)

3. **Other items for discussion as they pertain to quality:**

- **Personnel changes**
- **Instrument changes**
- **Upcoming Audits**
- **Certifications**
- **Quality System implementation activities and progress**

file:C\My Documents\Forms\Corrective Action Summary Report

3.0 Organization and Management

Microseeps is a small business as defined by Standard Industrial Classification 8734. Since Microseeps is a business that must compete in a marketplace characterized by merger and consolidation, it is even more important to have an efficient organization with well-defined responsibilities. The purpose of this section is to describe the organization and management structure of Microseeps, interdepartmental and intradepartmental relationships, and to detail employee responsibilities and qualifications.

3.1 Microseeps Organization

Organizations must have a framework within which to achieve their objectives. Organizational structure determines the configuration of positions, duties, and channels through which performance is controlled and authority is delegated. It is crucial that a company use a structure that enables it to realize competitive advantages.

In order for Microseeps to achieve its stated objectives for the quality of its analytical services, all employees must be able to function within a structure that provides an emphasis on quality from a technical standpoint. A functional organizational structure is specifically suited to this purpose.

3.1.1 Organizational Structure

Microseeps' organizational structure is functional. This type of structure is centralized, which restricts decision-making authority to higher levels of management. Company organization is departmental and is specialized and arranged according to function. Because of the nature of the individual departments, some authority is delegated to individuals who lead smaller groups within the larger departments.

The Quality Systems Department reports directly to the President and CEO. Sales, Finance, and other administrative departments are overseen by the Board of Directors. Microseeps' Organizational Chart is depicted in Figure 3-1.

3.1.2 Interdepartmental Relationships

Responsibility and dedication to quality laboratory practices and procedures begin at the highest level of management. It is the duty of the President to assure that the framework is in place to provide quality systems guidelines. It is the duty of Microseeps managers to implement the policies and procedures and to see that they drive the activities of the laboratory.

3.1.2.1 Operations Department

The Vice President of Operations oversees the following departments: 1) Operations; 2) Technical Services; 3) Customer Service; and 4) Information Technology. The Laboratory Manager oversees the analytical work groups in the laboratory. The Manager of Customer Service oversees all of the

Customer Service functions. The Manager of Technical Services is responsible for equipment maintenance and regulatory method development.

3.1.2.2 Quality Systems Department

The Quality Systems Department operates outside of the scope of the Operations Department in lines of authority. Responsibility for the Quality Systems Department falls upon the Quality Systems Manager who reports directly to the President and CEO. The Quality Systems Manager is responsible for the following work:

1) Quality Systems

The Quality Systems Coordinator is administratively responsible for quality assurance and quality control, and may serve as the Quality Systems Manager's representative in his absence.

3.1.3 Intradepartmental Relationships

The Quality Systems department has a quality assurance and quality control role to fulfill within each department at Microseeps. Discrepancies in quality in any department will be relayed from the Quality Systems Manager to the appropriate Department Head using a format that is appropriate for the type of discrepancy and recommending an appropriate corrective action. This may take the form of using an electronic "Help Desk" forum or using a Corrective Action Form.

3.2 Personnel Qualifications

All of Microseeps employees are responsible for complying with the applicable job specific quality assurance and quality control requirements. All staff are to familiarize themselves with the quality documentation and implement the policies and procedures contained in this Manual in their work. Each staff member, including contracted and additional technical and key support personnel should they be required, must demonstrate a combination of formal education and experience to satisfactorily perform their particular function, as well as, a general knowledge of laboratory operation, quality assurance, quality control, test methods, and records management. All personnel are placed in a work group or department with adequate supervision that ensures the employee works in accordance with the laboratory's Quality System. Documentation of employee proficiency for specific job functions and test methods is maintained in the Quality Systems Coordinator's Office.

The Laboratory Manager, based upon specific educational and experience requirements, makes laboratory job assignments. Laboratory analysts are assigned a job classification according to the level of formal education and related laboratory experience they possess.

Basic duties of key staff (those included in Figure 3-1) are discussed below. Complete job descriptions for key staff are contained in Appendix A of this Quality Systems Manual. Complete

documentation of all employee job descriptions is kept on file in Employee Personnel Files in the Senior Vice President's Office.

3.2.1 President and Chief Executive Officer

The President and Chief Executive Officer is responsible for overall company performance. He is responsible for new business development and the financial integrity of the company. The President and Chief Executive Officer represents Microseeps at trade shows and environmental conferences.

3.2.2 Senior Vice President

The Senior Vice President works closely with the President to provide overall direction for the laboratory operation. He is responsible for identifying potential new markets and shares the responsibility for the financial integrity of the laboratory with the President. Additionally, he approves capital expenditures and evaluates current market conditions to maintain the laboratory's competitiveness.

3.2.3 Vice President of Sales and Marketing

The Vice President of Sales and Marketing is responsible for meeting target sales goals, overseeing sales staff, and assists the President and Senior Vice President in developing sales and marketing strategies.

3.2.4 Vice President of Operations

The Vice President of Operations is responsible for implementing and enforcing laboratory policies and procedures. The Vice President of Operations delegates responsibility to the Department Heads, is responsible for the daily operation of each department of the laboratory, except Quality Systems, and is responsible for overseeing the routine expenditures and for maintaining the laboratory's budget.

3.2.5 Laboratory Manager

The Laboratory Manager is responsible for ensuring that Microseeps' goals of providing accurate and verifiable analyses are met. It is the Laboratory Manager's responsibility to ensure that all Operation's Department personnel have the required qualifications and training for their positions. Once qualified personnel are in place, the Laboratory Manager, in conjunction with the Quality Department, will be responsible for assuring that all employees are thoroughly familiar with the Quality Systems Manual and accepted laboratory practices. The Laboratory Manager oversees the daily management of the laboratory staff. A major component of this particular responsibility is the integrity of laboratory reports. The Laboratory Manager's qualified designee will review and approve all outgoing reports. The Laboratory Manager reports to the Vice President of Operations.

In the absence of the Laboratory Manager, the Vice President of Operations will assume these responsibilities.

3.2.6 Quality Systems Manager

The Quality Systems Manager is ultimately responsible for ensuring that the data produced by the laboratory are technically sound and of the highest quality possible. The Quality Systems Manager serves as the focal point for quality assurance and quality control and is responsible for the oversight and/or review of quality control data. This position notifies laboratory management of deficiencies in the quality system and monitors corrective action. The Quality Systems Manager reports directly to the Senior Vice President.

3.2.7 Manager of Technical Systems

The Manager of Technical Systems ensures that technical problems within the laboratory are resolved. This position is also responsible for ensuring instrument repairs are conducted in a timely and cost effective manner. The Manager of Technical Systems implements plans for method development and assists clients in data interpretation. The Manager of Technical Systems reports to the Vice President of Operations.

3.2.8 Manager of Customer Service

The Manager of Customer Service provides direction and supervision to ensure that clients receive the best service possible. This position reviews proposal submittals and proposes pricing strategies for potential projects. This position reports to the Vice President of Operations.

3.2.9 Quality Systems Coordinator

The Quality Systems Coordinator assists the Quality Systems Manager to ensure that Microseeps Quality Systems Policies and Procedures are being followed. This is accomplished by: (1) Reviewing data validation procedures; (2) Alerting the analysts should the need for corrective action exist; (3) Performing internal audits; (4) Establishing a periodic schedule for analyzing performance evaluation samples; and (5) Maintaining Quality Control records. The Quality Systems Coordinator reports to the Quality Systems Manager.

3.2.10 Sample Custodian

The Sample Custodian is responsible for properly receiving and logging-in all samples received at Microseeps and ensuring that storage and documentation requirements are met. This position also unpacks and marks the samples with the correct internal laboratory identification number so that the client's samples can be tracked through the laboratory. The Sample Custodian is responsible for documenting all discrepancies between samples received and accompanying chains of custody and for notifying customer service so that the client may be contacted. The Sample Custodian reports to the Laboratory Manager.

3.2.11 Lead Analyst

The Lead Analyst assists in the daily supervision and training of staff analysts and technicians. Lead Analysts ensure that safety policies are followed and that any unsafe conditions are reported to the Company Health and Safety Supervisor. The Lead Analyst performs a broad variety of duties within a specific group or division. The Lead Analyst ensures that analysts strictly adhere to procedures outlined in the Quality Systems Manual and Microseeps Standard Operating Procedures. The Lead Analysts report directly to the Laboratory Manager.

3.2.12 Laboratory Analyst

Laboratory Analysts are responsible for retrieving samples from Sample Receiving at the direction of the Lead Analyst and observing all internal custody requirements. The analysts shall ensure that aliquots analyzed are representative of the entire sample. All analyses shall be conducted according to Microseeps Standard Operating Procedures. Responsibilities also include following good laboratory practices in carrying out duties assigned, and complying with all safety regulations applicable to their respective laboratories. Analysts report directly to their Lead Analyst.

3.2.13 Project Manager/Customer Service Representative

The Project Manager (Customer Service Representative) is responsible for overseeing in-house analytical projects. It is the Project Manager's responsibility to accurately communicate project requirements to the Laboratory Manager so that projects are logged-in, analyzed, and reported in the format, timeframe, and within the project-specific protocols required by the client. This position reports to the Manager of Customer Service.

3.2.14 Bottle Preparation

The Bottle Preparation Technician is responsible for accurately preparing sample containers in a timely and safe manner according to client specification. The Bottle Preparation person also maintains records of standing orders and ensures they are prepared for the courier and for shipment when appropriate. This position also prepares purchase orders for ordering supplies as needed. The Bottle Preparation person also maintains the Bottle Preparation room and storage area in a neat and orderly manner. This position reports to the Manager of Customer Service.

3.3 Contract Review, Design Control, and Quality Planning

The Customer Service Department shall use the flow diagram shown in Figure 3-2 as an initial review of all new work that comes in to the laboratory. A project manager shall be assigned to the project if so designated by the flow diagram. When the contract or scope of work specifies two or more of the requests on the left side of the flow diagram, the assigned project manager shall initiate a Contract Review Checklist (see Figure 3-3) and forward it to the departments concerned.

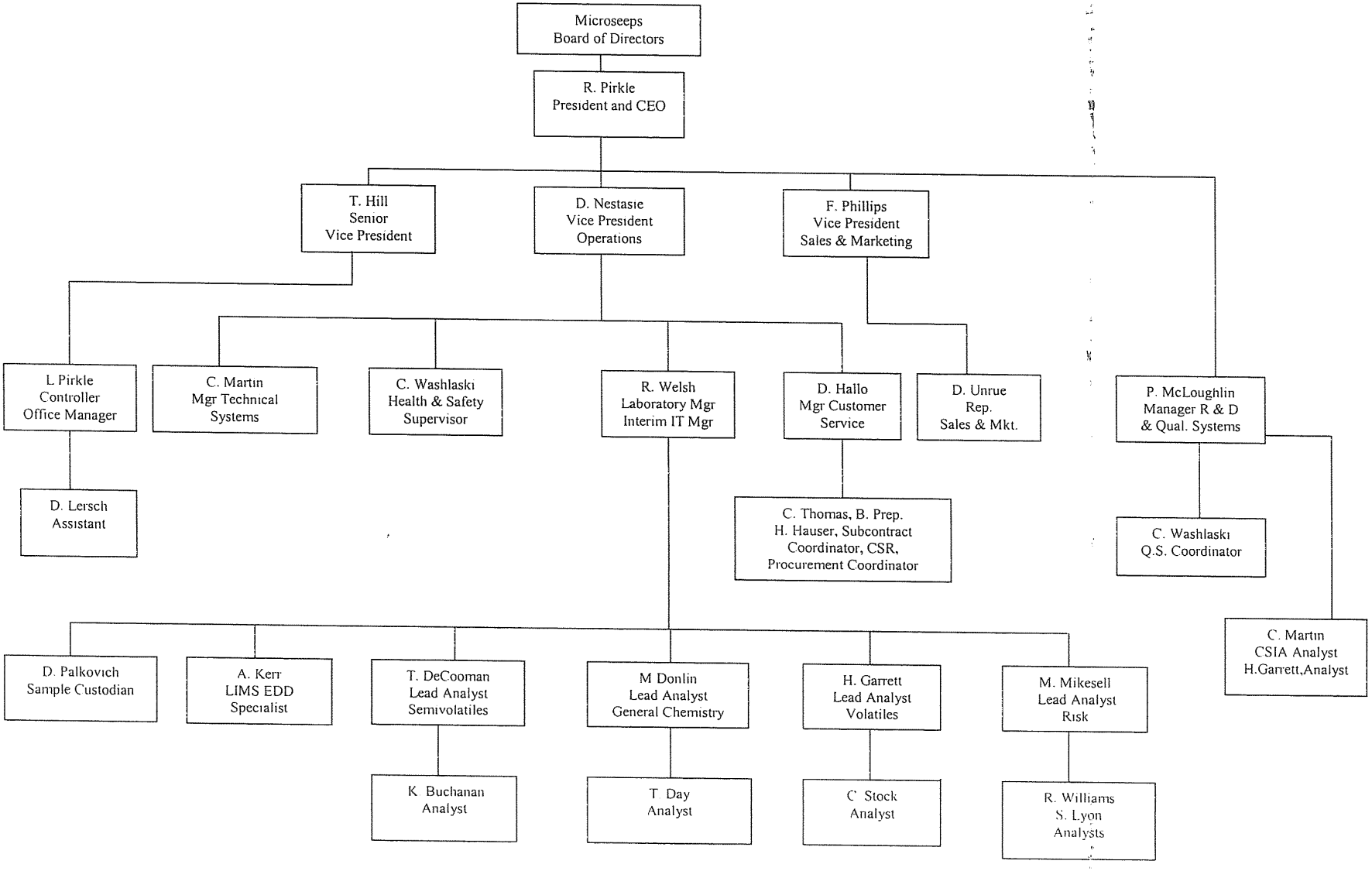
The contract and/or scope of work shall be reviewed by each department concerned to determine if Microseeps has adequate facilities and resources to complete the work in the contract appointed time frame. Each department is to indicate on the form whether their department can or cannot meet the contract requirements, initial and date the form, and return it to the Project Manager within 24 hours of receipt of the checklist. If a department cannot meet the requirements of the contract or scope of work, the department head is to indicate the reason(s) for that decision on the back of the Checklist prior to returning it to the Project Manager.

The client shall be informed and a resolution discussed if the laboratory review of capability indicates any potential conflict, deficiency, lack of accreditation status, or inability on the laboratory's part to complete the clients work. Any differences shall be resolved to the satisfaction of the laboratory and the client prior to commencement of work.

All records of Contract Reviews, including any significant changes, are maintained in the Customer Service Contract files. Customer service also maintains records of discussions with the client relating to the client's requirements and the results of the work during the period of the execution of the contract. Correspondence with the client pertaining to laboratory activities for a project are maintained in the Client Project File. The client shall be notified of any deviation from the contract, including accreditation changes that affect the contract.

In the event that the contract needs amended after work has commenced, the same contract review procedures shall be repeated and any changes shall be communicated to all affected personnel.

Figure 3-1
Microseeps' Organizational Chart



(C-2) C-705

Figure 3-2

Flow Diagram for Contract Review, Design Control, and Quality Planning

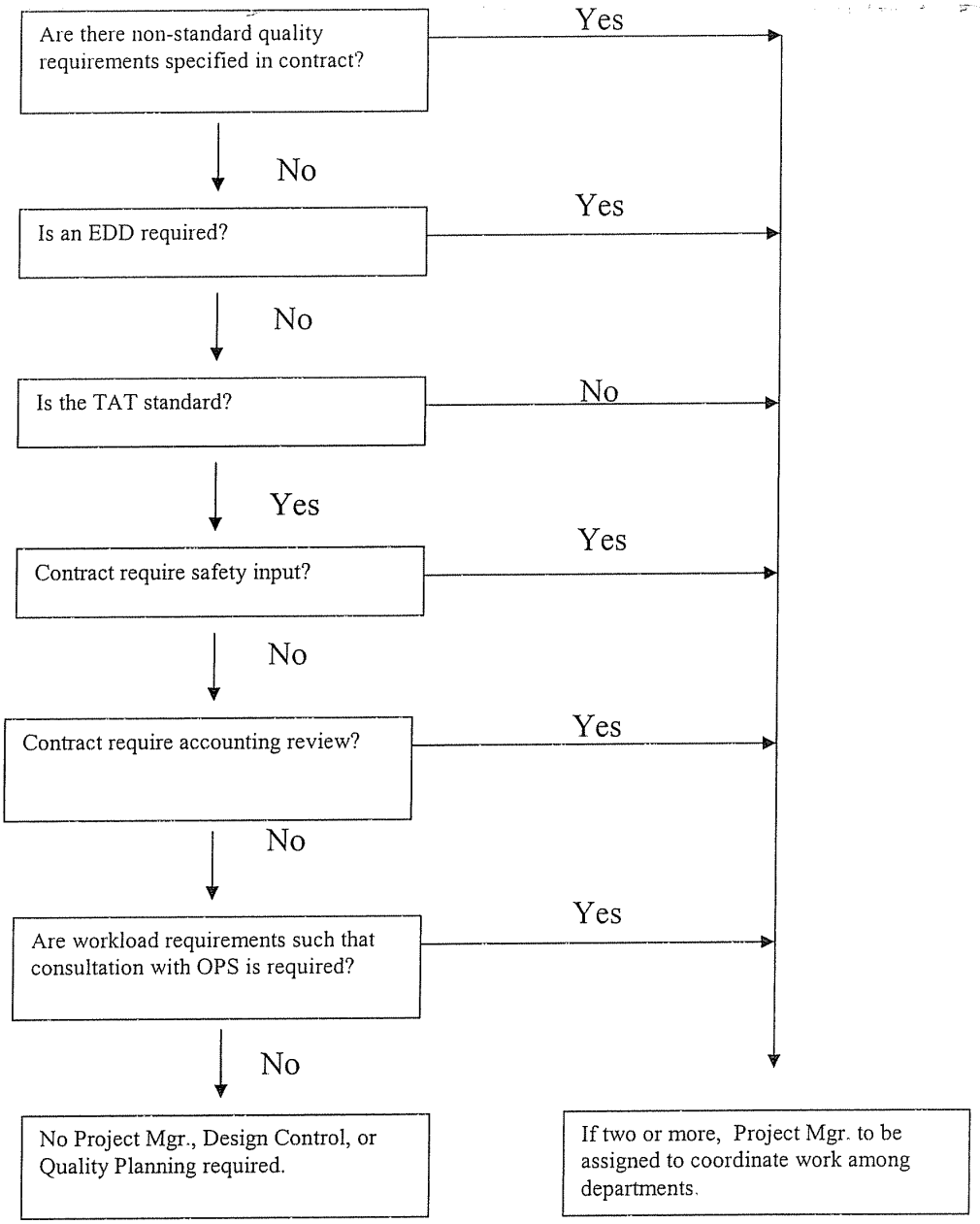


Figure 3-3

Contract Review Checklist

Please review the designated portion of the attached contract/scope of work and determine if your department/work group can or cannot meet all of the requirements of the contract.

Place an X in either the Can or Cannot column, date, and initial where indicated and return this form to the Project Manager within 24 hours of receipt.

Client Company: _____

Contact: _____

Microseeps PM: _____

Date Submitted	Department	Can	*Cannot	Date	Initials
	Operations: • Methods • Personnel • Detection Limits				
	Information Systems • EDD Format				
	Quality Systems • Certifications				
	Accounting • Payment terms				
	Safety • H & S Worksheet				
	Waste • Special requirements				

***Please note on the back of this page the specific reasons why your department may not be able to meet the requirements of the contract.**

4.0 Sample Management

The purpose of this section is to outline and reference procedures for the type and use of sample containers, the preservation of the samples, sample shipping, sample receipt, sample custody, sample log in, sample disposal, and analytical subcontracting. A list of analytical procedures conducted by Microseeps is found in Tables 4-1 through 4-3 at the end of this section. Certifications are found in Tables 4-4 and 4-5.

4.1 Sample Containers and Preservation

In order to provide the best possible service for the client and to obtain the required sample volume, Microseeps prefers to provide the sampling containers. Sample containers are constructed of either polyethylene or glass. Containers are purchased that are pre-cleaned and ready for use. Certified clean containers can be provided if project specifications require. Preservatives are added to most sample containers prior to shipping to the client, either by the bottle preparation technician, or the by the vendor. Clients are encouraged to completely fill each container in order to provide adequate sample volume.

For DoD projects: All bottles, reagents, solvents, and supplies used in DoD projects will be verified or certified by the supplier to meet or exceed standard specifications for environmental tests concerned. Verification must be kept on file to qualify each bottle, reagent, solvent and supply.

4.2 Sample Packing and Shipping

Samples are either hand carried to the laboratory or shipped using commercial carriers. Samples should always be shipped to the laboratory daily using a reliable overnight shipping service.

The chain of custody is used to establish the identity of samples and to provide proof of possession of the samples by Microseeps. Chains of custody are supplied to the client with the sample bottle order. The following information should be recorded, by the client, on the chain of custody.

- Client Name, Address, and Phone Number
- Project Name
- Project Number
- Sample identification, Collection Time, Number of Containers
- Specific Analytical Requirements
- Sample Matrix
- To whom the analytical data shall be submitted
- Relinquish signature

Ice should be added so that a temperature of $\leq 6^{o}C$ is maintained during shipment. Samples should be packed inside a large bag and the bag should be sealed. Bags of ice should be placed on top of the bag and should not be in direct contact with the sample containers. Cooler temperatures are taken electronically and recorded upon receipt by the laboratory.

The chain of custody should be enclosed in the zip lock bag provided by Microseeps, and taped to the inside lid of the cooler.

A copy of Microseeps' Sample Acceptance Policy is included with each client bottle order (see below). In order to expedite the sample log in process, please be sure to include and accurately complete all paperwork that should accompany samples to the laboratory.

Sample Acceptance Policy

1. Samples that are shipped to Microseeps must be accompanied by proper, full, and complete documentation. This documentation shall be marked on a chain of custody and shall include: sample identification, the location, date and time of collection, sampler's name, preservation type, sample type, specific parameters to be analyzed, and any special remarks concerning the sample.

2. Sample labels shall be supplied by Microseeps or the client. Those labels must be water resistant and completed using indelible ink. Each sample label must include a unique identification number that links it to the chain of custody documentation.

3. Samples shall be in the proper containers with the preservatives that are specific to the type of analysis required.

4. All samples must be received within specified holding times. Clients are requested to notify a Microseeps' Customer Service Representative if samples with short holding times are being shipped.

5. Samples must arrive at Microseeps with sufficient volume to conduct the requested analyses. All bottles should be filled completely.

6. When problems with samples or documentation are found during the sample receiving process, a Non-Conformance Form is completed by the Sample Custodian and forwarded to the Customer Service Office. A Customer Service Representative will make every attempt to contact the client as soon as possible to make decisions concerning those discrepancies. The Non-Conformance Form is kept as a permanent part of the project file.

7. If the client cannot be reached, a message will be left either on voice mail, or with a receptionist, or via email for the client to return the phone call. The samples will be placed in a storage refrigerator and held until a Microseeps' Customer Service Representative gets a response from the client. (Exceptions will be made when samples are received that have short holding times and the samples are from a client with whom Microseeps has regular and frequent dealings. Or when the samples have short holding times and the samples are from a client with whom Microseeps has a signed contract, work order, or purchase order.)

4.3 Sample Custody

Microseeps takes custody of the samples when they are received at the laboratory or picked up at a client site by a Microseeps' employee. All samples are maintained in access-controlled areas until work is started. The person responsible for either the sample preparation or analysis will retrieve the sample(s) from the storage area and return them when the function is complete.

4.3.1 Samples Requiring Secure Storage

For samples requiring locked storage and strict custody protocols (e.g. samples as evidence), the samples are locked in a temperature controlled sample storage cooler. When evidentiary samples are to be analyzed, the Sample Custodian initiates a sample tracking record (Figure 4-2), and the analyst signs them out using the complete sample number as generated by the LIMS to identify the samples taken.

After analysis, all remaining sample, sample extracts, or the empty sample container are returned to secure storage, signed back in on the sample tracking record, and placed back in the secure storage area. Entries are made to the form each time a sample is removed and returned to the storage areas. Whenever sample preparations are completed, the sample preparation group adds them to the tracking record. All records of evidentiary samples are maintained until the client authorizes destruction.

4.3.2 Sample Receipt Protocols

The Sample Custodian or designee signs for each shipment and a copy of the shipping documents is retained. Specific sample receipt procedures are addressed in Microseeps Standard Operating Procedure for Sample Receipt (SOP-S2). A brief outline follows:

- Shipment containers are inspected, opened, and monitored for temperature, if applicable.
- Temperature is recorded on the Chain of Custody.
- Shipment containers are unpacked and samples reconciled with the chain of custody.
- Chain of Custody is signed.
- Non-conformances are resolved with the client.
- Samples are logged in to Microseeps LIMS system.

Specific sample log in procedures are outlined in Microseeps Standard Operating Procedure for Sample Log-In using the Laboratory Information Management System (LIMS). Microseeps LIMS assigns a unique internal project number and sequential sample numbers. These numbers are used to track the project through the laboratory. The sample numbers are transferred to each sample container using a computer-generated label. These numbers are documented on the chain of custody form and verified by the sample custodian. The computer generates a cooler receipt form, which is printed and placed in the project file along with the chain of custody and other related documentation.

All documentation relating to the project is maintained in the project file and retained in the laboratory for five years.

4.3.3 Resolution of Non-conformances

Sample receipt non-conformances are resolved according to Microseeps Standard Operation Procedure for Sample Receipt (SOP-S2). In nearly all cases, the client is contacted for the resolution decision and documentation.

Several possibilities may exist for resolving sample receipt problems. All decisions are the client's responsibility. Once a resolution is determined, the solution is noted on the non-conformance form and one or more of the following actions will occur:

- The log-in process will continue
- Written documentation will be requested from the client
- The sample(s) will be returned
- The sample(s) will be disposed

Throughout the problem resolution process, the sample will either be kept in a secure area or will be in view of the sample receipt personnel. All records generated during this process become a part of the client's permanent file.

When a client decides to proceed with analyses of samples that do not meet acceptance criteria, that decision shall be fully documented on the non-conformance form and the analysis data shall be "qualified" using a narrative on the final data report.

4.4 Sample Storage and Recovery

Samples requiring refrigeration are placed into temperature-controlled coolers that are maintained at $\leq 6°C$. The cooler temperature is recorded each morning and afternoon. The walk-in cooler, volatiles storage cooler, and CSIA storage cooler, use a Min/Max thermometer, and are recorded once a day. Temperature logs are maintained for each cooler.

All samples for volatile analysis are segregated in a cooler away from other samples. All samples are stored separately from standards, reagents, food, and other potentially contaminating sources.

4.5 Sample Disposal

Once samples are analyzed, they are moved from a primary to a secondary storage area. Samples are stored in secondary storage areas for thirty days following the date an analytical report is generated. Samples are disposed or returned according to the procedures outlined in SOP-ADM 14, Microseeps Standard Operating Procedure for Waste Disposal.

4.6 Subcontracting Analytical Samples

Microseeps' Laboratory Manager or designee will notify the Project Manager when samples or extracts need to be sent to a subcontract laboratory, the number of samples to be sent, and the duration of the need for subcontract services for services routinely provided at the Microseeps facility. The Project Manager or Customer Service Representative will notify the client in writing of the intent to subcontract samples. The Subcontract Coordinator will schedule the work with the subcontract laboratory and arrange the specifics of shipping the samples.

The Subcontract Coordinator shall monitor the progress of the analytical work and receive the analytical data from the subcontract laboratory.

In the event of expedited turnaround that cannot be met by Microseeps, the Subcontract Coordinator shall initiate the subcontract laboratory procedure in order to meet the client's need.

4.6.1 Subcontract Laboratory Approval

The following procedures are in place to ensure that laboratories that are to be used for subcontracting analytical samples meet minimum requirements for quality as specified by the Quality Systems Department. Prior to approval of a subcontract laboratory, the Quality Systems Department shall request the following information from the laboratory:

- List of current certifications and expiration dates of each.
- Copy of the Quality Assurance Plan for the subcontract facility.

Once this material has been received, it shall be reviewed by the Quality Systems Department and a decision will be made concerning the approval of the subcontract laboratory.

For DoD projects: All subcontracting of DoD projects will be to approved DoD laboratories.

4.6.2 Client Notification of Subcontract Laboratory

There are various reasons why a subcontract laboratory may be used including, but not limited to, an expedited turnaround time, laboratory capacity, and special analysis. All clients shall be notified in writing when any samples are to be sent to a subcontract laboratory.

Where the laboratory subcontracts any part of the testing that is covered under NELAC, this work shall be subcontracted to a NELAC accredited laboratory. The laboratory performing the subcontracted work is indicated on the final report and non-NELAC accredited work is clearly identified.

TABLE 4-1

Container, Preservation, and Holding Time Requirements
(EPA Methods for Chemical Analysis of Water and Wastes Table I and SW-846 3rd ed. Revision 4, Tables 2-40A, 2-40B)

Parameter	Method	Container (1)	Preservative (2)(3)	Maximum Holding Time (4)
Alkalinity	SM2320B	P	Cool to ≤6°C	14 days
Anions by IC	SW846-9056	G, VOA with TLS	Cool to ≤6°C	48 hours for NO_2, NO_3 28 days for other anions
TOC/DOC	SW846-9060 SM 5310 C	G	Cool ≤6°C , H_2SO_4 to pH< 2	28 days
pH	SM 4500 H+ SW846-9040	P	Cool to ≤6°C	Immediate
Sulfide	SM4500 S-F	P	Cool, ≤6°C; Zinc Acetate & NaOH to pH>9	7 days
Purgeable Halocarbons	SW846-5030 8260B EPA 624	G with TLS	HCl to pH<2, Cool ≤6°C	14 days
Purgeable Aromatics	SW846-5030 8260B EPA 624	G with TLS	HCl to pH<2, Cool ≤6°C	14 days

1. AG - amber glass, G - glass, P - polyethylene, TLC - Teflon-lined cap, TLS - Teflon-lined septum
2. Sample preservation should be performed immediately upon sample collection. Composite samples may be preserved by maintaining at ≤6°C until sample splitting and collection is completed.
3. If the dissolved content is to be measured, samples should be filtered on site immediately before adding preservatives.
4. The holding times listed are the maximum times that samples may be held before analysis and still be considered valid under EPA regulations. Holding times are measured from sampling.

TABLE 4-2

Container, Preservation and Holding Time Requirements for Soil, Sediment, Sludge

Parameter	Method	Container[1]	Preservative	Maximum Holding Time
Volatiles	SW846-5035 8260B	G with TLC Encore	Cool ≤6°C MeOH & $Na(SO_4)_2$	14 days
Percent Solids	SM2540F	G with TLC	Cool ≤6°C	7 days
pH 1:1	SW846-9045C	G with TLC	Cool ≤6°C	Immediate

1. TLC = Teflon-lined cap, G = Glass

Table 4-3

Bioremediation Indicator Parameters and Risk Analysis Test Methods
These parameters are analyzed using Microseeps' Methods.

Parameter	Method	Container	Preservative	Maximum Holding Time
Hydrogen by Bubble Strip	SM9/AM20Gax	22 cc vapor vial with stopper septum	None	14 days
Light hydrocarbons by Bubble Strip	SM9/AM20Gax	22 cc vapor vial with stopper septum	None	14 days
Permanent gases by Bubble Strip	SM9/AM20Gax	22 cc vapor vial with stopper septum	None	14 days
Light hydrocarbons in water: Methane, Ethane Ethene	PM01/AM20Gax RSK-175	40 ml Amber VOA vial with mylar septum	Trisodium Phosphate or Benzalkonium Chloride & Cool to ≤6°C	14 days
Permanent gases in water: Oxygen, Nitrogen, Carbon Dioxide	PM01/AM20Gax	40 ml Amber VOA vial with mylar septum	Benzalkonium Chloride & Cool to ≤6°C	14 days
Light hydrocarbons in vapor	AM20Gax	22 cc vapor vial with flat septum	None	14 days
Permanent gases in vapor	AM20Gax	22 cc vapor vial with flat septum	None	14 days
Hydrocarbons in vapor	AM4.02	22 cc vapor vial with flat septum	None	Unspecified
Chlorinated hydrocarbons in vapor	AM4.02	22 cc vapor vial with flat septum	None	Unspecified
Total inorganic carbon in water	PM01/AM20Gax	40 ml Clear VOA vial with mylar septum	Cool to ≤6°C	14 days
Volatile Fatty Acids	AM21G	40 ml Clear	Cool to ≤6°C	21 days
Low Level VFA	AM23G	40 ml Amber with teflon septum	Cool to ≤6°C Benzalkonium chloride	14 days
AMIBA	WC-43	BAFeIII: 8oz plast. All by direct push: 2 oz glass soil jar All by drill. rig: 2 – 40 ml amber 2 oz glass soil jar	Freeze	3 months

Table 4-4
STATE CERTIFICATIONS

Parameter	Method	State Certification
Alkalinity	SM2320	WV, SC, CT
Chloride	SW846-9056	WV, SC, CT
Sulfate	SW846-9056	WV, SC, CT
Sulfide	SM 4500S	WV, SC, CT
TOC	SW846-9060, SM 5310C	WV, SC, CT
Nitrate	SW846-9056	WV, SC, CT
Nitrite	SW846-9056	WV, SC, CT
Volatiles (TCL)	SW846-8260B; EPA 624	WV, CT, SC
Preparations	SW846- 5030B, 5035	WV, CT, SC

Check with Microseeps Customer Service Office for state specific parameter lists.

Table 4-5

NELAC Accredited Parameters/Methods

Primary NELAC: Pennsylvania
Secondary NELAC: FL, NY, NJ, LA, TX
(Not all states accredit all parameters.)

Parameter	Method
Alkalinity	SM2320B
Chloride	SW846-9056
Nitrate	SW846-9056
Nitrite	SW846-9056
Sulfate	SW846-9056
Sulfide	SM 4500S-F
TOC	SW846-9060, SM 5310C
Volatile Organics	SW846-8260B, EPA 624
pH	SW 9040, SM 4500H+
Light Hydrocarbons	RSK175
Dissolve gases & Light Hydrocarbons	Microseeps SOP-AM20GAX
Hydrogen	Microseeps SOP-AM20GAX
Total Inorganic Carbon	Microseeps SOP-AM20GAX
Volatile Organics in Vapor	Microseeps SOP-AM 4.02
Volatile Fatty Acids	Microseeps SOP-AM23G, Microseeps SOP- AM21G
Fuel Oxygenates	SW846-8260, Mod. 524.2

Call Customer Service Office for state-specific analyte list.

Figure 4-1
Chain of Custody Form

CHAIN - OF - CUSTODY RECORD

Company : _____
Co. Address : _____
Proj. Manager : _____
Proj. Location : _____
Proj. Number : _____
Phone # : _____ Fax # : _____

Sampler's signature : _____

Parameters Requested

Results to : _____

Invoice to : _____

Cooler ID	Cooler Temp.

Sample ID	Sample Description	Date	Time	Comp.	Grab	# Cont.	Remarks

Relinquished by :	Company :	Date :	Time :	Recived by :	Company :	Date :	Time :
Relinquished by :	Company :	Date :	Time :	Recived by :	Company :	Date :	Time :
Relinquished by :	Company :	Date :	Time :	Recived by :	Company :	Date :	Time :

WHITE COPY : Accompany Samples YELLOW COPY : Laboratory File PINK COPY : Submitter

Figure 4-2
Sample Tracking Record

Sample Storage Location: _____

Samples placed in storage by: _____

Circle Bottle Type:	Sulfide	D-Gas	Hydrogen	
	IC-VOA	Vapor	G. Chemistry	
	TOC	VOA	Soil	VFA
	TIC	D-Gases	LLVFA	Fuel-Oxygenate

LIMS generated Sample Numbers	**Removed from Storage**			**Returned to Storage**		
	Initials	**Date**	**Time**	**Initials**	**Date**	**Time**

5.0 Facilities, Instrumentation, and Materials Procurement

5.1 Facility Description

Microseeps is located in the University of Pittsburgh's Applied Research Center (UPARC). The complex consists of 58 separate buildings, which house over 120 companies. The Oxford Development Company provides management for the UPARC facility including building maintenance and safety support. Oxford Development's maintenance personnel are on-site and respond quickly and efficiently to all internal environmental or air quality issues. Specialized maintenance staff is on-call to respond to ventilation, heating, cooling, lighting, or other problems that may occur.

Laboratory temperatures are controlled by the permanent heating and air-conditioning systems in the UPARC complex. Where the cooling systems have not been efficient enough to maintain correct temperature and humidity requirements for proper instrument function, Microseeps has installed additional units as needed.

When environmental conditions jeopardize the results of environmental tests, the Lead Analyst shall notify the Laboratory Manager immediately of the problem. If the problem cannot be resolved immediately the Laboratory Manager shall order a stoppage of all work until either the problem is resolved, or another area of the laboratory can be utilized to continue testing.

Microseeps is located in Building B-1 and has offices and laboratories on the first, second, and fourth floors.

5.1.2 Laboratory Areas

The following table specifies the locations of the individual laboratories and workspaces in the building in which Microseeps is housed.

First Floor

- Bottle Preparation — Room 108
- Sample Receiving — Room 115/117
- Volatiles Laboratory — Room 126/128
- Wet Chemistry — Room 127

Second Floor

- Dissolved Gas Laboratory — Room 220/222
- Vapor Laboratory — Room 221
- Instrument Laboratory — Room 213
- LIMS Center — Room 218

Fourth Floor

- CSIA Laboratory Room 424

5.1.3 Building and Laboratory Security

Employee access into the UPARC complex is controlled through key-card turn-styles where each individual that works in the complex has a unique code for entry. UPARC Security is aware of who is on-site or off-site at any given time. Microseeps' laboratory areas are controlled through keyed entry to prevent employees from other firms housed in the complex from gaining access to Microseeps' laboratories. Each employee is issued a key that will open doors to rooms occupied by Microseeps. Those keys do not access accounting or CEO offices. During normal working hours, the laboratory areas are kept unlocked. After normal business hours the rooms are locked to prevent unauthorized personnel entry.

A UPARC Security Force monitors the facility twenty-four hours a day with a series of video cameras. The guards also make rounds by foot and vehicle during afternoon and night shifts. Visitors cannot gain access to the complex except through the Main Security Gate. All visitors are required to register at the main gate and obtain a visitor's pass before entering the complex. UPARC Security notifies Microseeps upon the visitor's arrival to verify admittance. Visitors are directed to Microseeps Reception Office. The visitor is then escorted, by a Microseeps' employee to the Microseeps' front office to sign the visitors log, and is then directed to the employee or laboratory they intend to visit.

5.2 Instrumentation

Instrumentation must be properly calibrated and maintained to produce reliable and reproducible results. This section of the Quality Systems manual defines minimally acceptable standards for installation, calibration, and maintenance of analytical instruments used in the laboratory. Fully detailed procedures are instrument specific and are available in the individual instruments' Operator Manuals.

A list of Microseeps' Instrumentation is included in Exhibit 5-1. This list is updated whenever equipment is placed in service or removed from service.

5.2.1 Installation and Set-up

All new instrumentation must be included in the Quality Systems program prior to being used for sample analysis. When new equipment is ordered, the Laboratory Manager and the Manager of Technical Services determine the preparations that the laboratory must make to accommodate the equipment. This plan includes descriptions of facility modifications that may be required, personnel responsible for installation (manufacturer or Microseeps employee), performance criteria that need to be met, and training procedures that will be followed.

Data generated during installation and set-up will be included in the maintenance log for the instrument. This data may become important later for troubleshooting and diagnostic checks. Operator manuals supplied by the manufacturer are maintained in the laboratory for reference.

5.2.2 Calibration

Calibration procedures for instrumentation are thoroughly documented and routinely followed to provide assurance that the data produced are reliable and accurate. Specific calibration procedures and frequency are detailed in the individual Standard Operating Procedures.

Initial calibration involves comparing instrumental response to various concentration levels of the analytes of interest. The calibration curve will contain a minimum of five points (or better), excluding a blank. The lowest point of the curve should be equal to or lower than the reporting limit. The most concentrated standard should be below but near the upper concentration limit of the linear range. All standards are prepared from solutions of certified concentrations. All calibrations are checked against a "second source" obtained from, preferably a different vendor, but at least a different lot. All calibrations are followed by an initial calibration blank to verify that system contaminants and carry-over are not present. For organic analyses, surrogates are added to each blank and standard. In addition, internal standards are also added for some organic analyses.

If the initial instrument calibration results are outside established acceptance criteria, corrective actions are performed. These criteria and the corrective action are specified in individual Standard Operating Procedures. Data associated with unacceptable initial instrument calibration is not reported.

5.2.2.1 Calibration Verification

If an initial instrument calibration is not performed on the day of analysis, the validity of the initial calibration is verified prior to sample analysis by running continuing instrument calibration verification standards with each analytical batch. Continuing instrument calibration verifications must be run at the beginning and end of each analytical batch.

5.2.3 Instrument Maintenance

Maintenance of analytical instruments is carried out under the direction of the Manager of Technical Services and may include regularly scheduled preventive maintenance, or maintenance on an as-needed basis due to instrument malfunction. Maintenance activities for instrumentation are documented in Instrument Maintenance Logs. This documentation becomes a part of the laboratory's permanent records.

Regular maintenance of support equipment, such as balances, thermometers, and fume hoods is conducted annually and more often if required. Maintenance on other support equipment, such as ovens and refrigerators is conducted on an as needed basis. The Lead Analysts are responsible for ensuring that temperatures of ovens and refrigerators are checked and recorded twice daily. The

Laboratory Manager is notified if the temperature is outside of the range of use of the specific piece of support equipment, and the equipment is scheduled for maintenance.

Records of maintenance to support equipment are also documented in Maintenance Logbooks. Each piece of support equipment does not necessarily have it's own logbook. Maintenance logbooks may be shared with equipment that is housed in the same laboratory area.

5.2.3.1 Out of Service Instruments

In the event that an instrument cannot be calibrated or is determined to be out of order, an out of service or out of calibration tag is placed on it. The Lead Analyst is responsible for ensuring the equipment is tagged. This tag shall be removed when the instrument is repaired and ready for use.

5.2.3.2 Instrument Repair

Unexpected repairs resulting from instrument failure are scheduled immediately after the malfunction is observed. Instrument failures are detected through direct observations and by evaluation of the response of verification standards throughout the analytical run. The Manager of Technical Services is responsible for deciding if laboratory personnel can make the repair or if an outside contractor is required.

Data obtained during instrument failure are not entered into the LIMS for reporting to the client. Complete records of the repairs are maintained in the Instrument Maintenance Logbooks. These records may include notes taken by laboratory personnel during repair and a copy of the service call record. Acceptable instrument performance must be verified before samples can be analyzed.

5.3 Materials Procurement

The purpose of this section is to define requirements for the procurement of materials needed to support laboratory operations.

5.3.1 Purchase and Control of Standards, Reagents, and Materials

The Lead Analysts, or in the case of Customer Service, the Department manager, review and approve all material orders for their departments. The quality of purchased standards and reagents is determined as specified in approved analytical methods and further specified in Microseeps' Standard Operating Procedures. All standards and reagents are to be handled and treated in accordance with the Standard Operating Procedure for Analytical Standards and Reference Materials (SOP-ADM 15).

5.3.2 Placing Orders

The employee specifies the materials that need to be ordered on a Purchase Order Form that can be obtained from the Procurement Coordinator. The Purchase Order form is completed by filling in a suggested vendor's name and catalogue number, a description of the item, the quantity requested,

the date of the request, their name, and the accounting category. The form is forwarded through the Lead Analyst or Department Manager for approval. It is the Lead Analyst's or manager's responsibility to choose an acceptable vendor. Once the order is approved, the Procurement Coordinator places the order. One exception to this procedure is when an order needs placed with ThermoFisher on-line. Each Lead Analyst has a user name and password to order routine supplies on-line. If any supplies need a special quote or pricing, orders must then be placed by the Procurement Coordinator.

5.3.3 Receipt of Materials

Supplies or materials are received by the Sample Custodian, who in turn, calls the employee who the items are for to come pick them up. The employee compares the materials received to the packing list. Any discrepancies are noted on the packing list and is given to the Procurement Coordinator. A call is placed to the vendor to resolve any problems.

Once the order is reconciled with the purchase order, the packing list(s) is attached to the purchase order and is kept on file by the Procurement Coordinator. The materials and associated documentation (i.e. Certificates of Analysis or Purity) are forwarded to the Department that placed the order for storage until use.

All Material Safety Data Sheets are kept in the lab area where the supplies are being used.

Figure 5-1
Microseeps' Instrument and Equipment List

Volatiles

Hewlett Packard Chemstation Data System with NIST Chemical Structures; Hewlett Packard 6850Series II GC with EPC and 5973 Mass Spectrometer; Varion Archon Autosampler (Serial 14288) and Tekmar Velocity XPT Concentrator (Serial US04159007)

Thermo-Electron Focus GC (Serial 10603036) with DSQ II Mass Spetrometer (Serial MS1100132); Teledyne Aquatek 70 Autosampler (Serial US07003004) and Tekmar Velocity Concentrator (Serial US06335001)

Semi-volatiles

Dionex Ion Chromatograph Model LC25 (Serial #97040068) Autosampler Model AS40-1 (Serial 97050241), Eluent Generator Model EG50 (Serial 94050039): Conductivity Generator Model CD20-1 (Serial 97050142); Gradient Pump Model GP40-1 (Serial 97030784)

Dionex Ion Chromatograph Model ISC 2000 with Degasser; Gradient Eluent Generator; AS-40 Autosampler, Columns.

Varian 3400 Gas Chromatograph (Serial 10272) with Varian 8100 Autosampler (Serial 1371)

Risk Analysis

Hewlett Packard 5890 Series A Gas Chromatograph (Serial 2536A05842) with Tekmar 7000 Autosampler (Serial 91099014/91135007).

Agilent 6890 Gas Chromatograph (Serial US10347026) with Agilent G1888 Headspace Autosampler (serial IT40220036).

Hewlett Packard 5890 Series A Gas Chromatograph (Serial 2536A05842) with Tekmar 7000 Autosampler (Serial 91099014/91135007)

Hewlett Packard 5890 Series II Plus (Serial 3336A53505)

Hewlett Packard 5890 Series II (Serial 3336A51836) with Tekmar 7000/7050 Autosampler (Serial 91346008/91346016)

Three Proprietary GCs

Ohaus Discovery Analytical Balance Model # DV215CD (Serial 1128122704)

Wet Chemistry

Dionex 500 Ion Chromatograph with dual Autosamplers, columns and ovens, with electrochemical and UV-Vis Detectors.

Dionex ISC 3000 Ion Chromatograph with dual Autosamplers, columns, and ovens with conductivity and UV-VIS detectors.

Denver Instruments Model SI-4002 Top Loading Balance

Thermo Electron Hipertoc Model TOC Analyzer

Spectronic 20G Colorimeter

Spectronic 20D Colorimeter

Orion 601A pH Meter

Sartorius Model 1612 Analytical Balance

CSIA

Tekmar Aqua Tek 70 Autosampler (Serial US 06151001)

Tekmar Velocity XPT Purge and Trap (Serial US 06191003)

Entech 7100A Pre-concentrator (Serial 1304)

Thermo Trace GC Ultra Gas Chromatograph (Serial 200510408)

Thermo GC-Combustion III Interface (Serial 111201-175)

Thermo GC /TC Reactor OD (Serial 108520-349)

Thermo Delta V Plus Isotope Ratio Mass Spectrometer (Serial 8018)

Thermo-Electron GC (Serial 10603008) with DSQ II Mass Spectrometer (Serial 100442); Varian Archon Autosampler (Serial 14655) and Tekmar Velocity Concentrator (Serial US6047001)

6.0 Data Quality

6.1 Data Quality Objectives

Microseeps conducts analysis on environmental samples for clients who rely on the data in order to make decisions concerning environmental problems, environmental monitoring, and in many cases to investigate the feasibility of cutting edge remedial technologies. The necessity of high quality data is essential so that the best decisions can be made in the interest of both the environment and the client. Microseeps believes data quality objectives are applied to a project from the initial sampling to the final data validation process. On a laboratory scale, Microseeps' data quality objectives are reflected in individual Technical Standard Operating Procedures as quality control acceptance criteria.

Quality control sample acceptance criteria is generated using one of following three methods:

- A minimum of twenty data points are manually collected and entered into an EXCEL spreadsheet. The average percent recovery and/or relative percent difference (RPD) is calculated, whichever is applicable. Acceptance criteria are generated using the standard deviation of the average percent recovery and RPD. Three standard deviations comprise the acceptance range around the average percent recovery and above the RPD.
- Calculated electronically by the LIMS database and expressed in the Control Chart Program.
- Taken from EPA method-specific recommendations in the absence of laboratory-generated criteria.

Acceptance criteria generated from the implementation of control charts, either manually or LIMS generated are evaluated annually or more often. If it is determined that the acceptance criteria has changed to a broader set of limits, the reason for the change is evaluated for error to ensure the analytical method is still in control. When warranted, corrective action shall be instituted by the Quality Systems office or the Operations Department.

All analytical data including quality control results are checked in accordance with SOP-ADM 16 Standard Operating Procedure for Data Integrity, Review and Validation. Performance Evaluation studies are conducted twice a year and the results are reviewed by management. All failed PE samples are followed up with Corrective Actions.

To maintain the quality of laboratory data and to ensure that laboratory procedures are under control, a variety of internal batch quality control samples are analyzed. The data from those samples are used to calculate statistics that help determine precision, accuracy, and to track potential bias. In addition, performance evaluation studies are conducted regularly as well as random submission of blind quality control samples. Certified reference standards are used, as well, to ensure that quality is maintained.

6.2 Internal Batch Quality Control

Batch quality control sample types and frequencies are recommended in published methods and specified in Microseeps Technical Standard Operating Procedures. In general, a batch of samples consists of twenty samples or fewer (as recommended by analytical methods) that are analyzed at the same time. Typically, a set of internal quality control samples is analyzed once for each batch of clients' samples. The types of internal batch quality control samples are discussed below.

6.2.1 Initial and Continuing Calibration Verification Standards

Initial and continuing calibration standards verify the ratio of instrument response to the analyte amount. Typically, initial calibration verification and continuing calibration standards are made from stock solutions, which are different from the stock used to prepare the initial calibration standards.

6.2.2 Calibration Blank

A calibration blank is a 'clean' sample made from an appropriate matrix and/or solvent. The calibration blank is analyzed to insure that there is no contamination in any part of the analytical system, or to establish a baseline if that "contamination" is expected and known to be of consistent concentration. Calibration blanks are analyzed after each initial, continuing, and calibration verification standard. The analytical result of the blank must be below the laboratory's quantitation limit or project specific requirements in order for analysis to proceed. If the result of the analysis is above the acceptance limit, the source of contamination must be identified and eliminated. The one exception involves the presence of common laboratory solvents as defined by the EPA.

6.2.3 Method (Preparation) Blanks

Method blanks are reagent water or, for solid/waste matrices, sand, or other appropriate material, or an appropriate solvent carried through the entire analytical process to monitor potential contamination that may or may not be introduced during sample preparation and processing. For organic analyses, surrogates and internal standards are added to the method blank.

Method blanks are analyzed at the beginning of the batch and prior to sample analysis. If the analytes of interest are below the laboratory's quantitation limit or project specific limit, sample analysis can proceed. If analyte concentrations are found above the acceptance limits, the source of the contamination must be identified and corrected. The reagents used for sample preparation must be checked for contamination and the samples associated with the method blank must be prepared again and reanalyzed if necessary.

6.2.4 Duplicates

Duplicates are analyzed to assess precision of the analytical procedure. Samples for batch duplicate analyses are selected at random, ensuring that the selection is rotated among client samples so that various matrix problems may be noted and/or addressed. If the sample requires

that an aliquot be removed and placed in another container in order to conduct the duplicate analysis, a representative aliquot is collected using one of the following options discussed below in Subsection 6.3.4.

Precision of the analyses may vary due to the matrix effects of the sample. If the precision is outside established control limits, the duplicate analysis is repeated. If the precision is still outside established control limits and all other quality control checks are within control, a matrix effect is assumed.

6.2.5 Matrix Spike/Matrix Spike Duplicates

Matrix spike and spike duplicate samples are analyzed to determine the extent of matrix bias or matrix interference on analyte recovery and to determine sample-to-sample precision. Analytes stipulated by the method, by regulations, or by other requirements must be spiked into the sample. If not supplied by the client, the analyst may choose these samples at random. Percent recoveries are calculated for each of the analytes detected. The relative percent difference between the samples is calculated and used to assess analytical precision.

Recovery data is highly dependent upon matrix effects. If acceptable recoveries are observed, it is determined that matrix is having no significant affect on the analytical procedure and that sample preparation and analysis have been performed correctly. Whenever precision, calibration, and system quality control checks are acceptable, large or small matrix spike recoveries are attributed to matrix effects. Because samples are spiked prior to analysis, the concentration of the analyte of interest in the sample may be so high that the spike amount is insignificant. In these cases, spike recovery is meaningless and is not calculated.

6.2.6 Laboratory Control Samples

Laboratory control samples are samples that are spiked with a specific concentration of known reference materials, independent of the calibration standards that are carried through the entire analytical process. These standards are used to assess the accuracy of the analytical process. Where possible, acceptance limits are statistically based upon actual laboratory data. If results are outside acceptance limits, corrective action must be performed before sample analyses can proceed.

6.2.7 Surrogate Standards

Surrogates are organic compounds which are similar to analytes of interest in chemical composition, extraction, and chromatography, but which are not normally found in environmental samples. These compounds are spiked into all blanks, standards, samples and spiked samples prior to organic analysis.

If surrogate recovery is not within acceptance limits, corrective action is instituted and reanalysis of those samples occurs.

6.2.8 Insufficient Sample Volume

When there is insufficient client sample volume in a batch to conduct quality control samples for precision, then a laboratory control sample and laboratory control sample duplicate are analyzed.

6.3 Measurements of Data Quality

Data quality measurements vary from parameter to parameter, are represented as warning and control limits, and are displayed along with associated data on control charts. The following types of measurements are utilized by Microseeps to insure the highest quality data is being provided.

6.3.1 Precision

Precision is a measure of the degree of mutual agreement among individual measurements made under prescribed conditions. Precision of laboratory data is determined through duplicate analyses of samples or matrix spikes and spike duplicates, and is calculated for Microseeps purposes, as either the range or relative percent difference (RPD) of the measurements.

6.3.1.1 Range

Range is defined as the difference between the highest and the lowest value reported for a sample. Range is used in the laboratory as recommended in published regulatory methods. The formula for calculating range is as follows:

$$Range = HighestValue - LowestValue$$

6.3.1.2 Relative Percent Difference

Relative percent difference (RPD) is used for all of the analytical methods at the laboratory where sample duplicates are analyzed and where both matrix spikes and matrix spike duplicates are analyzed. If one or both measurements are less than the reporting limit, precision is not calculated. The formula for calculating relative percent difference is as follows:

$$RPD = \frac{|A - B|}{\frac{A + B}{2}} \times 100\%$$

6.3.2 Accuracy

Accuracy is the measurement of agreement between a measurement and the true value. It is calculated as the percent recovery of standards and spikes. Accuracy is calculated as the percent recovery of laboratory control samples, matrix spikes, and in organic chemistry surrogate recoveries.

6.3.2.1 Percent Recovery

Percent recovery is used for all of the analytical methods at the laboratory where laboratory control samples, matrix spikes, and/or surrogates are analyzed. The formula for calculating percent recovery is as follows:

$$\%\,\mathrm{Recovery} = \frac{Measurement}{True\,Value} \times 100\%$$

6.3.3 Bias

Bias is defined as a systematic error due to the experimental method that causes the measured values to deviate from the true value. Bias is determined by plotting the average percent recovery and the average relative percent difference on a control chart. A bias is suspected when seven successive data points are plotted on the same side of the average. This is considered an out of control event.

6.3.4 Representativeness

Representativeness is defined as data that accurately and precisely reflect the sampling points or environmental conditions. Numerous items throughout sampling and sample handling must be controlled to maximize representativeness. These include sample collection, preservation, and holding times. Since Microseeps does not perform sample collection, Microseeps cannot accept responsibility for representativeness of sample collection.

When an aliquot must be removed from the sample container for analysis or for making batch quality control samples, one of the following two options are used in order to obtain a representative aliquot:

1) If the analysis won't be compromised by agitation, then the sample is stirred, mixed, crushed, blended, as needed, and the aliquot is removed from sample container and placed in another container.

2) If the analysis may be compromised by shaking or mixing the sample, then portions of the aliquot are taken from different places within the original sample container ensuring that the aliquot is as representative as possible of the original sample.

In some cases where a sample cannot be homogenized, the client will be contacted and a course of action will be decided upon according to a mutually agreed upon decision.

6.3.5 Comparability

Comparability is the confidence level with which one set of data can be compared to a related set of data. Microseeps uses EPA recommended methodology, whenever feasible, participates in internal and external performance evaluation programs, and uses standard reference materials for sample analysis as means of enhancing comparability.

6.4 Validation of Methods

Validation is the confirmation by examination and the provision of objective evidence that the particular requirements for a specific intended use are fulfilled.

Microseeps validates non standard and laboratory-designed/developed methods, standard methods used outside their intended scope, and amplifications and modifications of standard methods to confirm that the methods are fit for the intended use. The laboratory keep records of results obtained, the procedures used, and a statement that the method is fit for the intended use.

Records of method development are kept in the Technical Director's Office.

6.5 Uncertainty

There are so many sources of uncertainty in a concentration measurement. Certainly, the collection of the sample is a very large source of uncertainty. The particular technique of collection is another, and the location of the points chosen for sampling is a third. Of course, there is uncertainty in analytical measurements as well, and all laboratory analytical procedures are specifically designed to minimize or otherwise control that uncertainty. As such, analytical uncertainty is very likely a minor contribution to the overall uncertainty of any measurement. However, analytical uncertainty is important. There are many potential causes. Some of the most common are enumerated below, along with a brief discussion on how Microseeps minimizes the effect of each:

- Human factors- by maintaining SOPs which are analysis-not analyst-dependant, and by engaging in training and yearly CDOP's, Microseeps tries to minimize the analyst-to-analyst variable.
- Accommodations and environmental conditions-Perhaps the biggest variable here is ambient temperature. In our wet chemistry, and one of our non-traditional laboratories, there is manually controlled heating and cooling. In one of our non-traditional analyses rooms there is additional, automated cooling. Since volatiles are extremely temperature sensitive, both our volatiles analysis laboratory and our CSIA laboratory are furnished with large capacity air conditioning systems.
- Environmental test calibration and method validation-The vast majority of our analytical activities are specifically designed to address these issues. Specifically, it should be pointed out that all of our tests are calibrated with externally certified standards, and then double-checked with a "second source" standard when possible. Additionally, since we utilize many of our own analytical tests, we have

strong emphasis on method validation. For a method to be validated here, it must either pass a successful calibration and then be verified against an external source. The method must be successfully brought through an IDOP and then regularly produce valid matrix spike, matrix spike duplicate and sample duplicate analyses, if applicable.

- Equipment-Before a new piece of equipment is routinely used, an IDOP must be successfully completed using that equipment. This minimizes the potential for un-controlled equipment fluctuations.
- Measurement traceability-If an analytical system is internally calibrated, we maintain records of that calibration, including the certificate of analysis of the standards used in that calibration. If the system was externally calibrated, the records of that external calibration are also supplied.

Finally, to measure the total analytical uncertainty we routinely perform LCS's. (LCS's are chosen because they are submitted to the entire analytical procedure.) The typical acceptance range for an LCS recovery is 80-120%, but the LCS performed with a batch and the SOP for the particular measurement technique should be consulted for specifics.

7.0 Data Handling

Microseeps maintains records that enable the re-creation of the specific conditions under which data are produced, and method specific Standard Operating Procedures outline procedures for data collection, reduction, validation, and reporting. This section discusses, in general, Microseeps data handling procedures.

On occasion when a client requests analyses that Microseeps is not equipped to perform, capacity issues prevent the laboratory from meeting requested turnarounds, or in the event of an instrument failure, a subcontract laboratory may be used. A list of routinely utilized subcontract laboratories is presented in Exhibit 1 at the end of this section.

For cases when the client requests analyses that Microseeps is unable to perform, the Project Manager notifies Subcontract Coordinator of the need to locate a subcontract lab. The Subcontract Coordinator is responsible for contacting potential subcontract laboratories to determine where the samples will be sent. For routinely requested analyses, standard agreements are in place with the subcontractor. Whenever applicable, subcontract laboratories will be NELAC-approved. Other factors that affect the decision include turnaround, cost, and ability to provide the required data deliverables. Once the decision is made, the Subcontract Coordinator notifies the Project Manager so that the client can be informed. For cases when the internal problems require the use of a subcontractor, the Laboratory Manager will notify the Project Managers of the situation, what samples are affected, and the proposed resolution. The Project Managers will then contact each affected client to obtain approval prior to samples being shipped to the subcontractor.

All samples that require subcontracted analyses are prepared for shipment by the Subcontract Coordinator. A chain of custody is prepared detailing the sample identification, collection date and time, as available, requested analyses, requested due date, and any other special instructions pertinent to the sample shipment. The samples are packed in a cooler with sufficient ice to maintain the appropriate temperature during shipment. All samples for subcontracted analyses are shipped so that the samples arrive the next business day.

All data received from the subcontract laboratory follow the same guidelines detailed in this section, with the Laboratory Manager filling the analyst's role and review performed by the Vice President of Operations.

7.1 Data Collection

All laboratory employees are responsible for maintaining laboratory records and documenting them in sufficient detail to recreate analyses. Manually entered data are made using permanent ink. Corrections are indicated by drawing a single line through the incorrect entry, dating and initialing the correction, and coding the reason for the correction. The use of correction fluid or tape, erasure, or other means of making corrections is prohibited. The following information is recorded at the bench either manually or printed out via the data system interfaced with the analytical instrument.

- Method performed.
- Analysis date.
- Analysis time.
- Analyst signature or initials on computer printouts.
- Instrument Identification and settings.
- Analytical sequence consisting of a chronological listing of the processing for each standard, quality control check, and sample recorded in an analytical sequence, run log, or data sheet.
- The laboratory sample number.
- Quality control sample type.
- Standard identification and volume used for all calibration standards.
- True value and lot number of all spiked quality control samples.
- Dilutions including actual initial and final volumes.
- Sample aliquot and final volume.
- Instrument reading.
- Final results with units.
- Calculations for all quality control checks.
- Narrative describing any unusual observances.

If an analysis extends over more than one shift or day, each person responsible for part of the analysis records the date and time their portion of the analysis was initiated.

7.2 Data Reduction

Reducing raw data into a presentable form is the responsibility of the analyst performing the determination. The actual equations used to calculate final results are found in the analytical methods. The following general rounding rules are used for the calculations:

- Data is not rounded until the final answer is obtained.
- To round a figure, the number of significant figures is determined. If the figure to the right of the immediate right-most significant figure is greater than 5, round up. If this figure is less than 5, truncate the result after the last reportable figure. If this figure is equal to 5 and there are non-zero digits to the right, round up. If the figure is equal to 5 and there are no non-zero digits to the right, round up when the preceding figure is odd, and truncate when the preceding figure is even.

7.3 Data Validation

Each analyst initials and dates the data that is generated in the laboratory. All data generated by the laboratory undergoes either an independent peer review or a review by a lead analyst or other designated individual to ensure compliance with accepted quality control standards prior to data entry. The purpose of this review is to check for precision, accuracy, and completeness. The following items are verified during this review. Not all items are applicable to each test.

- Holding times.

- Proper measurement units.
- Instrument tune and initial calibration criteria.
- Proper number of calibration standards and blanks.
- Surrogate and spike percent recoveries.
- Comparison of quality control sample results to acceptance criteria.
- Corrective action for out of control conditions.

After this review, all data that is not instrument-interfaced directly with the Laboratory Information Management System (LIMS), is manually entered into the database. All manually entered data undergoes a review upon entry. The Laboratory Director attempts to review approximately 10% of all laboratory data. Either the Quality Systems Manager or his designee will review 10% of all DoD data packages. This review is part of the oversight program and does not have to be completed in "real time." Project Managers complete the data validation process by reviewing final reports for completeness prior to submission to the client.

7.4 Laboratory Information Management System

The LIMS is the point of collection for all of the laboratory data. The integrity of laboratory data is of the utmost importance. The LIMS system has built-in security levels that keep individual access on an "as needed" basis and does not allow for access beyond what is necessary for the completion of individual duties. Specific operation and management of the LIMS system is outlined in LIMS Standard Operating Procedures that are maintained by the Laboratory.

7.5 Report Preparation

After all of the data has been entered into the LIMS, a draft copy of the final report is generated and the following items are reviewed by the Laboratory Manager or designee:

- Client name and address.
- Analytical results, units, and reportable figures.
- Appropriate data qualifiers are applied as required according to the following table.
- Inter-parametric relationships.
- Data reasonableness in respect to sample information.

Table A

Data Qualifiers

Qualifier	Description
J	Estimated value-result is >MDL but <PQL
U	Component was analyzed for but not detected
R	Surrogate recoveries are outside control limits
M	Percent recoveries or RPD outside of control limits for MS/MSD, sample/dup analyses
B	Component was detected in blank
L	Analyses were performed by a subcontract lab
N	NELAC certified anayses

Following the review, if results are acceptable the Laboratory Manager, or designee makes the determination to generate a final report. The report is forwarded to a Project Manager who conducts a general review for completeness, signs the report, and releases it to the client. The draft copy of the report is placed in the client file for storage.

Final Reports are sent to clients through the U.S. Postal Service unless the client requests an electronic copy. Electronic copies are placed into Portable Document Format (pdf) or EXCEL spreadsheets and password protected prior to transmission.

7.5.1 Final Report Modifications

Once a laboratory final report is generated and sent to the client, it can only be modified under the following circumstances:

- A copy of the original unedited report is kept on file in the laboratory. In the case of electronic records and/or files, either a pdf is generated of the original report prior to editing, or a paper copy of the original report is placed in the project file.
- The reissued report must have a statement in the comments section that specifies the modification(s) and indicates the date of report reissue.
- A copy of the reissued report must be filed with the original report.

Data review of report modifications is conducted in accordance with Microseeps Standard Operating Procedure for Data Review and Validation (SOP-ADM 16).

7.6 Record Retention

A signature record of all employees is maintained in the Training Records Manual kept in the Quality Systems Coordinator's Office.

All raw data and reports for analytical projects are kept for a full five (5) years. After this time the records are destroyed. Records are kept onsite in a locked storage area and maintained in accordance with Microseeps Standard Operating Procedure for Document Control (SOP-ADM 05). The area is inspected monthly.

The LIMS database, which retains all electronic records that have been entered from the date of the LIMS inception, is backed up each day. The tape on which this backup is stored is maintained in a secure location off-site.

All critical data on personal computers is backed up on the internal network S drive. The network is backed up on tape and stored at a secure location off-site.

7.7 Service to the Client and Confidentiality

Clients are welcome to an on-site visit to Microseeps Laboratories in order to discuss client needs or requests, and also to monitor the laboratory's performance in relation to ongoing client projects. All efforts are made to maintain client confidentiality while providing service to other clients.

All client data, whether from privately owned or government organizations, and all correspondence is considered confidential information and shall not be released to anyone other than the client without the expressed written permission of the client. These transactions shall be handled by the Customer Service Office.

7.8 Records Dispensation

In the unlikely event that the laboratory transfers ownership or goes out of business, all clients will be notified in writing and requested to notify Microseeps concerning the dispensation of their records.

Exhibit 1

Subcontractor Laboratories

Name	Requested Analyses
Pace Analytical Services	Semivolatile organics, Pesticides, PCB's, Herbicides, EDB by 8011,Gasoline and Diesel Organics, TO-14, Metals, General Chemistry,Parameters,Total and Fecal Coliform,Oil & Grease.
Analytical Laboratory Services	TOX, EOX, PAH's by 525
Alternative Testing Laboratory	Silicon, Carbon, Percent Chloride on Ash, Total Carbon
Microbial Insights	Dehalococcoides, Phospholipid Acids
Pace Analytical Services Seattle, Washington location	All analyses for DoD projects

8.0 Measurement Traceability

Traceability of measurements and standards is insured in the laboratory by using balance calibration weights that are traceable to national standards, calibrating thermometers using an NIST calibrated thermometer, and by the use of certified standard solutions.

8.1 Reagents and Standards

The purity of the materials required in analytical chemistry varies with the type of analysis, the parameter being measured, and the sensitivity of the detection system. In general analytical reagent grade is satisfactory for most inorganic analyses. Other analyses, such as trace organic, may require special ultra-pure reagents. In cases were the method does not specify the purity of the reagent, it is intended that analytical reagent grade be used. The labels on the container are checked and the contents examined to verify that the purity of the reagents meets the needs of the particular method involved.

8.1.1 Reagent and Standard Preparation

Reagents are prepared and standardized with the utmost of care against reliable primary standards. They are re-standardized or prepared fresh as often as required by their stability as specified by method and other reference sources. Stock and working standard solutions are checked regularly for signs of deterioration.

Standard preparation procedures are specific to the analytical determination being made and are defined in detail in specific technical Standard Operating Procedures, regulatory methods, and in the laboratory's Standards Logbooks.

8.1.2 Reagent and Standard Labeling and Storage

Standard solutions are labeled with the compound name, lot number, preparation date, and expiration date. The analysts store reagents and solvents in a manner that prevents contamination and deterioration prior to their use. Standard solutions are stored in compatible containers.

Microseeps Standard Operating Procedure for Analytical Standards and Reference Materials (SOP-ADM 15) gives detailed instructions for handling, storing, labeling, and documenting standards and reference materials.

8.1.3 Standards Preparation Logbook

Standard Preparation Logbooks are issued as controlled documents to every analyst or laboratory in which they will be used. These logbooks are used to document standard preparation procedures, dates, lot numbers, concentrations, manufacturer, expiration date, and any other information that

may be necessary in order to re-create or track a particular standard. The compound or element name and/or formula are documented along with the final concentration or normality. The description of how reagents and standards are prepared may be referenced from a previous description if the exact procedure is used.

8.1.4 Traceability of Standards

The traceability of each purchased stock standard is easily accessible. Certificates of analysis of each standard are maintained in a binder in the laboratory until the standard is depleted or disposed. The certificate is then given to the quality assurance manager for archiving. The traceability of each laboratory prepared standard is entered into standard logbooks.

For DoD projects: All bottles, reagents, solvents, and supplies used in DoD projects will be verified or certified by the supplier to meet or exceed standard specifications for environmental tests concerned. Verification must be kept on file to qualify each bottle, reagent, solvent and supply.

8.2 Thermometers

All of the laboratory thermometers in use are calibrated annually using a thermometer that is traceable to NIST. Thermometer calibration is outlined in Microseeps Standard Operating Procedure for Calibration of Thermometers (SOP-ADM 12).

8.3 Weights and Balances

All balances are calibrated before use with Class I NIST traceable weights that are calibrated annually. Balances are serviced and calibrated by an outside contractor annually. Additional information is outlined in Microseeps Standard Operating Procedures for Calibration of Weights and Balances (SOP-ADM 11).

9.0 Training Program

Training for laboratory personnel is accomplished at several levels. Areas for which training is conducted include new employee orientation, laboratory safety, specific task training, analytical procedure training, laboratory ethics, and other technical training courses as the need arises.

Analytical training, while addressed in general in this manual, is addressed specifically in the Standard Operating Procedure for Administering and Documenting Training in Laboratory Procedures and Instrumentation (SOP-ADM 02).

9.1 New Employee Orientation

Orientation is conducted to familiarize new employees with company policies, quality system procedures, facilities, coworkers, laboratory ethics, and laboratory safety. The Department Managers are responsible for notifying the various departments of the start date of all new employees.

9.2 Laboratory Safety

Upon hire, each employee is required to read the laboratory's Chemical Hygiene Plan (safety manual), is issued a pair of safety glasses, and is instructed in basic laboratory safety requirements. Safety Meetings are mandatory for all employees and are held on a monthly basis by the Safety Coordinator. Documentation of safety training is updated and maintained by Microseeps' Safety Coordinator.

9.3 Task Training

Task training must be successfully completed for employees to perform the following tasks without direct supervision:

- Sample Receiving
- Bottle Preparation
- Customer Service
- Data Entry

Task training is conducted by the Department Manager of the department concerned. During this training, the trainer works closely with the trainee to ensure that all pertinent points of procedures are addressed. If the procedure is outlined in a Standard Operating Procedure (SOP), the trainee is charged with reading the SOP and the trainer will ensure that the training covers all aspects of the SOP. For training to be considered complete, proficiency in the task must be demonstrated to the trainer.

9.4 Analytical Method Training

Prior to conducting analysis on client samples, all analysts must demonstrate their proficiency through initial technical training. Analytical proficiency must be demonstrated annually thereafter. All analytical method training must be conducted by an analyst who is certified in the method for which training is required.

For individual analyst training in sample preparation and analysis, initial training and proficiency is demonstrated through the analysis of a set of 4 consecutively run mid-range standards or an Initial Demonstration of Proficiency (IDOP). When an analysis involves a work group (i.e. semivolatile preparation and analysis), a team approach to the IDOP applies. When each individual completes their part in the successful analysis of the IDOP, the analytical team is considered competent to conduct the analysis on client samples.

For continued demonstrations of capability for individuals and work groups, one of the following procedures may be used:

1) Another IDOP
2) Four consecutively run laboratory control samples that fall within laboratory/method acceptance criteria.
3) Acceptable analysis of Performance Evaluation samples.
4) Acceptable analysis of blind quality control sample.

Training documentation is maintained in the Quality Systems Office. See Exhibit 9-1 for a flow diagram of analytical method training. A Demonstration of Capability Certification Statement (Exhibit 9-2) shall be maintained for each method in which an employee is certified. This documentation is kept in the Employee Training Records in the Quality Systems Office.

9.5 Laboratory Ethics Training

All employees shall receive initial and annual ethics training. The training shall be conducted by Microseeps' management personnel. The training shall include the contents of the Ethics Program (see Appendix C) in its entirety, including employee and supervisory responsibilities, examples of unethical behavior, disciplinary action for unethical actions, and a means to report unethical actions.

Documentation of Ethics Program Training is kept in the Quality Systems Office. All employees are required to sign and date the Ethics and Data Integrity Agreement (see Exhibit 9-3). This form shall be placed in the employees personnel file.

9.6 Other Technical Training

Other types of technical training are conducted on an as-needed basis and may include training on new instruments, new procedures, or new equipment. Training may be conducted by Microseeps' employees

9.7 External Training

Employees are encouraged to continue their education through the use of symposia and seminars conducted by professional societies, regulatory agencies, and equipment manufacturers. These courses serve as one way for laboratory personnel to remain current on regulatory trends, analytical procedures, and advances in instrumentation. Documentation of external training will be added to the analysts' training records.

Exhibit 9-1

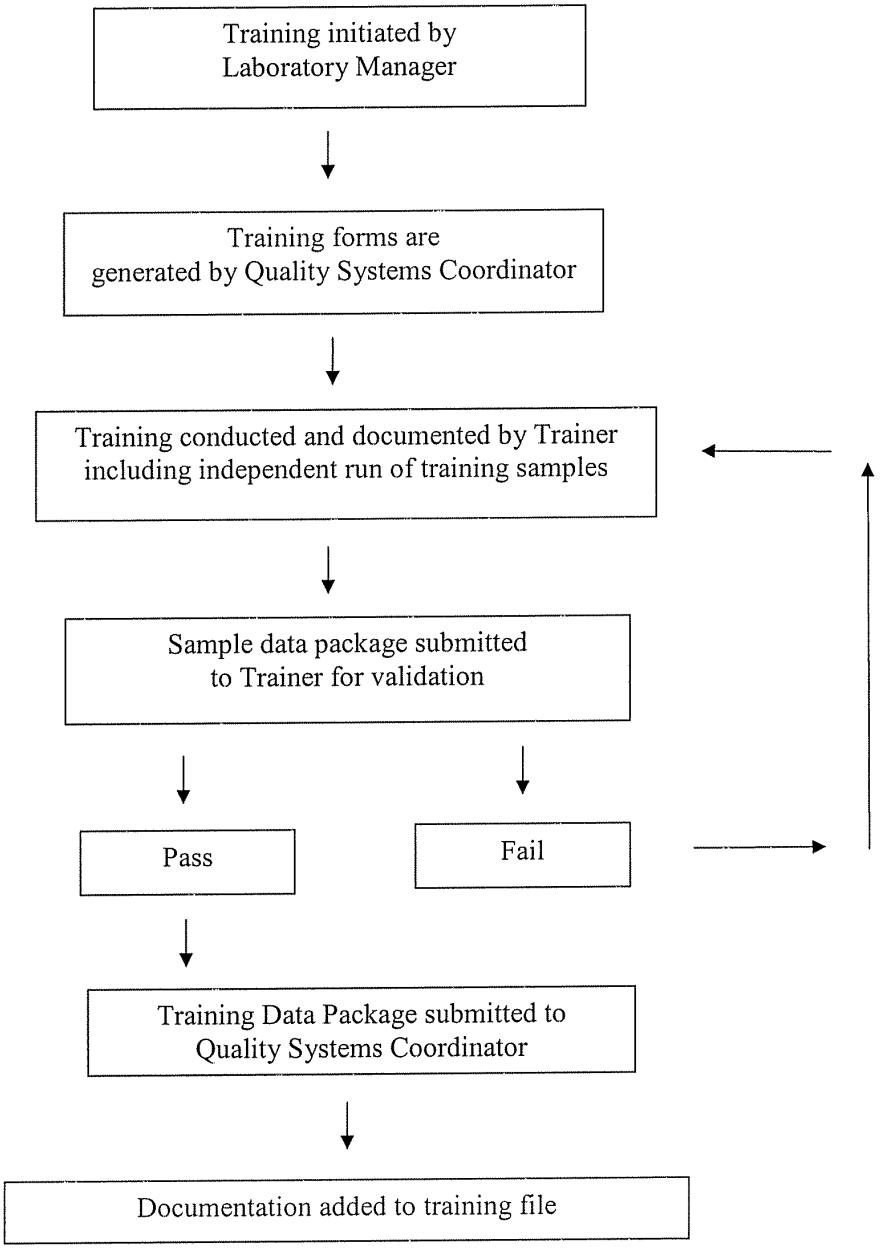

Training initiated by Laboratory Manager
Training forms are generated by Quality Systems Coordinator
Training conducted and documented by Trainer including independent run of training samples
Sample data package submitted to Trainer for validation
Pass
Fail
Training Data Package submitted to Quality Systems Coordinator
Documentation added to training file

Exhibit 9-2

Demonstration of Capability Certification Statement

Date: _____

Microseeps, Incorporated
220 William Pitt Way
Pittsburgh, PA 15238

Analyst Name: _____

Matrix: (laboratory pure water, soil, air, matrix spike)

Analyte (or group)	**Method Number**	**SOP #**	**Revision #**

I, the undersigned, CERTIFY that:

1. The analyst(s) identified above, using the cited test method (s), which is in use at this facility for the analyses of samples under the National Environmental Laboratory Accreditation Program, have met the Demonstration of Capability.

2. The test method was performed by the analyst(s) identified on this certification.

3. A copy of the test method and the laboratory-specific SOPs are available for all personnel on-site.

4. The data associated with the demonstration of capability are true, accurate, complete, and self-explanatory.

5. All raw data (including a copy of this certification form) necessary to reconstruct and validate these analyses have been retained at the facility, and that the associated information is well-organized and available for review by authorized assessors.

_____ _____
Patrick McLoughlin, Ph.D. Date
Technical Director

Exhibit 9-3

Microseeps, Incorporated

ETHICS AND DATA INTEGRITY AGREEMENT

I, _____, state that I understand the high standards of integrity required of me with regard to the duties I perform and the data I report in connection with my employment at **Microseeps, Incorporated.**

I agree that in the performance of my duties at Microseeps:

I. I shall not intentionally report data values that are not the actual values obtained;

II. I shall not intentionally report the dates and times of data analyses that are not the actual dates and times of data analyses; and

III. I shall not intentionally represent another individual's work as my own.

I agree to inform Microseeps of any accidental reporting of non-authentic data by myself in a timely manner.

I agree to inform Microseeps of any accidental or intentional reporting of non-authentic data by other employees.

I have read, acknowledge, and understand my personal ethical and legal responsibilities including potential punishments and penalties for improper, unethical, or illegal actions.

_____ _____

Signature Date

Appendix A

Key Personnel Job Descriptions

President and Chief Executive Officer

General Description: This exempt position is responsible for the overall administrative, technical, and financial direction of the corporation.

Educational Requirements: This position requires a graduate degree and/or fifteen or more years of experience in business management.

Functional Job Description

1. Performs the duties of the Chief Operational Officer as required.
2. Assumes financial responsibility and financial liability for the corporation.
3. Approves company policies and procedures.
4. Represents the company at trade shows.
5. Writes and presents research papers at environmental conferences.
6. Represents the corporation in negotiating major contracts.
7. Researches and considers the growth potential for future markets.
8. Keeping the company's focus on the technical edge of the environmental laboratory market.
9. Serves on the Board of Directors.
10. Committed to insure a framework is in place for the company to provide quality systems guidelines of the highest standard.
11. Other responsibilities as assigned by the Board of Directors.

Senior Vice President

General Description: This exempt managerial position is responsible for providing corporate management of the organization.

Educational Requirements: This position requires a graduate degree and/or ten or more years of experience in business management.

Functional Job Description

1. Administers and enforces company policies and procedures.
2. Produces plans and administrative procedures to maximize the efficiency of the corporation within established guidelines.
3. Oversees outsourced Human Resource, Information Technology, and Health and Insurance Benefits contracts.
4. Oversees Finance Department.
5. Maintains budgetary restraints for corporate profitability.
6. Supports the role of Quality Systems within the corporation.
7. Coordinates the administration of the Corporate Ethics Program and Policy.
8. Serves on the Board of Directors.
9. This position has contractual liability for the corporation.
10. Other duties and responsibilities as assigned by the President.

Vice President of Sales and Marketing

General Description: This exempt managerial position is responsible for directing the activities of the Sales and Marketing Staff. This position is ultimately responsible for meeting sales goals and reports directly to the President.

Educational Requirements: This position requires a Bachelor's Degree in Business or a related discipline and a minimum of ten years of business management experience.

Functional Job Description

1. Provide leadership and direction for the Sales and Marketing Staff.
2. Propose the yearly Sales and Marketing Budget to the Board of Directors.
3. Set and communicate reasonable target sales goals for sales staff.
4. Hold sales staff accountable for meeting target sales.
5. Assist the President and Senior Vice-President in developing short and long term sales and marketing strategies.
6. Communicate staffing needs for all related positions so that the sales division can efficiently handle workloads.
7. Responsible for sales staff's code of conduct and disciplinary action.
8. Act as liaison with other departments to ensure interdepartmental communication.
9. Serves on the Board of Directors.
10. Other duties and responsibilities as assigned by the President.

Vice President of Operations

General Description: This exempt managerial position is responsible for providing overall administration of the Operations Department.

Educational Requirements: This position requires a bachelor's degree and/or ten or more years of experience in operations management.

Functional Job Description

1. Oversee the duties and responsibilities of the department managers as required.
2. Administer and enforce company policies and procedures.
3. Develop administrative procedures to maximize the efficiency of operations within established company guidelines.
4. Oversee implementation of procedures to maximize the quality and efficiency of work performed.
5. Oversee effective subordinate development including but not limited to, training and career path development.
6. Maintains budgetary restraints for operational profitability and turnaround schedules.
7. Ensures that all technical assignments are carried out consistently within applicable professional and company quality standards.
8. Maximizes business opportunities within the current client base.
9. Supports the role of Quality Systems within the corporation.
10. Serves on the Board of Directors.
11. Other duties and responsibilities as assigned by the President.

Laboratory Manager

General Description: This exempt managerial position is responsible for overseeing the daily management of the laboratory operations and operations' staff. This position reports to the Vice President of Operations.

Educational Requirements: This position requires a Bachelor's Degree in Chemistry or a related field with a minimum of ten years of experience.

Functional Job Description

1. Provide supervision and direction to all Operations Department personnel.
2. Manage the size of the Operations Department efficiently in accordance with available workload and communicate staffing requirements to the Vice President Operations.
3. Responsible for laboratory code of conduct and staff disciplinary action.
4. Responsible for scheduling work and laboratory personnel to ensure efficient use of time and resources.
5. Assist the Vice President of Operations in developing short and long-range plans for laboratory improvement and growth.
6. Responsible for ensuring that the highest degree of technical quality possible is represented in outgoing final analytical data reports.
7. Ensure that laboratory work assignments are coordinated with work assignments of other groups in the company.
8. Represent the Operations Department in weekly meetings with other managers.
9. Work in conjunction with the Manager of Technical Services to provide input and support for instrumentation needs.
10. Coordinate with the Quality Systems Manager to develop and implement Standard Operating Procedures.
11. Support and enforce the specific methods, policies, and procedures outlined in Microseeps' Standard Operating Procedures and Quality Systems Manual.
12. Assist in the writing and review of laboratory Standard Operating Procedures.

13. Ensure MDLs, and PQLs are updated in the LIMS as required.

14. Responsible for the administration of the Chemical Hygiene Plan within the laboratory.

15. Perform other duties and responsibilities as assigned by the Vice President of Operations.

Quality Systems Manager

General Description: This exempt managerial position carries the responsibility for providing leadership and direction to ensure that the laboratory function at the highest level of quality possible in accordance with regulatory and certification requirements. The term Technical Director is use synonymously with the Quality Systems Officer.

Educational Requirements: This position requires a Bachelor's degree in chemistry or a related discipline and a minimum of ten years of laboratory experience.

Functional Job Description

1. Provide leadership and direction for the laboratory's Quality Systems Program.
2. Provide supervision and delegate tasks to the Quality Systems Coordinator.
3. Keep current on regulatory requirements to ensure that Standard Operating Procedures for analyses are in compliance with applicable regulations.
4. Conduct laboratory quality audits according to the annual schedule.
5. Serve as liaison between Microseeps and Regulatory Agencies.
6. Provide leadership and direction for developing and maintaining all Laboratory Standard Operating Procedures and the Quality Systems Manual.
7. Work with the Quality Systems Coordinator and the Laboratory Manager to discern and correct issues leading to poor quality and, if deemed necessary stop unsatisfactory work or prevent reporting of unjustifiable results.
8. Serve as client contact for questions that cannot be answered by the Customer Service Department or the Manager of Technical Services.
9. Other duties as assigned by the President and Chief Operating Officer.

Manager of Research and Development

General Description: The Manager of Research and Development is responsible for directing and managing an ongoing research and development program that insures the laboratory is ready to respond to leading edge technology needs.

Educational Requirements: This position requires a graduate degree in chemistry or a related discipline and a minimum of ten years of laboratory experience.

Functional Job Description

1. Provide assistance with interpretation of client data needs and data reports in cooperation with the Customer Service Department.

2. Assist clients in defining data needs, constructing sampling plans and interpreting data reports via non-binding suggestions and selected references to published documents.

3. Prepare and present at least two presentations per year at seminars, workshops, or conferences.

4. Develop written material for the use of Customer Service personnel to provide client assistance and education.

5. Identify emerging analytical needs and upon agreement with the Board of Directors, develop conceptual models for analytical methods to support those emerging client needs.

6. Develop conceptual analytical methods into routine procedures for bench chemists to implement.

7. Ensure that new method development is fully documented by compiling a complete package of experimental data in a form that is organized, summarized, and that can be validated.

8. When new analytical methods have become routine procedures, transfer responsibility to the Laboratory Manager.

9. Establish and maintain an awareness of the literature and the community of scientists and engineers that drive the market identified by the President and Chief Executive Officer.

10. Other duties as assigned by the President and Chief Executive Officer.

Manager Customer Service

General Description: This exempt managerial position is responsible for directing the activities of the Customer Service Staff, Bottle Preparation, and Sample Courier. This position is ultimately responsible for ensuring customer service satisfaction. This position reports directly to the Vice President of Operations.

Educational Requirements: This position requires a Bachelor's Degree and a minimum of four years of Customer Service experience.

Functional Job Description

1. Provide leadership and direction for the Customer Service Office, Bottle Preparation, Sample Courier, and EDD Specialist.
2. Assist the Vice President Sales and Marketing in developing short and long-term sales and marketing strategies.
3. Communicate staffing needs for all related positions so that the divisions can efficiently handle workloads to better ensure customer satisfaction.
4. Responsible for customer service code of conduct and disciplinary action.
5. Perform all of the functions of a Project Manager/Customer Service Representative.
6. Provide review of contracts.
7. Oversee purchasing for Bottle Preparation.
8. Act as liaison with other departments to ensure interdepartmental communication.
9. Ensure customer complaints are resolved to the customer's satisfaction.
10. Represent Customer Service at weekly Operations Meetings.
11. Communicate the importance of Customer Service within the company.
12. Review client projects and Lab Project Summary Sheets after projects are logged in, on a daily basis.
13. Other duties and responsibilities as assigned by the Vice President of Operations.

Manager of Technical Services

General Description: This exempt managerial position is responsible for overseeing the use of the laboratory facilities and instrumentation, and implementing new method development. This position reports to the Vice President of Operations.

Educational Requirements: This position requires a Bachelor's Degree in Chemistry or a related field with a minimum of ten years of experience.

Functional Job Description

1. Provide assistance for the technical review of client requests for chemical analysis as needed.

2. Respond to daily technical problems and questions on unusual samples at the request of the Laboratory Manager.

3. Evaluate the technical aspects and justify the cost of newly proposed instrumentation or existing instrument upgrades.

4. Maintain and repair of equipment and apparatus being used at Microseeps.

5. Provide support for equipment and data interfacing to the LIMS.

6. Recommend upgrades to laboratory apparatus to meet method requirements and to maintain Microseeps position as a leading-edge technology provider.

7. Reviews and participates in new hardware and software purchases for technical instrumentation.

8. Provide ongoing facility (plant) recommendations.

9. Any other duties as assigned by the Vice President of Operations.

Quality Systems Coordinator

General Description: This exempt position is responsible for assisting the Quality Systems Manager to ensure that the Quality Systems Policies and Procedures are implemented in accordance with the Quality Systems Manual and Microseeps Standard Operating Procedures. This position reports to the Quality Systems Manager.

Educational Requirements: This position requires a Bachelor's Degree and 2 years of laboratory experience.

Functional Job Description

1. Administratively responsible for the Quality Systems Program as directed by the Quality Systems Manager.
2. Prepare an annual schedule of laboratory audits.
3. Prepare audit reports and necessary corrective action to the Laboratory Manager.
4. Follow up on all corrective action reports until appropriate action has been taken.
5. Establish a schedule, order supplies, coordinate, and submit final analytical data for performance evaluation studies.
6. Maintain, control, and update the Quality Systems Manual and all of Microseeps' Standard Operating Procedures.
7. Ensure new Operation's Department employees receive required training and orientation and maintain the documentation of the training.
8. Ensure appropriate studies are conducted when necessary i.e. Initial Demonstrations of Proficiency and MDL's.
9. Calibrate all laboratory thermometers once a year.
10. Annually send out scale weights, radiation screening instrument, and NIST thermometer for calibration.
11. Coordinate annual inspections for certifications of balances and fume hoods.
12. Other duties as assigned by the Quality Systems Manager.

Procurement Coordinator

General Description: This exempt position is responsible for all aspects of purchasing and supply receipt and reconciliation. This position reports to the Vice President of Operations.

Education Requirements: This position requires a high school diploma and two years of laboratory experience. A basic knowledge of chemistry and laboratory analytical procedures is required.

Functional Job Description

1. Purchase, track, receive, and distribute supplies for the Operations Department.

2. Performs other duties and responsibilities as assigned by the Vice President of Operations.

Sample Custodian

General Description: This exempt position is responsible for all aspects of sample custody from sample receipt to storage until final disposal. This position reports directly to the Laboratory Manager.

Education Requirements: This position requires a high school diploma and two years of laboratory experience. A basic knowledge of chemistry and laboratory analytical procedures is required.

Functional Job Description

1. Receive and inspect samples and sample containers and sign appropriate documents according the Standard Operating Procedure for Sample Receiving and the Quality Systems Manual.

2. Record all necessary information on chain of custody.

3. In the event of any discrepancies or non-conformance issues with the above procedures, immediately complete a non-conformance form and submit it to customer service.

4. Notify Project Manager upon receipt of client samples when requested.

5. Accurately log samples into LIMS ensuring that all required fields are completed for the level of analysis required.

6. Label all sample containers.

7. Initiate transfer of samples as soon after receipt as possible to appropriate storage areas, ensuring that all samples are maintained at the appropriate temperature at all times.

8. Notify Lead Analysts immediately upon receipt of samples with short holding times. Complete non-conformance form for all samples that do not get picked up by analysts within the specified holding time.

9. Control and monitors access to and storage of samples in secure storage.

10. Performs other duties and responsibilities as assigned by the Laboratory Manager.

Lead Analyst

General Description: This exempt supervisory position assists in the daily supervision and training of staff analysts and technicians. This position reports directly to the Laboratory Manager.

Educational Requirements: This position requires a Bachelor of Science Degree in Chemistry or a related field and five years of related work experience, or a combination of college coursework in chemistry with a minimum of eight years of related work experience.

Functional Job Description

1. Conduct routine day to day technical training and supervision of analysts including proper Quality Assurance and Quality Control implementation and training.

2. Ensure that samples arriving with short holding times are analyzed within the applicable time frame.

3. Schedule analysis to efficiently meet holding times, and expedited and priority project schedules. Follow up with analysts to ensure turnaround times are met.

4. Review analytical data for completeness and accuracy. Institute corrective action when quality is found to be below the standards specified in the Standard Operating Procedures.

5. Properly collect, label, and store hazardous waste streams that are generated from the laboratory under the position's direct supervision. Coordinate with the Waste Coordinator for waste pick-up.

6. Ensure that the following logbooks are completed as required by Standard Operating Procedures: Temperature Logs, Balance Logs, Standards Preparation Logs, Run Logs, Sample Preparation Logs and Instrument Maintenance Logs.

7. Work with the Manager of Technical Systems to ensure preventative maintenance is conducted according to the maintenance schedule. Conduct daily preventative maintenance and minor repairs if able.

8. Keep laboratory stocked with supplies by submitting purchase orders to Procurement Coordinator in a timely manner.

9. Assure that all personnel under direct supervision practice quality control procedures in accordance with the Laboratory Quality Systems Manual and specific Standard Operating Procedures.

10. Enforce Health and Safety Procedures in the laboratory in accordance with the Chemical Hygiene Plan.

11. Work with the Health and Safety Officer to help correct suspected unsafe practices, situations, or working spaces.

12. Responsible for ensuring that all workspaces are maintained in a clean and well-organized fashion.

13. Performs other duties and responsibilities as assigned by the Laboratory Manager.

Staff or Laboratory Analyst

General Description: This exempt position is responsible for the timely analysis and custody of client samples. Job responsibilities cover a broad range of duties and responsibilities depending on employee education and experience. This position reports directly to the Lead Analyst.

Educational Requirements: This position covers a range of educational levels from a high-school education with on-the-job training to a Ph.D. in Chemistry or a related field. Analysts must pass Initial Demonstrations of Proficiency for every analytical method before independent analysis can be conducted.

Physical Requirements: This position requires the ability to lift 20 pounds and the ability to stand for extended periods of time.

Functional Job Description

1. In matters concerning order and priority of analysis, this position is accountable to, and will take direction from the Lead Analyst.
2. Keep all workspaces neat, clean, and organized.
3. Obtain samples from Sample Receiving as directed by the Lead Analyst.
4. Observe internal chain of custody requirements for all samples taken from Sample Receiving.
5. Ensure all aliquots analyzed are representative of the entire sample.
6. Analyze samples according to Microseeps Standard Operating Procedures (SOP) and or the applicable Standard or EPA Methods.
7. Observe and practice applicable quality control procedures in accordance with the Laboratory Quality Systems Manual and specific SOPs. This includes analyzing required quality control samples, ensuring results are within specified acceptance limits, and initiating corrective action when results are outside of acceptable parameters.
8. Analyze samples arriving with short holding times within the applicable time frame.
9. Review peer analytical data for completeness and accuracy where possible.
10. Responsible for making accurate entries into logbooks.

11. Conduct equipment preventative maintenance according to the Equipment Maintenance SOP.

12. Maintain supply inventory and notify lead analyst when laboratory supplies are needed.

13. Comply with all Health and Safety Procedures outlined in the Chemical Hygiene Plan.

14. Work with the Health and Safety Officer to help correct suspected unsafe practices, situations, or working spaces.

15. Identify any problems with samples, equipment, or procedures that will affect the integrity of analysis and report them to the Lead Analyst.

16. Performs other duties and responsibilities as assigned by the Lead Analyst or the Laboratory Manager.

Project Manager/Customer Service Representative

General Description: This exempt position is responsible for marketing, inside sales, and project management. This position reports to the Manager of Customer Service.

Educational Requirements: This position requires a minimum of five years of customer service experience in the laboratory, environmental, or chemistry field.

Functional Job Description

1. Generates quotations utilizing the LIMS.
2. Serves as the liaison between clients and the technical departments.
3. Provides supervision and direction to Bottle Preparation and Courier.
4. Takes bottle orders from clients.
5. Enter projects into the LIMS.
6. Manage projects as required according to client contracts.
7. Signs final data reports acknowledging that the reports are being sent.
8. Contact clients as required by Non-Conformance Forms.
9. Arrange rush and special analytical projects with Laboratory Manager.
10. Participate in conferences, trade shows, and other marketing activities.
11. Responsible for updating client databases with current information.
12. Provide initial review of contracts.
13. Serve as Microseeps representative to the client and as such, conduct themselves with the highest standard of ethics, is responsive to clients' needs, and portrays a level of professionalism for which Microseeps is known.
14. Create, generate, and disseminate company information to clients as requested.
15. Review invoices for accuracy.
16. Other duties as assigned by the Manager of Customer Service.

EDD Analyst/Customer Service Assistant

General Description: The EDD analyst position is responsible for the overall development, testing, maintenance, documentation, and support of all Electronic Data Deliverable (EDD) related processes, code and transactions. The Customer Service Assistant aspect of the position is responsible for specific customer service activities as they relate to the coordination of project work and consistently provides work on a timely basis.

Educational Requirements: This position requires High School education plus an Associate degree in Information Technology. Two years experience working with Electronic Data Interchange processes. Must also possess strong skills with SQL scripting and SQL Stored Procedures, as well as abilities with Microsoft SQL Server, Enterprise Manager, and Query Analyzer.

Functional Job Description

1. Provide technical support for all EDD related implementations.
2. Analyze client requirements, specify design, and develop new EDD solutions.
3. Modify and enhance existing EDD related code or procedures.
4. Provide customer support and correct reported problems with EDDs.
5. Utilize and develop procedures for EDD checking utilities, formatting applications and other tools as necessary.
6. Develop and publish documentation and procedures regarding proper uses and operations of the EDD environment as well as individual EDD requirements, structure, code, and results.
7. Perform training for responsible parties on the use of EDD related processes.
8. Work with customers, staff, external IT consultants and managers for both new development and problem determination/resolution.
9. Assure the highest levels of stability, integrity, reliability, performance, security, and availability of EDDs consistent with the resources available and the established change control policies, and prescribed procedures.

10. Use established user request and problem management tool to track requests and follow up to resolution and closure.

11. Perform special projects or other duties as required.

12. Review and check project files when received from login for correctness and completeness.

13. Review and prepare data packages by paginating data, scanning reports, and burning CDs.

14. Prepare and send Excel reports and invoices to clients electronically.

15. Receive and fill out bottle orders for clients when taking client calls.

16. Answering the telephone when required for customer satisfaction.

Bottle Preparation Technician

General Description: This non-exempt position consists of up to forty hours of work per week in completing bottle preparation activities. This position reports to the Customer Service Office.

Educational Requirements: High School Diploma with knowledge in basic chemistry. Computer skills are required, as well as, competencies in basic math, reading, and writing.

Physical Requirements: The position requires a maximum lifting capacity of 40 pounds with the ability to lift and carry large and bulky objects. The job skills also require a full range of motion for bending over and straightening.

Functional Job Description

1. Properly assemble, prepare, preserve, package and ship orders following Company Standard Operating Procedures in a cost effective and timely manner.

2. Inventory, order, track, and stock all necessary bottle preparation supplies.

3. Track, maintain, and fill all standing bottle orders for pick-up, delivery, and shipment.

4. Keep accurate records and files of standing orders, supply orders, and end of month inventories.

5. Track client packages that were lost or late via the telephone or by using the available software. (When given a computer/telephone)

6. Practice open communication in all instances with the Customer Service department in order to assure clients get the best possible service.

7. Maintain a neat, clean, and organized work and storage area.

8. Wear safety glasses while in bottle preparation room, and other protective clothing such as gloves and smocks when using chemicals, handling samples, and cleaning coolers.

9. Assist courier in sample collection, pick-up, or delivery when necessary.

10. Clean and dry all coolers that arrive prior to placing them in storage.

11. Obtain stock preservatives.

12. Other duties as designated by the Manager of Customer Service.

Appendix B

References

REFERENCES

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U.S. Environmental Protection Agency, 1983, Methods for Chemical Analysis of Water and Wastes, EPA-600/4-79-020, Environmental Monitoring and Support Laboratory, Cincinnati, Ohio.

Standard Methods for the Examination of Water and Wastewater, 20th ed., 1998.

National Environmental Laboratory Accreditation Conference, Quality Systems National Environmental Laboratory Accreditation Program, Rev. 16, July 12, 2002, Chapter 5.

Department of Defense Quality Systems Manual For Environmental Laboratories. Version 3.

Appendix C

Microseeps Ethics Program

Microseeps, Incorporated

Ethics Program

Approved by: _____ Date: _____
Thomas W. Hill

Reviewed by: _____ Date: _____

1.0 Introduction

At Microseeps, we are committed to operating with integrity. We value the integrity of the individual and the institution. We value honesty and trust in the way we treat one another and in the way we meet our commitments. Consistent with our commitment to integrity, our Ethics Program is based on values. For the program to succeed, each employee must use his or her own values to make decisions that reflect well on the employee and on the company.

This Program will encompass defining improper, unethical, and illegal actions, outline responsibilities, and ways in which the laboratory can prevent and/or detect these types of actions. This Program shall identify specific examples of improper, unethical, or illegal actions and establish potential punishments and penalties for the same. This Program shall include initial and annual ethics training.

The Statement of Ethical Responsibility (see Exhibit 1) shall be read and signed by every Microseeps' employee. This Program shall designate one individual to whom company personnel can report improper, unethical, or illegal practices.

2.0 Definitions

Improper Actions: Deviations from contract-specified or method-specified analytical practices and may be intentional or unintentional.

Unethical or Illegal Actions: The deliberate falsification of analytical or quality assurance results, where failed method or contractual requirements are made to appear acceptable (also known as laboratory fraud).

3.0 Responsibility

3.1 Laboratory Managers and Supervisors

The Laboratory management and supervisors at all levels are responsible for:

- Implementing the Ethics Program
- Providing clear guidelines on matters of everyday conduct to all employees.
- Stressing to all employees the need for a commitment in word and deed to the ethics policy and practices.
- Demonstrating their own commitment to the ethics policy and practices in the management of their area of responsibility and the activities of all employees under their supervision.

- Maintaining a workplace environment that encourages frank and open communication, free from the fear of reprisal, concerning the upholding of the ethics policy and practices.
- Distributing the Ethics Program to all employees.
- Developing and presenting training to ensure that all employees understand the laboratory's ethics principles and practices.
- Provide continuing counsel on laboratory practices, policies, rules, and regulations to any employee who seeks assistance.
- Maintaining working conditions supportive of employee responsibilities.
- Enforcing compliance with the ethics policy and practices.
- Recognizing employees who make exemplary efforts to implement and uphold the ethics policy and practices.
- Reviewing and investigating all allegations of wrongdoing.
- Ensuring that all current and new employees under their supervision have received a copy of the Ethics Policy and are trained in its meaning and applications.
- Reviewing the level of Ethics Policy knowledge and understanding of the employees under their supervision.

3.2 Employees

All employees are responsible for:

- Reviewing regularly their knowledge and understanding of the ethics policy and practices.
- Upholding the ethics policy and practices as demonstrated in their daily conduct.
- Contributing to a workplace environment that is conducive to the maintenance of the ethics policy and practices in daily activities.
- Seeking help when the proper course of action is unclear or unknown to them.
- Remaining alert and sensitive to situations that could result in actions by any employee that are improper, illegal, unethical, or otherwise in violation of the ethics policy and practices.
- Counseling fellow employees when it appears that they are in danger of violating the ethics policy and practices.
- Reporting violations of the ethics policy and practices to their supervisor, or higher-level management.
- Writing thorough case narratives explaining why analytical data may or may not be useful due to anything in the analytical process that may have been wholly or partially deficient.

4.0 Prevention and Detection

A key element in preventing fraud is the adoption of an ethics policy that is strictly enforced. Having employees that understand the difference between a mistake and improper behavior and

that are trained to make ethical decisions is one of the best prevention strategies. A strongly reinforced ethics-training program is one of the key cornerstones to an effective total ethical process.

4.1 Examples of Unethical Actions

There are a number of laboratory practices, which may constitute fraud. The following are some examples:

- Falsification of results to meet method requirements.
- Reporting of results without analyses to support (i.e. dry-labbing).
- Selective exclusion of data to meet QC criteria (initial calibration points dropped without technical or statistical justification).
- Misrepresentation of laboratory performance by presenting calibration data or QC limits within data reports that are not linked to the data set reported.
- Notation of matrix interference as a basis for exceeding acceptance limits (typically without implementing corrective actions) in interference-free matrices (e.g. method blanks or laboratory control samples).
- Unwarranted manipulation of computer software (e.g. improper background subtraction to meet ion abundance criteria for GC/MS tuning, chromatographic baseline manipulations).
- Improper alteration of analytical conditions (e.g. modifying EM voltage, changing GC temperature program to shorter analytical run time) from standard analysis to sample analysis.
- Misrepresentation of QC samples (e.g. adding surrogates after sample extraction, omitting sample preparation steps for QC samples, over-spiking or under-spiking).
- Reporting results from the analysis on one sample for those of another.

4.2 Prevention and Detection

The Quality Inquiry Form (QIF) can be used as a means to report improper, unethical, or illegal actions. The QIF should be completed and submitted anonymously (if desired) to the Technical Director. An immediate investigation shall be conducted by a member of Microseeps' management to ascertain if a violation did occur.

Other means of prevention and detection include an Internal Audit Program, an ethics-training program, and a requirement for analyst notation and sign-off on manual integration changes to data.

4.3 Disciplinary Action for Unethical Actions

Because the implications of unethical actions are substantial, swift and decisive disciplinary action shall be carried out for violations of the Ethics Policy. If an investigation reveals knowing and willful violation of laboratory ethics by an employee, that employee shall be terminated. Other violations can result in disciplinary actions that range from verbal warnings to written warnings with time off. The severity of the discipline shall match the severity of the offense. All ethics violations shall be documented in employee's personnel files.

5.0 Training

All employees shall receive initial and annual ethics training. The training shall be conducted by Microseeps' management personnel. The training shall include the contents of this policy in its entirety, employee and supervisory responsibilities, examples of unethical behavior, disciplinary action for unethical actions, and a means to report unethical actions.

5.1 Ethics Training Documentation

Training documentation shall be maintained in the Quality Systems Office. All employees are required to sign and date Exhibit 1, the Ethics, and Data Integrity Agreement. This form shall be placed in the employees personnel file.

5.2 Training Frequency

Initial training shall be conducted within two weeks of all initial hires and annually thereafter.

Microseeps, Incorporated

ETHICS AND DATA INTEGRITY AGREEMENT

I, _____, state that I understand the high standards of integrity required of me with regard to the duties I perform and the data I report in connection with my employment at **Microseeps, Incorporated.**

I agree that in the performance of my duties at Microseeps:

I. I shall not intentionally report data values that are not the actual values obtained;

II. I shall not intentionally report the dates and times of data analyses that are not the actual dates and times of data analyses; and

III. I shall not intentionally represent another individual's work as my own.

I agree to inform Microseeps of any accidental reporting of non-authentic data by myself in a timely manner.

I agree to inform Microseeps of any accidental or intentional reporting of non-authentic data by other employees.

I have read, acknowledge, and understand my personal ethical and legal responsibilities including potential punishments and penalties for improper, unethical, or illegal actions.

_____ _____

Signature Date

Microseeps, Incorporated

Analytical Method AM20GAx
Standard Operating Procedure for the Analysis of Biodegradation Indicator Gases

Controlled Copy No. _____

Ruth Welsh
Laboratory Manager

Signature of Final Approval:

Patrick McLoughlin, Ph D.
Technical Director

10-15-10
Effective Date

1.0 Scope and Application

Method AM20GAx is used to determine the concentration of biodegradation indicator gases in vapor samples. Specifically, Method AM20GAx is used to determine the dissolved concentration of the following gases:

Gases	CAS Number
Acetylene	74-86-2
Carbon dioxide	124-38-9
Oxygen	7782-44-7
Nitrogen	7727-37-9
Hydrogen	1333-74-0
Methane	74-82-8
Ethane	74-84-0
Ethene	74-85-1
Propane	74-98-6
Propene	115-07-1
n-Butane	106-97-8
i-Butane	75-28-5
Carbon Monoxide	630-08-0
Total Inorganic Carbon*	

*Total inorganic carbon (TIC) is converted to carbon dioxide using the steps outlined in SOP-PM01. The sample is then analyzed for carbon dioxide according to this SOP. Any differences in method are specified in the appropriate section.

This method is recommended for use by, or under the supervision of, analysts experienced in sample preparation, the operation of gas chromatographs and in the interpretation of chromatograms.

2.0 Method Summary

The sample gas is analyzed with a gas chromatograph capable of simultaneous analysis of all of the target analytes from a single gas sample. A single injection of gas from integral, simultaneously filled sample loops is used to assure consistent injection volume. The permanent gases are analyzed using a thermal conductivity detector (TCD). The light hydrocarbons are analyzed using a flame ionization detector (FID). Hydrogen is analyzed using a reduction gas detector (RGD). The data are transferred to a microcomputer, converted to digital format, stored, and processed using a chromatography data system.

2.1 Definitions

Batch: A batch consists of twenty or fewer samples.

Instrument Flush: The front end of the sample loop is flushed with ultra high purity helium injected into the loop directly from the cylinder to remove possible interference by ambient air and to avoid cross contamination between samples.

Method Blank: An injection analyzed by all three detectors that consists of ultra high purity helium. The method blank is free from the analytes of interest

Laboratory Control Sample: A sample of laboratory grade deionized water spiked with verified known amounts of analytes. A LCS is used to assess the performance of the measurement system.

Matrix Spike and Matrix Spike Duplicate: A sample prepared by adding a known concentration of target analyte to a specific amount of sample. Matrix spikes are used to determine the effect of sample matrix on a method's recovery efficiency.

3.0 Apparatus and Materials and Operating Conditions

3.1 Apparatus

Gas Chromatograph: The chromatographs designed and built by Microseeps are equipped with multiple packed columns and multi-port valves, a TCD, a FID, a RGD, and multiple sample loops. The FIDs, which were also built by Microseeps, are of a special design that allows considerably more sensitivity than commercially available models. This instrument provides rapid turn-around for consecutive analyses and a clean baseline for accurate, reproducible results.

3.2 Materials

- Sample vials (Supelco, Inc, Bellefonte, PA or equivalent)
- Syringe: locking gas tight (Hamilton/Alltech, 3, 5, 10, 30 and 60 ml or equivalent)
- Syringes: Disposable (60mL)

3.3.1 Interferences

The most likely source of "interference" is ambient air. Due to the relatively high concentrations of oxygen and nitrogen, a very small amount of air as a contaminant will dramatically affect the results. The analyst must take great care to ensure that air is flushed from the gas tight syringe

before sample preparation and that no air has entered the syringe or needle prior to injection of the sample into the gas chromatograph.

Contamination by carryover can occur whenever high-level and low-level samples are sequentially analyzed. An unrestricted flow (Instrument flush) of pure carrier gas from a 10 psig source should be allowed to flow through each sample loop for 30 seconds prior to each analysis.

As required, the analyst should demonstrate the absence of carryover contamination by analysis of the contents of the sample loop when purged with carrier gas. This demonstration should be performed when carryover contamination is suspected (after high samples). In the event that 'ghost peaks' (peaks similar to previous sample) appear when a pure carrier gas sample is analyzed (method blank), measures should be taken to eliminate the carryover contamination.

4.0 Reagents

- Helium (UHP Gas)
- Nitrogen (UHP Gas)
- Certified Commercial Gas Standards
- Benzalkonium chloride (BAK) solution – Prepared by dissolving 12.08 g into 1L DI water.
- Tri-sodium phosphate (TSP) – purchased as the dodecahydrate

4.1 Standard Preparation Procedures

Calibration standards are prepared by using the procedures below:

4.1. Vial Preparation

Headspace vials used for instrument calibration standards for this method are prepared as follows:

- Crimp and cap each vial, with stopper septa.
- Evacuate each vial to vacuum.
- Flush each vial to atmospheric pressure with the vial balance gas appropriate for the detector being calibrated. (See Table 4.1)

Table 4.1

Detector	Vial Balance Gas
FID	Nitrogen
TCD	Helium

RGD	Nitrogen

4.1.1 Preparing Calibration Standards

The instrument is initially calibrated (ICAL) using dilutions of custom certified gas mixes.

- Prepare the correct number of vials for the detector being calibrated.
- Each of the three detectors is calibrated with a gas mix from a commercial source.
- Remove the specified amount of standard by extracting it from the standard mix gas cylinder using a gas-tight syringe and injecting it into a prepared vial.
- Add the specified amount of vial balance gas to the same vial.

The dilution factor of one is achieved by directly injecting the standard gas mix from the cylinder into the GC.

4.1.2 Calibration Standard Concentrations

Calibration standards are made up in the following concentrations as specified in Tables 4.1.2 A, B, C, D, and E. The true values of the calibration standards vary slightly from cylinder to cylinder. The values below are very close approximations. All standards are prepared using headspace vials with stopper septum or serum bottles.

Table 4.1.2 A
Light Hydrocarbons by FID
(Methane, Ethane, Ethene, Butane, Propane, Propene)

Stock-1000ppmv Hydrocarbon Mix in Nitrogen from Matheson Tri-Gas, or equivalent.

Std Level	Conc. (PPMV)	Std	Make-up Gas
Working Std #2	40.0	8cc Stock	192cc (w/serum bottle)
Working Std #3	5.00	1cc Stock	199cc (w/serum bottle)
Level 1	1000	As received from cylinder	0
Level 2	200	10cc Stock	40cc
Level 3	40.0	2cc Stock	48cc
Level 4	8.00	10cc Working Sol #2	40cc

Level 5	2.00	2.5cc Working Sol #2	47.5cc
Level 6	0.500	5cc Working Sol #3	45cc
Level 7	0.125	1cc Working Sol #3	39cc
Std Level	**Conc. (PPMV)**	**Std**	**Make-up Gas**
Level 8	0.040	2cc Working Sol #3	248cc (w/serum bottle)

Table 4.1.2 B
Acetylene by FID

Stock-1000 PPMV Acetylene in Nitrogen, Matheson Tri-Gas, or equivalent.

Std Level	Conc. (PPMV)	Std	Make-up Gas
Working Sol #1	20.0	1cc Stock	49cc
Level 1	100	5cc Stock	45cc
Level 2	25	1cc Stock	39cc
Level 3	5.00	10cc Working Sol #1	30cc
Level 4	1.00	2.0cc Working Sol #1	38cc
Level 5	0.200	0.5cc Working Sol #1	49.5cc

Table 4.1.2 C
Hydrogen by RGD

Stock-100 PPMV Hydrogen in Nitrogen, Matheson Tri-Gas, or equivalent.

Level	Conc.	Std	Make-up Gas
Working Sol #4	2.00	1cc Stock	49cc
Level 1	50.0	21cc Stock	21cc
Level 2	20.0	10cc Stock	40cc
Level 3	10.0	5cc Stock	45cc
Level 4	5.00	2cc Stock	38cc
Level 5	2.00	1cc Stock	49cc

Level 6	0.500	10cc Working Sol #4	30cc
Level 7	0.200	4cc Working Sol #4	36cc

Table 4.1.2 D
Permanent Gases by TCD
(Oxygen, Carbon Dioxide, Nitrogen, Methane, Carbon Monoxide)

Stock-Multi-component Mix at various conc. in Nitrogen, Matheson Tri-Gas, or equivalent.

Level	Std	Make-up Gas
Working Sol #5	1cc Stock	49cc
1	As received from cylinder	0
2	21cc Stock	21cc
3	5.0cc Stock	45cc
4	1.0cc Stock	49cc
5	0.5cc Stock	49.5cc
6	10cc Working Sol #5	40cc

Table 4.1.2 E
Permanent Gases by TCD
(Carbon Dioxide, Methane, Ethane, Ethene)

Stock-Single component sources, 100% Stock by Matheson Tri-Gas, or equivalent.

Std Level	Conc. (PPMV)	Std	Make-up Gas
Working Sol #6	20,000	5cc each comp	230cc (w/serum bottle)
Level 1	200,000 CO_2 100,000 MEE	10cc CO_2 5cc MEE	25cc
Level 2	50,000	2.5cc each comp	40cc
Level 3	10,000	25cc Working Sol #6	25cc
Level 4	2,000	5.0cc Working Sol #6	45cc
Level 5	400	1.0cc Working Sol #6	49cc

4.2 Quality Control Sample Preparation

Quality control samples are prepared as indicated below.

4.2.1 Initial Calibration Verification (ICV) and Continuing Calibration Verification (CCV)

The ICV and CCV are prepared from a source different from the source used to prepare the ICAL standards. The concentration of the ICV and CCV is in the middle of the calibration range and is close to that of the ICAL midpoint, but because of the nature of gas standard it is not at exactly that concentration.

4.2.2 Laboratory Control Sample (LCS) and LCS Duplicate (LCSD)

The LCS and LCSD are prepared at a mid-range concentration. The type of LCS/LCSD depends upon the original matrix of the sample. For samples that arrive as vapors, the LCS/LCSD is injected as a gas. For samples that arrive as waters, DI water is spiked with a gas mixture of target analytes and prepared the same as the samples. Water that is free of the principle atmospheric components of nitrogen and oxygen is very difficult to make and similarly difficult to store. Toward that end, LCS/LCSD results for nitrogen or oxygen will not be reported with client data. Table 4.2.2 below gives the true values of the LCS/LCSDs.

4.2.2.1 Total Inorganic Carbon LCS

Mix approximately 0.20g $NaHCO_3$ into 200ml laboratory grade DI water, prepare according to the TIC procedures outlined in PM01 and analyze in duplicate as a sample. The true value of the spike is calculated as follows:

$$\text{mg/L } CaCO_3 = \frac{Mass(g)NaHCO_3}{H_2O(L)} X \frac{100.09}{84.01} X (1{,}000{,}000)$$

4.2.3 Matrix Spike (MS) and Matrix Spike Duplicate (MSD)

- For water samples, MS and MSDs are prepared, analyzed, and reported when clients' request and send sufficient numbers of aliquots to prepare them (e.g. one 40 ml vial each for the MS and another for the MSD).

Table 4.2.2

Compound	Vapor LCS/LCSD (ppmv)	Water LCS/LCSD & MS/MSD
Methane	300.0	825 µg/L
Ethane	100.0	45 µg/L
Ethene	100.0	41 µg/L
Propane	100.0	67 µg/L
Propene	100.0	60 µg/L
iso-Butane	100.0	82 µg/L
n-Butane	100.0	85 µg/L
Acetylene	-------	36 µg/L
Carbon dioxide	50,000	130 mg/L
Oxygen	20,000	***
Nitrogen	balance gas	balance gas
Hydrogen	25.00	69 nM
Carbon Monoxide	-------	2.2 mg/L

Notes on Table 4.2.2

- Since oxygen is an ubiquitous "contaminant", it is not monitored in either the LCS or MS.
- Actual values vary slightly from lot to lot of cylinders of calibration gases.
- MS/MSD prepared by using a standard gas mix instead of He in the headspace prep. procedure.

4.2.3.1 Total Inorganic Carbon MS and MSD

Mix approximately 0.04g $NaHCO_3$ directly into client samples (when provided and requested), prepare according to the TIC procedures outlined in PM01 and analyze in duplicate as a sample. The true value of the spike is calculated as follows:

$$\text{mg/L } CaCO_3 = \frac{Mass(g)NaHCO_3}{H_2O(L)} X \frac{100.09}{84.01} X (1{,}000{,}000)$$

4.2.4 Method Blank

Method blanks are made up of ultra high purity helium injected into a vial and then into the instrument.

4.2.4.1 Total Inorganic Carbon Method Blank

The method blank for TIC is made up of deionized water in a 40 ml vial, prepared according to the TIC procedures outlined in PM01, and analyzed as a sample.

4.3 Glassware and Storage Requirements for Reagents and Standards

Reagents are stored at room temperature (70°F ±5°) and all standards are prepared fresh for each use immediately prior to each analysis. Standards are made up from compressed gas cylinders. Those standards expire after 2 years.

5.0 Procedure

Water samples should be cooled upon collection and stored at a temperature of just above freezing but below 6°C.

Gas samples are shipped and received at a positive pressure, which eliminates a cross-contamination issue during sample shipment. It is preferable that gas samples be shipped without cooling. However, it is not a sample receipt non-conformance if received vapor samples are packed in ice (sample may experience slight loss in pressure.) Gas samples are stored in the laboratory at room temperature (70°F ±5°). The pressure in gas vials is not checked upon receipt in the laboratory because of the inherent risk of losing sample, or inadvertently introducing atmospheric gases, when the septum is pierced. The number of times the septum is pierced should be as few as absolutely possible. See Section 5.2.2 for a discussion on how the laboratory checks and documents vial pressure. Holding time for both gas and water samples is fourteen days.

Water samples for light hydrocarbon analyses only (methane, ethane, ethane, propane, propene, n-butane, i-butane, acetylene) are collected in 40ml VOA vials with zero headspace and preserved with tri-sodium phosphate (TSP). TSP is added as the dodecahydrate at 200 mg/40 ml vial. This results in a sample pH > 10. Water samples collected for either permanent gases only or permanent gases and light hydrocarbon analyses are collected in 40ml amber VOA vials with zero headspace and preserved with four drops of BAK solution.

Analysts who use this method have been certified for the method by running Initial Demonstration of Proficiency (IDOP) Samples in accordance with Microseeps Standard Operating Procedure for Administering and Documenting Training in Laboratory Procedures and

Instrumentation (SOP ADM 02). IDOPs are run any time there is significant change to an instrument, method, or in the training procedure for training a new analyst.

Because the results from this method frequently require the analyst to use manual integration, manual integration is included as part of the training. Because of the nature of the instrument, the range of interrogated concentrations and the low specificity of the detectors, it is often necessary to perform manual integration even on the laboratory control samples. As part of the training, the analysts must:

- Retain an electronic copy of the original chromatogram that was integrated by the automated settings of the instrument software. (This is done automatically by the chromeleon software.)
- Document on the hard copy Case Narrative a justification for the manual integration and circle "YES" in the box in the lower right corner of the narrative sheet.
- The analyst shall present all the data to the Lead Analyst (if not applicable, then the Laboratory Manager) for review.
- The Lead Analyst or Laboratory Manager shall thoroughly examine the data and when satisfied, check the appropriate box on the case narrative form and place their initials where designated.
- If there are questions about the manual integration, the data reviewer shall review the original chromatogram from the data system.
- If agreement is obtained from the Lead Analyst or Laboratory Manager that the manual integration was indeed necessary, the Lead Analyst or Laboratory Manager shall document on the same hard copy Case Narrative (lower left corner) that the manual integration was reviewed and the justification stands. If the other criteria of the training are met, the training is deemed successful.
- If the reviewer disagrees with either the necessity of the integration or the specific manipulations done in the integration, the specifics objections should be discussed with the trainee and the training should be repeated (4 new samples must be analyzed).

5.1 Sample Preparation

Samples that are collected using the Bubble Strip Sampling Technique, Microseeps Sampling Method SM9, do not require additional preparation prior to analysis.

Samples that are collected as waters and are to be analyzed for dissolved gases (methane, ethane, ethene, acetylene, CO_2, N_2, O_2, propane, propene, iso-butane, n-butane, TIC), must be prepared using Microseeps Standard Operating Procedure PM01G.

Samples that are collected as gases, for example from a soil gas survey or from the headspace of a microcosm sample, need not be collected by a Microseeps sampling method, nor do they require additional preparation.

5.2 Analysis

5.2.1 If the sample is prepared via SOP-PM 01, it can be injected from the gastight syringe in which it is prepared by inserting the needle of the syringe through the septum on the "sample in" port. If the sample is a calibration standard, a bubble strip sample (SM9), or a gas, the septum inlet to the "sample in" port of the GC must be removed and a luer-lock needle receptacle is plumbed to the "sample in" port in place of the needle. A needle is attached to the luer-lock receptacle and inserted through the septa of the calibration standard, bubble stripped sample, or gas sample.

5.2.2 In order to initiate analysis and introduce the sample into the GC sample loop, a needle is attached to the entry port on the GC and inserted through the sample septum. The flow through the sample loop is monitored by a flow meter connected to the sample-loop vent-port on the gas chromatograph.

When a vial is sufficiently filled, the ball in the flow meter will shoot to the top of the column. This indicates that there is sufficient pressure in the vial to fill the sample loop. If the loop is not properly pressurized, this is reflected on the flow meter immediately. The ball in the flow meter will go up the column part way and drop back to the bottom. This indicates there is not sufficient pressure in the sample vial. If this happens, the analyst will remove the vial from the inlet port as quickly as possible and withdraw 10 – 12ccs of sample from the sample vial using a locking syringe. This is then injected into the instrument. The lack of sufficient pressure in the vial and the means of sample injection are then documented on the case narrative.

5.2.3 Once the flow out of the sample loop ceases (3 seconds if SOP-PM 01 is used) the sample loop valves are activated.

5.2.4 Once the sample loop valves are activated, the ports to and from the sample loop are flushed with ultra high purity helium injected into the loop directly from the cylinder to remove any interference from ambient air and to avoid cross contamination between samples.

5.3 Calibration and Results

5.3.1 The standard calibration gas should be introduced in the same manner as described in section 5.2.1 above. Measured peak areas are converted to concentrations using certified commercial gas standards. Dilutions are made to achieve multi-point calibration curves for each detector.

5.3.2 Initial calibration is accomplished by analyzing multiple standards of appropriate calibration ranges.

Note: Due to the nature of preparing custom gas standards, the component concentration can fluctuate between purchased lots. This is accounted for during method/calibration development. These results will be used to establish a multi-point calibration curve.

Acceptance Criteria: A linear fit to an area response versus concentration plot is formed with the origin forced to zero, and the calibration is accepted for use if r^2, the coefficient of determination is ≥ 0.995. If this criterion can not be met using a linear fit, a quadratic can be used. For the quadratic fit, the acceptance criteria is also $r^2 \geq 0.995$.

Corrective Action: If the acceptance criteria specified above is not met, the reason is determined and a new set of calibration standards are analyzed.

5.3.3 An Initial Calibration Verification (ICV) standard immediately follows the initial calibration. Acceptance criterion for the ICV is an instrument response within ± 15% drift. Since the instrumentation used at Microseeps routinely monitors the percent recoveries and in this instance percent drift is equal to percent recovery less 100%, the control limits are 85%-115% recovery for the ICV.

$$Percent\ \mathrm{Re}\,\mathrm{cov}\,ery = \frac{MeasuredValue}{TrueValue} \times 100\%$$

Acceptance Criteria and Corrective Action: If the instrument response for the ICV standard is outside the acceptance window of 85-115%, the analyst will not analyze samples until either the reason is determined and the problem is corrected, or a new multi-point calibration is analyzed and an acceptable ICV is run using that calibration.

5.3.4 An initial calibration blank follows the ICV. The blank is made up of the carrier gas. Compounds must not be detected above the reporting limits. For DoD projects the results of the ICB must be < ½ RL.

Corrective Action: If the blank does not meet the acceptance criterion, another blank is injected until the results are within the acceptance criterion.

5.3.5 The analytes of this method are indicators. Every attempt to achieve and deliver precise results is made. However, it is realized that for indicator parameters measuring the range of the analyte concentration (*i.e.* is the concentration of methane gas >1 mg/l or < 0.1 mg/l) is the primary goal of employing these analyses. The calibration range is chosen to extend over most of the bio-indicator concentration range. If the concentration of an analyte exceeds that of the highest calibration standard, but does not saturate the instrument response, the concentration is calculated by assuming detector response linearity and using an extrapolation of the calibration plot. If the instrument response is saturated the sample is diluted to bring the analyte concentration into the calibration range.

5.4 Quality Control

The following quality control samples shall be analyzed with each analytical batch of fifteen or fewer samples.

5.4.1 A Continuing Calibration Verification: The CCV is made up from a source other than what was used to make up the initial calibration. The acceptance criterion for the CCV is a percent recovery of 85-115%. The CCV is also analyzed at the beginning and end of each analytical shift and after every 15 samples.

Corrective Action: If the CCV fails, a new CCV is prepared and analyzed. If the new CCV falls within the acceptance criterion, analysis continues. If the new CCV fails, the instrument shall be recalibrated, and all samples since the last acceptable calibration verification shall be reanalyzed, provided sufficient sample volume is present and the samples have not been compromised by exposure to air.

5.4.2 A Continuing Calibration Blank: A CCB follows each CCV. The blanks are made up of the carrier gas. The acceptance criterion for the blank is the result must be less than the reporting limits for all compounds. For DoD projects the results for the CCB must be < ½ RL.

Corrective Action: If the blank does not meet the acceptance criterion, another blank is injected until the results are within the acceptance criterion.

5.4.3 Laboratory Control Sample and Laboratory Control Sample Duplicate: The LCS and LCSD are prepared and analyzed at a mid-calibration range. Both an LCS and an LCSD are to be run with each batch.

Acceptance Criteria: Percent recovery is required to be between 80-120%, inclusive. An acceptance criterion is based upon the percent recovery and the RPD as calculated by:

$$Percent\,\mathrm{Recovery} = \frac{MeasuredValue}{TrueValue} \times 100\%$$

$$RPD = \frac{|C1 - C2|}{\frac{C1 + C2}{2}} \times 100\%$$

Where**:** C1=LCS
C2=LCSD

RPD (Relative Percent Difference) is required to be less than or equal to 20%.

Corrective Action: If the LCS fails, a new LCS is prepared and analyzed. If the new LCS falls within the acceptance criterion, analysis continues. If the new LCS fails, analysis is stopped and the instrument is checked with a series of standards to determine the cause. Once the cause is determined and the instrument repaired, calibration is conducted and analysis continues.

5.4.4 Matrix Spike and Matrix Spike Duplicate: Matrix spikes and spike duplicates are analyzed for water samples only when requested by a client and sufficient sample aliquots are provided. Acceptance criterion is a percent recovery between 70% and 130% and a relative percent difference (RPD) of less than or equal to 20%.

Corrective Action: If the matrix spike and spike duplicate fail but all the other quality control samples are within the acceptance criteria, matrix interference is noted in the Case Narrative.

5.4.5 Method Blank: A method blank is analyzed with each sample batch. The blanks are made up of UHP helium for all of the gases except for blanks for TIC. TIC blanks are made up of deionized water. The acceptance criterion for the blank is the result must be less than the reporting limits for all compounds. For DoD projects the results for the method blank must be < ½ RL.

Corrective Action: If the blank does not meet the acceptance criterion, another blank is injected until the results are within the acceptance criterion.

5.4.6 Contingency for Handling Out of Control or Unacceptable Data

If the requirements set forth in section 5.4 are not met, the analytical program will be terminated until the cause is determined and a solution is affected. All samples associated with out of control quality control samples (with the exception of matrix interference) must be reanalyzed provided another vial of sample has been provided by the client. If quality control acceptance criteria cannot be met using the corrective action above, a detailed check of the analytical system is made. Reagents, standards, and other quality control samples are re-prepared and analyzed. If problems persist, sample analysis will be halted and the Laboratory Manager shall be contacted immediately to determine the cause and implement corrective action.

Any data submitted with unacceptable quality control sample results shall be qualified in a case narrative. The narrative should indicate the out of control event that occurred, the corrective action that was taken, and any other pertinent information to inform the client of exactly what occurred.

5.4.7 An experienced analyst shall examine all chromatograms.

5.4.8 Through out analysis the gas samples are injected mechanically into the GC flow path utilizing a sample loop to achieve a uniform sample size from a flow directly from the sample preparation syringe. The uniform sample size achieved using the sample loop assures consistent and accurate results. Table 5.4.8 (see next page) gives example data from a study performed via this analysis. That data can also be used for accuracy and precision estimates.

Table 5.4.8
Example Data for Precision and Accuracy Studies

	Carbon Dioxide	Oxygen	Nitrogen	Methane	Hydrogen	Methane	Ethane	Ethylene	Propane	Propylene	Iso-Butane	N-Butane
REP. #	(%v)	(%v)	(%v)	(%v)	(PPMV)	(PPMV)	(PPMV)	(PPMV)	(PPMV)	(PPMV)	(PPMV)	(PPMV)
1	0.1221	0.0670	0.5744	0.0410	0.1118	0.2512	0.0525	0.0453	0.0461	0.0581	0.0473	0.0358
2	0.1267	0.0690	0.6020	0.0428	0.1122	0.2608	0.0518	0.0468	0.0521	0.0465	0.0439	0.0407
3	0.1207	0.0657	0.5838	0.0446	0.1247	0.2812	0.0509	0.0485	0.0529	0.0588	0.0436	0.0405
4	0.1193	0.0667	0.6036	0.0444	0.1244	0.2779	0.0549	0.0460	0.0461	0.0536	0.0549	0.0476
5	0.1261	0.0703	0.5860	0.0439	0.1120	0.2894	0.0551	0.0497	0.0520	0.0549	0.0417	0.0460
6	0.1193	0.0665	0.5861	0.0478	0.0943	0.2970	0.0515	0.0467	0.0458	0.0542	0.0435	0.0514
7	0.1227	0.0732	0.5748	0.0353	0.1296	0.3053	0.0532	0.0473	0.0485	0.0584	0.0483	0.0535
AVERAGE	0.1224	0.0683	0.5872	0.0428	0.1156	0.2804	0.0528	0.0472	0.0491	0.0549	0.0462	0.0451
KNOWN	0.1500	0.0700	0.6649	0.0450	0.0999	0.1500	0.0500	0.0500	0.0500	0.0500	0.0500	0.0500
STD. DEV.	0.003	0.003	0.012	0.004	0.012	0.019	0.002	0.001	0.003	0.004	0.004	0.006

5.4.9 The gas matrix for this analysis minimizes the opportunity for matrix effects. If the gas is prepared from a matrix other than that which is injected into the GC (*e.g.* prepared through headspace extraction via Microseeps SOP-PM01), the client should request that matrix spike (MS) and matrix spike duplicate (MSD) analyses be conducted and should supply sufficient sample volume. Since matrix effects are extremely site dependent, the MS and MSD are not part of the regular analytical quality assurance program.

5.4.10 All of the target analytes gases are at room temperature so the opportunity for carry over is small. This is further reduced by the flushing of the sample loop, by the "backflush" configuration of the GC plumbing, and by the nightly bake-out procedure. These combine to keep carry-over concentrations to less than half of the reporting limits.

5.5 Capturing and Submitting Data

The output of the chromatograph is directed to a microcomputer where the signal is converted to digital format, stored, and processed using a chromatography data system.

Automated valve control: Digital control is provided by the microcomputer though the chromatography data-system software. This control provides constant start and stop times for directing carrier gas flow. The event times are programmed and saved using the method editor module of the software.

5.5.1 Total Inorganic Carbon Result Calculation

The total inorganic carbon result is calculated as follows:

TIC as mg/L $CaCO_3$= (%CO_2)((Volume headspace)(2.08)+43.3)

This analysis produces concentration of the analyzed gas in % V.

5.5.2 Retention Time Windows

Retention time studies have been conducted for this analysis. These studies are kept on file in the Quality Systems Office. The exact retention times will vary as a function of column type, column age, and column history. For the instruments that use this method, true retention times and retention time windows are taken from the most recent standard analyzed.

5.5.2.1 Determination of Retention Time Windows

Inject a standard a total of 3 times over a 72 hour period. Record the retention time for each component to a minimum of 3 decimal places. Calculate the mean and standard deviation of the three absolute retention times for each component. If the standard deviation for a target compound is 0.000, use a default standard deviation of 0.01 minutes. The width of the retention time window for each analyte is ±3 times the standard deviation of the mean absolute retention

time. If the default standard deviation 0.01 minutes is used, the width of the window will be 0.03 minutes.

Establish the center of the retention time window for each analyte by using the absolute retention time for each analyte from the calibration verification standard at the beginning of the batch.

Retention time windows must be calculated for each instrument and column used. New retention time windows must be established when a new column is installed.

5.6 Bake-out Procedure

Either overnight, through the weekend or whenever the instrument is not going to be used for several hours, the instrument is put in "bake-out". With carrier gas continuous flushing through the GC, the temperature on the oven is manually turned up to 210 degrees or as high as the instrument column oven can maintain.

6.0 Secondary Data Review

All analytical data must undergo a minimum of a two-tiered review. The analyst first reviews the data for completeness and accuracy. The data is then submitted to the Group Lead Analyst for final review and the data is entered into the LIMS. Once approved at this level, the data is uploaded into the LIMS.

7.0 Reporting Limits

The reporting limits for this analysis are listed in Table 7.0 below. Method detection limit studies are run annually in accordance with Microseeps Standard Operating Procedure for the Determination of Method Detection Limits and PQLs (SOP-ADM 18).

Those MDLs must be less than the reporting limits specified below. MDL studies are also performed when there is reason to suspect that method sensitivity has changed. The MDL studies are kept on file in the Quality Systems Office.

Reporting Limits
Table 7.0

Parameter	Reporting Limit	Units
Carbon Dioxide	0.2	%V
Oxygen	0.1	%V
Nitrogen	0.1	%V
Hydrogen	0.5	ppmv
Parameter	**Reporting**	**Units**

	Limit	
Acetylene	0.1	ppmv
Methane	0.2	ppmv
Ethane	0.02	ppmv
Ethene	0.03	ppmv
Propane	0.05	ppmv
Propene	0.1	ppmv
n-Butane	0.07	ppmv
i-Butane	0.05	ppmv

7.1 Conversion Factors

This procedure is used to measure the volume concentration of the analytes in a gas. Two methods are used to extract that gas from the groundwater. The conversion factors that are used to convert the concentration of the analytes in the water from the concentration of the analytes as they are measured using this method, are specific to the collection or preparation method and can be found in either SOP-SM9 or SOP-PM 01.

8.0 Safety

Gloves, proper eye protection, and a laboratory coat shall be worn when handling samples and standards. The major hazard in this laboratory area is stick from needles. All needles must be capped when not in use and when moving about the laboratory. The proper way of capping a needle is to place the cap on the laboratory bench and direct the needle into the cap. A needle is never to be directed into a cap while the cap is being held.

All compressed gases are to be moved using a dolly made for transporting gases and shall be chained in place when in the laboratory. The chain shall be tightened sufficiently to keep the cylinder upright if jostled.

9.0 Laboratory Waste

Samples are kept for 30 days following analysis. Samples are disposed according to Microseeps Standard Operation Procedure for Waste Disposal (SOP-ADM 14).

9.1 Waste Minimization

Where possible, Microseeps takes steps to minimize the amount of waste generated in the laboratory by using substitution, where possible, and good chemical handling procedures. For specific information on waste minimization consult SOP-ADM 14.

10.0 References

Citing a reference does not imply that all of the recommendations and/or requirements in those cited methods is required in this Standard Operating Procedure. This section simply refers to sources that were consulted to gather information or knowledge in order to write an informed technical procedure.

U.S. Environmental Protection Agency, Test Methods for Evaluating Solid Waste. SW-846, 3rd ed., Office of Solid Waste and Emergency Response, Washington, DC. 1986.

Newel, B.S., RSK-SOP-175, Sample Preparation and Calculations for Dissolved Gas Analysis in Water Samples using GC Headspace Equilibration Technique. Revision No. 0, August 1994.

American Society for Testing and Materials, Standard Practice for Analysis of Reformed Gas by Gas Chromatography. Annual Book of ASTM Standards. Vol. 14.02, 1994.

Kampbell, D.H. and Vandegrift, S.A., Analysis of Dissolved Methane, Ethane, and Ethylene in Ground Water by a Standard Gas Chromatographic Technique. Journal of Chromatographic Science. Vol. 36, May 1998.

SOP Review Date: October 12, 2010

Appendix D

SW-846 QC Requirements from the Department of Defense Quality Systems Manual, Version 4.2

Table F-4. Organic Analysis by Gas Chromatography/Mass Spectrometry (Methods 8260 and 8270)					
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action**	**Flagging Criteria**	**Comments**
Demonstrate acceptable analytical capability	Prior to using any test method and at any time there is a significant change in instrument type, personnel, test method, or sample matrix.	QC acceptance criteria published by DoD, if available; otherwise, method-specific criteria.	Recalculate results; locate and fix problem, then rerun demonstration for those analytes that did not meet criteria (see Section C.1.f).	NA.	This is a demonstration of analytical ability to generate acceptable precision and bias per the procedure in Appendix C. No analysis shall be allowed by analyst until successful demonstration of capability is complete.
LOD determination and verification (See Box D-13)					
LOQ establishment and verification (See Box D-14)					
Tuning	Prior to ICAL and at the beginning of each 12-hour period.	Refer to method for specific ion criteria.	Retune instrument and verify. Rerun affected samples.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be accepted without a valid tune.
Breakdown check (DDT Method 8270 only)	At the beginning of each 12-hour period, prior to analysis of samples.	Degradation ≤ 20% for DDT. Benzidine and pentachlorophenol should be present at their normal responses, and should not exceed a tailing factor of 2.	Correct problem then repeat breakdown check.	Flagging criteria are not appropriate.	No samples shall be run until degradation ≤ 20%.

Table F-4. Organic Analysis by Gas Chromatography/Mass Spectrometry (Methods 8260 and 8270) (continued)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Minimum five-point initial calibration (ICAL) for all analytes	ICAL prior to sample analysis.	1. Average response factor (RF) for SPCCs: VOCs ≥ 0.30 for chlorobenzene and 1,1,2,2-tetrachlorolethane; ≥ 0.1 for chloromethane, bromoform, and 1,1-dichloroethane. SVOCs ≥ 0.050. 2. RSD for RFs for CCCs: VOCs and SVOCs ≤ 30% and one option below: Option 1: RSD for each analyte ≤ 15%; Option 2: linear least squares regression r ≥ 0.995; Option 3: non-linear regression–coefficient of determination (COD) $r^2 \geq$ 0.99 (6 points shall be used for second order, 7 points shall be used for third order).	Correct problem then repeat ICAL.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until ICAL has passed. Calibration may not be forced through the origin.
Second source calibration verification (ICV)	Once after each ICAL.	All project analytes within ± 20% of true value.	Correct problem and verify second source standard. Rerun second source verification. If that fails, correct problem and repeat ICAL.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until calibration has been verified.
Retention time window position establishment for each analyte and surrogate	Once per ICAL.	Position shall be set using the midpoint standard of the ICAL curve when ICAL is performed. On days when ICAL is not performed, the initial CCV is used.	NA.	NA.	

Table F-4. Organic Analysis by Gas Chromatography/Mass Spectrometry (Methods 8260 and 8270) (continued)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Evaluation of relative retention times (RRT)	With each sample.	RRT of each target analyte within ± 0.06 RRT units.	Correct problem, then rerun ICAL.	Flagging criteria are not appropriate.	Laboratories may update the retention times based on the CCV to account for minor performance fluctuations or after routine system maintenance (such as column clipping). With each sample, the RRT shall be compared with the most recently updated RRT. If the RRT has changed by more than ±0.06 RRT units since the last update, this indicates a significant change in system performance and the laboratory must take appropriate corrective actions as required by the method and rerun the ICAL to reestablish the retention times.
Continuing calibration verification (CCV)	Daily before sample analysis and every 12 hours of analysis time.	1. Average RF for SPCCs: VOCs ≥ 0.30 for chlorobenzene and 1,1,2,2-tetrachlorolethane; ≥ 0.1 for chloromethane, bromoform, and 1,1-dichloroethane. SVOCs ≥ 0.050. 2. %Difference/Drift for all target compounds and surrogates: VOCs and SVOCs ≤ 20%D (Note: D = difference when using RFs or drift when using least squares regression or non-linear calibration).	DoD project level approval must be obtained for each of the failed analytes or corrective action must be taken. Correct problem, then rerun calibration verification. If that fails, then repeat ICAL. Reanalyze all samples since last acceptable CCV.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to all results for the specific analyte(s) in all samples since last acceptable CCV.	Problem must be corrected. Results may not be reported without a valid CCV. Flagging is only appropriate in cases where the samples cannot be reanalyzed.

Table F-4. Organic Analysis by Gas Chromatography/Mass Spectrometry (Methods 8260 and 8270) (continued)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Internal standards verification	Every field sample, standard, and QC sample.	Retention time ± 30 seconds from retention time of the midpoint standard in the ICAL; EICP area within -50% to +100% of ICAL midpoint standard.	Inspect mass spectrometer and GC for malfunctions. Reanalysis of samples analyzed while system was malfunctioning is mandatory.	If corrective action fails in field samples, apply Q-flag to analytes associated with the non-compliant IS. Flagging criteria are not appropriate for failed standards.	Sample results are not acceptable without a valid IS verification.
Method blank	One per preparatory batch.	No analytes detected > ½ RL and > 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater). Blank result must not otherwise affect sample results. For common laboratory contaminants, no analytes detected > RL (see Box D-1).	Correct problem, then see criteria in Box D-1. If required, reprep and reanalyze method blank and all samples processed with the contaminated blank.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch.	Problem must be corrected. Results may not be reported without a valid method blank. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
LCS containing all analytes to be reported, including surrogates	One per preparatory batch.	QC acceptance criteria specified by DoD, if available. Otherwise, use in-house control limits. In-house control limits may not be greater than ± 3 times the standard deviation of the mean LCS recovery. See Box D-3 and Appendix G.	Correct problem, then reprep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available (see full explanation in Appendix G).	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.	Problem must be corrected. Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Matrix Spike (MS)	One per preparatory batch per matrix (see Box D-7).	For matrix evaluation, use LCS acceptance criteria specified by DoD, if available. Otherwise, use in-house LCS control limits.	Examine the project-specific DQOs. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met.	For matrix evaluation only. If MS results are outside the LCS limits, the data shall be evaluated to determine the source of difference and to determine if there is a matrix effect or analytical error.

Table F-4. Organic Analysis by Gas Chromatography/Mass Spectrometry (Methods 8260 and 8270) (continued)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Matrix spike duplicate (MSD) or sample duplicate	One per preparatory batch per matrix (see Box D-7).	MSD: For matrix evaluation, use LCS acceptance criteria specified by DoD, if available. Otherwise, use in-house LCS control limits. MSD or sample duplicate: RPD ≤ 30% (between MS and MSD or sample and sample duplicate).	Examine the project-specific DQOs. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met.	The data shall be evaluated to determine the source of difference.
Surrogate spike	All field and QC samples.	QC acceptance criteria specified by DoD, if available. Otherwise, use in-house control limits.	For QC and field samples, correct problem then reprep and reanalyze all failed samples for failed surrogates in the associated preparatory batch, if sufficient sample material is available. If obvious chromatographic interference with surrogate is present, reanalysis may not be necessary.	Apply Q-flag to all associated analytes if acceptance criteria are not met.	Alternative surrogates are recommended when there is obvious chromatographic interference.
Results reported between DL and LOQ	NA.	NA.	NA.	Apply J-flag to all results between DL and LOQ.	

Table F-7. Inorganic Analysis by Inductively Coupled Plasma (ICP) Atomic Emission Spectrometry and Atomic Absorption Spectrophotometry (AA) (Methods 6010 and 7000 Series)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Demonstrate acceptable analytical capability	Prior to using any test method and at any time there is a significant change in instrument type, personnel, test method, or sample matrix.	QC acceptance criteria published by DoD, if available; otherwise, method-specified criteria.	Recalculate results; locate and fix problem, then rerun demonstration for those analytes that did not meet criteria (see Section C.1.f).	NA.	This is a demonstration of analytical ability to generate acceptable precision and bias per the procedure in Appendix C. No analysis shall be allowed by analyst until successful demonstration of capability is complete.
LOD determination and verification (See Box D-13)					
LOQ establishment and verification (See Box D-14)					
Instrument detection limit (IDL) study (ICP only)	At initial set-up and after significant change in instrument type, personnel, test method, or sample matrix.	IDLs shall be ≤ LOD.	NA.	NA.	Samples may not be analyzed without a valid IDL.
Linear dynamic range or high-level check standard (ICP only)	Every 6 months.	Within ± 10% of true value.	NA.	NA.	

Table F-7. Inorganic Analysis by Inductively Coupled Plasma (ICP) Atomic Emission Spectrometry and Atomic Absorption Spectrophotometry (AA) (Methods 6010 and 7000 Series) (continued)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Initial calibration (ICAL) for all analytes **ICP: minimum one high standard and a calibration blank;** **GFAA: minimum three standards and a calibration blank;** **CVAA: minimum 5 standards and a calibration blank**	Daily ICAL prior to sample analysis.	If more than one calibration standard is used, $r \geq 0.995$.	Correct problem, then repeat ICAL.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until ICAL has passed.
Second source calibration verification (ICV)	Once after each ICAL, prior to beginning a sample run.	Value of second source for all analyte(s) within ± 10% of true value.	Correct problem and verify second source standard. Rerun ICV. If that fails, correct problem and repeat ICAL.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until calibration has been verified.
Continuing calibration verification (CCV)	After every 10 field samples and at the end of the analysis sequence.	ICP: within ± 10% of true value; GFAA: within ± 20% of true value; CVAA: within ± 20% of true value.	Correct problem, rerun calibration verification. If that fails, then repeat ICAL. Reanalyze all samples since the last successful calibration verification.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to all results for the specific analyte(s) in all samples since the last acceptable calibration verification.	Problem must be corrected. Results may not be reported without a valid CCV. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Low-level calibration check standard (ICP only)	Daily, after one-point ICAL.	Within ± 20% of true value.	Correct problem, then reanalyze.	Flagging criteria are not appropriate.	No samples may be analyzed without a valid low-level calibration check standard. Low-level calibration check standard should be less than or equal to the reporting limit.

Table F-7. Inorganic Analysis by Inductively Coupled Plasma (ICP) Atomic Emission Spectrometry and Atomic Absorption Spectrophotometry (AA) (Methods 6010 and 7000 Series) (continued)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Method blank	One per preparatory batch.	No analytes detected > ½ RL and greater than 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater). Blank result must not otherwise affect sample results. For common laboratory contaminants, no analytes detected > RL (see Box D-1).	Correct problem, then see criteria in Box D-1. If required, reprep and reanalyze method blank and all samples processed with the contaminated blank.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch.	Problem must be corrected. Results may not be reported without a valid method blank. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Calibration blank	Before beginning a sample run, after every 10 samples, and at end of the analysis sequence.	No analytes detected > LOD.	Correct problem. Re-prep and reanalyze calibration blank. All samples following the last acceptable calibration blank must be reanalyzed.	Apply B-flag to all results for specific analyte(s) in all samples associated with the blank.	
Interference check solutions (ICS) (ICP only)	At the beginning of an analytical run.	ICS-A: Absolute value of concentration for all non-spiked analytes < LOD (unless they are a verified trace impurity from one of the spiked analytes); ICS-AB: Within ± 20% of true value.	Terminate analysis; locate and correct problem; reanalyze ICS, reanalyze all samples.	If corrective action fails, apply Q-flag to all results for specific analyte(s) in all samples associated with the ICS.	
LCS containing all analytes to be reported	One per preparatory batch.	QC acceptance criteria specified by DoD, if available; see Box D-3 and Appendix G.	Correct problem, then reprep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available (see full explanation in Appendix G).	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.	Problem must be corrected. Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed.

Table F-7. Inorganic Analysis by Inductively Coupled Plasma (ICP) Atomic Emission Spectrometry and Atomic Absorption Spectrophotometry (AA) (Methods 6010 and 7000 Series) (continued)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Matrix spike (MS)	One per preparatory batch per matrix (see Box D-7).	For matrix evaluation, use QC acceptance criteria specified by DoD for LCS.	Examine the project-specific DQOs. If the matrix spike falls outside of DoD criteria, additional quality control tests are required to evaluate matrix effects.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met.	For matrix evaluation only. If MS results are outside the LCS limits, the data shall be evaluated to determine the source of difference and to determine if there is a matrix effect or analytical error.
Matrix spike duplicate (MSD) or sample duplicate	One per preparatory batch per matrix (see Box D-7).	MSD: For matrix evaluation use QC acceptance criteria specified by DoD for LCS. MSD or sample duplicate: RPD ≤ 20% (between MS and MSD or sample and sample duplicate).	Examine the project-specific DQOs. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met.	The data shall be evaluated to determine the source of difference.
Dilution test (ICP and GFAA only)	One per preparatory batch.	Five-fold dilution must agree within ± 10% of the original measurement.	ICP: Perform post-digestion spike (PDS) addition; GFAA: Perform recovery test.	Flagging criteria are not appropriate.	Only applicable for samples with concentrations > 50 x LOQ.
Post-digestion spike (PDS) addition (ICP only)	When dilution test fails or analyte concentration in all samples < 50 x LOD.	Recovery within 75-125% (see Table B-1).	Run all associated samples in the preparatory batch by method of standard additions (MSA) or see flagging criteria.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met.	Spike addition should produce a concentration of 10 – 100 x LOQ.
Recovery test (GFAA only)	When dilution test fails or analyte concentration in all samples < 25 x LOD.	Recovery within 85-115%.	Run all associated samples in the preparatory batch by method of standard additions (MSA) or see flagging criteria.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met.	
Method of standard additions (MSA)	When matrix interference is confirmed.	NA.	NA.	NA.	Document use of MSA in the case narrative.
Results reported between DL and LOQ	NA.	NA.	NA.	Apply J-flag to all results between DL and LOQ.	

APPENDIX C-3
Data Management Plan

Former Charlotte Naval Ammunition Depot
Charlotte, North Carolina

December 2011

DERP-FUDS Project No. I04NC080301
Contract No.:W912DY-10-D0025
Delivery Order No.: 0007

PREPARED FOR:

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Data Management Plan

Former Charlotte Naval
Ammunition Depot
Charlotte, North Carolina

Prepared for:
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Our Reference:
DERP-FUDS Project No. I04NC080301
Contract No.: W912DY-10-D0025
Delivery Order No.: 0007

Date:
December 2011

Table of Contents

Acronyms and Abbreviations

ADR	Automated Data Review
CNAD	Charlotte Naval Ammunition Depot
COC	Chain of Custody
DMP	Data Management Plan
EDD	Electronic Data Deliverable
ESRI	Environmental Systems Research Institute, Inc.
FSP	Field Sampling Plan
GIS	Geographical Information System
GPS	Global Positioning System
ID	Identification
JV	Joint Venture
MNA	Monitored Natural Attenuation
PIKA	PIKA International, Inc.
PIKA-PIRNIE JV Team	PIKA International, Inc./Malcolm Pirnie, Inc. Joint Venture LLC Team
Pirnie	Malcolm Pirnie, Inc.
PM	Project Manager
QAPP	Quality Assurance Project Plan
QA/QC	Quality Assurance/Quality Control
QC	Quality Control
RAWP	Remedial Action Work Plan
SEDD	Staged Electronic Data Deliverable
U.S.	United States
USACE	United States Army Corps of Engineers, Huntsville Center
USEPA	United States Environmental Protection Agency
WERS	Worldwide Environmental Remediation Services

1. Introduction

Environmental management encompasses many tasks, priorities, and decisions. Accountability for effectively managing the collection and utilization of information according to well-defined processes within standardized software environments is essential to effective environmental management. The PIKA International, Inc. (PIKA)/Malcolm Pirnie, Inc. (Pirnie) Joint Venture (JV), LLC (the PIKA-PIRNIE JV Team), on behalf of the United States (U.S) Army Corps of Engineers, Huntsville Center (USACE), has prepared this Data Management Plan (DMP) for groundwater remediation activities at the Former Charlotte Naval Ammunition Depot (CNAD) site. The DMP was prepared in a manner consistent with the following reference and guidance document.

- USACE. *Munitions Constituents Chemical Data Quality Deliverables.* Worldwide Environmental Remediation Services (WERS) 009.01. (USACE, 2010).

Challenges with collection, storage and presentation of data can be mitigated with a DMP. This DMP will specify how data is to be labeled and categorized, the format that the data is to be stored in, how to handle data collected over a period of time or over a significant geographic area, and procedures to account for changes in the investigation. The advantage to having a data management plan is that it allows the users to collect, label and record data in a consistent manner. Consistent terms, units, methods and procedures, will allow any user to collect or retrieve data accurately and quickly.

The DMP is a dynamic document accounting for the changing nature of the investigation. Additionally, the DMP is intended to complement the existing Quality Assurance Project Plan (QAPP) and QAPP addendums, prepared to describe overall Quality Assurance and Quality Control (QA/QC) protocols and requirements for the Former CNAD site, as it relates to the management of electronic data.

1.1 Data Management Software Platform

The PIKA-PIRNIE JV Team will use its in-house data management system (Earthsoft's EQuIS) to store and manage environmental data collected as part of the remedial action activities at the Former CNAD site. The PIKA-PIRNIE JV Team database is a comprehensive geo-environmental data management database designed to store analytical test results and related data obtained during environmental site

investigations, routine site monitoring, and hazardous waste remediation projects. The database can be used for report and chart generation and is integrated with multiple statistical, numerical modeling and data visualization tools. The PIKA-PIRNIE JV Team will use Automated Data Review (ADR) and will submit Staged Electronic Data Deliverables (SEDDs) as outlined in USACE WERS-009.01.

1.2 Migration of Existing Data

The PIKA-PIRNIE JV Team will integrate the limited historical data, as needed, into the data management strategy outlined in this plan. Sampling nomenclature in the historical data will be maintained as practical, but will be modified as necessary to comply with data quality requirements. Modifications would involve the concatenation of additional information to the end of the existing sample identification (ID) to ensure uniqueness within the database. Examples of typical modifications would be the addition of a date code or depth interval. Suitable reference values will be generated and incorporated into the PIKA-PIRNIE JV Team database reference values table. Data collected from other consultants and subcontractors working at the Former CNAD site may also be migrated into the PIKA-PIRNIE JV Team database, as dictated by project requirements.

1.3 Data Management Roles and Responsibilities

As part of the data management system, the PIKA-PIRNIE JV Team has identified individuals who will have data management roles for the Former CNAD site. A list of data management personnel is provided below.

Company/Organization	Title	Name	Phone Number
PIKA-PIRNIE JV Team	Data Management Lead	Dak Patel	484.688.0364
	Database Manager	Maribel Vital	303.471.3425

2. Data Acquisition

2.1 Analytical and Field Data Recording

Analytical data includes groundwater samples that will be collected during baseline, performance, and monitored natural attenuation (MNA) monitoring events. These samples will be collected according to the procedures set forth in the Remedial Action Work Plan (RAWP) and submitted to a laboratory with chain-of-custody (COC) documentation for analysis of specific constituents. Sample information recorded by field scientists will include: sample ID, date and time, container and preservative, sample location, types of requested analysis, method of shipment, required validation level, and turn-around time. This information will be recorded in field log books and on the COC.

Data collected from the field will be transcribed from the field log book to a standard electronic template. This template will be used to populate the PIKA-PIRNIE JV Team database.

2.2 Spatial Data

Spatial data for each sampling location will be collected via a global positioning system (GPS) unit with the required accuracy as soon as possible and transmitted to the Geographic Information System (GIS) Specialist. Locations collected with a GPS unit will be post-processed prior to uploading to the central GIS and analytical database. Where vertical elevations are needed for sample locations such as monitoring wells, professional surveys will be performed. Horizontal coordinates obtained from the surveys will be uploaded to the GIS database for spatial representation, as well as the analytical database. Consistent units and coordinate system for the x, y, and z coordinates will be used.

2.3 Groundwater Gauging Data

Groundwater levels will be collected from monitoring wells, piezometers, sumps, streams, and/or rivers. This information may be included in the PIKA-PIRNIE JV Team database and can be used to determine the average direction of groundwater flow, horizontal and vertical gradients, and other properties of the watershed or aquifer. Water level information will be entered into a standard spreadsheet template to facilitate importing to the database.

3. Data Warehousing

3.1 Data Transmittal

Data transmittal from the field log book to a standard template and COC will be the responsibility of the person or persons who committed the entries to the field log book to avoid interpretation issues. Additionally, the standard template and COC will be checked by the Data Manager or Project Chemist (for completeness and legibility issues with the field personnel prior to the data being entered into the database. Daily Quality Control (QC) Reports will be prepared and copies sent to the Project Manager (PM), Task Manager, and Project Chemist as detailed in USACE WERS-009.01.

For transmittal of electronic data deliverables (EDDs), the analytical laboratory will have the responsibility to transmit the data with the final hardcopy data package electronically. This EDD will be entered into the project record, upon review by the Data Manager to ensure that the EDDs are free from errors.

Prior to submission to the PIKA-PIRNIE JV Team, each data file must also be reviewed by the laboratory to ensure that the sample IDs, dates, times, EDD specifications, and other inter-related information is consistent and complete. All parameters that are subcontracted to other laboratories must be included in the EDD for a specific Sample Delivery Group or Laboratory Project Number. It is not acceptable to submit separate EDDs for subcontract parameters. Manual review of the files may be necessary to complete this review.

It is imperative that the EDD results match the hard copy results. This includes issues involving various rounding routines for different electronic data management programs within the laboratory (*i.e.,* Laboratory Information Management System vs. U.S. Environmental Protection Agency (USEPA) Contract Laboratory Program). Significant figures must also match hard copy and be consistent from one sampling event to the next. Reporting limits must be consistent between events as well and must be in compliance with the Laboratory Task Order or Project Statement of Work. There may be instances where diluted surrogates and unrecovered spike compounds will require population of the EDD with numeric values, in lieu of data flags in the hard copy report. The PIKA-PIRNIE JV Team Data Manager will provide project-specific guidance for these conditions.

Prior to submitting an EDD to the PIKA-PIRNIE JV Team, the data checker must be run to check and verify the EDD structure, format, and reference value compliance in accordance with our EQuIS standard operating procedure (included in QAPP). The

checker report must be submitted for each file with each EDD set. The data checker error report, which demonstrates that the EDD files were successfully checked, must be electronically submitted with the four EDD files to the PIKA-PIRNIE JV Team.

3.2 Data Tracking

Tracking the variety of information and data associated with each sample, including status of data availability, is important for any environmental project. The communication between the Data Manager, laboratory, Project Chemist, and various members of the project team is critical to the success of the project.

The data will be tracked using an internal tracking sheet to track data from receipt to final reporting. It will include information as EDD and hard copy received dates, lot or batch number of the EDDs, data source, any re-submissions, when the EDD was verified against the hard copy report, and laboratory turn-around times. Data that is qualified by the laboratory in accordance with the prescribed validation criteria will be entered into a specified field (lab_qualifier) in the EQuIS database.

Data that is qualified through the ADR validation process (according to the validation procedures discussed in the QAPP) will be tagged with a qualifier in the validator_qualifier field. An update query will be used to move the applicable qualifiers to a third field (interpreted_qualifier), which will be used for reporting purposes. In general, the validator_qualifier will supersede the lab_qualifier.

3.3 Data Storage and Retrieval

The project database will be stored on the PIKA-PIRNIE JV Team secured server with access limited (via password) to Data Management Specialists designated by the PM. The PIKA-PIRNIE JV Team secured server has nightly back-up routines that will enable the recovery of data, which may become corrupted or lost due to instrument, computer, and/or power failures. Electronic media will be stored in climate-controlled areas to minimize potential for degradation. Access to storage areas will also be limited.

4. Data Verification/Validation

4.1 Historical Data

Verification of historical data will be performed and checked against original lab reports, if available. Any discrepancies or data inconsistencies will be documented in the PIKA-PIRNIE JV Team database.

4.2 Analytical Data

A certificate of analysis and summary sheets shall be generated as a deliverable by the analytical laboratory. The sheets shall contain information about analytical tests performed, date and condition of samples received, results, methodology, and quality of data reported.

Data verification for the project is defined as review of the data package for completeness and comparison of the EDD to the analytical report for consistency. EDDs found to contain significant defects in format or information will be returned to the laboratory for correction and re-submittal.

Any data that is hand entered into a spreadsheet for management in the project database is compared to the original field notes and subsequently peer reviewed prior to upload to minimize the potential for transcription errors.

The Data Manager is responsible for verification that the laboratory EDD matches the analytical report. Any identified discrepancies will be communicated to the Project Chemist for determination of required corrective action and resolution with the laboratory. Field duplicate parent samples are added to the Field Sample table and the Data Manager confirms that all fields are correctly populated with sample IDs, sample types, and matrix codes. The location codes associated with each sample are entered into the Field Sample table.

Data validation protocols are defined in the QAPP and subsequent addenda.

4.3 Field Measurements

Field measurements shall be reported on applicable forms specified as specified in the Field Sampling Plan (FSP). Data shall be verified for accuracy by a person other than the one responsible for entering the data. The PM, or designee, shall be responsible for checking and approving the final presentation of reported data to ensure that project-specific requirements are met.

4.4 Spatial Data

Spatial data will be managed in a central geodatabase utilizing the Environmental Systems Research Institute, Inc. (ESRI) suite of tools. This geodatabase will include a variety of data types pertinent to the project, for example: site features, well locations, sample locations, and regional features. The GIS Specialist will be responsible for managing and updating this data as needed, as well as maintaining the appropriate metadata, and creating figures representing the data for reporting or other purposes.

A comparison between the locations provided through the surveys or GPS data collection and the locations in the GIS will be completed upon each upload. Specific to the locations collected with the GPS units, draft maps will be produced for the field crew to perform a final QC of the locations after they are post-processed, in the event there was a weak signal or canopy cover that prevented the point to be collected in the intended location. In addition, a cross-check of the sample locations in the database and sample locations in GIS will be performed prior to any reporting effort to ensure completeness and consistency.

4.5 Geologic Data

Geologic description data will be recorded on field soil sampling logs. This information will be incorporated into the PIKA-PIRNIE JV Team database. The geologic data will then be exported to generate formal boring logs for reports.

5. Data Presentation

5.1 Tables

Draft tables will be delivered, as needed, each time that data is loaded to the database. Frequency of database maintenance will depend on the rate of delivery of electronic data from the lab, and project needs as communicated to the Data Manager.

Final tables will be delivered upon request. Content of the table(s) should be established via email communication, and clarification will be requested by the Data Manager as needed. Structure and formatting will be provided by the PIKA-PIRNIE JV Team, preferably by including a sample Excel file to be used as a template.

Quality checking will initially be performed by the Data Manager to ensure that the table includes the data that was requested, and that the data conforms to the database contents. Final quality checking and report formatting will be performed by the project staff, or other staff designated by the PIKA-PIRNIE JV Team.

5.2 Maps (GIS)

Analytical data and the applicable geographic information will be provided as updates to the GIS Specialist for updating the central geodatabase. The updates will be provided to the GIS Specialist in the form of exports from the EQuIS database.

Content of the figure(s) should be established via email communication, and clarification will be requested by the GIS Specialist as needed. Direction on site-specific symbology and formatting will be provided by the PIKA-PIRNIE JV Team where needed.

Figures will be reviewed by the GIS Specialist to ensure the figure includes features and themes requested, and represents the most up-to-date spatial data available for the project. Final quality checking and report formatting will be performed by the project staff, or other staff designated by the PIKA-PIRNIE JV Team.

6. Data Submittals

Daily electronic QC Reports will be prepared and sent to the PM and Project Chemist, at a minimum.

The PIKA-PIRNIE JV Team will submit EDDs as outlined in USACE WERS-009.01, in format SEDD version 5.2 (or most recent version). The PIKA-PIRNIE JV Team will use the USEPA Stage 2A checker to insure the files successfully pass prior to submission. The deliverables will be reviewed, and when found acceptable they will be submitted for approval.

The submittal will also include post-review ADR files and error log files (if any), including other submissions as outlined in USACE WERS-009.01.

7. Collaboration

7.1 Team Site

A project portal for this project will be in place for the internal collaboration of the home team. An archive of information, such as analytical lab reports, that needs to be available to all team members will also be established in various subfolders. Draft versions of documents will be stored and checked out.

7.2 GIS Portal

The spatial data can be accessed through a web-based GIS portal. This secure site will require a username and password for each individual, and can be accessed through a standard web browser. New user requests will need to be sent to the GIS Specialist to be set up with the proper access. Data content will be based on the established GIS and analytical datasets for the project.

Appendix D

Project Management Plan (PMP)

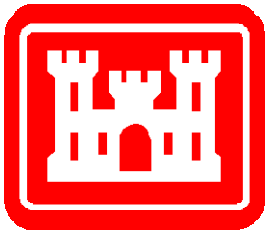
APPENDIX D
Project Management Plan

Former Charlotte Naval Ammunition Depot
Charlotte, North Carolina

December 2011

DERP-FUDS Project No. I04NC080301
Contract No.:W912DY-10-D0025
Delivery Order No.: 0007

PREPARED FOR:



U.S. Army Corps of Engineers, Huntsville Center
U.S. Army Engineering and Support Center
4820 University Square
Huntsville, Alabama 33816-1822

I have reviewed this document and certify that it contains accurate content and is sufficient to guide project execution.

Ray Livermore
USACE Project Manager

Jywanya Dillinger
USACE Contracting Officer's Representative

Mark Albe, PMP
Corporate Quality Manager

Patrick Shirley
PIKA-PIRNIE JV Team Certified Project Manager

Project Management Plan

Former Charlotte Naval Ammunition Depot
Charlotte, North Carolina

Prepared for:
U.S. Army Corps of Engineers,
Huntsville Center

Prepared by:
PIKA-PIRNIE JV, LLC
12723 Capricorn Drive
Suite 500
Stafford, Texas 77477

Our Reference:
DERP-FUDS Project No. I04NC080301
Contract No.: W912DY-10-D0025
Delivery Order No.: 0007

Date:
December 2011

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Acronyms and Abbreviations

APP	Accident Prevention Plan
bgs	Below Ground Surface
CERCLA	Comprehensive Environmental Response, Compensation and Liability Act
CFR	Code of Federal Regulations
CNAD	Charlotte Naval Ammunition Depot
COD	Chemical Oxygen Demand
COR	Contracting Officer's Representative
CQCP	Construction Quality Control Plan
CSM	Conceptual Site Model
DID	Data Item Description
DO	Delivery Order
DQO	Data Quality Objective
ERD	Enhanced Reductive Dechlorination
FFP	Fixed Firm Price
FFS	Focused Feasibility Study
FTP	File Transfer Protocol
FUP	Fixed Unit Price
H&S	Health and Safety
HSU	Hydrostratigraphic Unit
IT	Information Technology
JV	Joint Venture
KO	Contracting Officer
mm	Millimeter
MNA	Monitored Natural Attenuation
NCDENR	North Carolina Department of Environment and Natural Resources
NCP	National Oil and Hazardous Substances Contingency Plan
NTP	Notice to Proceed
O&M	Operation and Maintenance

OSHA	Occupational Safety & Health Administration
PDF	Portable Document Format
PIKA	PIKA International, Inc.
PIKA-PIRNIE JV Team	PIKA International, Inc./Malcolm Pirnie, Inc. Joint Venture LLC Team
Pirnie	Malcolm Pirnie, Inc.
PIP	Public Involvement Plan
PM	Project Manager
PMP	Project Management Plan
PWR	Partially Weathered Rock
PWS	Performance Work Statement
QA/QC	Quality Assurance/Quality Control
QC	Quality Control
RA	Remedial Action
RAO	Remedial Action Objective
RAR	Remedial Action Report
RAWP	Remedial Action Work Plan
RFP	Request for Proposal
RI	Remedial Investigation
RTC	Response to Comments
SARA	Superfund Amendments and Reauthorization Act
SSHP	Site Safety and Health Plan
TCE	Trichloroethene
UIC	Underground Injection Control
U.S.	United States
USACE	U.S. Army Corps of Engineers, Huntsville Center
USAEC	U.S. Army Environmental Command
USEPA	U.S. Environmental Protection Agency
WERS	Worldwide Environmental Remediation Services

1. Introduction

In accordance with the Performance Work Statement (PWS), PIKA International, Inc. (PIKA)/Malcolm Pirnie, Inc. (Pirnie) Joint Venture (JV), LLC (the PIKA-PIRNIE JV Team) has developed this Project Management Plan (PMP) for the Remedial Action (RA) at the Former Charlotte Naval Ammunition Depot (CNAD) in Charlotte, North Carolina. This Task Order contains both a firm fixed price (FFP) and a fixed unit price (FUP) tasks, issued as Delivery Order (DO) 0007 under the United States (U.S.) Army Corps of Engineers, Huntsville Center (USACE) Worldwide Environmental Remediation Services (WERS) Contract W912DY-10-D-0025 (USACE, 2011). This DO was issued and administered by the Huntsville Center.

This PMP is a living document that specifies the management, administrative, and technical details of work execution, including project organization, staffing, status reporting, deliverables, schedule, and payment milestones. A current version of the PMP with an updated schedule of milestones will be maintained throughout the project.

1.1 Installation Setting and Status

In June 1942, the Department of Navy signed a contract with the U.S. Rubber Company for the construction of 40-millimeter (mm) anti-aircraft ammunition shell loading and assembly plant in Charlotte, North Carolina known as the CNAD. In 1945, planned production was cut and operation of the facility was transferred to the U.S. Navy. In 1956, the CNAD status was changed from Maintenance to Inactive. At the time of operation, the entire CNAD complex occupied approximately 2,266 acres of land. In 1959, the former CNAD complex was sold to a local partnership and is currently occupied by light industrial and commercial businesses.

Two areas (1 and 2) of the site were used for the production of 40-mm anti-aircraft munitions. Area 1 consisted of anti-aircraft ammunition loading lines. This area was dedicated to the assembly of final rounds and was composed of 22 buildings. The largest of the buildings in Area 1 (1-60 and 1-70) were used for final assembly, packaging, and shipping of munitions.

The operations carried out in Area 2 were reportedly identical to those conducted in Area 1. Area 2 was also used to process returned ammunition after World War II. Only Area 2 was used after 1945 for reconditioning of returned munitions. A trichloroethene (TCE) vapor-degreasing operation was located on the southeast corner

of building 2-30. The unit was used to remove cutting oil and preservatives from the exteriors of returned shells. Refer to **Figure 1-1** for the Site Location Map.

Environmental remediation activities at the Former CNAD are performed in accordance with the provisions of the Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA), 42 USC §9601 *et seq*., as amended by the Superfund Amendments and Reauthorization Act of 1986 (SARA), and to the extent practicable, the National Oil and Hazardous Substances Pollution Contingency Plan (NCP), 40 Code of Federal Regulations (CFR) Part 300 *et seq*., as amended. The North Carolina Department of Environment and Natural Resources (NCDENR) concurrs with the selected remedy, as outlined in the Decision Document (USACE, 2011a).

1.2 Project Scope of Work and Performance Objectives

The PIKA-PIRNIE JV Team has contracted with the USACE to complete RA at the Former CNAD in Charlotte, North Carolina (PIKA-PIRNIE JV, LLC, 2011). The proposed approach is designed to meet all performance objectives detailed in the July 12, 2011 Request for Proposal (RFP) from the USACE, the revised Performance Work Statement (PWS) (USACE, 2011b), and question and answer sets under the WERS Contract. A summary of the Contractual Performance Requirements has been provided in **Table 1-1**.

The PIKA-PIRNIE JV Team has entered the project flow after the approval of the Decision Document (USACE, 2011a). As such, project efforts have focused on the aspects of implementing the proposed remedial design.

1.3 Technical Approach

The general remediation approach is based on prior proven approaches that have been successful in the Piedmont region. The technical approach for the site was developed to adopt the approved remedy for in-situ enhanced reductive dechlorination (ERD). Implementation of this remedy will include a limited number of injection events to reduce the site-related constituents (TCE) to a level where monitored natural attenuation (MNA) is feasible. This section outlines the technical approach for implementing an in-situ RA capable of meeting these objectives.

Development of the technical approach for the site included a comprehensive review of the available site data presented in the Remedial Investigation (RI) Reports and the Focused Feasibility Study (FFS) (SAIC, 2009). The first element that was reviewed in

these documents was the Conceptual Site Model (CSM). In order to properly develop a technical approach, the subsurface conditions, contaminants present, migration pathways, and the influence of these factors must be understood. The following is a summary of key elements that were considered during the development of the technical approach.

- The hydrostratigraphic framework beneath the site is composed of three units, which include the saprolite (shallow), transition zone and bedrock.

- The transition zone is defined as a combination of coarse gained sand, partially weathered rock (PWR) and the upper portion of the fractured bedrock.

- The transition zone is serving two roles in groundwater fate and transport:

    - The presence fine grained saprolitic soils intermixed with the coarse sand and PWR provides a reservoir for contaminant mass storage within the transition zone. This mass storage increases the potential for additional diffusion to groundwater as the remediation proceeds (*i.e.,* rebound).

    - The permeable zones in the transition zone act as a pathway for lateral contaminant migration.

- The fracture density in the bedrock decreases with depth with the primary fracture sets observed from 15 to 90 feet below ground surface (bgs); however, significant water bearing fractures have been observed in the vicinity of the site at depths up to 600 feet bgs.

- The primary bedrock fracture orientation is generally in a north/south direction. Groundwater flow in bedrock is dominated by the orientation of these fracture sets, resulting in a southern flow component. This southern component of groundwater flow was verified by the chemical oxygen demand (COD) distribution observed during the pilot study.

- Groundwater flow directions, and subsequently contaminant migration directions in the transition zone, are significantly influenced by the characteristics of the bedrock surface and the orientation of the shallow fractures.

- The estimated source area for impacts to groundwater is located in the vicinity of a bedrock topographic high, which represents an area of groundwater recharge.

- Vertical hydraulic gradients in the vicinity of the bedrock topographic high were observed to be in a downward direction from the transition zone to the bedrock, under ambient conditions; however based on the available data for the bedrock hydrostratigraphic unit (HSU), the magnitude and direction , may vary across the area.

- The natural migration of the groundwater from the transition zone to the bedrock is an important consideration for developing the remedial approach for the site. Reagent injected in the transition zone will provide indirect treatment of the underlying bedrock as the reagent naturally distributes post injection. A similar reagent distribution is anticipated in the bedrock. The deeper groundwater impacts, although not directly targeted by the injection, can be indirectly treated by natural groundwater flow in the fracture network.

These elements of the CSM were used to determine a technical approach for a remedial alternative to address the TCE impacts in the transition zone and bedrock HSUs.

1.4 Risk Approach

Table 1-2 identifies risks associated with implementing the proposed RAs and approaches to managing the risks in compliance with the remedial action objectives (RAOs) and the schedule presented in **Appendix A** to this PMP.

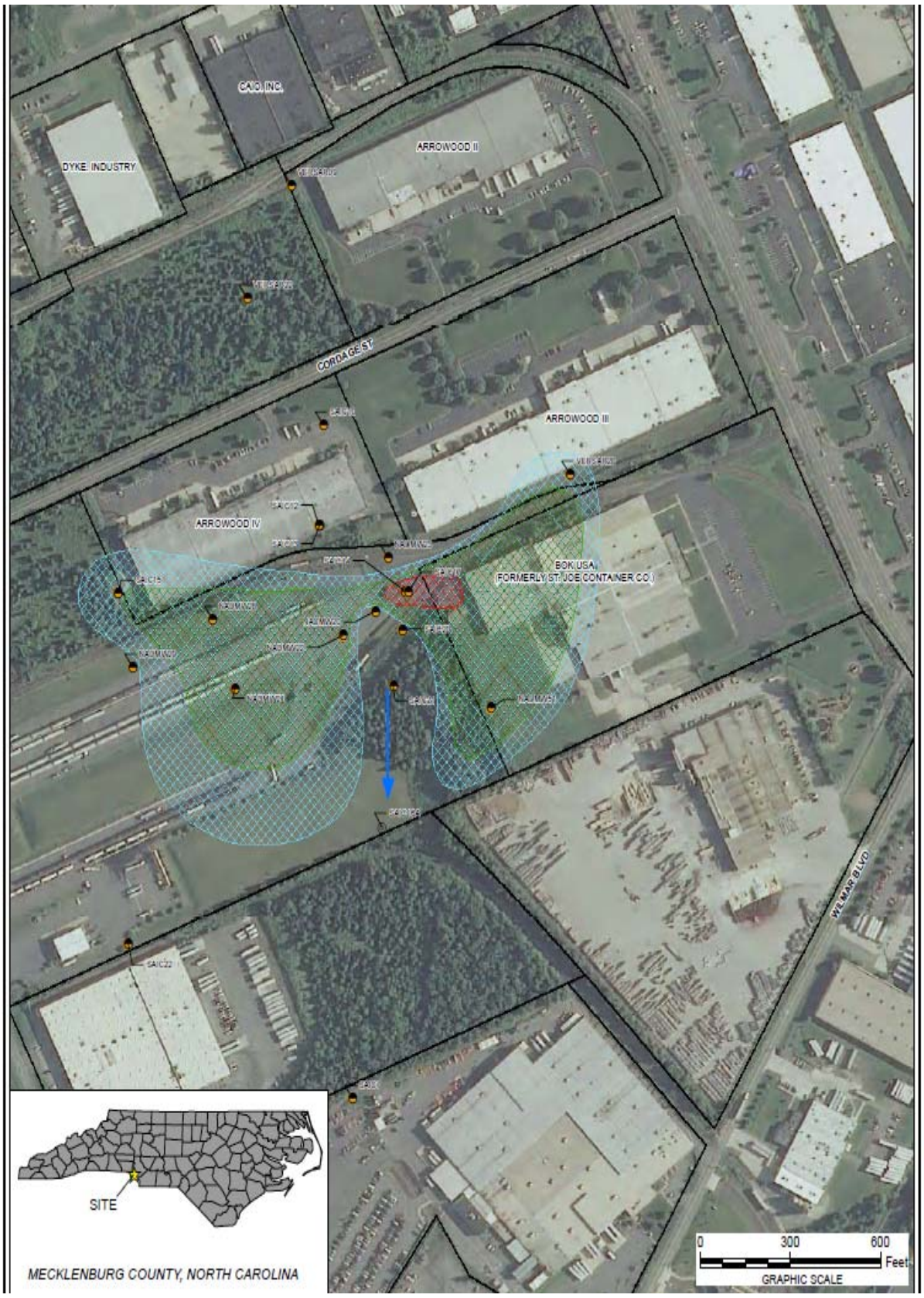
Table 1-1
Performance Requirements
Former Charlotte Naval Ammunition Depot – Charlotte, North Carolina

Performance Objective	Performance Standards
Preparation of a RAWP covering all requirements of the PWS • Draft RAWP within 45 calendar days of the Notice to Proceed (NTP). • Final RAWP submittal within 87 days of NTP. • Revised Final RAWP within 115 days of NTP.	Prepared in accordance with PWS, Data Item Descriptions (DIDs) and all applicable regulations. Final approval through NCDENR.
Implementation of activities identified in the RAWP Submittal of quarterly progress reports on the operation of the RA	Successful implementation of the RAOs as stated in the RAWP. Field work, data quantity and quality, and analysis of said data provides the following results in the RAR. Demonstrate that the work was performed in accordance with the applicable laws, regulations, and guidance documents. Meet the project data quality objectives (DQOs)
Monitoring Event Reports (Baseline Sampling Report, Performance Monitoring Reports, Annual MNA Monitoring Report)	Report and documents prepared in accordance with DIDS, the RAWP, and all applicable Federal, State and Local Regulations.
Remedial Action Report (RAR)	Acceptance of the Final RAR by NCDENR with no more than 20 minor comments and 3 major comments.

Table 1-2
Identified Risks with Mitigation Measures and Contingencies
Former Charlotte Naval Ammunition Depot – Charlotte, North Carolina

Risk	Impact	Mitigation Strategy/Contingency Plan
H&S Incident During Fieldwork (Low)	Stop Work to investigate root cause and implement mitigating actions to prevent reoccurrence (High)	Develop SSHP prior to field mobilization with Activity Hazards Analyses and clear communication with all site employees and subcontractors to ensure H&S is primary focus during field operations. If necessary, increase onsite H&S resources to provide greater oversight and support to field crews.
Regulators Will Not Accept Proposed Remediation Strategy (Medium)	Optimization will be delayed or not achieved within period of performance (High)	Identify regulatory issues with proposed plan and develop examples to demonstrate superiority of approach.
Dispute w/ Property Owners over Site Use or Access (Low)	Project Delays and/or modification to approach (Medium)	Maintain open lines of communication with each property owner to keep stakeholders engaged in proposed plans and approach. Coordinate with USACE as required to address site access agreements.
Cannot inject in to transition zone wells or the flow rates are significantly lower than estimated (Medium)	Untreated portions of the TCE plumes (Medium)	May require aggressive redevelopment and/or replacement injection wells to provide a well capable of delivering reagent to the transition zone.
Uncontrolled methane generation during ERD operation (High)	Increased potential for physical hazards, such as explosion (High)	Methane monitored will be included as part of the remedial action implementation to provide an ongoing evaluation of the potential risks. Should the routinely monitored methane concentrations in the soil gas reach established action levels, the USACE will be notified and an appropriate response will be developed.
Potential vapor intrusion issues related to untreated vinyl chloride produced during ERD process (Medium for only eight injection events)	Potential human health concerns and additional regulatory interest and scrutiny (High).	The greater risk is associated with a lower number of injection events, as TCE may be reduced to the targeted treatment concentration within that timeframe, daughter products generated during the ERD process are likely to remain elevated, potentially posing a higher human health risk (in the case of vinyl chloride) than the parent product. The mitigation strategy implemented is to increase the proposed number of injections (8) until vinyl chloride is transformed in to ethane, an innocuous end product.
Obtaining Underground Injection Control (UIC) permit (Medium)	Project Delays and/or modification to approach (Medium)	Identify issues with proposed plan. Discuss the alternatives with the NCDENR UIC administrator and modify the plan as necessary to alleviate concerns and demonstrate expertise and experience for installing these systems.
PWS defined injection volumes are not adequate to treat significant portions of the plume (High)	Untreated portions of the plume will continue to act as an ongoing source in groundwater (High)	Increase injection volumes to provide adequate radius of influence.

Figure 1-1
Site Location Map



2. Management Approach

The PIKA-PIRNIE JV Team will work with the project stakeholders to achieve project objectives. The management approach for the PIKA-PIRNIE JV Team consists of providing USACE a proven project management team consisting of a Program Manager, Project Manager (PM), Corporate Quality Manager, and support staff. The Program Managers, Mr. Bill Davis and Mr. Mark Albe, will ensure that the best resources are applied to this project, quality plans are implemented, contractual requirements are met, and issues are resolved quickly. The PM, Mr. Patrick Shirley, retains the ultimate responsibility for the project, ensuring it is being consistently completed on schedule, within budget, and of acceptable quality. The PM will also manage and integrate team members and oversee the preparation of reports.

The management team is supported by Health and Safety (H&S) and Quality Control (QC) personnel. The PIKA-PIRNIE JV Team maintains a dedicated staff of H&S and QC professionals to provide the project with oversight, guidance, and support resources. These personnel will conduct H&S and QC inspections to determine whether construction and operations are being conducted in accordance with plans, USACE requirements, Occupational Safety and Health Administration (OSHA) regulations, and other applicable requirements.

Project execution is directed by the management team located in the Greenville, South Carolina office of the PIKA-PIRNIE JV Team. The PIKA-PIRNIE JV Team organizational chart for the project is presented in **Figure 2-1**. Each position within the team organization carries with it a defined set of responsibilities and authorities. The roles and responsibilities of key The PIKA-PIRNIE JV Team project personnel are summarized in **Table 2-1**. The contact information for personnel responsible for ensuring the execution of the project, including names, titles, and contact information, is provided in Section 2.1.

2.1 Points of Contact

Points of contact for USACE and the PIKA-PIRNIE JV Team project management personnel are provided in **Table 2-2**.

2.2 Project Stakeholders

The following project stakeholders, at a minimum, are anticipated to be involved with this project:

- USACE
  - Huntsville Center
  - Savannah District
  - Wilmington District
- PIKA-PIRNIE JV Team
- NCDENR
- Norfolk Southern and surrounding property owners
- Public

Table 2-1
Roles and Responsibilities of Key Personnel
Former Charlotte Naval Ammunition Depot – Charlotte, North Carolina

Project Personnel	Roles and Responsibilities
Bill Davis, PE **Mark Albe, PMP** *Program Managers/Corporate Quality Manager*	• Responsible for monitoring overall project progress • Responsible for overall project quality • Ensure PM has the resources available to efficiently and safely complete project tasks • Programmatic communication and information distribution will occur through the PM • Execute task order in accordance with PWS and all applicable regulations as well as meeting the expectations of USACE • Conduct and document quality control reviews • Identify, document, report, and ensure completion of all corrective actions to ensure compliance with QC Plan • Stop, amend, or curtail work for quality or H&S deficiencies
Patrick Shirley, PG *Project Manager*	• Single point of contact for the task order activities • Execute task order in accordance with PWS and all applicable regulations • Direct day-to-day project operations and preparation of all deliverables • Overall quality of work performed • Assignment of project resources/coordinate workflow • Compliance with project scope and budget • Comprehensive focus on overall project schedule • Responsible for the final quality and technical review of all submittals • Prepare/submit monthly/weekly progress and cost reporting and periodic reports • Review all invoices and cost details • Meet contractual obligations • Implement procedures to eliminate conflicts, errors, and omissions and ensure the accuracy of all output • Supervise all site activities, overseeing normal or emergency work • Perform any emergency notifications • Stop, amend, or curtail work for quality, H&S, regulatory, or operational deficiencies • Maintain communication and coordination with USACE
David A. Lamoureux, PE *Senior Contracts Manager*	• Acquisition and contract management • Subcontract management • Federal, state, and local regulations pertaining to contract management and acquisition
Bill Mener, CSP *Corporate Health and Safety Officer*	• Overall responsibility for project safety management • Stop, amend, or curtail work for quality or H&S deficiencies • Certified Safety Professional designation

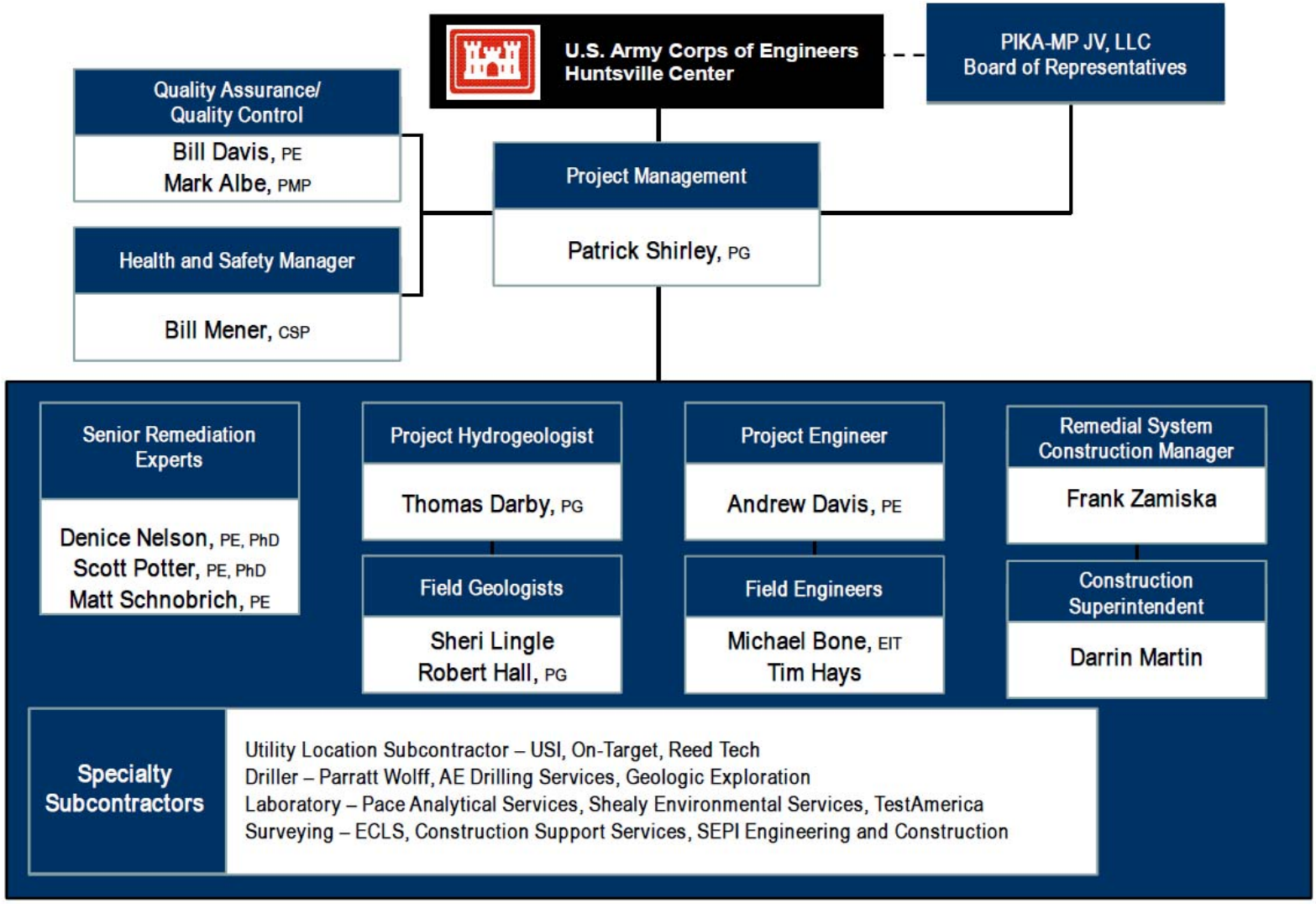
Table 2-1
Roles and Responsibilities of Key Personnel
Former Charlotte Naval Ammunition Depot – Charlotte, North Carolina

Project Personnel	Roles and Responsibilities
Matt Schnobrich, PE *Senior Remediation Expert*	• Evaluate existing monitor well network and propose locations for new wells • Evaluate existing groundwater data and develop a layout for injection transects • Provide design of an injection system to be implemented at the site • Provide CQC for system construction • Provide analysis of system performance monitoring and develop any modifications to the system to maximize the effectiveness of injection events
Andrew Davis, PE *Project Engineer*	• Provide Engineering support for Work Plan preparations • Provide injection system design and CQA oversight for construction • Obtain Permits associated with project activities • Evaluate monitoring data and prepare performance reports
Tim Hays **Michael Bone** *Field Engineers*	• Responsible for oversight of field activities including monitor well installation, injection well installation • Remedial system construction oversight • Execution of ERD injection • Preparation of Monitoring Reports
William Mener, CSP *Certified Safety Professional*	• Responsible for developing, implementing, and overseeing all H&S aspects of a project during investigation and remediation operations per approved site plans and federal, state, and local regulations to ensure a safe workplace is maintained; he is available for emergencies and on-site consultation. • Develops and/or approves all project accident prevention and safety and health plans, activity hazard analyses, and emergency response plans. • Will evaluate toxicological risks and define response action levels, leads site-specific supervisory training for on-site personnel in proper confined space entry procedures, exposure assessment, and air monitoring techniques.

Table 2-2
Points of Contact Information
Former Charlotte Naval Ammunition Depot – Charlotte, North Carolina

Name	Title/Project Function	Address	Contact Information
USACE			
Julie Hiscox	USACE Program Manager	U.S. Army Corps of Engineers, Savannah District 100 W. Oglethorpe Ave Savannah, GA 31402	912-652-5363 (office) julie.a.hiscox@usace.army.mil
Frank Burwell	USACE Technical Manager	U.S. Army Corps of Engineers, Savannah District 100 W. Oglethorpe Ave Savannah, GA 31402	Frank.e.burwell@usace.army.mil
Ray Livermore	USACE Project Manager	U.S. Army Corps of Engineers, Wilmington District 69 Darlington Ave Wilmington, NC 28403	910-251-4702 (office) raymond.r.livermore@usace.army.mil
PIKA-PIRNIE JV Team			
William T. Davis, PE	Program Manager	PIKA-PIRNIE 12723 Capricorn Drive Suite 500 Stafford, TX 77477	281-299-5022 (office) bdavis@pikainc.com
Mark Albe, PMP	Program Manager/Quality Control Manager	PIKA-PIRNIE 300 East Lombard Street Suite 1510 Baltimore, MD 21202	410-230-9965 (office) 443-825-2800 (mobile) 410-230-0491 (fax) mark.albe@arcadis-us.com
Patrick Shirley, PG	Project Manager	PIKA-PIRNIE 30 Patewood Drive Suite 155 Greenville, SC 29615	864-987-3909 (office) 864-238-0065 (mobile) 864-987-1609 (fax) patrick.shirley@arcadis-us.com
Matt Schnobrich, PE	Senior Technical Advisor	PIKA-PIRNIE 640 Freedom Business Center, Suite 310 King of Prussia, PA 19406	610-768-5813 (office) 651.226.4666 (mobile) 610-226-4666 (fax) matthew.schnobrich@arcadis-us.com
Andrew Davis, PE	Project Engineer	PIKA-PIRNIE 30 Patewood Drive Suite 155 Greenville, SC 29615	864-987-3917 (office) 864-561-5833 (mobile) 864-987-1609 (fax) andrew.davis@arcadis-us.com
Thomas Darby, PG	Project Geologist	PIKA-PIRNIE 30 Patewood Drive Suite 155 Greenville, SC 29615	864-987-3918 (office) 304-685-7739 (mobile) 864-987-1609 (fax) thomas.darby@arcadis-us.com
Richard Collins	Regulatory Specialist	PIKA-PIRNIE 1114 Benfield Boulevard Suite A Millersville, MD 21108	410-923-7765 (office) 410-987-4392 (fax) richard.collins@arcadis-us.com

Figure 2-1
Project Organizational Chart



3. Coordination and Communication

This section describes the coordination of and communication with stakeholders that is necessary to ensure the successful completion of the remedial implementation at the Former CNAD. Key stakeholders will be kept informed of project status, existing or potential problems, and changes required to manage the project.

3.1 Monthly Progress Reports

The PIKA-PIRNIE JV Team will provide monthly progress reports to update USACE on the status of the project. Monthly Progress Reports will be submitted by the 15th day of the following month. Monthly progress reports will be submitted via email to the USACE PM.

Monthly Progress Reports will provide summary information that includes, but is not limited to, work completed during the reporting period, milestones achieved, work scheduled, technical issues, regulatory challenges/issues, issues that may hamper project schedule, and any other project-related issues raised by any of the stakeholders, as well as their means of resolution. Monthly Progress Reports will also provide an updated schedule to reflect changes as a result of conditions encountered.

3.2 Phone Conferences/Informal Site Meetings

Within five days of occurrence of all discussions, verbal directions, and telephone conversations on topics relevant to this project, a Confirmation Notice shall be prepared in accordance with the Task Order requirements. Notices will document participating personnel, subject(s) discussed, and conclusions reached.

3.3 Regulatory Negotiations

All regulatory coordination will be approved by the USACE and the contracting officer's representative (COR). The PIKA-PIRNIE JV Team will provide the necessary support to initiate, schedule, and address all applicable regulatory aspects of the project (*e.g.,* organizing discussions with regulators concerning remedial action work plans (RAWPs), execution, and performance monitoring).

The USACE PM, or designee, will attend and represent the Army at all meetings with the regulators. The PIKA-PIRNIE JV Team will prepare and submit minutes for both the Post RAWP review meeting with the NCDENR and the Post RA Report review meeting with NCDENR. With approval of the COR, the PIKA-PIRNIE JV Team may

also informally discuss project issues with regulators and provide an after-action report back to the COR and the USACE PM.

The Army will be the signature authority for all regulatory agreements and regulatory documentation associated with this DO.

3.4 Public Involvement

The PIKA-PIRNIE JV Team will not make available or publicly disclose any data or report generated under this contract unless specifically authorized by the contracting officer (KO) through the COR. If any person or entity requests information pertinent to the scope of work or work being conducted hereunder, the PIKA-PIRNIE JV Team will refer them to the COR. All reports and other information generated during this project will be the property of the USACE, and the PIKA-PIRNIE JV Team will not distribute to any other entity unless authorized by the KO.

All public participation coordination shall be approved by the USACE and the PIKA-PIRNIE JV Team PMs. The PIKA-PIRNIE JV Team recognizes that successful project execution requires the involvement of community stakeholders. The PIKA-PIRNIE JV Team will prepare a Public Involvement Plan (PIP) that describes a strategy and plan for public participation as well as coordination with current property owners. If requested by the Army, the PIKA-PIRNIE JV Team will provide high-level strategy development, deliver materials and activities such as website updates and factsheets; and design and facilitate meetings to manage issues, keep the facts straight and accessible to lay audiences, and steer the emotional energy around those issues to win favorable outcomes.

4. Deliverables, Milestones, and Schedule

4.1 Deliverables

Documents included in Section 4.8.3 of the PWS (Monthly Progress Reports, Minutes of Meetings and Conferences, Draft/Final/Revised Final Monitoring Reports) will be produced in draft (USACE Internal Draft), draft-final, and final versions. Each document will be submitted as a hard and electronic (Portable Document Format [PDF]) copies with two copies of each document submitted to USACE – Savannah, Wilmington, and Huntsville for each version. All other documents will be submitted to the USACE Savannah District office only. The electronic format will prepared in accordance with the requirements provided in the PWS, Section 3.2.4. With USACE concurrence, the PIKA-PIRNIE JV Team may coordinate with appropriate regulatory agencies to determine if fewer versions of each deliverable are sufficient for review.

The COR will provide consolidated comments on draft documents to the PIKA-PIRNIE JV Team within thirty (30) calendar days. The PIKA-PIRNIE JV Team will submit draft and final Responses to Comments (RTCs) by email before submitting subsequent document versions. All documents will be identified as draft or draft-final until accepted by the COR, when they will be signed and finalized.

The PIKA-PIRNIE JV Team will follow the substantive requirements for all subject areas of the USACE guidance applicable to deliverables required for achievement of performance objectives identified in the PWS. If versions of Engineer Manuals, DIDs, etc. are updated, the substantive requirements of the most recently approved version will apply.

The deliverables included under this FFP DO are summarized in **Table 4-1.**

4.2 Milestones

The COR will be responsible for contract management, inspection, oversight, review, and approval activities. Certification and approval of project milestones by the COR is necessary before distribution of milestone payment(s).

The payment schedule for major milestones and additional interim milestones is summarized in **Table 4-2**. The purpose of interim milestones is to minimize the time between significant incurrence of expenses (such as for field work) and the next milestone (such as approval of a report) which can often take months or even years to

achieve. The PIKA-PIRNIE JV Team understands the USACE's intent to verify progress before making milestone payments. Thus, successful completion of a milestone is defined as follows:

- Submittal of Draft, Draft Final, or Final versions of reports to the USACE. While milestones may be achieved, the final completion of a report will be defined as having comments on the all previous versions addressed and approved, and the document has received USACE and, if appropriate, regulatory concurrence.

- Completion of fieldwork means that (1) the scope of work defined in the PWS has been completed and the USACE concurs with the completion; or (2) the field logs for sampling have been submitted to the USACE and received approval by the PM.

Final acceptance of milestone completion will include appropriate acceptance of activities associated with site remediation by regulators and USACE, where applicable. Certification by the Army is contingent upon the PIKA-PIRNIE JV Team performing in accordance with the terms and conditions of the contract, the PWS, and all amendments/options.

4.3 Schedule

A complete activity-based schedule that fully supports the technical approach and outlines the due dates for all milestones and payable deliverables is included as **Appendix A**. The project schedule will be updated and submitted with the monthly status report or as necessary when significant changes are made. The schedule will include all events that impact the project and will be updated for actual starts and finishes. For instance, the field work schedule may vary based on H&S reasons (*e.g.,* heat) inclement weather, access restrictions or other events that would alter the proposed dates.

Table 4-1
Key Project Deliverables
Former Charlotte Naval Ammunition Depot – Charlotte, North Carolina

Deliverable	Site ID (Site Name)	Versions
Remedial Action Work Plan (RAWP)	Not Applicable (N/A)	Draft, Final, Revised Final
Baseline Sampling Monitoring Report	N/A	Draft, Final, Revised Final
Remedial Action Report (RAR)	N/A	Draft, Final, Revised Final
Performance Monitoring Report #1	N/A	Draft, Final, Revised Final
Performance Monitoring Report #2	N/A	Draft, Final, Revised Final
Performance Monitoring Report #3	N/A	Draft, Final, Revised Final
Performance Monitoring Report #4	N/A	Draft, Final, Revised Final
Performance Monitoring Report #5	N/A	Draft, Final, Revised Final
Performance Monitoring Report #6	N/A	Draft, Final, Revised Final
Performance Monitoring Report #7	N/A	Draft, Final, Revised Final
Performance Monitoring Report #8	N/A	Draft, Final, Revised Final
Annual MNA Monitoring Report #1	N/A	Draft, Final, Revised Final

Table 4-2
Major Performance-Based Milestones
Former Charlotte Naval Ammunition Depot – Charlotte, North Carolina

Milestone	Acceptance Criteria	Due Date
Draft RAWP	Draft submittal to USACE	10/28/2011
Revised Final RAWP	Revised Final Submittal to USACE/ NCDENR	1/6/2012
Installation of Monitor Wells Mobilization	Mobilization for well installation	1/10/2012
Installation of Monitor Wells 100% Complete	Well Installation 100% complete	3/4/2012
Baseline Groundwater Monitoring	Completion of sampling	3/18/2012
Installation of Injection Wells Mobilization	Mobilization for well installation	3/26/2012
Installation of Injection Wells 50% Complete	Well Installation 50% complete	5/12/2012
Installation of Injection Wells 100% Complete	Well Installation 100% complete	6/17/2012
Mobilization and Remedial System Materials	Mobilization for system installation	4/1/2012
Construction of Remedial System	Completion of component installation, start up test	6/5/2012
Injection Event #1	Acceptance by USACE	7/29/2012
Performance Monitoring Event #1	Completion of sampling	9/4/2012
Injection Event #2	Acceptance by USACE	10/28/2012
Performance Monitoring Event #2	Completion of sampling	12/4/2012
Injection Event #3	Completion of event	1/27/2013
Performance Monitoring Event #3	Completion of sampling	
Injection Event #4	Completion of event	4/28/2013
Performance Monitoring Event #4	Completion of sampling	
Injection Event #5	Completion of event	7/28/2013
Performance Monitoring Event #5	Completion of sampling	
Injection Event #6	Completion of event	10/27/2013
Performance Monitoring Event #6	Completion of sampling	
Injection Event #7	Completion of event	1/26/2014
Performance Monitoring Event #7	Completion of sampling	
Injection Event #8	Completion of event	4/27/2014
Performance Monitoring Event #8	Completion of sampling	

Table 4-2
Major Performance-Based Milestones
Former Charlotte Naval Ammunition Depot – Charlotte, North Carolina

Milestone	Acceptance Criteria	Due Date
MNA Monitoring – Quarter #1	Completion of sampling	9/6/2014
MNA Monitoring – Quarter #2	Completion of sampling	12/5/2014
MNA Monitoring – Quarter #3	Completion of sampling	3/5/2015
MNA Monitoring – Quarter #4	Completion of sampling	6/3/2015
MNA Monitoring – Quarter #5	Completion of sampling	9/1/2015
MNA Monitoring – Quarter #6	Completion of sampling	11/30/2015
MNA Monitoring – Quarter #7	Completion of sampling	2/28/2016
MNA Monitoring – Quarter #8	Completion of sampling	5/28/2016
Baseline Groundwater Monitoring Report	Draft report submittal to USACE	5/5/2012
Performance Monitoring Report #1	Submittal of Monitoring Report to USACE	10/31/2012
Performance Monitoring Report #2	Submittal of Monitoring Report to USACE	1/30/2013
Performance Monitoring Report #3	Submittal of Monitoring Report to USACE	5/1/2013
Performance Monitoring Report #4	Submittal of Monitoring Report to USACE	7/31/2013
Performance Monitoring Report #5	Submittal of Monitoring Report to USACE	10/30/2013
Performance Monitoring Report #6	Submittal of Monitoring Report to USACE	1/29/2014
Performance Monitoring Report #7	Submittal of Monitoring Report to USACE	4/30/2014
Performance Monitoring Report #8	Submittal of Monitoring Report to USACE	7/30/2014
MNA Reporting – Annual Report (Year #3)	Submittal of Monitoring Report to USACE	10/1/2015
Draft RAR	Submittal of Monitoring Report to USACE	1/25/2013
Revised Final RAR	Acceptance by USACE	4/5/2013
Meeting 2 – after RAWP review by NCDENR		1/15/2012
Meeting 2 – after RAR review by NCDENR		4/30/2013
PIP		1/15/2012
Public Meeting		4/30/2013

5. Records and Data Management

5.1 Administrative Records and Information Repository

Project-related information includes all documentation developed by the PIKA-PIRNIE JV Team in order to achieve the performance objectives specified in the PWS will be maintained in multimedia form. At the completion of the project, the PIKA-PIRNIE JV Team will provide the USACE PM a complete set of project records including correspondence, memorandums, trip reports, confirmation notices, test results, submittals, photographs and any other records or documents generated as a result of the project. This will be submitted in both hard copy format and electronic format.

6. Quality Assurance/Quality Control (QA/QC) Program

To ensure the success of the project and meet client goals, all work performed under this DO will adhere to the PIKA-PIRNIE JV Team's comprehensive Quality Management System and project-specific requirements. The PIKA-PIRNIE JV Team has developed and submitted, as part of the RAWP, a Construction Quality Control Plan (CQCP) and Operation and Maintenance (O&M) Plan (Appendices G and E respectively) in order to assure proper construction and operation of equipment and processes as defined in the PWS and the PIKA-PIRNIE JV Team proposed scope. All work conducted by the PIKA-PIRNIE JV Team at the Former CNAD will require concurrence from the Army COR and be consistent to meet the evaluation criteria set forth within the CQCP/O&M Plan.

The PIKA-PIRNIE JV Team will work directly with the USACE and NCDENR to determine project expectations. All project plans and deliverables will be reviewed to ensure they meet contractual requirements and that changes to existing documents will be communicated to project personnel. Lessons learned and corrective actions taken will be submitted to the USACE as part of monthly status reporting.

7. Health and Safety

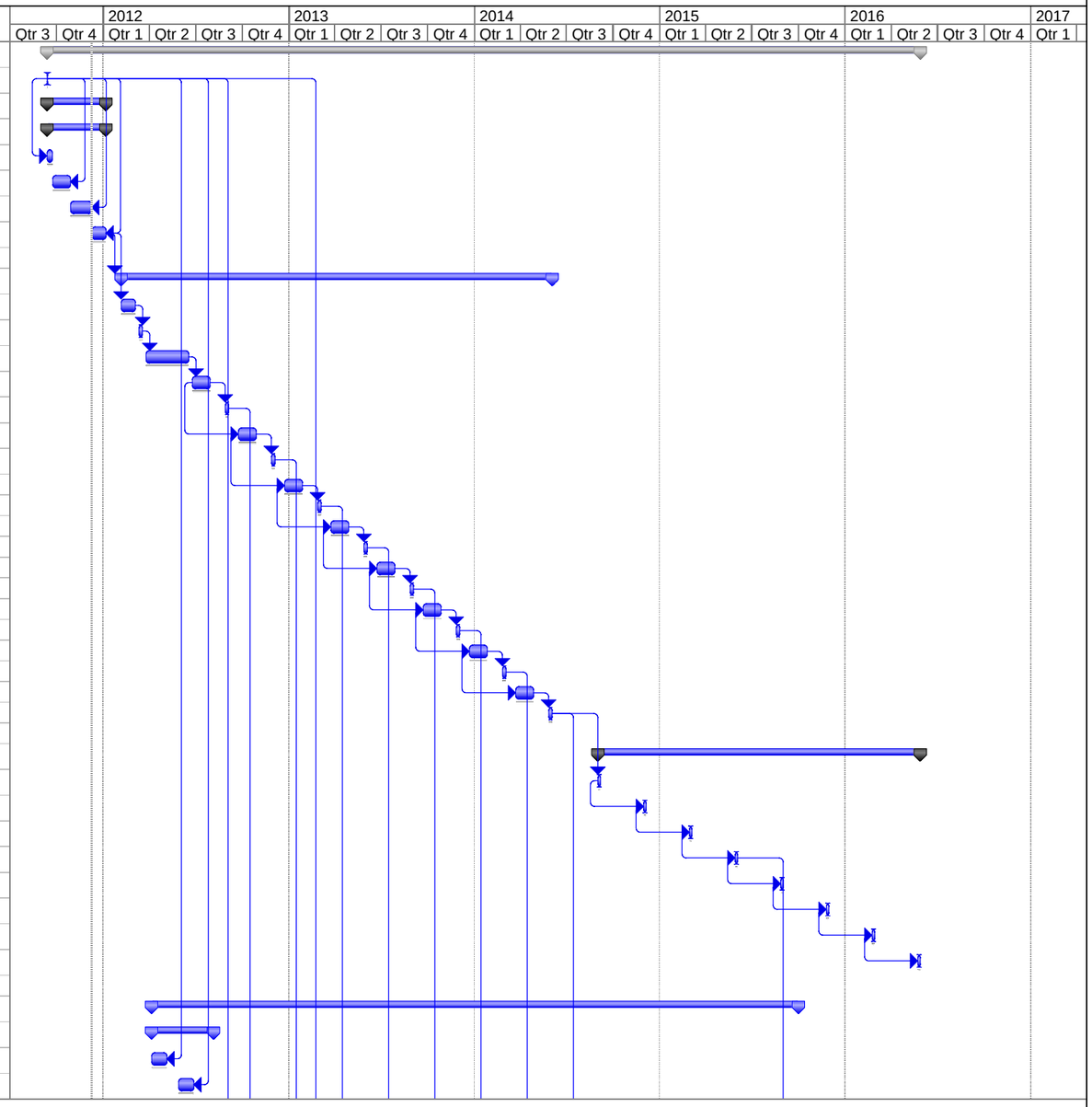
The H&S of all employees and subcontractors is a vital non-negotiable element of all work activities. All project personnel have the authority and responsibility to stop unsafe work and to conduct work activities in compliance with applicable USACE regulations and guidance. The PIKA-PIRNIE JV Team has prepared a Site-Specific Safety and Health Plan (SSHP) and Accident Prevention Plan (APP) (Appendix B of the RAWP) for work conducted under this DO and will ensure that its subcontractors, suppliers, and support personnel follow all H&S provisions established in the approved SSHP.

Appendix A

Project Schedule

Charlotte Naval Ammunition Depot
Project Schedule

ID	Task Name	Duration	Start	Finish	Predecessors
0	Charlotte Naval Ammunition Depot	1720 days	Tue 9/13/11	Sat 5/28/16	
1	Notice to Proceed	1 day	Tue 9/13/11	Tue 9/13/11	
2	Task 1	116 days	Tue 9/13/11	Fri 1/6/12	
3	Remedial Action Work Plan	116 days	Tue 9/13/11	Fri 1/6/12	
4	Progress Chart	10 days	Tue 9/13/11	Fri 9/23/11	1SS
5	Draft	35 days	Sat 9/24/11	Fri 10/28/11	1FF+45 days
6	Final	42 days	Sat 10/29/11	Fri 12/9/11	1FF+87 days
7	Revised Final	28 days	Sat 12/10/11	Fri 1/6/12	1FF+115 days
8					
9	Task 2: Remedial Action Implementation	849 days	Mon 2/6/12	Tue 6/3/14	7
10	Installation of transition zone and bedrock MWs	28 days	Mon 2/6/12	Sun 3/4/12	7FS+30 days
11	Baseline sampling event	7 days	Mon 3/12/12	Sun 3/18/12	10FS+7 days
12	Installation of Injection wells (bedrock / transition)	84 days	Mon 3/26/12	Sun 6/17/12	11FS+7 days
13	Injection Event #1	35 days	Mon 6/25/12	Sun 7/29/12	12FS+7 days
14	Performance Monitoring Event #1	7 days	Wed 8/29/12	Tue 9/4/12	13FS+30 days
15	Injection Event #2	35 days	Mon 9/24/12	Sun 10/28/12	13SS+91 days
16	Performance Monitoring Event #2	7 days	Wed 11/28/12	Tue 12/4/12	15FS+30 days
17	Injection Event #3	35 days	Mon 12/24/12	Sun 1/27/13	15SS+91 days
18	Performance Monitoring Event #3	7 days	Wed 2/27/13	Tue 3/5/13	17FS+30 days
19	Injection Event #4	35 days	Mon 3/25/13	Sun 4/28/13	17SS+91 days
20	Performance Monitoring Event #4	7 days	Wed 5/29/13	Tue 6/4/13	19FS+30 days
21	Injection Event #5	35 days	Mon 6/24/13	Sun 7/28/13	19SS+91 days
22	Performance Monitoring Event #5	7 days	Wed 8/28/13	Tue 9/3/13	21FS+30 days
23	Injection Event #6	35 days	Mon 9/23/13	Sun 10/27/13	21SS+91 days
24	Performance Monitoring Event #6	7 days	Wed 11/27/13	Tue 12/3/13	23FS+30 days
25	Injection Event #7	35 days	Mon 12/23/13	Sun 1/26/14	23SS+91 days
26	Performance Monitoring Event #7	7 days	Wed 2/26/14	Tue 3/4/14	25FS+30 days
27	Injection Event #8	35 days	Mon 3/24/14	Sun 4/27/14	25SS+91 days
28	Performance Monitoring Event #8	7 days	Wed 5/28/14	Tue 6/3/14	27FS+30 days
29					
30	MNA Events for Transition and Bedrock wells	635 days	Tue 9/2/14	Sat 5/28/16	
31	Quarter #1	5 days	Tue 9/2/14	Sat 9/6/14	28FS+90 days
32	Quarter #2	5 days	Mon 12/1/14	Fri 12/5/14	31SS+90 days
33	Quarter #3	5 days	Sun 3/1/15	Thu 3/5/15	32SS+90 days
34	Quarter #4	5 days	Sat 5/30/15	Wed 6/3/15	33SS+90 days
35	Quarter #5	5 days	Fri 8/28/15	Tue 9/1/15	34SS+90 days
36	Quarter #6	5 days	Thu 11/26/15	Mon 11/30/15	35SS+90 days
37	Quarter #7	5 days	Wed 2/24/16	Sun 2/28/16	36SS+90 days
38	Quarter #8	5 days	Tue 5/24/16	Sat 5/28/16	37SS+90 days
39					
40	Task 3: RA Report / Monitoring Report	1274 days	Fri 4/6/12	Thu 10/1/15	
41	Baseline Sampling Event Report	122 days	Fri 4/6/12	Sun 8/5/12	
42	Draft	30 days	Fri 4/6/12	Sat 5/5/12	1FF+235 days
43	Final	30 days	Tue 5/29/12	Wed 6/27/12	1FF+288 days



Project: Charlotte Naval Ammunition D
Date: Thu 12/8/11

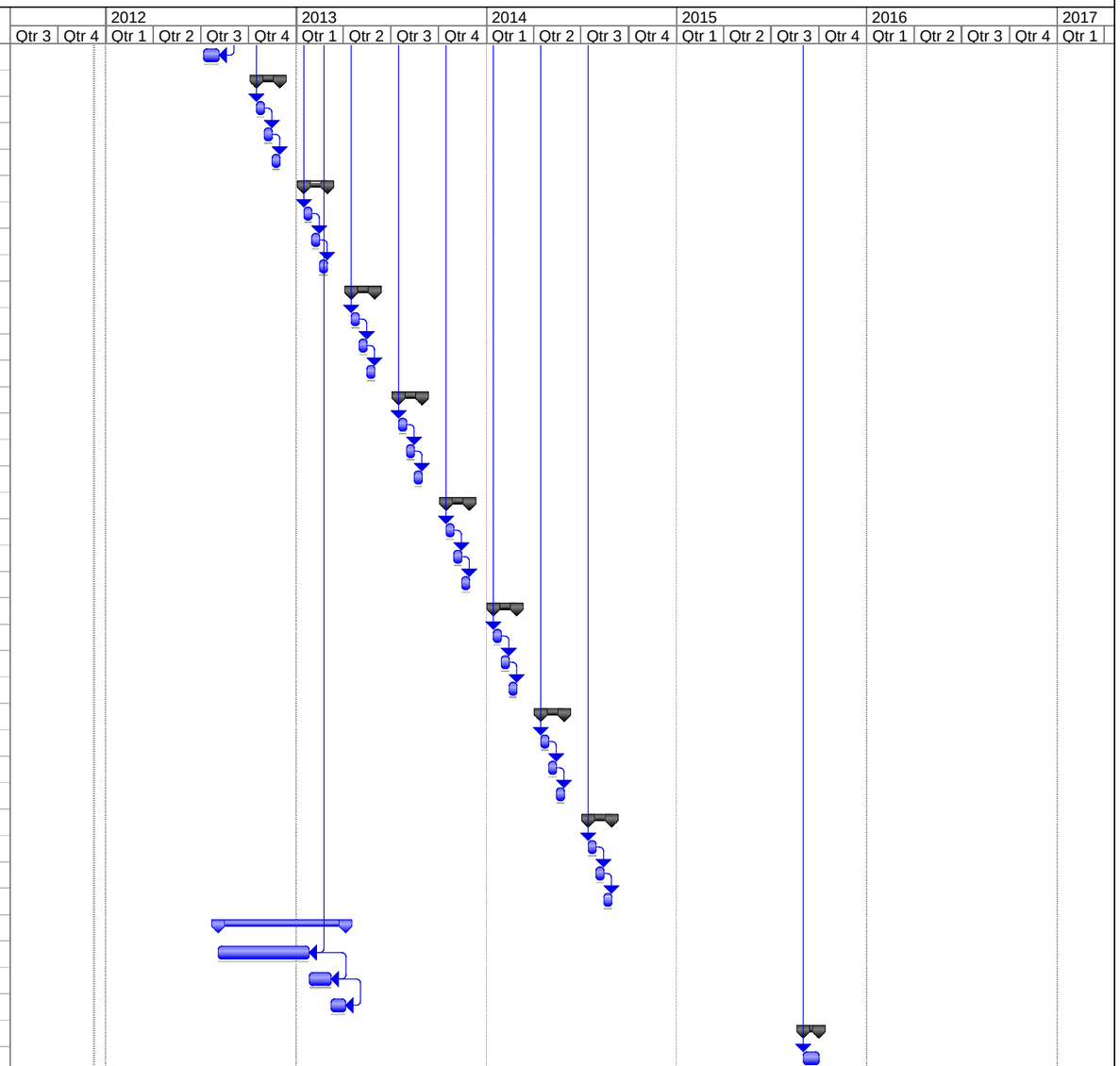
Task | Progress | Summary | External Tasks | Deadline
Split | Milestone | Project Summary | External Milestone

PIKA INTERNATIONAL, INC. — MALCOLM PIRNIE — A JOINT VENTURE

Contract No. W912DY-10-D0025, DO 0007
December 2011

Charlotte Naval Ammunition Depot
Project Schedule

ID	Task Name	Duration	Start	Finish	Predecessors
44	Revised Final	30 days	Sat 7/7/12	Sun 8/5/12	1FF+327 days
45	1st Performance Monitoring Report	45 days	Wed 10/17/12	Fri 11/30/12	
46	Draft	15 days	Wed 10/17/12	Wed 10/31/12	14FS+42 days
47	Final	15 days	Thu 11/1/12	Thu 11/15/12	46
48	Revised Final	15 days	Fri 11/16/12	Fri 11/30/12	47
49	2nd Performance Monitoring Report	45 days	Wed 1/16/13	Fri 3/1/13	
50	Draft	15 days	Wed 1/16/13	Wed 1/30/13	16FS+42 days
51	Final	15 days	Thu 1/31/13	Thu 2/14/13	50
52	Revised Final	15 days	Fri 2/15/13	Fri 3/1/13	51
53	3rd Performance Monitoring Report	45 days	Wed 4/17/13	Fri 5/31/13	
54	Draft	15 days	Wed 4/17/13	Wed 5/1/13	18FS+42 days
55	Final	15 days	Thu 5/2/13	Thu 5/16/13	54
56	Revised Final	15 days	Fri 5/17/13	Fri 5/31/13	55
57	4th Performance Monitoring Report	45 days	Wed 7/17/13	Fri 8/30/13	
58	Draft	15 days	Wed 7/17/13	Wed 7/31/13	20FS+42 days
59	Final	15 days	Thu 8/1/13	Thu 8/15/13	58
60	Revised Final	15 days	Fri 8/16/13	Fri 8/30/13	59
61	5th Performance Monitoring Report	45 days	Wed 10/16/13	Fri 11/29/13	
62	Draft	15 days	Wed 10/16/13	Wed 10/30/13	22FS+42 days
63	Final	15 days	Thu 10/31/13	Thu 11/14/13	62
64	Revised Final	15 days	Fri 11/15/13	Fri 11/29/13	63
65	6th Performance Monitoring Report	45 days	Wed 1/15/14	Fri 2/28/14	
66	Draft	15 days	Wed 1/15/14	Wed 1/29/14	24FS+42 days
67	Final	15 days	Thu 1/30/14	Thu 2/13/14	66
68	Revised Final	15 days	Fri 2/14/14	Fri 2/28/14	67
69	7th Performance Monitoring Report	45 days	Wed 4/16/14	Fri 5/30/14	
70	Draft	15 days	Wed 4/16/14	Wed 4/30/14	26FS+42 days
71	Final	15 days	Thu 5/1/14	Thu 5/15/14	70
72	Revised Final	15 days	Fri 5/16/14	Fri 5/30/14	71
73	8th Performance Monitoring Report	45 days	Wed 7/16/14	Fri 8/29/14	
74	Draft	15 days	Wed 7/16/14	Wed 7/30/14	28FS+42 days
75	Final	15 days	Thu 7/31/14	Thu 8/14/14	74
76	Revised Final	15 days	Fri 8/15/14	Fri 8/29/14	75
77	Remedial Action Report	245 days	Sat 8/4/12	Fri 4/5/13	
78	Draft	175 days	Sat 8/4/12	Fri 1/25/13	1FF+500 days
79	Final	42 days	Sat 1/26/13	Fri 3/8/13	78FF+42 days
80	Revised Final	28 days	Sat 3/9/13	Fri 4/5/13	79FF+28 days
81	MNA / Reporting	30 days	Wed 9/2/15	Thu 10/1/15	
82	1st Annual MNA Monitoring Report	30 days	Wed 9/2/15	Thu 10/1/15	34FS+90 days



Project: Charlotte Naval Ammunition D
Date: Thu 12/8/11

Task | Progress | Summary | External Tasks | Deadline
Split | Milestone | Project Summary | External Milestone

Appendix E

ERD Injection System Operation and Maintenance (O&M) Plan

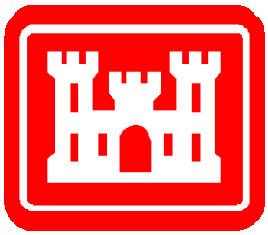
APPENDIX E
Enhanced Reductive Dechlorination Injection System Operation and Maintenance Plan

Former Charlotte Naval Ammunition Depot
Charlotte, North Carolina

December 2011

DERP-FUDS Project No. I04NC080301
Contract No.:W912DY-10-D0025
Delivery Order No.: 0007

PREPARED FOR:



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Enhanced Reductive Dechlorination Injection System Operation and Maintenance Plan

Former Charlotte Naval Ammunition Depot
Charlotte, North Carolina

Prepared for:
U.S. Army Corps of Engineers,
Huntsville Center

Prepared by:
PIKA-PIRNIE JV, LLC
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Our Reference:
DERP-FUDS Project No. I04NC080301
Contract No.: W912DY-10-D0025
Delivery Order No.: 0007

Date:
December 2011

Table of Contents

Acronyms and Abbreviations

APP	Accident Prevention Plan
ASIP	Arrowood Southern Industrial Park
bgs	Below Ground Surface
BOD	Biological Oxygen Demand
cis-DCE	cis-1,2-dichloroethene
CMUD	Charlotte Mecklenberg Utilities Department
CNAD	Charlotte Naval Ammunition Depot
CSM	Conceptual Site Model
CVOC	Chlorinated Volatile Organic Compound
ERD	Enhanced Reductive Dechlorination
FFS	Focused Feasibility Study
FSP	Field Sampling Plan
GPM	Gallons per Minute
H_2	Molecular Hydrogen
HDPE	High-Density Polyethylene
HSU	Hydrostratigraphic Unit
IDW	Investigation-Derived Waste
IRZ	In-situ Reactive Zone
JV	Joint Venture
mm	Millimeters
MNA	Monitored Natural Attenuation
MSDS	Material Data Safety Sheet
µg/L	Micrograms per Liter
µm	Micron
NCAC	North Carolina Administrative Code
O&M	Operation and Maintenance Plan
PIKA	PIKA International, Inc.
PIKA-PIRNIE JV Team	PIKA International, Inc./Malcolm Pirnie, Inc. Joint Venture LLC Team
Pirnie	Malcolm Pirnie, Inc.

PWS	Performance Work Statement
QAPP	Quality Assurance Project Plan
QC	Quality Control
psi	Pounds per Square Inch
PVC	Polyvinyl Chloride
RAO	Remedial Action Objective
RAWP	Remedial Action Work Plan
ROI	Radius of Influence
SAP	Sampling and Analysis Plan
SOW	Scope of Work
SSHP	Site Safety and Health Plan
TCE	Trichloroethene
TOC	Total Organic Carbon
TZ	Transition Zone
U.S.	United States
USACE	United States Army Corps of Engineers, Huntsville Center
VC	Vinyl Chloride
VOC	Volatile Organic Compounds

1. Introduction

This Operation and Maintenance (O&M) Plan presents the details of the selected remedy implemented for the former Charlotte Naval Ammunition Depot (CNAD) Site (the Site), located in Charlotte, North Carolina. This O&M Plan describes activities associated with the enhanced reductive dechlorination (ERD) remedial components as detailed in the Remedial Action Work Plan (RAWP). This plan includes details on system O&M procedures to be followed for proper remedy implementation. All work will be conducted in accordance with the Sampling and Analysis Plan (SAP) (RAWP Appendix C), which includes the Field Sampling Plan (FSP) and the Quality Assurance Project Plan (QAPP).

Data and other observations obtained from the installation, testing, startup and shakedown of the remedial systems will be used to further refine this O&M Plan and to develop optimal operational parameters and system efficiency. The collected data and observations from injection and monitoring events and other Site O&M activities will be compiled and reported in quarterly performance monitoring reports submitted to the United States (U.S.) Army Corps of Engineers, Huntsville Center (USACE). These reports will include the relevant system operational observations, a summary of field activities completed and discussion regarding adjustment/replacement of system components.

The objectives of this O&M Plan are to outline and present the methods and procedures for the achievement of the full-scale remedies detailed in the RAWP.

1.1 Site Location and History

The former CNAD complex is located in Charlotte, Mecklenburg County, North Carolina (see Figure 1-1 of the RAWP). At the time of operation, the entire CNAD complex encompassed approximately 2,266 acres of land. This area, now known as the Arrowood Southern Industrial Park (ASIP), is currently occupied by light industrial and commercial businesses. The former CNAD complex is bound by Cordage Street to the north, ASIP Building III to the east, Frito-Lay, Inc. to the south, and Nevada Boulevard to the west.

In June 1942, the Department of Navy signed a contract with the U.S. Rubber Company for the construction of 40-millimeter (mm) anti-aircraft ammunition shell loading and assembly plant in Charlotte, North Carolina known as the Charlotte CNAD. In 1945, planned production was cut and operation of the facility was transferred to the

U.S. Navy. In 1956, the Naval Depot status was changed from Maintenance to Inactive. At the time of operation, the entire CNAD complex occupied approximately 2,266 acres of land. In 1959, the former CNAD complex was sold to a local partnership and is currently occupied by light industrial and commercial businesses.

Two areas (1 and 2) of the site were used for the production of 40-mm anti-aircraft munitions. Area 1 consisted of anti-aircraft ammunition loading lines. This area was dedicated to the assembly of final rounds and was composed of 22 buildings. The largest of the buildings in Area 1 (1-60 and 1-70) were used for final assembly, packaging, and shipping of munitions.

The operations carried out in Area 2 were reportedly identical to those conducted in Area 1. Area 2 was also used to process returned ammunition after World War II. Only Area 2 was used after 1945 for reconditioning of returned munitions. A trichloroethene (TCE) vapor-degreasing operation was located on the southeast corner of building 2-30. The unit was used to remove cutting oil and preservatives from the exteriors of returned shells.

1.2 Remedial Action Objectives (RAOs)

RAOs are the desired outcome of the groundwater cleanup action. The following RAOs for the former CNAD complex, as specified in the Performance Work Statement (PWS), include:

- Address TCE at concentrations exceeding 500 micrograms per liter (µg/L) in groundwater through enhanced bioremediation using a dilute molasses solution injections in both the bedrock and transition zones (TZ); and

- Monitored Natural Attenuation (MNA) to reduce volatile organic compound (VOC) concentrations to below the North Carolina Administration Code (NCAC) 2L standard 2.8 µg/L.

The RAOs described in this RAWP focus on the design, construction, implementation, and operation of the in-situ ERD remedial system.

1.3 Plan Organization

- Section 1 – Introduction: This section presents the Site background information, plan objectives, and organization of the O&M Plan.

- Section 2 – ERD System Description: This section presents a description of the components of the groundwater remedy including a summary of the ERD technology, the injection infrastructure required to deliver carbohydrate solution to the subsurface, and the selected carbohydrate substrate.

- Section 3 – ERD System Operations: This section describes the field operating procedures for mixing and injecting the dilute molasses solution. The section contains relevant information to the mixing process, the target injection volumes, the estimated injection timeframes, and the operational monitoring activities conducted during each field event.

- Section 4 – General Equipment Maintenance: This section describes the manufacturer-recommended maintenance and the PIKA International, Inc.(PIKA)/Malcolm Pirnie, Inc.(Pirnie) Joint Venture (JV) LLC (PIKA-PIRNIE JV Team) best practices for ensuring system equipment integrity.

- Section 5 – Regularly Scheduled Maintenance: This section describes regularly scheduled maintenance to maintain system operation and prevent potential system problems.

- Section 6 – Troubleshooting: T his section outlines issues that may arise during injection activities and a summary of the procedures to facilitate continued operation.

- Section 7 – Spill Prevention/Contingency Planning: This section describes the best practices available for cleanup in the event that unintended release of bulk or dilute carbohydrate solution occurs.

2. ERD System Description

The ERD technology will be implemented at the Site to address areas where TCE concentrations in groundwater exceed 500 µg/L. The ERD system is designed to inject a dilute carbohydrate solution into injection wells located within the TZ and bedrock hydrostratigraphic units (HSUs) to support development of highly anaerobic, reducing conditions necessary for ERD.

The selected reagent for ERD at the Former CNAD Site is molasses. The bulk molasses solution will be delivered by tanker to the Site prior to and as needed during injection events and will be stored on-property for two months or less prior to use. During injection events, the bulk molasses will be diluted to the specified injection strength via an in-line mixer with the available potable water supply. After mixing has been completed, the dilute molasses solution will be delivered into target injection wells via below-grade manifolds and a subsurface conveyance distribution line (see Appendix A of the RAWP). The steps involved in implementing the ERD technology are discussed in further detail below.

2.1 ERD Technology

As detailed in the RAWP, the ERD anaerobic bioremediation technique involves the delivery of a degradable source of organic carbon into the impacted groundwater to achieve four basic goals:

1. *Overcome the continuous electron acceptor supply:* This includes oxygen, nitrate, and other electron acceptors that tend to support a more aerobic microbial community.

2. *Produce molecular hydrogen (H_2) through fermentation:* Molecular H_2 is a product of fermentation and is used as an electron donor by dechlorinating bacteria.

3. *Achieve complete dechlorination of the target compounds:* Dechlorinating bacteria use the hydrogen produced through fermentation as an electron donor and chlorinated volatile organic compounds (CVOCs) as electron acceptors. Hydrogen atoms are substituted for chlorine atoms in the dehalorespiration process resulting in a step-wise chemical reduction of the CVOC, which for TCE follows the pathway:

TCE → cis-DCE → VC → Ethene

4. *Achieve dissolution of nonaqueous phase contaminant mass:* Under natural conditions, the dissolution of hydrophobic organic compounds (making them available for treatment) is very slow, allowing groundwater plumes to persist for many decades if the dissolution rate cannot be enhanced. With ERD, this enhancement is achieved through a variety of mechanisms.

The key to successful implementation of ERD is delivering, distributing and sustaining an adequate supply of organic carbon donor to create strongly-reducing conditions, while maintaining the pH above 5. As demonstrated by the ERD pilot test, sufficient volumes of carbohydrate solution can be delivered to the bedrock and TZ units at the Site to reduce CVOC concentrations. Throughout the pilot test, total organic carbon (TOC) concentrations were sustained above background, methanogenic conditions were established and ERD was observed that was distinguishable from the ongoing naturally occurring dechlorination elsewhere at the Site.

2.2 Injection Well Network

As described in the RAWP, ERD implementation will include installation of an injection well network (see Figures 3-1 and 3-2 of the RAWP) to support delivery of organic carbon reagent for treatment of TCE concentrations in the TZ and bedrock HSUs. Injection areas were determined based on the area where TCE concentrations exceed 500 µg/L.

Fifty-three (53) injection wells will be completed in the TZ HSU in the four identified plume areas where elevated TCE concentrations have been observed (see Figure 3-1 of the RAWP). Well locations will be installed in nine transect lines oriented approximately north-south or perpendicular to groundwater flow in the TZ. Injection wells in each transect will be spaced approximately 35-feet apart in order to achieve lateral distribution and coverage along the transects based on the expected organic carbon radius of influence (ROI).

Each TZ injection well will be installed to an approximate total depth of 25 feet below ground surface (bgs) with a 17 foot screened interval from 8 to 25 feet bgs. Each well will be installed via a combination of hollow stem auger and air rotary drilling methods and will be completed with 2-inch diameter Schedule 80 polyvinyl chloride (PVC) casing with stainless steel wire-wrapped well screens. A coarse sand pack will be installed to 2 feet above the top of screen; 2 feet of fine sand will be placed on top of

the coarse sand pack to separate the sand pack from the well seal. The remainder of the well annulus will be sealed with a neat cement grout. Grain size analysis will be performed on select TZ monitor wells installed in order to confirm design of the appropriate filter pack of the TZ injection wells. All final well construction details will be determined in the field.

Twenty-five (25) injection wells will be completed in bedrock (see Figure 3-2 of the RAWP) to address TCE impacts greater than 500 µg/L in the bedrock HSU. Injection wells will be placed along three transect lines oriented from east to west and perpendicular to the bedrock fracture planes and understood groundwater flow direction in bedrock. Based on results collected during the pilot test, injection wells in each transect will be spaced approximately 80-feet apart in order to achieve complete lateral distribution and coverage based on the expected organic carbon ROI.

Each bedrock injection well will be installed to an approximate total depth of 100 feet bgs with a screened interval from 25 to 100 feet bgs. Each well will be installed via rotary combination of hollow stem auger and air rotary drilling methods and constructed out of 2-inch diameter Schedule 80 PVC casing with stainless steel wire-wrapped well screens. A coarse sand pack will be installed to 2 feet above the top of screen; 2 feet of fine sand will be placed on top of the coarse sand pack to separate the sand pack from the well seal. Similar to the TZ injection wells, the remaining well annulus will be sealed with a neat cement grout. All final well construction details will be determined in the field.

Prior to installation of the proposed injection wells, a baseline groundwater sampling event will be conducted on a combination of existing and newly installed monitoring wells to confirm the orientation of the injection well network. The data will be utilized to review the distribution of TCE within shallow and intermediate bedrock, supplement the historical site-wide data collected from the site (most recently conducted during 2006), and refine the injection well network as necessary. In addition, an iterative injection well installation program will be conducted to confirm injection transect positioning. Approximately one injection well from each transect will be sampled during well drilling and samples will be submitted to the laboratory for expedited analysis. Data collected will be used to confirm the orientation of the well transects while drilling activities are underway. The actual locations of the injection wells will be revised, as needed, based on the baseline sampling event and following review of results collected during the iterative well installation and sampling activities. The iterative program will be designed to increase delineation within these areas and ensure that the installed injection infrastructure will allow for maximum remedial benefit. Therefore, the well locations

presented as part of the RAWP are subject to change based on the results of the baseline monitoring event, the injection well installation sampling results, and/or revisions to the conceptual site model (CSM).

2.3 ERD Distribution Line

Subsurface 1-inch high-density polyethylene (HDPE) conveyance piping will connect the injection wells to centrally located distribution vaults. Two inch header piping will connect the distribution vaults to the injection systems. The dedicated piping runs will minimize handling and manipulation of above-grade distribution hose during injection events and ensure minimal disruption to current property activities during the injection events. As presented on Drawing C3 in Appendix A of the RAWP, the conveyance piping will be installed in trenches excavated to 3 feet below grade. The dedicated pipe for each injection well will be placed on a minimum of 3-inch centers within the trench, bedded and backfilled with the native soil removed to create the trench. Native material removed from the trench will be moisture conditioned and screened before being used as pipe bedding and backfill material. The moisture screening process used during conveyance piping installation will include visual screening to prevent the use of saturated soil materials as trench backfill. Based on the depth of the trench, it is not expected that significant quantities of saturated soil materials will be encountered during the trenching process. Physical soil screening will include the removal of larger aggregate or construction materials from the native soils prior to use in backfill. During backfill, water utility locating tape will be placed along the length of the injection trench to ensure that the location can be identified during future utility inspections. Following backfilling, the trench will be completed to pre-installation conditions, with asphalt or seeded soils based on location.

Conveyance piping routes are based on the injection well locations and current understanding of site constraints. Three portions of the trenched delivery piping will be installed via horizontal boring underneath the railroad spur to connect the reagent delivery lines to injection wells located between the two rail spurs (injection wells IW-44 through IW-53, BIW-14 through BIW-25, and BIW-1 through BIW-9). One additional horizontal boring will be used to connect the mixing system to the injection wells north of Cordage Street (IW-1 through IW-11 and IW-14). During system installation, the selected locations of the conveyance piping may be revised, as needed, based on underground utilities or other existing Site constraints or based on revisions to the injection transects identified during baseline groundwater and/or well installation sampling activities.

Each of the 13 remote distribution manifolds will be installed in a subsurface, traffic-rated concrete vault, and will contain totalizing flow meters and pressure gauges required to monitor injection flow rates and wellhead pressures during injection events. Based on the available water supply, injection wells within the northern portion of the Site will be divided into three different well sets. Flow meters and pressure gauges within the distribution manifolds will be configured to control flow to multiple (3) injection wells. During injection startup, valves within the distribution manifold will be configured such that flow is directed to one of the three injection well legs, with the other two valves being closed. Injections to the first set of wells will continue until the target injection volumes are achieved, following which the on-Site operator will manually reconfigure the control valves to direct flow to the second injection well distribution line. As wells are completed, select wells will be brought offline and the system will be reconfigured to convey the organic carbon reagent to injection wells within the subsequent well set. This arrangement will allow detailed monitoring of flow rates, injection volumes, and wellhead pressures to each of the injection wells. Construction details for the distribution vaults are included on Drawing M1 in Appendix A of the RAWP. The concrete vault will be approximately 3 feet wide by 4 feet long. The vault will be constructed with an open bottom to allow entrance for the 1-inch conveyance lines. The vault will be covered with a spring-assisted aluminum cover, which can be locked during and between injection events. As shown on Drawing M1 in Appendix A of the RAWP, the 1-inch PVC pipe will be welded such that it turns approximately 90 degrees from the trench into the vault. Within the vault, the end will be fitted with a threaded steel nipple and a 2-inch female cam-lock fitting.

2.4 Molasses Mixing Injection System

Two fixed injection systems will be utilized at the Site in order to minimize the number of under railroad bores and the overall lengths of piping and trenching that will be needed. One of the systems will be positioned north of the railroad yard and one will be positioned to the south (see Figures 3-1 and 3-2 of the RAWP).

The injection systems will be used to facilitate semi-automated mixing and distribution of the dilute molasses solution to injection wells (Drawing PFD and PID1 in Appendix A of the RAWP). Each of the two injection systems will be comprised of one potable water equalization tank used to equalize the flow coming from the potable water supply (T-103 and T-104), a molasses transfer feed pump (P-101 and P-103), a dilution water feed pump (P-102 and P-104), and a bag filter assembly (BF-201A/B and BF-202A/B). These components will be connected via 2-inch PVC pipe and valves to control flow within the system. The flow control on the molasses feed pump (P-101 and P-103)

and the dilution water feed pump (P-102 and P-104) will be automated during system operation to meter the necessary flow rates and facilitate mixing of the target dilute molasses concentration within the static mixer prior delivery through the bag filter assembly and to the injection wells.

The pumps, flow meters and pressure gauges within the two mixing stations will be tied into a control panel located within each of the injection systems. System controls will allow for automated adjustment and will be constructed to allow operation via remote telemetry (Drawing PID1 in Appendix A of the RAWP). Remote system control will allow for system operation and monitoring during injection events while field operators are off Site.

2.5 Selected Reagent

The selected reagent for ERD application at the Site is feed-grade molasses. The material safety data sheet (MSDS) is included in **Appendix A**. The bulk (100 percent) molasses solution will be delivered to the Site, mixed to achieve the targeted injection strength (1 percent), and injected into the subsurface to sustain TOC concentrations over time. Ongoing process monitoring results (*i.e.,* VOC trends, TOC concentration, and pH) will be used to evaluate subsurface conditions within the treatment areas. Based on these results and the rate of observed TOC degradation and/or flushing from the injection area, adjustments to the injection frequency, target volumes and molasses solution strength may be recommended to increase system efficiency and optimize remedial performance.

3. ERD System Operation

Injection events will be conducted on a quarterly basis for a period of two years. This frequency is based on the observed longevity of TOC within the subsurface, washout of the TOC from the injection area, and the required dosing frequency to achieve the necessary subsurface reducing conditions to support treatment to the remedial objectives. TOC samples collected during the performance monitoring events will be used to evaluate the required injection frequency and adapt the injection program, as necessary. Operation of typical in-situ reactive zone (IRZ) systems relies on sustaining TOC concentrations above baseline levels to promote development of reducing conditions and support dechlorination, therefore, the necessary injection frequency and total injection volumes will be adjusted to ensure optimal remedial performance.

The following section includes a description of the injection mixing system and a summary of the logistics associated with implementation of the ERD technology.

3.1 Molasses Mixing and Delivery

Approximately 21,000 gallons of 100 percent food-grade molasses will be delivered to the Site via tanker trucks during each injection event. The molasses will be offloaded to the external vented and jacketed carbon storage tanks located adjacent to the conex boxes housing the injection system equipment and controls. The storage tanks to the north and south of the railroad tracks will have capacity for 4,100 and 11,500 gallons, respectively. Offloading of molasses to the tanks will be supervised by the PIKA-PIRNIE JV Team personnel during all injection events.

To facilitate mixing, the bulk molasses solution will be pumped via the molasses transfer pump (P-101 and P-103) from the external storage tank into the conex box through a 2-inch PVC, heat-traced pipeline. Concurrent delivery of molasses from the storage tank (T-101 and T-102) and dilution water from the equalization tank (T-103 and T-104) will produce the diluted molasses solution in-line static mixing. Automated controls will adjust the operational speeds of the molasses feed pump and dilution water feed pump to achieve the target injection solution strength.

Flow meters will monitor the effluent of the molasses feed pump (P-101 and P-103) and dilution water feed pump (P-102 and P-104) during injection events to ensure that a consistent molasses solution strength is being produced over the course of each injection event. Flow meters will be tied into the control panel where current solution strength, effluent flow rate, and the total volume of solution injected will be monitored.

Use of these controls will enable quality control (QC) monitoring of the system while field personnel are off Site. During each injection event, field personnel will conduct approximately two daily visits (*e.g.,* Monday and Friday) while injections are ongoing. Inspections during these visits will be conducted to ensure consistent operation of the system and to collect injection volume and pressure readings. The control panel will also provide notification of any alarms, system failures, or shutdowns. Additional troubleshooting or inspections will be conducted during more frequent Site visits, as necessary. Automation will enable full operation of the systems without the PIKA-PIRNIE JV Team personnel on Site.

3.1.1 Molasses Injection and Monitoring

Molasses solution from the static mixer will be directed through a bag filter assembly (BF-201A/B and BF-202A/B) and into the wellhead distribution manifolds and conveyance lines. The bag filter assembly (BF-201A/B and BF-202A/B) will be used to remove any particulate material within the molasses solution prior to delivery to the injection wells. Two bag filters will be connected in parallel and each unit will contain one 100 micron (µm) filter. The paired bag filters will be used to allow for periodic filter change out and cleaning, as necessary, without interrupting the dilute molasses solution injection. Pressure gauges on either side of the bag filters will be monitored during injection events. A significant drop in pressure (relative to clean filter baseline) on the downstream side of the filter will indicate that filter cleaning is required.

3.1.1.1 Molasses Distribution

From the injection systems, the molasses solution will be directed to the wells through approximately 2,200 linear feet of 2-inch diameter pipe that will serve as conveyance lines. Conveyance piping will be connected to the thirteen remote distribution manifolds locate d across the Site (see Drawing C1 in Appendix A of the RAWP). Approximately 6,500 linear feet of individual one-inch lengths of subsurface conveyance piping will connect the distribution manifolds and wells. Solution will be delivered to the 53 injection wells installed in the TZ and 25 injection wells installed in the bedrock zone.

Injections will be conducted over the course of each event within the limits of the available water supply. Using the design injection flow rates given in the Focused Feasibility Study (FFS) (1.5 gallons per minute [gpm] for transition wells and 6 gpm for bedrock wells), anticipated water supplies of 195 gpm and 35 gpm would be required for the northern and southern injection areas, respectively. Based on previous discussion with the Charlotte Mecklenberg Utilities Department (CMUD), the Cordage

Street water supply line (north injection system) has an available flow of 70 gpm and the Nevada Boulevard water supply line has an available flow of 100 gpm. Based on the dilution water required for continuous operation of the northern and southern injection systems injections within each area will be separated into three different groups of injection wells. Under this arrangement, injections will proceed within the first set of wells until the target injection volume is achieved, following which flow controls would be reconfigured by operators to facilitate injection into the subsequent set of injection wells. Specific groupings of wells may be adjusted over the course of the program to optimize overall efficiency.

Injection volumes and wellhead pressures are discussed in further detail in Sections 3-2 and 3-3, respectively.

3.2 Injection Volumes

Based on a review of the pilot test data, it is anticipated that approximately 12,000 gallons and 58,000 gallons will be injected into the TZ and bedrock zone wells, respectively. These target volumes were estimated based on the target ROI of 17.5 feet in the TZ and 40 feet in the bedrock unit (half the distance between injection wells), the proposed length of vertical well screen and an estimated mobile porosity. The method for estimating these volumes is presented below:

$$V_{inj} = \pi \times r_{inj}^{\ 2} \times h \times \theta_m$$

Where:

r_{inj} = radius of influence = 17.5 feet in TZ and 40 feet in bedrock

h = target interval thickness = 17 feet in TZ and 75 feet in bedrock

θ_m = the mobile porosity (percentage of interconnected pore spaces providing flow) of the formation, assumed to be 10 percent and 2 percent in the TZ and bedrock, respectively

V_{inj} = volume of injection = 12,000 gallons in TZ and 58,000 gallons in bedrock

This calculation was used to estimate the initial target volumes for the wells completed in the TZ and bedrock. Based on these estimates, the target volumes are 12,000 gallons for TZ wells (17-foot screen) and 58,000 gallons for bedrock wells (75-foot

screen). These volumes will be refined as necessary over the active injection period based on TOC concentrations observed during process monitoring events.

Based on pilot test results, it has been assumed that the flow rates in TZ and bedrock zone injection wells are 1.5 and 6 gpm, respectively. The achievable flow rates will be evaluated and adjusted in the field during injection events. Flow rates may vary significantly between well locations due to subsurface heterogeneity. Therefore, low wellhead pressures will be applied to injection wells to increase the rate of delivery to the extent possible to achieve the target injection rate and to minimize overall injection time. These pressures are discussed in greater detail in Section 3.3. The estimated durations (in 24-hour days) for TZ and bedrock injection wells are presented below:

- TZ injection wells – based on a target injection rate of 1.5 gpm and a target volume of 12,000 gallons, the injection duration is approximately 5 – 6 days.

- Bedrock injection wells – based on a target injection rate of 6 gpm and a target volume of 58,000 gallons, the injection duration is 6 – 7 days.

3.3 Injection Pressures

Solution delivery to each injection well will be controlled via manual manipulation of the valves included in the manifold assemblies. While valves will be opened to promote solution flow into the well at the target injection flow rates (1.5 gpm in TZ, 6 gpm in bedrock), the flow rate will be balanced to ensure that the injection pressure applied to the wellhead does not result in the development of preferential flow fractures within the subsurface, does not result in solution daylighting, and protects the overall integrity of the aquifer matrix and injection well construction. To maintain protectiveness of the injection wells and to ensure that the injection solution is distributed within the target injection interval, gravity-feed delivery or low pressure (less than 5 pounds per square inch [psi]) will be applied. These pressures will be re-evaluated based on the observed performance of the system and will be further refined (*i.e.,* increased) based on field observations during system startup for the TZ and bedrock zone injection wells.

4. General Equipment Maintenance

4.1 Molasses Feed Pump

A Moyno cast iron positive displacement gear pump (or approved equal) (P-101 and P-103) will be used to transfer molasses from the large volume tanks (T-101 and T-102) during injection events. Flow rate from the pump will be visually monitored during on-Site inspections and will via telemetry when field personnel are off Site. Flow rates will also be evaluated during each of the injection events to confirm that the pump operation is consistent with the specifications. Automated controls will be used to adjust the operating speed of the pump so that a steady flow rate of the desired molasses solution concentration is being injected from the system to the wells. A service manual for the pump is included in **Appendix B.** Detail regarding cleaning and flushing of the pump following each injection event is presented in Section 5.

4.2 Dilute Water Feed Pump

A Goulds™ centrifugal pump, model 3296 EZMAG (or approved equal) (P-102 and P-104) will be used to transfer the water from the dilution water storage tank (T-103 and T-104). Automated controls will allow flow adjustments to control the flow rate at which the feed pump is operating, so that the pump works with the molasses transfer pumps (P-101 and P-103) to achieve the correct system flow rate and solution strength. The flow from the feed pump will be monitored during each injection event by monitoring the flow readings and will be used as part of system QC. Additional instructions on O&M activities are included in **Appendix B**.

4.3 Static Mixer

A Koflo Corporation static mixer will be used to mix molasses and water in-line to create the dilute molasses solution. Static mixers contain fixed mixing elements which create mixing of the molasses and water from the energy of the flow stream. During injections, the effluent from the mixer will be visually inspected to ensure that adequate mixing of the solution is occurring. Aside from flushing the mixer after injection events, discussed in greater detail in Section 5.4, minimal maintenance should be required.

4.4 Filtration System

The filtration system is composed of duplex Hayward Flow Control Systems bag filters (BF-201A/B and BF-202A/B). The bag filters will be aligned in parallel and will each

contain a 100 µm bag filter. The bag filter housings are designed to facilitate easy replacement via swing bolts and banded closure located at the top of the housing. The injection system is plumbed to allow flow through a single bag filter while change outs are being conducted. One pressure gauge is located on the upstream and downstream sides of each filter housing to allow monitoring of filter performance during injection events. General maintenance for the bag filter assembly will be conducted by closing the influent and effluent valves to the filter, depressurizing the filter housing, and then inspecting and/or replacing the bags. Pressure gauges on either end of the bag filters will be connected to the automated system controls to allow evaluation of filter performance while field personnel are off Site.

4.5 Pressure Gauges

Pressure gauges located within the ERD distribution system will be used to monitor the mixing and injection processes to ensure that the system is functioning as designed. Pressure gauges are used to monitor the differential pressure at the bag filter assembly and injection pressures on each distribution line and injection well manifold. General maintenance is not required for these gauges, but working conditions will be documented during startup inspections prior to beginning each injection event. Parts that are not in working condition (no observed pressure response or fluid leaking from inside the gauge, for example) will be replaced as necessary.

5. Regularly Scheduled Maintenance

Regularly scheduled maintenance is designed to maintain the integrity of system infrastructure and to prevent potential O&M problems by proactively preparing the system for use or shutdown. The general Site maintenance, injection system maintenance, and well maintenance are outlined below.

5.1 Injection System Maintenance

Injection system inspections and general system maintenance will be completed at the beginning and end of each injection event. Observations will be made regarding the operation and appearance of system components. Details of the system cleaning and maintenance activities are detailed below.

5.2 Pump Maintenance

During system startup, the flow rating from the molasses pumps (P-101 and P-103) will be determined to ensure accurate mixing of the dilute molasses solution. The flow rating will be determined by recording the length of time required to fill a receptacle of known volume. During the injection, visual observation and daily flow reading measurements will be taken and recorded in the field logs to ensure that pump operation is consistent and to determine if maintenance is required. Following each injection event, the pump will be rinsed during the clean water flush activities presented in Section 5.4.

The dilute molasses water feed pumps (P-102 and P-104) will be monitored during system startup to ensure that pump performance is consistent with design flow rates and previous field events. Changes in pump performance will be noted in the system inspection logs and O&M activities will be conducted as necessary. Following each injection event, the pump will be rinsed during the clean water flush activities presented in Section 5.4.

5.3 Well Maintenance and Redevelopment

Wellhead pressures and injection flow rates collected over the course of the active ERD injection program will be used to evaluate the long-term integrity of the injection well network. Over the length of the active remediation period, decreased well performance (indicated by increased wellhead pressures and decreased flow rates) will be used to evaluate whether well redevelopment is required. Field observations and

detail included in the injection logs will be used to support these determinations over time. Well redevelopment may entail standard jetting and/or brushing procedures or addition of a reagent (*i.e.,* biocide, hydrogen peroxide) to remove retained residues and/or biofilms that build up or are retained within the well casing and filter pack. USACE approval will be obtained prior to using any reagents for well redevelopment.

Following achievement of the target injection volume in all wells, the clean water rinse cycle will serve to push residual dilute molasses within the mixing system, conveyance pipelines, and injection wells into the formation. It is anticipated that a total rinse volume equivalent to at least 50 gallons of water will be used per well (4,400 gallons) for this process, but the required rinse volumes will vary based on the volume of water required to flush the above-grade distribution system. This process is presented in detail in Section 5.4.

5.4 ERD System Cleaning

Removal of the bulk and dilute molasses solution from the above-grade ERD mixing and distribution infrastructure following each injection event is essential to ensure the long-term integrity of system components. The objective of the clean water rinse cycle is to use a sufficient volume of water to rinse the mixing and delivery infrastructure such that clean water arrives at the injection wells at the end of the cycle. To accomplish this, following completion of all injection activities, water from the on-Site potable source will be used to rinse residual bulk molasses from the molasses storage tanks (T-101 and T-102) to the point at which clean water exits the tanks and flows through the molasses gear pumps (P-101 and P-103). Once the tanks and gear pump have been rinsed (*i.e.,* clean water is observed entering the bag filters), they will be removed from system operation. Additional clean water will be used to supply a continuous stream of clean water through the bag filters, conveyance pipelines, distribution manifold assemblies, and to the injection wells. Well caps on select injection wells will be removed and rinse water flowing through the subsurface conveyance lines will be visually inspected to ensure that a sufficient volume of water has been flushed through the 1-inch conveyance lines to remove residual molasses solution.

Flow measurements will be recorded during the water rinsing cycle to track the specific volume of water delivered to each of the injection wells. The distribution infrastructure will be operated such that all wells receive at least 50 gallons of clean water during the cycle (4,400 gallons total). As described above, the total volume of water required to rinse the system during the first full-scale injection event will be recorded and will be

used for planning subsequent rinse cycles. Modification to the rinse volume will be conducted to ensure that residual molasses is rinsed from delivery system components. Depending on overall timing and field logistics, individual injection lines and wells that are part of the first or second well sets may be rinsed following delivery of the target injection volumes prior to removal from operation.

6. Troubleshooting

The following table presents a general description of potential problems that may be encountered during normal operation of the ERD injection equipment. This section describes the probable solution(s) required to alleviate the problem once detected by on-Site field personnel. An adequate volume of molasses will be ordered prior to each injection event.

Problem Identification	Probable Cause	Probable Solution
Elevated Mixing/Injection Line Pressure	Bag Filter(s) (BF-201A/B, BF-202A/B) fouled	1. Remove suspect piece of equipment from service. Clean out debris and other accumulated solids. 2. Replace bag filters as necessary.
	Injection well is fouled	1. Flush well with clean water. Two well volumes. 2. Well redevelopment required (jetting and swabbing techniques). 3. Introduction of chemical reagent (*e.g.,* biocide or hydrogen peroxide).
	Subsurface conveyance infrastructure is fouled	1. Evaluate whether increased pressure is observed within distribution vault or in 1-inch conveyance lines. 2. Flush line with clean water.
	Inline valve not fully open	1. Verify position of all valves during operation.

Problem Identification	Probable Cause	Probable Solution
Molasses Solution not at target concentration	Gear pump malfunction	1. Service pump. 2. Replace pump.
	Flow meter malfunction: Water Flow Meter Injection Line Flow Meter	1. Verify flow meter operation. Clean out debris or other accumulated solids. 2. Replace as necessary.
	Molasses Lines Fouled	1. Purge molasses lines with clean water.
	On-Site Water Flow Rate too low/high	1. Verify flow meter operation, correct as needed.
Foaming in Molasses Storage Tank (T-101 and T-102)	Molasses Solution Fermentation	1. Drain molasses from tank. 2. Purge all residual material to ensure no biomass is left in tank. 3. Flush tanks with clean water. 4. Flush with clean water.
Low Mixing/Injection Line Pressure	System Leak	1. Determine and isolate source of leak. Repair as needed.
Poor Injection (No pressure or volume into well)	Formation Pressurization	1. Verify molasses injection / daylighting observation. 2. If daylighting observed stop injection and reevaluate injection parameters.

7. Spill Prevention and Contingency Measures

In accordance with the Accident Prevention Plan (APP) and Site Safety and Health Plan (SSHP) (Appendix B of the RAWP), all efforts will be made to prevent spills from occurring during the work activities. This section discusses spill prevention and contingency measures associated with O&M of the ERD System.

7.1 Potential Impacts

The proposed scope of work (SOW) involves off-loading of molasses and dilution with potable water prior to injection. If released, molasses is a non-hazardous substance and expected to have minimal impact to the environment. If an unanticipated spill or release does occur, appropriate measures will be taken to minimize the discharge to waterways or sewers that may result in high biological oxygen demand (BOD) levels and oxygen depletion in aquatic systems.

7.2 Spill Prevention

The following spill prevention measures will be followed during work activities:

- Take adequate measures to prevent spills during off-loading of the molasses. These measures will include storage of molasses in a vented tank suitable to handle products with specific gravity of at least 1.3 and the use of properly sized equipment and hosing, designated unloading areas and lockable ball valves to secure the poly tanks.

- Take adequate measures to prevent spills during injections resulting from pumps, hoses, poly tanks and other injection operations. These measures will include routine inspections of pumps, hoses and connections and routing hoses away from vehicular traffic, railroad tracks, and operational areas.

- Utilize practices that will reduce the potential for spills. These practices include not overloading containers (by volume or weight), drivers traveling at slow speeds and maintaining driving surfaces in clean and good condition.

7.3 Contingency Measures

7.3.1 Daylighting

In the event that a molasses solution penetrates the vadose zone or the grouted well annulus and pools on the surface, injection activities will be temporarily shut down and evaluated with injection volumes and pressures documented for reference. The injection rate and/or pressure will be modified and surface conditions and possible subsidence will be monitored. If daylighting continues, the injection at the respective location will cease and the total injection volume will be noted. Future injections at that location will be monitored for daylighting or discontinued altogether.

7.3.2 Spills

Although molasses is non-hazardous and readily biodegradable, a molasses spill, should one occur, will be responded to immediately. A PIKA-PIRNIE JV Team employee will be on Site during each molasses tank delivery and approximately two days a week during injection events. In the event of a spill, the PIKA-PIRNIE JV Team field staff will follow these contingency measures:

- Stop the source of the spill.
- Isolate the spill area, keep all unnecessary personnel out of the area and estimate the volume of the spill(s).
- Use engineering controls such as sand bags or dirt berms to prevent migration to waterways or sewers.

Additional spill response information can be found in the Molasses MSDS (**Appendix A**). In the event of a true emergency, the emergency action plan procedures in the SSHP (Appendix B-1 of the RAWP) should be consulted and the PIKA-PIRNIE JV Team Project Manager should be contacted as soon as possible.

8. Management of Investigation-Derived Waste (IDW)

IDW will be managed in accordance with the IDW Management Plan – Appendix F of the RAWP.

Appendix A

Molasses MSDS

MATERIAL SAFETY DATA SHEET
MOLASSES/MOLASSES BLENDS/BINDERS

1. CHEMICAL PRODUCT AND COMPANY IDENTIFICATION

Chemical Name	Chemical Formula	Molecular Weight
N/A	Mixture of liquid Agricultural commodities	No data

Trade Name – Molasses/Molasses Blends

Synonyms: DOT Identification No.

Liquid animal supplement: N/A

Company Identification:
Westway Trading Corporation
365 Canal Street, Suite 2900
New Orleans, Louisiana 70130
(504) 525-9741

2. COMPOSITION, INFORMATION ON INGREDIENTS

Component(s), Chemical Name	CAS Registry No.	%(Approx.)	ACGIH TLV-TWA
Proprietary **See ingredient tag**	NA	No data	No data

3. HAZARDS IDENTIFICATION

Emergency Overview

This material should be stored in a vented tank designed to contain a material with a specific gravity of 1.3 or greater. Material can ferment if excessive moisture contamination is allowed. Fermentation can yield carbon dioxide with possible traces of ethanol or volatile fatty acids (e.g. acetic, propionic, lactic, or butyric) and if exposed to a spark or flame may result in an explosion. These conditions should be avoided. If maintenance of tank requires entry by personnel, OSHA's Confined Space standard (29CFR1910.146) shall be complied with. If welding is to be performed, the tank should be gas freed and only certified welders shall perform welding operations.

Potential Health Effects

Eyes - Mild irritant

Skin - None

Inhalation – Insufficient oxygen may be present in vessels containing the product due to the generation of carbon monoxide during fermentation

4. FIRST AID MEASURES

Eyes: Flush eyes for 15 minutes.

Skin: Wash with soap and water.

Ingestion: No data

5. FIRE FIGHTING MEASURES

Flashpoint (Method used)	Flammable Limits in Air
Non-flammable Non-combustible	Non-flammable Non-combustible

Extinguishing Agents - NA

Unusual Fire and Explosion Hazards – Fermentation occurs when diluted with water and is accelerated by heat. During fermentation, carbon monoxide with possible traces of ethanol or volatile fatty acids (e.g., acetic, propionic, lactic, or butyric) is given off, which produces inhalation hazards and possible explosion hazards.

6. ACCIDENTAL RELEASE MEASURES

Steps to be Taken in Case Material is Released or Spilled

Small spills - Stop the source of the spill. Recover as much product as possible for reuse. Absorb remaining spill and dispose solids in waste container.

Large spills - Stop the source of the spill. Create diversionary structures to minimize the extent of the release. Prevent the release from entering a waterway or sewer. Recover useable product. Absorb remaining spill and dispose of at an approved facility such as a municipal landfill or land application site.

7. HANDLING AND STORAGE

This material should be stored in a vented tank designed to contain a material with a specific gravity of 1.3 or greater. Material can ferment if excessive moisture contamination is allowed.

8. EXPOSURE CONTROLS, PERSONAL PROTECTION

Respiratory Protection - None

Ventilation – Provide adequate ventilation to prevent accumulation of vapors.

Skin Protection - Rubber gloves

Eye Protection - Safety glasses

Hygiene - Wash any exposed area promptly with soap and water. Launder contaminated clothing.

Other Control Measures - None

9. PHYSICAL AND CHEMICAL PROPERTIES

Appearance Dark brown syrupy liquid	Odor Sweet
Physical State Liquid	Specific Gravity 1.45
Boiling Point Very high	Freezing/Melting Point Varies
Vapor Pressure Low	% Volatile, by Volume No data
Evaporation Rate No data	Vapor Density in Air Water vapor only
Solubility in Water Soluble	pH 2.25 to 6.0

10. STABILITY AND REACTIVITY

Chemical Stability - Stable

Conditions to Avoid – Excess moisture or heat. Unventilated containers.

Incompatibility with Other Materials - Reacts with concentrated nitric acid or concentrated Sulphuric acid. Ferments when diluted with water.

Hazard Decomposition Products – Carbon monoxide, alcohol or fatty acid vapors

Hazardous Polymerization - NA

11. ECOLOGICAL INFORMATION

Prevent releases to land or water. Results in high Biological Oxygen Demand (BOD) and potential oxygen depletion of aquatic systems.

12. DISPOSAL CONSIDERATIONS

Dispose of waste material at an approved municipal landfill or land application site.

13. TRANSPORT INFORMATION

Hazardous Materials Description/ Proper Shipping Name - NA

DOT Hazard Class - NA

DOT Identification Number - NA

X This product is not a DOT hazardous material.

14. REGULATORY INFORMATION

Discharges to a water of the U.S. are regulated by the Environmental Protection Agency.

15. OTHER INFORMATION

None.

Date of Preparation: 3/15/96 **REVISED: 10/12/01**

Prepared by: Jane Besch, Director - HSE

Disclaimer:
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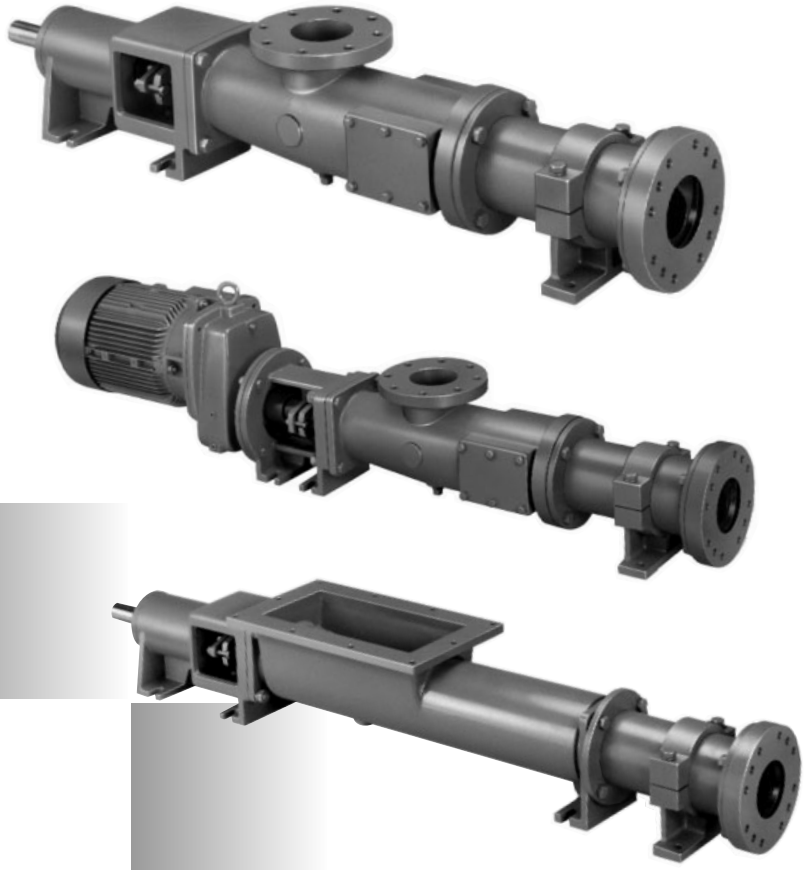
Appendix B

Service Manuals

SERVICE MANUAL

MOYNO®
1000 Pumps



MOYNO

Always the Right Solution™

TABLE OF CONTENTS

SERVICE MANUAL
MOYNO® 1000 PUMPS

1-1. INTRODUCTION

1-2. GENERAL

The Moyno 1000 pump is the most versatile positive displacement pump available. Its design parameters have been proven in thousands of applications over the past 60 years, and it is backed by this same half century-plus of experience in application and manufacturing know-how.

The Moyno 1000 pump is a progressing cavity pump. The pumping action is created by a single helical rotor rolling eccentrically in the double-threaded helix of the stator. In its revolution, the rotor forms, in conjunction with the stator, a series of sealed cavities 180 degrees apart. As the rotor turns, the cavities progress from the suction to the discharge. As one cavity diminishes, the opposing cavity increases at exactly the same rate. Thus, the sum of the two discharges is a constant volume. The result is a pulsationless, positive displacement flow.

1-3. SCOPE

This service manual covers the standard, close-coupled, and open throat configurations of the Moyno 1000 pump line. Disassembly and assembly procedures are also covered in this manual.

1-4. NAMEPLATE DATA

The pump nameplate, located on the bearing housing, or drive adaptor, contains important information relative to the operation and servicing of the pump. This information includes the direction of rotation arrow and the pump model and serial numbers.

The model and serial numbers must be used when ordering spare parts. To facilitate parts ordering, the nameplate data for your pump has been recorded on the nameplate drawing on the front cover of this manual.

1-5. Pump Rotation. The direction of rotation is indicated by a rotation arrow on the nameplate. Standard rotation of Moyno 1000 pumps is clockwise, when viewed from the driven end of the pump. Close-coupled models only, are not to be run in reverse.

1-6. Model Number. The pump model number is a series of letters and numbers which identifies the pump's basic design and materials of construction. A typical model number, for example, might be A2E CDQ3AAA, as shown on the nameplate in Figure 1-1.

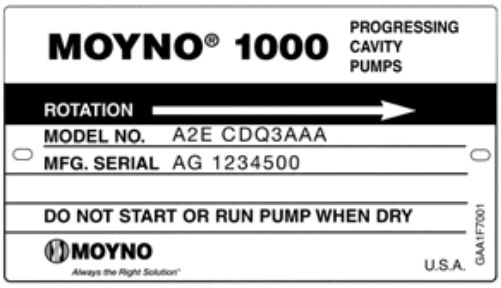


Figure 1-1. Typical nameplate showing rotation arrow, model, and manufacturing serial numbers.

The first three letters and numbers identify the pump's basic design characteristics.

In the first space, a letter designates the pump type. Letters used and their corresponding design types are as follows:

A = Standard
B = Close-coupled
C = Open throat
D = High Abrasion, Standard
E = High Abrasion, Close-coupled

The second position number identifies the number of stages in the pumping elements. This will generally be a 1, 2, or 4.

The third position is a letter, A through K, which identifies the pump's capacity in terms of gallons (gal.) per 100 revolutions. Sometimes the third position is followed by the letter "E" which denotes the pumping element is our Ultra Pro 23 geometry.The letters, with their corresponding capacities, are:

A – .38 gal./100 revs.
B – .75 gal./100 revs.
C – 1.5 gal./100 revs.
D – 3.0 gal./100 revs.
E – 6.0 gal./100 revs.
F – 12.0 gal./100 revs.
G – 22.0 gal./100 revs.
H – 36.0 gal./100 revs.
J – 48.0 gal./100 revs.
K – 62.0 gal./100 revs.
L – 115.0 gal./100 revs.

The next 3 positions, always letters, describe the pump's "Materials of Construction" in component groups of parts.

The first letter in this group identifies the material of the suction chamber casting.

The second letter indicates the material used in the rotating parts, i.e., the drive shaft, connecting rod, rotor,

and other metallic parts in contact with the material being pumped.

The third letter indicates the material of the stator. It identifies only the stator material and not that of the tube in which the stator is placed. The tube, a non-wetted part, is always alloy steel.

A typical designation such as the CDQ used in our example would result in the following:

C = Cast iron suction chamber
D = Hardened alloy steel internals including drive shaft, connecting rod, pins, and rotor
Q = Nitrile (NBR) stator (70 durometer hardness)

The following letters identify the materials used in standard construction:

C = Cast iron
D = Hardened alloy steel
S = Stainless steel, Type 316
Q = Nitrile (NBR), 70 durometer hardness
B = EPDM
F = Fluoroelastomer

The next position is a number identifying the current pump revision, this manual corresponds to revision 3.

The last three letters indicate the trim code and denote internal variations in a pump. The first letter identifies sealing variations. The second letter indicates internal variations. The third letter indicates rotor variations.

A typical trim code is AAA, designating the following:

A = Standard black packing
A = Standard plated shaft
A = Standard size chrome-plated rotor

The variations available are:

Sealing:
A – Standard black packing
C – Teflon7 white packing (not food grade)
S – single mechanical seal
D – Double mechanical seal

Internal variations:
A – Standard plated shaft
B – Non-plated shaft
P – Two-piece shaft or pinned close coupled

Rotor variations:
A – Standard plated rotor
B – Non-plated rotor
C – Standard undersize
E – Standard oversize
X – Special to order

*Teflon is a registered trademark of E.I. duPont de Nemours & Co., Inc.

2-1. INSTALLATION

2-2. GENERAL

Accessibility to the pump and adequate clearance should be prime considerations in any installation. Enough space should surround the unit so that maintenance can be performed with ease.

2-3. PIPING

2-4. Suction piping should be as short as possible. Normally, the suction line should be the same diameter as the pump suction; however, conditions such as high viscosity or required minimum flow velocities may dictate otherwise. Long-sweep 90 degree elbows or 45 degree elbows should be used instead of the standard elbow. Avoid using suction piping loops which trap air.

2-5. Discharge piping diameter should generally be as large as the discharge port unless fluid conditions indicate otherwise.

An easily-removable section of piping, at least twice as long as the stator, should be mated to the discharge port. This will allow the rotor and stator to be removed without having to remove the complete pump from the base.

2-6. FOUNDATION

For maximum pump-driver unit life, each unit should be mounted on a strong steel baseplate. The baseplate should be mounted on a firm foundation. The motors should be supported on close-coupled configurations above 1 HP.

2-7. SHAFT ALIGNMENT

After the base has been bolted down to the foundation, check the following conditions:

2-8. Coupling connected units. Be sure that the pump and drive shafts are aligned before the coupling is connected. Care should be exercised to ensure that all components are level and mounted in a direct line.

Check the gap between coupling halves (refer to coupling manufacture's recommendations). Adjustment can usually be made by loosening the mounting bolts on either the pump or driver and moving the loosened component into alignment with the fixed component. Do not use a hammer! On couplings with equal diameter hubs, it may be helpful to lay a straight edge across the coupling halves to check alignment.

2-9. Belt drive units. Be sure that sheaves or sprockets are in alignment. Check belts for proper tension. Tension requirements will vary with type of belt, center distances, and belt speeds. Consult belt manufacturer for specific recommendations.

3-1. OPERATION

3-2. INTIAL CHECK

Before putting the pump into operation, the following items should be checked to ensure that each piece of equipment is installed correctly:

-Pump, driver, coupling, or sheave alignment.
-Electrical connections.
-Gauges and other instruments.
-Pump rotation. Rotation is indicated on the pump nameplate.
-Belt tension on belt driven units. There should be no appreciable deflection when first starting up.
-All valves should be open on both suction and discharge sides of pump.
-Seal flush systems if required should be operational. Double seals require flushing between faces.

CAUTION: This is a positive displacement pump. Do not operate it against a closed valve.

3-3. START-UP

CAUTION: DRY OPERATION IS HARMFUL TO THE PUMP! Never allow the pump to operate without liquid, as dry operation will cause premature wear of the stator and possible damage. The stator is lubricated by the liquid which is being pumped.

1. Before operating the pump for the first time, fill it with liquid to lubricate the stator for the initial start-up.

Note: If the pump is shut down temporarily, enough liquid will remain in the system to provide lubrication upon restarting. It is advisable to maintain the suction piping at a higher elevation than the centerline of the pump in order to contain some liquid in the pump at time of shutdown.

2. Once the pump has been filled with liquid, check for direction of pump rotation by momentarily starting and stopping the drive. See pump nameplate for correct rotation.

3. Start seal flush water if so equipped.

4. Start pump.

3-4. PACKING LEAKAGE

The packed stuffing box is designed to control leakage, not stop it completely. Leakage is necessary to reduce friction and dissipate heat.

In a new pump, before the packing has had a chance to seat properly, excessive leakage through the stuffing box is common. Frequent adjustments of the packing gland may be necessary during the first few hours of operation in order to compress and seat the packing. See Section 4-3.

4-1. MAINTENANCE

4-2. GENERAL

The Moyno 1000 pump has been designed for a minimum of maintenance, the extent of which is routine adjustment of the packing, and infrequent lubrication of the bearings in tapered roller bearing models. The pump is one of the easiest to work on because the main elements are very accessible and require few tools to disassemble.

4-3. PACKING ADJUSTMENT

Packing gland nuts (see Figure 4-1) should be evenly adjusted. Overtightening the packing gland may result in premature packing failure and possible damage to the shaft and gland.

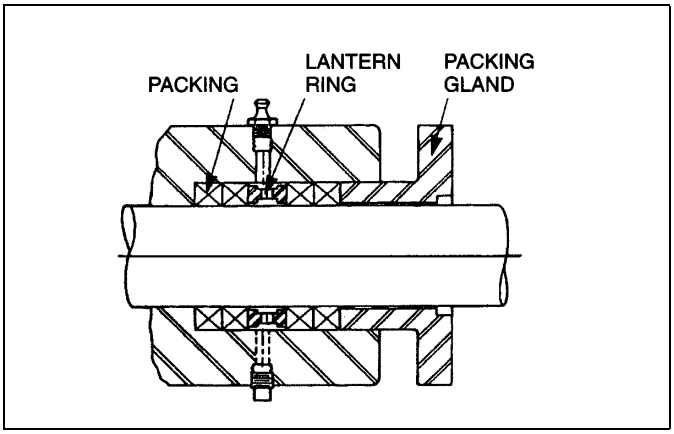


Figure 4-1. Cross Section of Packing Retainer

When packing is new, frequent minor adjustments during the first few hours of operation are recommended in order to compress and seat the packing.

1. Upon initial start-up of the pump, adjust the gland nuts for a leakage rate of 50-100 drops per minute until the packing has seated and adjusted to the operating temperature (approximately 10-15 minutes).

2. If leakage is excessive after 15 minutes of operation, tighten the gland nuts ¼ of a turn.

3. Tighten the gland nuts ¼ of a turn after an additional 15 minutes if necessary and repeat this procedure until a desired leakage of 1-2 drops per minute is obtained. Adding grease may also reduce leakage by providing a barrier at the lantern ring.

CAUTION: Do not tighten until zero leakage is obtained. Overtightening the packing gland may result in accelerated wear on the packing and damage to the shaft. In those situations where no packing leakage can be tolerated, consult your Moyno Authorized Service Distributor.

4-4. PACKING REPLACEMENT

Note: In this section, the first reference to each pump part will be followed by a number or a letter in parentheses ( ). These numbers and letters are those used to identify the pump parts and hardware items in the Exploded Views in Section 4-43.

When leakage can no longer be regulated by tightening the gland nuts, remove and replace the packing. The entire pump need not be disassembled to replace the packing. Briefly, replace as follows:

1. Remove packing gland nuts and lock washers and slide gland (0900) and slinger ring (6800) back along drive shaft (6000).

2. Use a packing puller tool (see Figure 4-2) to remove the packing (6900).

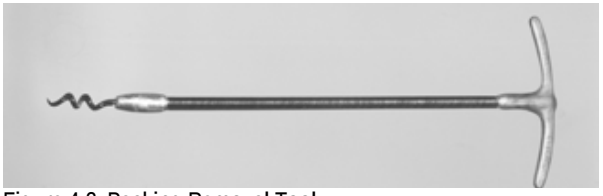


Figure 4-2. Packing Removal Tool

3. Inspect surface of drive shaft for excessive wear or grooves due to packing rub. If shaft is worn, or is badly scored or grooved, it should be replaced.

4. If drive shaft is not worn, install a lantern ring and 4 packing rings, lubricating them before installation with a good grade of packing grease. Be sure to stagger the packing ring joints at 90 degrees increments.

Note: The stuffing box is supplied with 4 rings installed, a fifth ring may be added after initial compression.

CAUTION: ALWAYS USE A PROPER PACKING TAMPER TOOL TO INSTALL PACKING. Do not use a pointed or sharp tool, as damage to the packing material or drive shaft could result. To assure proper shaft lubrication, never use a one-piece spiral wrap packing.

5. Replace packing gland and secure with packing gland screws (H), lock washers, and nuts.

6. Adjust packing per Section 4-3.

4-5. BEARING LUBRICATION (BEARING MODELS ONLY)

There are two types of bearings used in Moyno 1000 pumps. The smaller models utilize ball bearings which are lubricated and permanently sealed by the bearing manufacturer. These bearings cannot be lubricated in service and generally, due to their low cost and availability, are changed periodically during routine maintenance operations.

The larger pumps, including all open throat models, utilize tapered roller bearings which can be relubricated. Under normal operating conditions, bearings should not require replacement or relubrication for at least 15,000 hours or every 2 years.

To lubricate tapered roller bearings:

1. Remove the drive shaft assembly and the bearings in accordance with DESASSEMBLY instructions, Sections 4-14 and 4-15.

2. Clean bearing cups and cones and the shaft assembly to remove all old grease.

3. Use a good grade of EP (Extreme Pressure) Lithium soap-base grease such as Mobilux EP2 (Mobil Chemical Co.), Shell Alvania EP2 (Shell Oil Co.), or equivalent, to lubricate bearings.

4. Reassemble in accordance with the ASSEMBLY instructions, Section 4-26.

4-6. DRIVE SHAFT AND ROTOR-EXTENDED LIFE PROVISION

The heads on the drive shaft and rotor of Moyno 1000 pumps are manufactured with two sets of drive pin holes located 90 degrees apart.

If, after many hours of service, pin hole wear is encountered, the drive shaft and/or rotor may be rotated 90 degrees and the second set of pin holes utilized.

4-7. DISASSEMBLY

Note: In this section and in following sections on CLEANING, INSPECTION, and ASSEMBLY, the first reference to each pump part will be followed by a number or letter in parentheses ( ). These numbers and letters are those used to identify the pump parts and hardware items in the Exploded Views in Section 4-43.

4-8. Disconnect Pump

1. Disconnect the power source.

2. Close suction and discharge valves to isolate the pump from the line.

3. Remove drain plug (N or P) in bottom of suction chamber (1100) to drain any fluid remaining in pump and suction line.

4-9. Stator Removal

1. Remove section of discharge pipe attached to discharge flange (1400).

2. Remove discharge flange (1400) by unbolting from stator clamp ring (1800) and remove stator gasket (1200). Remove stator retaining ring (R) and stator clamp ring (1800) from stator (6500).

3. Remove top half of stator support (1700).

4. Unbolt stator clamp ring (1800) from suction housing, remove stator from rotor, turning stator while removing will ease disassembly. Use a screwdriver tip to carefully remove the stator retaining ring. Remove stator clamp ring (1800) from stator (6500). See Figure 4-3 for the typical retaining ring removal procedure.

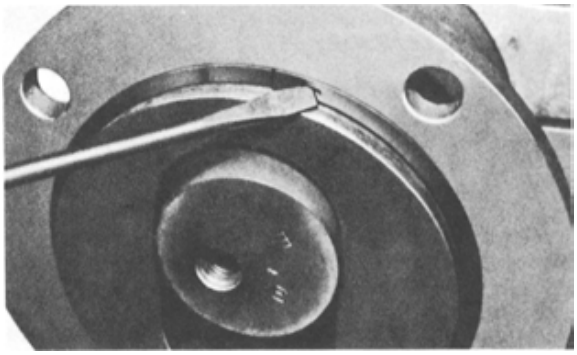


Figure 4-3. Typical Retaining Ring Removal

Note: On some four-stage models, a stator adaptor (1500) and gasket (1210) will be installed between the stator and suction chamber.

4-10. Suction Chamber Removal

1. On standard and close-coupled models, remove four suction chamber bolts and lock washers (M) holding suction chamber to bearing housing (0100). On open throat models, studs (O) screwed into the bearing housing are used in place of the suction chamber bolts. Remove four suction chamber nuts and lock washers (O) holding suction chamber to bearing housing. Remove stator gasket from housing.

2. Remove suction chamber and suction chamber gasket (1220) over the connecting rod (6200) and rotor on standard models or auger assembly (6200) and rotor on open throat models.

4-11. Rotor Removal

1. With snap ring pliers, remove the rotor head snap ring (J), sliding it down over the rotor. Some models may use spiral type rings (see Figure 4-3).

2. Carefully tap the retaining ring (6100) towards the rotor end exposing the edge of the universal joint seal (6400).

3. Remove the edge of the universal joint seal from the groove in the rotor head and fold the seal back.

4. Carefully tap the retaining ring back towards the universal joint seal until the drive pin (6300) is exposed.

5. Push the drive pin through the rotor head and remove the rotor.

6. Remove head O-ring (K) from rotor head.

4-12. Connecting Rod or Auger Assembly Removal

1. Remove the drive shaft head snap ring (J) sliding it back towards the bearing housing. Some models may have spiral type rings (see Figure 4-3).

2. Tap the retaining ring towards the bearing housing, exposing the edge of the universal joint seal.

3. Remove the edge of the universal joint seal from the groove and fold the seal back.

4. Slide the retaining ring back towards the seal until the drive pin is exposed.

5. Push the drive pin through the drive shaft head and remove the connecting rod or auger assembly from the drive shaft (6000).

6. Remove head O-ring from drive shaft head.

4-13. Packing Removal

To remove packing without removing the drive shaft and bearing assembly, refer to Section 4-4. If the drive shaft and bearing assembly are to be removed, proceed directly to Sections 4-14 and 4-15.

4-14. Drive Shaft Removal

Shaft Drive Models (one-piece shaft, not close-coupled)

The Moyno 1000 pump is designed so that the stuffing box (1000), packing gland, packing, and bearings (D or E) are removed as an assembly with the drive shaft. **For Close-Coupled models, skip to Step 17. For two-piece Shaft Drive models, skip to Step 6.**

1. Remove the drive shaft key (I).

2. Remove bearing cover screws and lock washers (A).

Note: Ball bearing models do not have a bearing cover. A bearing housing snap ring (G), located at the drive shaft end of the bearing housing, is used to position the drive shaft and bearings. This snap ring need not be removed.

3. Slide bearing cover (0300) with grease seal (B) and bearing shims (6700) off of drive shaft.

4. Using snap ring pliers, remove the bearing housing snap ring (G) located at the stuffing box end of the bearing housing.

5. Slide drive shaft assembly from bearing housing.

Shaft Drive Models (two-piece shaft, not close-coupled)

The Moyno 1000 pump is designed with a two-piece drive shaft available that allows for removal of the drive shaft head for easy seal maintenance.

6. Remove drive shaft key (I).

7. Move slinger/pin retainer (6800) toward packing or seal housing, exposing shaft pin.

8. Remove shaft pin (2000).

9. Pull intermediate shaft from bearing housing assembly (2100).

10. Inspect the sealing O-ring (2200) and replace if worn or damaged.

11. Remove mechanical seal and seal housing from bearing housing, or remove packing stuffing box from bearing housing.

12. Remove bearing cover screws and lock washers.

Note: Ball bearing models do not have a bearing cover. A bearing housing snap ring, located at the drive shaft end of the bearing housing, is used to position the drive shaft and bearings. Remove this snap ring.

13. Slide bearing cover (0300) with grease seal (B) and bearing shims (6700) off of drive shaft.

14. Slide the drive shaft/bearing assembly out of the bearing housing toward the bearing cover end.

15. Remove, if desired, the bearing housing snap ring (G) located at the stuffing box end of the bearing housing.

16. This allows removal of the grease seal housing. Inspect and replace grease seal if worn or damaged.

Close-Coupled Models Only

The Moyno 1000 close-coupled pump is designed so that the stuffing box, packing gland, and packing are removed as an assembly with the drive shaft.

17. Pull back slinger ring on pinned versions, and push out the pin.

18. On keyed versions, remove set screws (D) in locking ring (1600). Rotate locking ring 90 degrees. Slide drive shaft assembly from drive adaptor, uncoupling from output shaft of drive.

Note: There is a flange on the gearbox side of the locking ring that catches on the back end of the drive shaft key. By rotating 90 degrees the set screw holes should be 90 degrees from the key. This will align a clearance in this locking ring for removal.

4-15. Bearing Removal (Not Close-Coupled)

1. Remove shaft snap rings (F).

2. Use an arbor press to press bearings from drive shaft. The first bearing is pressed off with its accompanying grease retainer (0500). Slide the second grease retainer off the shaft and then remove the second bearing.

Note: When replacing drive shaft or bearings on tapered roller bearing models, it is recommended that both grease seals be replaced.

3. Remove the grease seal housing (0700) and grease seal, bearing housing snap ring, slinger ring, packing gland, packing, and stuffing box from drive shaft, on one-piece shaft models.

4-16. Gearbox/Gearmotor Removal
Close-Coupled Models Only - Pinned and keyed

1. To remove the gearbox/gearmotor from the pump, remove the bolts holding the gearmotor to the drive adaptor.

2. Pull back slinger ring on pinned versions, and push out the pin.

3. On keyed versions, loosen and remove two set screws in the locking ring. **Rotate locking ring 90 degrees in either direction.**

4. Disengage gearmotor shaft from the pump drive shaft.

Note: Rotating locking ring aligns the slots in locking ring with shaft key to allow disengagement.

4-17. CLEANING

Clean parts in a suitable cleaning solvent.

4-18. INSPECTION

4-19. Bearings. As described in Section 4-5 on Bearing Lubrication, ball bearings (E) are sealed by the manufacturer and are not designed to be relubricated and reused. The following inspection procedure applies to tapered roller bearings (D).

1. After cleaning, rotate bearings very slowly under hand pressure to feel for smooth and even action. Never spin a dry bearing. Check for cracks, galling, pitting, burns, etc. Replace bearing if there is any doubt concerning complete serviceability; bearings should be readily available from any bearing source.

4-20. Drive Shaft and Intermediate Shaft. Inspect drive shaft (6000) and intermediate shaft (2100) if so equipped for scoring, burrs, cracks, etc. Replace as necessary. The drive shaft head is equipped with two sets of pin holes. When one set becomes worn, rotate shaft 90 degrees and use second set.

4-21. Seals. It is sound practice to always replace grease seals (B) whenever the drive shaft and tapered roller bearings are removed.

4-21a. Mechanical Seals. It is sound practice to replace mechanical seals when the pump is disassembled. Extreme care should be taken to protect the seal faces from damage. These are fragile; avoid touching the faces and keep them clean.

The rubber bellows (Type 43) or equal seals will adhere to the shaft after assembly and must be replaces if

removed from the shaft. The (Type 680) metal bellows seals or equal use O-rings to seal against the shaft and may be reused if their condition does not dictate replacement.

The rubber bellows (Type 43) or equal seals are friction-driven which makes adhesion to the shaft a necessity. The metal bellows (Type 680) seals or equal are positive driven and locked to the shaft with set screws that must be loosened to remove seal.

4-22. Packing. It is sound practice to always replace packing (6900) whenever the pump is disassembled.

4-23. Rotor

1. To check for excessive rotor (5000) wear, measure the rotor crest-to-crest diameter (see Figure 4-4) and compare with the following chart:

Rotor Size	Standard Crest-to-Crest Dia. (inches)
A	.886
B	1.061
C	1.327
D	1.671
E	2.100
F	2.676
G	3.428
H, J, K	4.015

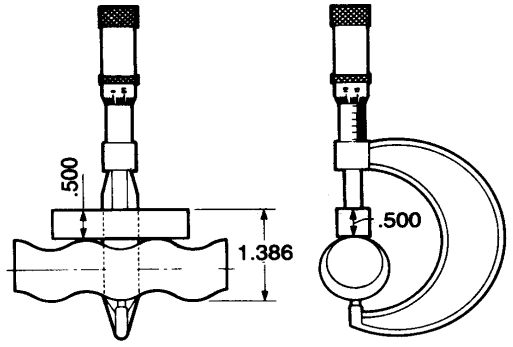
The rotor size is designated by the third letter in the Model Number (i.e., A2ECDQ3AAA).

2. If the measured crest-to-crest diameter is within .010 in. of the standard value, the rotor is reusable provided that:

a. the rotor pin holes are not excessively worn.

b. the rotor surface is not cracked, pitted or deeply grooved (.030 in. or more).

3. Rotors with crest-to-crest diameters greater than .010 in. under the standard value should generally be replaced.



To check any rotor, place .500 in. bar across the crest on one side of the rotor. The micrometer reading minus .5 in. equals the rotor crest-to-crest diameter. Example: 1.383 in. – .500 in. = .883 in. crest-to-crest.

Figure 4-4. Measuring Rotor Diameter

4-24. Stator. The best indication of stator wear (6500) and the need for replacement is a drop in pump performance. Stators with interior surfaces that are pitted, grooved or gouged should also be replace.

4-25. All Other Parts. Check for cracks, excessive wear, damage to threaded holes, burrs, etc. Replace as necessary. Replace O-rings (K) and all gaskets (1200, 1210, and 1220) at each disassembly and reassembly.

4-26. ASSEMBLY

Bearing Housing (One-Piece Shaft Models)

NOTE: For two-piece shaft models, go to 4-27. For close-coupled models, go to 4-28.

1. Slide stuffing box (1000) on to drive shaft (6000), large flanged end first.

2. Slide packing gland (0900) on to shaft so that the round portion fits into the stuffing box.

3. Place slinger ring (6800) on shaft, adjacent to the packing gland.

4. Place bearing housing snap ring (G) on the shaft.

4-26a. Ball Bearing Models

1) Press sealed bearing (E) on drive shaft.

2) Install shaft snap ring (F) in groove on shaft. Seat bearing against snap ring.

3) Install second shaft snap ring.

4) Press second sealed bearing on shaft and seat against snap ring.

5) If not already in place, install bearing housing snap ring (G) in groove inside drive end of bearing housing (0100).

6) Place assembled drive shaft into bearing housing. Secure by inserting second bearing housing snap ring in groove in stuffing box end of bearing housing.

4-26b. Tapered Roller Bearing Models

1) Press grease seal (B) into grease seal housing (0700) and place assembly on shaft with chamfered side of grease seal housing facing keyway end of shaft.

2) Pack the bearings (D) **thoroughly** with grease, Mobilux EP2 or equal.

3) Place bearing cup on shaft and press bearing cone on shaft approximately .150 in. beyond snap ring groove.

4) Place grease retainer (0500) on shaft and install shaft snap ring (F) in groove on shaft.

5) Seat bearing and grease retainer against snap ring.

6) Install second shaft snap ring in groove on shaft and place second grease retainer on shaft.

7) Press bearing cone on shaft, and seat bearing and grease retainer against snap ring.

8) Fill grease seal housing with grease.

9) Slide drive shaft assembly into bearing housing. Secure by inserting bearing housing snap ring (G) in groove in bearing housing at rear of stuffing box area.

10) Fill area around the bearing in the drive end of the bearing housing with grease.

11) Complete bearing assembly by sliding bearing cup into bearing housing.

12) Press grease seal in bearing cover (0300) and place bearing cover on shaft. Secure with bearing cover screws and lock washers (A). (see Section 4-29.)

4-27. ASSEMBLY (Two-Piece Shaft Models)
NOTE: For close-coupled models, go to 4-28.

4-27a. Ball Bearing Models

1) Press sealed bearing on drive shaft.

2) Install shaft snap ring in groove on shaft. Seat bearing against snap ring.

3) Install second shaft snap ring.

4) Press second sealed bearing on shaft and seat against snap ring.

5) If not already in place, install bearing housing snap ring in groove inside stuffing box end of bearing housing.

6) Place assembled drive shaft into bearing housing. Secure by inserting second bearing housing snap ring in groove in drive end of bearing housing. Proceed to 4-30.

4-27b. Tapered Roller Bearing Models

1) Install bearing housing retaining ring in bearing housing groove.

2) Press grease seal into grease seal housing and assemble in housing with chamfered side of grease seal housing facing bearing location.

3) Slide bearing cup into the bearing housing against the seal housing.

4) Press bearing cone on shaft approximately .150 in. beyond snap ring groove.

5) Place grease retainer on shaft and install shaft snap ring in groove on shaft.

6) Seat bearing and grease retainer against snap ring.

7) Install second shaft snap ring in groove on shaft and place second grease retainer on shaft.
8) Press bearing cone on shaft, and seal bearing and grease retainer against snap ring.

9) Fill grease seal housing with grease.

10) Slide drive shaft assembly into bearing housing.

11) Fill area around the bearing in the drive end of the bearing housing with grease.

12) Complete bearing assembly by sliding bearing cup into bearing housing.

13) Press grease seal in bearing cover (0300) and place bearing cover on shaft. Secure with bearing cover screws and lock washers Proceed to 4-29.

4-28. Close-Coupled Models - Pinned versions are similar to two piece shaft versions. Locking ring version is listed below.
1) Place locking ring on drive shaft with key clearance slot aligned with keyslot in drive shaft.

2) Mount drive to the drive adaptor (0100) using four bolts, lock washers, and nuts.

3) Install drive shaft assembly into the drive adaptor while coupling drive shaft to output shaft of drive.

4) **Rotate locking ring 90 degrees and secure one set screw over the key and the other set screw at 180 degrees through the hole in the drive shaft, securing on the output shaft of the drive.**

4-29. Adjusting Bearing End Play
(Tapered Roller Bearings Only)

1. Tighten bearing cover screws around bearing cover firmly, to the point the shaft will not turn. (Screws should be tightened evenly, opposite each other.)

2. Measure the gap between the bearing cover and the bearing housing.

3. Remove bearing cover and add shims (6700) to equal gap measured in step 2 plus an additional .010 in.

4. Install bearing cover with shims and tighten screws evenly, opposite each other.

5. Tap lightly on the shaft head or end using a soft mallet. Rotate shaft to “free up” assembly. You should be able to rotate the shaft by hand.

6. Shims may be added or subtracted to get the proper setting.

7. Slide the rubber pin retainer on the drive shaft pushing past the pin hole.

4-30. Intermediate Drive Shaft Installation (Mechanical Seal Pumps Proceed to 4-30a.)

1. Position the packing in the stuffing box and install the gland studs, if removed.

2. Position the stuffing box in the bearing housing, sliding the packing gland on packed models onto the gland studs. Rotate the stuffing box so that the zerk fitting is on the side.

3. Install the intermediate shaft seal ring (2200) on the intermediate shaft.

4. Insert the intermediate shaft through the stuffing box, seating the end of the intermediate drive shaft into the drive shaft. Rotate the shafts to align the pin holes and insert the pin.

5. Slide the rubber pin retainer in place on the drive shaft over the pin.

4-30a. Mechanical Seal Installation (Single Seal)

1. Install the stationary component (seat and O-ring) of mechanical seal (6950) in seat of seal retainer (1000).

2. Position the seal housing in the bearing housing (1).

3. Slide the rotating component (spring and rotating seat) onto the drive shaft. It may be necessary to wipe a small amount of lubricant around inside diameter of rotating component.

4. Lock mechanical seal set screws onto shaft if mechanical seal is positively driven, locating the end of the seal flush with the end of the drive shaft.

5. Install the intermediate shaft O-ring (2200) on the intermediate shaft.

6. Insert the intermediate shaft into the drive shaft. Rotate the shafts to align the pin holes and insert the drive shaft pin, compressing the mechanical seal spring on single spring friction drive seals.

7. Locate slinger/pin retainer on the shaft covering the pin.

4-30b. Mechanical Seal Installation (Double Seal)

1. Press the stationary seat into the gland for a double seal installation, taking care not to damage the face. Put gland on shaft with care.

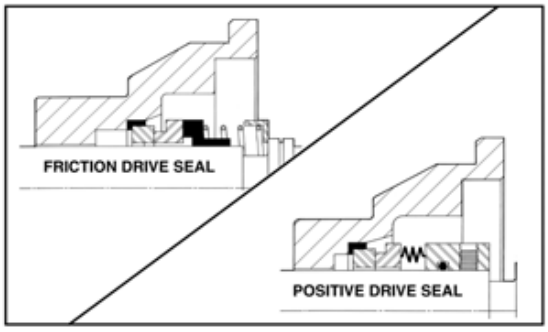
2. Install the seal on the shaft.

3. On double seal models the seal housing (modified packing stuffing box) is slid on the shaft, with stationary seat fitted into place. The seal assembly is then compressed by bolting the gland to the seal housing. This will bring the faces into contact.

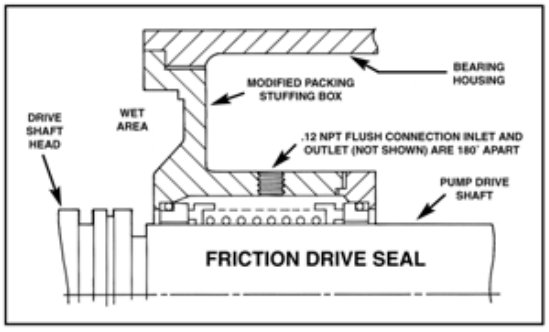
4. Install the intermediate shaft O-ring (2200) on the intermediate shaft.

5. Insert the intermediate shaft into the drive shaft. Rotate the shafts to align the pin holes and insert the drive shaft pin.

6. Locate the slinger/pin retainer on the shaft covering the drive shaft pin.



Cross Section of Typical Single Seal Installations



Cross Section of Typical Double Seal Installation

4-31. Connecting rod or Auger Assembly

1. Install universal joint seal (6400) on connecting rod (or auger assembly) (6200), making sure the raised ridge inside the universal joint seal is placed in the matching groove on the connecting rod.

2. Fold other end of seal back towards center of connecting rod.

3. Spread head snap ring (J) ends and place on drive shaft between drive shaft head and stuffing box.

4. Place head O-ring (K) in matching groove on drive shaft head.

5. Fill shaft head with a good grade of EP (Extreme Pressure) lithium soap-base grease such as Shell Alvania EP1 (Shell Oil Co.), Texas Refinery Corp. 880 Crown and Chassis grease, or equivalent.

6. Slide retaining ring (6100), chamfered end first, over universal joint seal on to connecting rod.

7. Place end of connecting rod in drive shaft head and align holes. Coat drive pin (6300) with grease and insert in pin hole.

8. Slide retaining ring back over the universal joint seal and place on the drive shaft head, exposing universal joint seal groove.

9. Unfold the seal and snap the lip in matching groove on the shaft head.

10. Slide the retaining ring back, compressing the lip of the seal and exposing the snap ring groove on the shaft head. Install snap ring in groove to secure the retaining ring.

11. Using similar procedures outlined in previous steps 1 through 10, attach rotor (5000) to connecting rod or auger assembly.

4-32. Suction Chamber

1. Screw drain plug (N or P) into bottom of suction chamber.

2. Before installing the suction chamber (1100), make sure that the stuffing box and packing gland bolt holes are in a horizontal plane parallel to the pump base. If not, turn stuffing box in either direction until bolt holes are horizontal.

3. Place suction chamber gasket (1220) over projection on end of suction chamber.

4. Slide suction chamber over the rotor and connecting rod or auger assembly and fasten to the bearing housing.

On standard models, fastening is by means of suction chamber bolts and lock washers (M). On open throat models, the suction chamber slides onto studs (O) protruding from the bearing housing and is secured with nuts and lock washers.

4-33. Stator

1. Put stator gasket (1200) in place in the exposed end of suction chamber.

Note: 1. Before installation of stator, lubricate rotor and/or inside of stator with water or glycerine to facilitate installation. Be sure to use a lubricant that is compatible with the stator elastomer.

2. On four-stage models, a stator adaptor (1500) will be used. Place stator adaptor gasket (1210) in suction housing, install stator adaptor and place stator gasket in stator adaptor before installing stator.

2. Slide stator clamp rings (1800) on both ends of the stator (6500) and secure in position with retaining rings (R).

3. Slide stator (6500) over the rotor.

Note: Turning the rotor counterclockwise while inserting into stator makes assembly easier.

4. If the stator is firmly seated against the stator gasket in the suction housing recess, align the holes in clamp ring with the threaded holes in the suction housing. Thread the four hex head bolts (M) with lock washers through hole in clamp ring into the threaded holes. Tighten bolts evenly.

4-34. Stator Support/Discharge Flange Assembly

1. Place top of stator support over stator. Fasten to bottom half of stator support using hex head bolts.

Note: Some smaller models have a fabricated steel support that attaches to the stator clamp ring at the discharge end.

2. Place stator gasket in recess in discharge flange and position discharge flange on end of stator. Align holes in stator clamp ring with threaded holes in discharge flange. Install and evenly tighten the hex bolts.

4-35. Packing

1. Lubricate inside diameter of packing (6900) in accordance with manufacturer's instruction.

2. Install 4 rings of packing in stuffing box, staggering joints at 90 degrees intervals. The lantern ring should be in between with 2 rings on each side.

Note: On new packing, typically 4 rings are installed with a 5^{th} being added after adjustments.

3. Secure packing gland with packing gland screws, lock washer and nuts (H).

4. Adjust packing per Section 4-3.

4-36. Pump Connections

1. Install shaft key (I) in drive shaft keyway for installation of coupling or sheave.

2. Connect piping to pump.

3. Check complete pump installation per INSTALLATION instructions. (Sections 2-1 through 2-9)

4. Review OPERTAION instructions per Sections 3-1 through 3-4.

4-37. STORAGE

4-37a. Short-Term Storage. Storage of six months or less will not damage the pump. However, to ensure the best possible protection, the following is advised:

1. Store pump inside whenever possible or cover with some type of protective covering. Do not allow moisture to collect around pump.

2. Remove drain plug to allow the pump body to drain and dry completely.

3. See drive manufacturer's instructions for motor and/or drive storage.

4. Every 2 or 3 weeks, rotate the pump manually a few revolutions to avoid a "set" condition of rotor in stator elastomer. This will prevent hard starting and excessive torque requirements when pump is again put into operation.

5. See OPERATION Sections 3-1 through 3-4 before start-up. Be sure all lubricants are in good condition.

4-37b. Long-Term Storage. If pump is to be in storage for more than six months, perform the above short-term storage procedures with the exception of Step 4. In its place, it is suggested that the stator be removed to avoid developing a "set" condition. In addition:

1. Apply rust inhibitor to all unpainted cast iron and machined carbon steel surfaces.

2. Remove drive belts if applicable.

4-38. STANDARD PACKING SPECIFICATION

Packing is of the braided, non-asbestos type, fully impregnated throughout with lubricant and graphite. Service is to be for medium-to-high-speed rotary shaft applications (to 10 mps or 2000 fpm) and excellent for use in low or medium pressure pumps as well as valves. Temperatures to 350 degrees (175 degrees C). pH range: 4-10. Recommended packing grease Mobilux MPG 2 or equal.

4-39. RECOMMENDED SPARE PARTS

The Moyno 1000 pump has been designed and built to minimize overall operating costs. All wearable parts are replaceable. A recommended inventory of spare parts is dependent upon the application and the importance of continued operation.

To minimize down time, we recommend the following quantities of spare parts be stocked.

1 – Rotor
2 – Head O-Rings
2 – Head Snap Rings
1 – Stator
2 – Stator Gaskets
1 – Connecting Rod
2 – Universal Joint Seals*
2 – Drive Pins
1 – Packing Set

*For models utilizing a one-piece seal, a single universal joint seal should be stocked.

The above is a suggested list. For further assistance in determining what you will need for your application, contact your Moyno representative.

4-40. HOW TO ORDER SPARE PARTS

When ordering spare parts, the pump model and serial number should be supplied. The part numbers and letters shown on the exploded views apply to all models.

The following information must be supplied when ordering parts:

1. Pump model number

2. Manufacturer's serial number

3. Refer to the Exploded Views (Section 4-43 through 4-47) to locate the parts needed.

4. Refer to your particular pump model in Section 4-48 and identify the actual part number needed.

CLAMP RING BOLT CHART

Ref. No. Description	QTY [ ]	A2A B2A A1B B1B A2B B2B D4B E4B	A4A B4A	A4B B4B	A1C B2C A2C D4C B1C E4C	A4C B4C	A1D A1E D4D A2D A2E E4D B1D B1E D4E B2D B2E E4E	A4D B4D	A4E B4E
Suction End Bolts	[4]	M10x30 (AA076)	M12x50 (AA105)	M12x55 (AA106)	M12x35 (AA102)	M12x55 (AA106)	M12x40 (AA103)	M16x70 (AA159)	M16x80 (AA161)
Lockwashers	[4]	M10 (AE075)	M12 (AE100)	M12 (AE100)	M12 (AE100)	M12 (AE100)	M12 (AE100)	M16 (AE150)	M16 (AE150)
Discharge End Bolts	[4]	M10x30 (AA076)		M16x35 (AA162)			M12x35 (AA102)		
Lockwashers	[4]	M10 (AE075)		M16 (AE150)			M12 (AE100)		
Discharge End Bolts	[1]		M12x30 (AA101)		M12x30 (AA101)				
Lockwashers	[1]		M12 (AE100)		M12 (AE100)				
Discharge End Bolts	[3]		M12x35 (AA102)		M12x35 (AA102)				
Lockwashers	[3]		M12 (AE100)		M12 (AE100)				
Discharge End Bolts	[8]					M12x35 (AA102)		M16x40 (AA153)	M16x45 (AA154)
Lockwashers	[8]					M12 (AE100)		M16 (AE150)	M16 (AE150)

Ref. No. Description	QTY [ ]	A1F A2F B2F B1F D4F	A4F	A1G B1G A2G D4G	A1H A1J A2H A2J D4H A1K	C1E C2E	C1F C2F	C1G C2G	C1H C2H C2J C1J C1K
Suction End Bolts	[4]	M16x45 (AA154)	M20x90 (AA188)	M16x45 (AA154)	M20x55 (AA181)	M12x35 (AA102)	M16x40 (AA153)	M16x40 (AA153)	M20x45 (AA179)
Lockwashers	[4]	M16 (AE150)	M20 (AE175)	M16 (AE150)	M20 (AE175)	M12 (AE100)	M16 (AE150)	M16 (AE150)	M20 (AE175)
Discharge End Bolts	[4]	M16x35 (AA152)		M16x40 (AA153)	M20x45 (AA179)	M12x35 (AA102)	M16x35 (AA152)	M16x40 (AA153)	M20x45 (AA179)
Lockwashers	[4]	M16 (AE150)		M16 (AE150)	M20 (AE175)	M12 (AE100)	M16 (AE150)	M16 (AE150)	M20 (AE175)
Discharge End Bolts	[8]		M20x50 (AA180)						
Lockwashers	[8]		M20 (AE175)						

4-41. STANDARD HARDWARE LIST - STANDARD AND OPEN THROAT MODELS

NOTE: It is suggested that the standard hardware below be purchased at your local hardware outlet. Items can be purchased from Moyno Inc. The number in ( ) is Moyno's reference number only.

Ref. No. Description	Qty [ ]	MODELS										
		A2A	A1B A2B D4B	D4C A1C A2C A4A	A4B	D4D A1D A2D	D4E A1E A2E A4C C1E C2E	D4F A1F A2F A4D C1F C2F	D4G A1G A2G A4E C1G C2G	A4F	A1K A1H C1H A2H C2H D4H C1J A1J C2J A2J C1K
A. Bearing Cover Screw (Steel hex head socket, ISO metric fully threaded)		----			M8x1.25x30 [4] (AM051)			M10x1.5x30 [4] (AM078)		M10x1.5x30 [6] (AM078)	
Lock Washer*		----			M8 (4) (AE050)			M10 (4) (AE075)		M10 (6) (AE075)	
B. Grease Seal (Lip contact sealing with press fit O.D.)		----			48 O.D.x32 I.D. [2] (DA061)			68 O.D.x45 I.D. [2] (DA086)		78 O.D.x55 I.D. [1] (DA097) 78 O.D.x60 I.D. [1] (DA098)	
C. Name Plate Drive Screw (Steel, round head, type U)	[4]	No. 2 x 3" (AN005)			No. 2 x 3" (AN005)			No. 2 x 3" (AN005)		No. 2 x 3" (AN005)	
D. Tapered Roller Bearing	[2]				LM67048/LM67010 (BL005)			25580/25520 (BL010)		JM207049/JM207010 (BL015)	
E. Ball Bearing	[2]	6205 Dbl. Seal (BD005)			----			----		----	
F. Shaft Snap Ring (Basic external retaining ring)	[2]	Sft. ϕ 25 (EB004) 23.1 I.D.x1.1w			Sft. ϕ 32 (EB012) 29.6 I.D.x1.3w			Sft. ϕ 45 (EB024) 41.6 I.D.x1.6w		Sft. ϕ 55 (EB031) 50.6 I.D.x2.0w	
G. Bearing Housing Snap Ring (Basic internal retaining ring)		52 Bore (EA015) 57.9 O.D.x2.0w [2]			58 Bore (EA022) 64.4 O.D.x2.0w [1]			82 Bore (EA035) 91.1 O.D.x2.8w [1]		95 Bore (EA041) 105.6 O.D.x2.8w [1]	
H. Packing Gland Screw	[2]	M6x1.0x45 (AL029)			M8x1.25x55 (AL056)			M10x1.5x60 (AL082)		M10x1.5x70 (AL084)	
Packing Gland Stud***	[2]	4220416001			4220417001			4220418001		4220418001	
Hex Nut (Steel, ISO metric coarse thread)	[2]	M6x1 (AB025)			M8x1.25 (AB050)			M10x1.5 (AB075)		M10x1.5 (AB075)	
Lock Washer*	[2]	M6 (AE025)			M8 (AE050)			M10 (AE075)		M10 (AE075)	
I. Shaft Key	[1]	6x6x40 (GA007)			8x7x70 (GA012)			12x8x80 (GA021)		14x9x100 (GA024)	
J. Head Snap Ring (Basic external retaining ring)H	[2]	Sft. ϕ 28 25.8 I.D.x1.3w (EB007)	Sft. ϕ 35 32.3 I.D.x1.3w (EB015)		Sft. ϕ 40 36.75 I.D.x1.6w (EB020)	Sft. ϕ 48 44.4 I.D.x1.6w (EB027)		Sft. ϕ 65 60.4 I.D.x2.0w (EB036)	Sft. ϕ 82 76.4 I.D.x2.4w (EB045)	Sft. ϕ 100 92.5 I.D.x2.8w (EB049)	
K. Head O-Ring	[2]	25 I.D.x 2.65 ϕ (CA137)	30 I.D.x 2.65 ϕ (CA141)		34.5 I.D.x 2.65 ϕ (CA145)	40.0 I.D.x 3.55 ϕ (CA175)		58.0 I.D.x 3.55 ϕ (CA188)	75.0 I.D.x 3.55 ϕ (CA197)	90 I.D.x3.55 ϕ (CA228)	
L. Clamp Ring Bolt (Steel hex, ISO metric bolt coarse thread)		See Chart on Page 11									
Lock Washer*											
R. Stator Ring**	[2]	RST-162 (EC022)	RS-187 (EC028)	RS-236 (EC034)		RS-287 (EC039)	RS-343 (EC046)	RS-450 (EC055)	RS-551 (EC060)		RS-662 (EC065)
S. Stator Support Bolt (Steel hex, ISO metric coarse thread)	[2]	----			M10x40 (AL078)	M10x50 (AL080)		M16x80 (AL161) (A4D & A4E M10x50) (AL080)		M16x80 (AL161)	M20x90 (AL188)
Lock Washer*	[2]	----			M10 (AE075)	M10 (AE075)		M16 (AE150)		M16 (AE150)	M20 (AE175)
T. Optional Insp. Plate Bolt		----			----	M6x16 (AA023)		M8x20 (AA049)	M10x25 (AA075)	M10x25 (AA075)	
Lock Washer*		----			----	M6 (AE025)		M8 (AE050)	M10 (AE075)	M10 (AE075)	
STANDARD MODELS ONLY											
M. Suction Chamber Bolt (Steel hex head, ISO metric coarse thread)	[4]	M10x1.5x35 (AA077)			M10x1.5x40 (AA078)			M16x2x55 (AA156)		M20x2.5x70 (AA184)	
Lock Washer*	[4]	M10(AE075)			M10 (AE075)			M16 (AE150)		M20 (AE175)	
N. Drain PlugH	[1]	.5 NPT (FB012)			.5 NPT (FB012)			.75 NPT (FB018)		.75 NPT (FB018)	
OPEN THROAT MODELS ONLY											
O. Suction Chamber Stud (Steel, double end ISO metric coarse thread)	[4]	----			----	M10x1.5x30 (AS076)		M16x2x45 (AS154)		M20x2.5x50 (AS180)	
Hex Nut (Steel, ISO metric coarse thread)	[4]	----			----	M10x1.5 (AB075)		M16x2 (AB150)		M20x2.5 (AB175)	
Lock Washer*	[4]	----			----	M10 (AE075)		M16 (AE150)		M20 (AE175)	
P. Drain PlugH	[1]					1" NPT (FB020)		1" NPT (FB020)		2" NPT (FB023)	

* All lock washers are steel, single coil, helical spring.

H Stainless steel required on stainless steel models. Change the third digit of the part number to "5", i.e., Drain Plug (FB512).

** 4-stage models use the same stator rings as 2-stage models of the same element size.

*** Stuffing boxes with tapped holes use the studs.

LEGEND:
O.D. = Outer Diameter
I.D. = Inner Diameter
ϕ = Diameter
W = Wide
" = Inches
NPT = American National Standard Taper Pipe
Sft = Shaft

4-42. STANDARD HARDWARE LIST – CLOSE-COUPLED MODELS

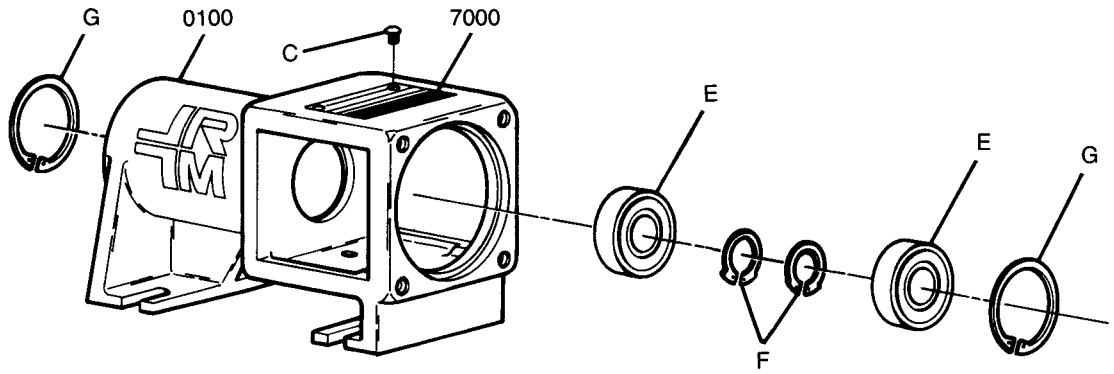
Ref. No. Description	Qty [ ]	MODELS									
		B2A	E4B B1B B2B	B1C B4A B2C E4C	B4B	B1D B2D E4D	B1E B4C B2E E4E	B1F B2F B4D	B1G B2G B4E	B4F B4G	B1H, B2H B1J, B2J B1K,B2K B1L
C. Name Plate Drive Screw (Steel, round head, type U)	[2]	No. 2 x 3" (AN005)			No. 2 x 3" (AN005)			No. 2 x 3" (AN005)		No. 2 x 3" (AN005)	
D. Locking Ring Set Screw (Hex socket flat Point)	[2]	M6x1.0x6 (AK079)			M6x1.0x8 (AK180)			M8x1.25x10 (AK106)			
H. Packing Gland Screw	[2]	M6x1.0x45 (AL029)			M8x1.25x55 (AL056)			M10x1.5x60 (AL082)		M10x1.5x70 (AL084)	
Packing Gland Stud***	[2]	4220416001			4220417001			4220418001		4220418001	
Hex Nut (Steel, ISO metric coarse thread)	[2]	M6x1.0 (AB025)			M8x1.25 (AB050)			M10x1.5 (AB075)		M10x1.5 (AB075)	
Lock Washer*	[2]	M6 (AE025)			M8 (AE050)			M10 (AE075)		M10 (AE075)	
J. Head Snap Ring (Basic external retaining ring)H	[2]	Sft. ϕ 28 25.8 I.D.x1.3w 5100-112 (EB007)		Sft. ϕ 35 32.3 I.D.x1.3w 5100-137 (EB015)	Sft. ϕ 40 36.75 I.D.x1.6w 5100-156 (EB020)		Sft. ϕ 48 44.4 I.D.x1.6w 5100-187 (EB027)	Sft. ϕ 65 60.4 I.D.x2.0w 5100-255 (EB036)	Sft. ϕ 82 76.4 I.D.x2.4w 5100-325 (EB045)	Sft. ϕ 100 92.5 I.D.x2.8w (EB049)	
K. Head O-RingHH	[2]	23.6 I.D.x 2.65 ϕ (CA137)		30 I.D.x 2.65 ϕ (CA141)	34.5 I.D.x 2.65 ϕ (CA145)		40.0 I.D.x 3.55 ϕ (CA175)	58.0 I.D.x 3.55 ϕ (CA188)	75.0 I.D.x 3.55 ϕ (CA197)	90 I.D.x3.55 ϕ (CA228)	
L. Clamp Ring Bolt (Steel hex, ISO metric coarse thread) Lock Washer*		**See Chart on Page 11**									
M. Suction Chamber Bolt (Steel hex head, ISO metric coarse thread)	[4]	M10x1.5x50 (AA080)			M10x1.5x50 (AA080)			M16x2x75 (AA160)		M20x2.5x70 (AA184)	
Hex Nut	[4]	M10x1.5 (AB075)			M10x1.5 (AB075)			M16x2 (AB150)			
Lock Washer*	[4]	M10 (AE075)			M10 (AE075)			M16 (AE150)		M20 (AE175)	
N. Drain PlugH	[1]	.5 NPT (FB012)			.5 NPT (FB012)			.75 NPT (FB018)		.75 NPT (FB018)	
R. Stator Ring	[2]	RST-162 (EC022)	RS-187 (EC028)	RS-243 (EC034)	RS-281 (EC039)		RS-343 (EC046)	RS-450 (EC055)	RS-551 (EC060)		RS-662 (EC065)
S. Stator Support Bolt (Steel hex nut, ISO metric coarse thread)	[2]	----			M10x40 (AL78)	M10x50 (AL 080)		M16x80 (AL161) (A4D & A4E M10x50) (AL080)		M16x80 (AL161)	M20x90 (AL188)
Lock Washer	[2]	----			M10 (AE075)	M10 (AE075)		M16 (AE150)		M16 (AE150)	M20 (AE175)
T. Optional Insp. Plate Bolt		----			----		M6x16 (AA023)	M8x20 (AA049)	M10x25 (AA075)	M10x25 (AA075)	
Lock Washer		----			----		M6 (AE025)	M8 (AE050)	M10 (AE075)	M10 (AE075)	

* All lock washers are steel, single coil, helical spring.
H Stainless steel required on stainless steel models (XX5XX).
HH Requires fluoroelastomer (CFXXX) on pumps with RF stators
** 4-stage models use the same stator rings as 2-stage models of the same element size.
*** Stuffing boxes with tapped holes use the studs.

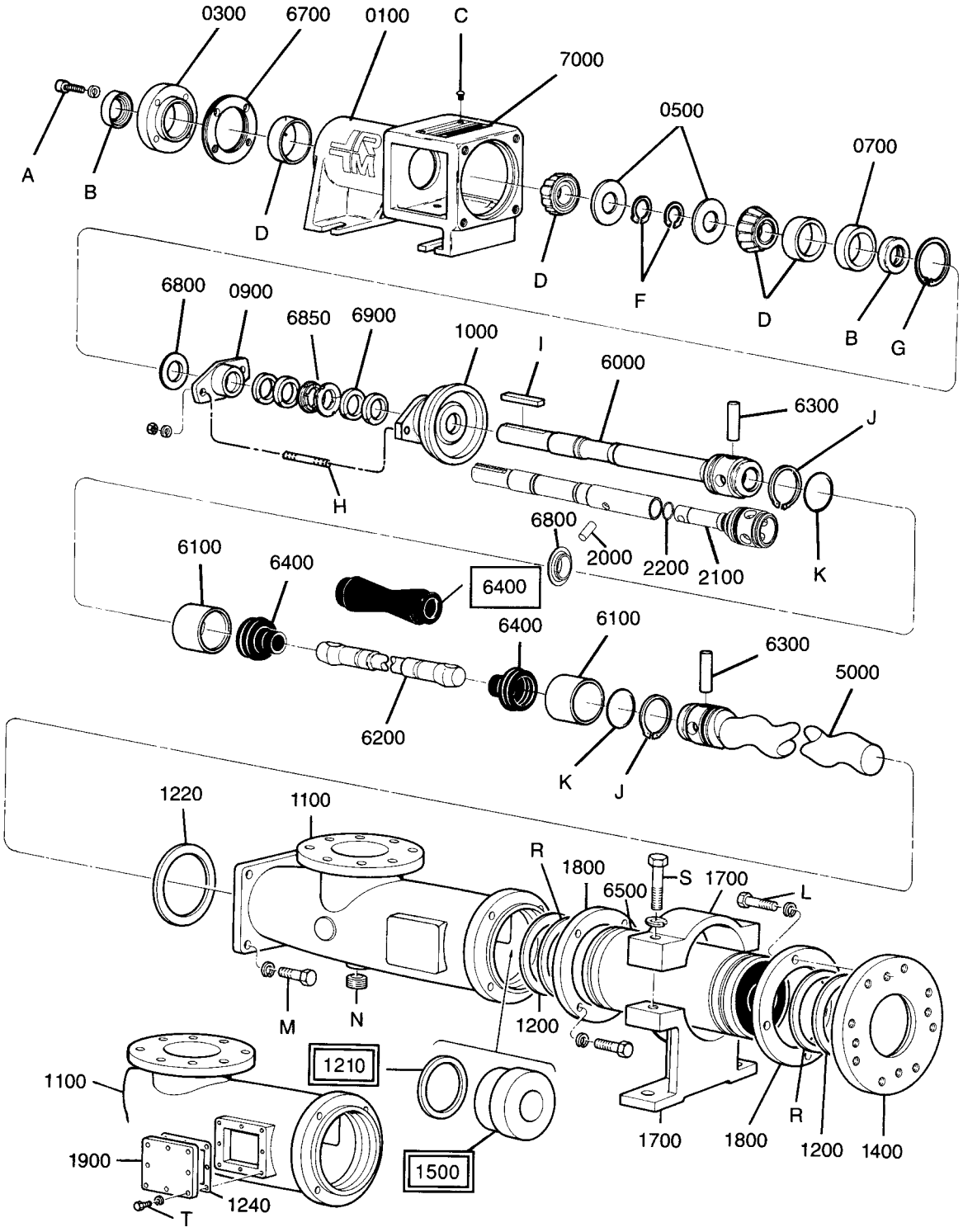
LEGEND:
O.D. = Outer Diameter
I.D. = Inner Diameter
ϕ = Diameter
W = Wide
" = Inches
NPT = American National Standard Taper Pipe
Sft = Shaft

4-43. EXPLODED VIEWS

4-44. STANDARD MODEL – BALL BEARING DESIGN DRIVE END



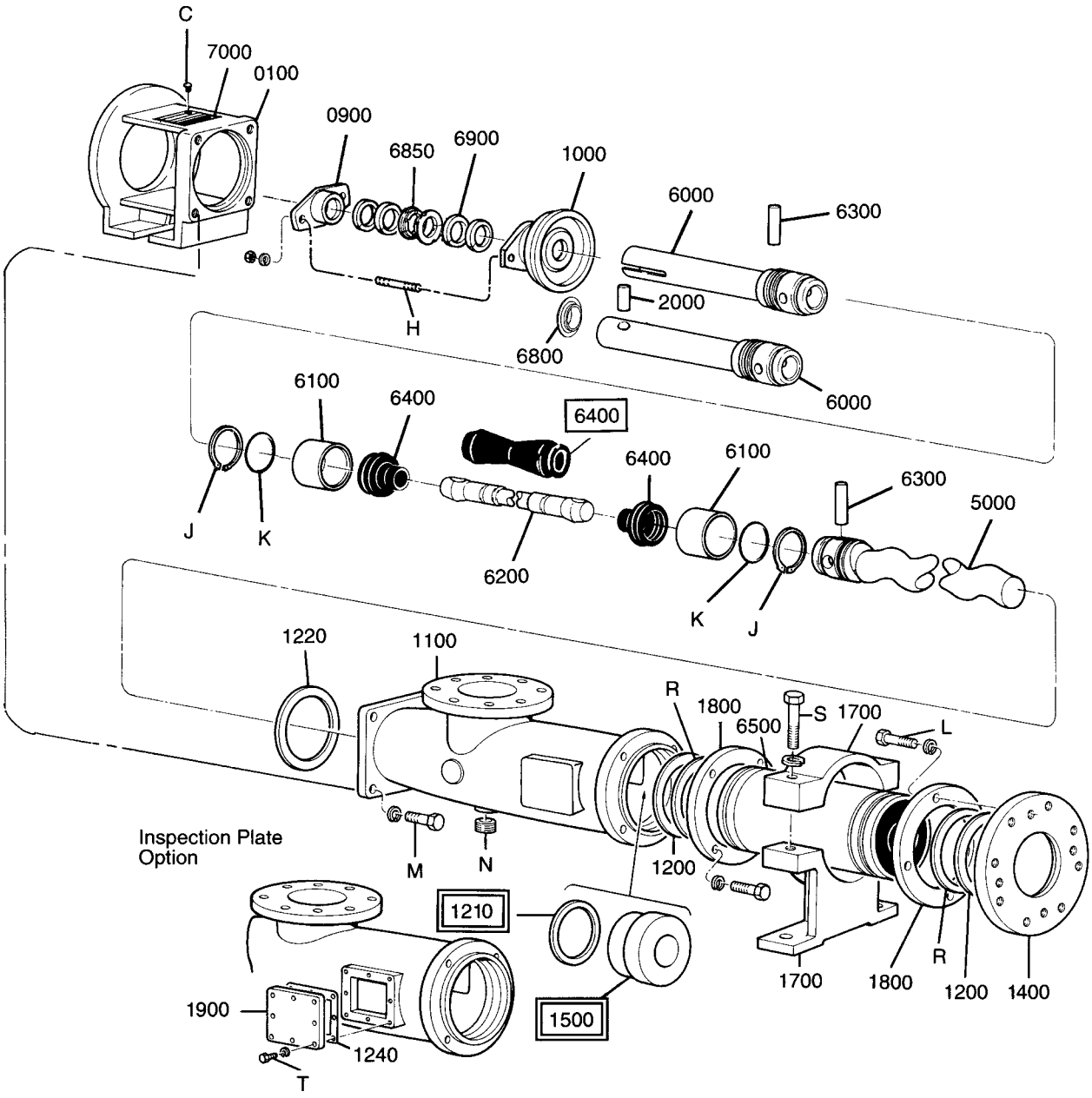
4-45. STANDARD MODEL – ROLLER BEARING DESIGN DRIVE END



= Used on Models A4A, A4B, A4C, A4D, A4E, A4F

= Used on Models A1B, A1C, A1D, A2A, A2B, A2C, A2D, A4A, A4B, D4B, D4C, D4D

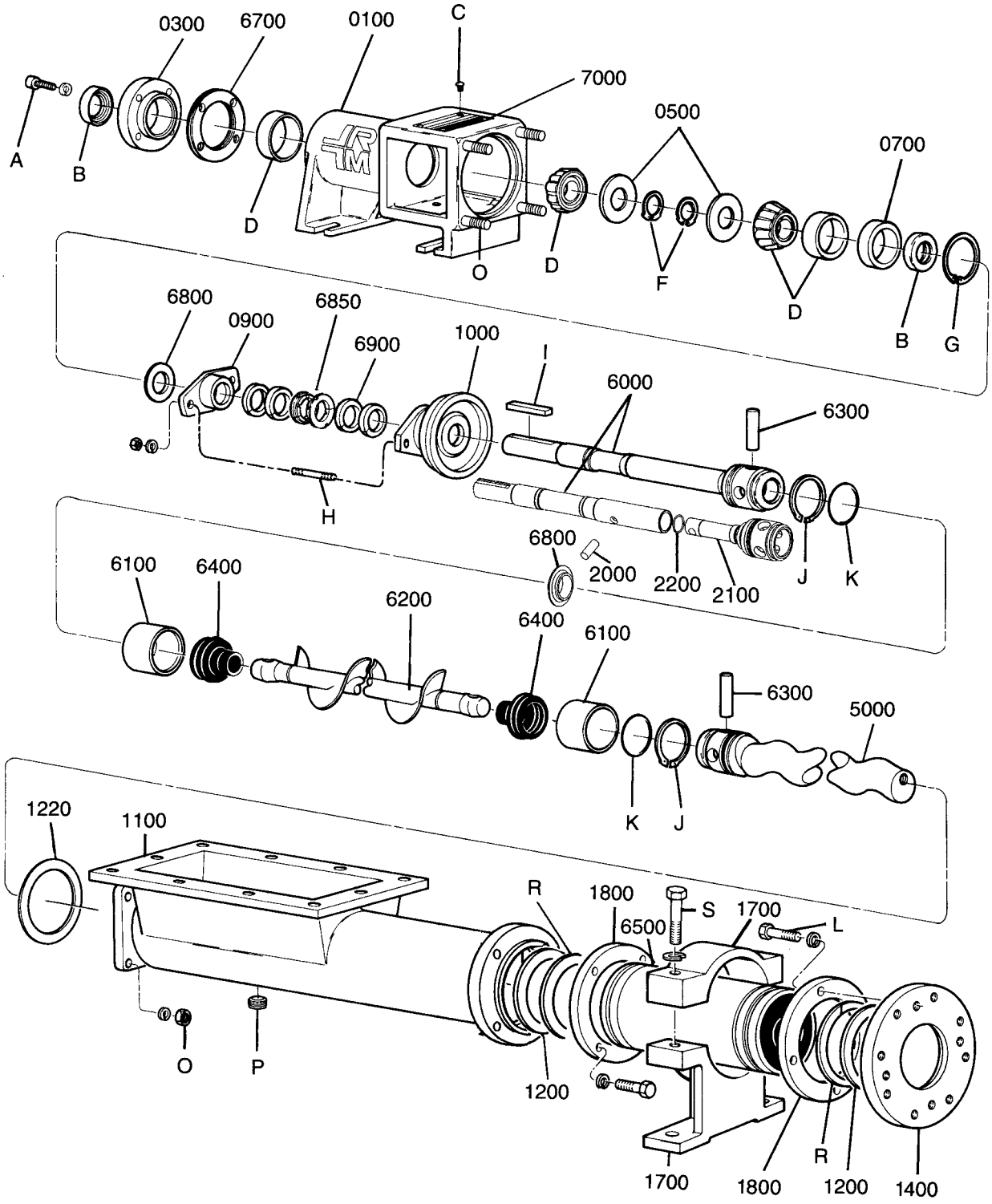
4-46. CLOSED-COUPLED MODELS



= Used on Models B4A, B4B, B4C, B4D, B4E

= Used on Models B1B, B1C, B1D, B2A, B2B, B2C, B2D, B4A, B4B, E4B, E4C, E4D,

4-47. OPEN THROAT MODELS



4-48. PARTS LISTS

REF. DESCRIPTION		TYPE	A2A PART NO.	A1B PART NO.	A2B PART NO.	A1C PART NO.	A2C PART NO.
0100 BEARING HOUSING		CD/SS	CC A2A0110	CC A2A0110	CC A2A0110	CC A2A0110	CC A2A0110
0900 PACKING GLAND		CD/SS	SC A2A0910	SC A2A0910	SC A2A0910	SC A2A0910	SC A2A0910
1000 STUFFING BOX		CD	CC A2A1010	CC A2A1010	CC A2A1010	CC A2A1010	CC A2A1010
		SS	SC A2A1010	SC A2A1010	SC A2A1010	SC A2A1010	SC A2A1010
1000 SEAL	*	CD/SS	SC A2A1011	SC A2A1011	SC A2A1011	SC A2A1011	SC A2A1011
HOUSING	**	CD/SS	4240833007	4240833007	4240833007	4240833007	4240833007
1100 SUCTION		CD	CC A2A1110	CC A1B1110	CC A1B1110	CC A1C1110	CC A1C1110
CHAMBER		SS	SC A2A1110	SC A1B1110	SC A1B1110	SC A1C1110	SC A1C1110
1200 STATOR GASKET		Q/R	GG A2A1200	GG A1B1200	GG A1B1200	GG A1C1200	GG A1C1200
		F	GF A2A1200	GF A1B1200	GF A1B1200	GF A1C1200	GF A1C1200
		B	GB A2A1200	GB A1B1200	GB A1B1200	GB A1C1200	GB A1C1200
1220 SUCTION		Q/R	GG A2A1220	GG A2A1220	GG A2A1220	GG A2A1220	GG A2A1220
CHAM. GASKET		F	GF A2A1220	GF A2A1220	GF A2A1220	GF A2A1220	GF A2A1220
		B	GB A2A1220	GB A2A1220	GB A2A1220	GB A2A1220	GB A2A1220
1400 DISCHARGE FLANGE		CD	MS A2A1410	MS A1B1410	MS A1B1410	MS A1C1410	MS A1C1410
		SS	SS A2A1410	SS A1B1410	SS A1B1410	SS A1C1410	SS A1C1410
1700 STATOR SUPPORT		CD/SS	MS A2A1710	MS A2A1710	MS A2A1710	MS A1C1710	MS A1C1710
1800 CLAMP RING		CD/SS	MS A2A1810	MS A1B1810	MS A1B1810	MS A1C1810	MS A1C1810
2000 SHAFT PIN		CD/SS	4220487017	4220487017	4220487017	4220487017	4220487017
2100 INTERMEDIATE		CD	4250392001	4250392001	4250392001	4250393001	4250393001
SHAFT		SS	4250392017	4250392017	4250392017	4250393017	4250393017
2200 SEAL		Q/R	3207902206	3207902206	3207902206	3207902206	3207902206
RING		F	3207905206	3207905206	3207905206	3207905206	3207905206
		B	3207904206	3207904206	3207904206	3207904206	3207904206
5000 ROTOR		CD	TS A2A5000	TS A1B5000	TS A2B5000	TS A1C5000	TS A2C5000
		SS	SS A2A5000	SS A1B5000	SS A2B5000	SS A1C5000	SS A2C5000
6000 DRIVE SHAFT		CD	AS A2A6000	AS A2A6000	AS A2A6000	AS A1C6000	AS A1C6000
		SS	SS A2A6000	SS A2A6000	SS A2A6000	SS A1C6000	SS A1C6000
6000 DRIVE SHAFT		CD	4250378001	4250378001	4250378001	4250378001	4250378001
(2-PIECE OPTION)		SS	4250378017	4250378017	4250378017	4250378015	4250378015
6100 RETAINING RING		CD/SS	ST A2A6100	ST A2A6100	ST A2A6100	ST A1C6100	ST A1C6100
6200 CONNECTING ROD		CD/SS	AS A2A6200	AS A2A6200	AS A2A6200	AS A1C6200	AS A1C6200
6300 DRIVE PIN		CD/SS	TR A2A6300	TR A2A6300	TR A2A6300	TR A1C6300	TR A1C6300
6400 JOINT SEAL		Q/R	RD A2A6400	RD A2A6400	RD A2A6400	RD A1C6400	RD A1C6400
		F/B	RF A2A6400	RF A2A6400	RF A2A6400	RF A1C6400	RF A1C6400
6500 STATOR		Q	RQ A2A6510	RQ A1B6510	RQ A2B6510	RQ A1C6510	RQ A2C6510
		R	RR A2A6510	RR A1B6510	RR A2B6510	RR A1C6510	RR A2C6510
		F	RF A2A6510	RF A1B6510	RF A2B6510	RF A1C6510	RF A2C6510
		B	RB A2A6510	RB A1B6510	RB A2B6510	RB A1C6510	RB A2C6510
6800 SLINGER RING		CD/SS	RZ A2A6800	RZ A2A6800	RZ A2A6800	RZ A2A6800	RZ A2A6800
6800 SLINGER/PIN RETNR (2-PIECE OPTION)		CD/SS	423052800	423052800	423052800	423052800	423052800
6850 LANTERN RING		CD/SS	GR A2A6850	GR A2A6850	GR A2A6850	GR A2A6850	GR A2A6850
6900 PACKING		STD.	PC A2A6901	PC A2A6901	PC A2A6901	PC A2A6901	PC A2A6901
		PTFE	3403655001	3403655001	3403655001	3403655001	3403655001
7000 NAME PLATE		CD/SS	GA A2A7000	GA A2A7000	GA A2A7000	GA A2A7000	GA A2A7000
K O-RING		Q/R	CA137	CA137	CA137	CA141	CA141
		F/B	CF137	CF137	CF137	CF141	CF141
J SNAP RING		CD	EB007	EB007	EB007	EB015	EB015
		SS	EB507	EB507	EB507	EB015	EB015
R STATOR RING		CD/SS	EC022	EC028	EC028	EC034	EC034

*FRICTION DRIVEN SEAL, TYPE 43 OR EQUAL
**POSITIVE DRIVE SEAL, TYPE 680 OR EQUAL
NOTE: CONTACT FACTORY FOR REPLACEMENT MECHANICAL SEAL PART NUMBERS

PARTS LIST (Cont.)

REF. DESCRIPTION	TYPE	A4A PART NO.	A4B PART NO.	A4C PART NO.	A4D PART NO.	A4E PART NO.	A4F PART NO.
0100 BEARING HOUSING	CD/SS	CC A2A0110	CC A1D0110	CC A1D0110	CC A1F0110	CC A1F0110	CC A1H0110
0300 BEARING COVER	CD/SS		CC A1D0300	CC A1D0300	CC A1F0300	CC A1F0300	CC A1H0300
0500 GREASE RETAINER	CD/SS		MS A1D0500	MS A1D0500	MS A1F0500	MS A1F0500	MS A1H0500
0700 GREASE SEAL HSG.	CD/SS		MS A1D0700	MS A1D0700	MS A1F0700	MS A1F0700	MS A1H0700
0900 PACKING GLAND	CD/SS	SC A1A0910	SC A1D0910	SC A1D0910	SC A1F0910	SC A1F0910	SC A1H0910
1000 STUFFING BOX	CD	CC A2A1010	CC A1D1010	CC A1D1010	CC A1F1010	CC A1F1010	CC A1H1010
	SS	SC A2A1010	SC A1D1010	SC A1D1010	SC A1F1010	SC A1F1010	SC A1H1010
1000 SEAL *	CD/SS	SC A2A1011	SC A1D1011	SC A1D1011	SC A1F1011	SC A1F1011	SC A1H1011
HOUSING **	CD/SS	4240833007	4240834007	4240834007	4240835007	4240835007	4240836007
1100 SUCTION CHAMBER	CD	CC A1C1110	CC A1D1110	CC A1E1110	CC A1F1110	CC A1G1110	CC A1H1110
	SS	SC A1C1110	SC A1D1110	SC A1E1110	SC A1F1110	SC A1G1110	SC A1H1110
1200 STATOR GASKET	Q/R	GG A2A1200	GG A1B1200	GG A1C1200	GG A1D1200	GG A1E1200	GG A1F1200
	F	GF A2A1200	GF A1B1200	GF A1C1200	GF A1D1200	GF A1E1200	GF A1F1200
	B	GB A2A1200	GB A1B1200	GB A1C1200	GB A1D1200	GB A1E1200	GB A1F1200
1210 STATOR ADAPTOR GASKET	Q/R	GG A1C1200	GG A1D1200	GG A1E1200	GG A1F1200	GG A1G1200	GG A1H1200
	F	GF A1C1200	GF A1D1200	GF A1E1200	GF A1F1200	GF A1G1200	GF A1H1200
	B	GB A1C1200	GB A1D1200	GB A1E1200	GB A1F1200	GB A1G1200	GB A1H1200
1220 SUCTION CHAM. GASKET	Q/R	GG A2A1220	GG A1D1220	GG A1D1220	GG A1F1220	GG A1F1220	GG A1H1220
	F	GF A2A1220	GF A1D1220	GF A1D1220	GF A1F1220	GF A1F1220	GF A1H1220
	B	GB A2A1220	GB A1D1220	GB A1D1220	GB A1F1220	GB A1F1220	GB A1H1220
1240 INSP. PLATE GSKT.	Q/R					GG A1G1240	GG A1H1240
	F					GF A1G1240	GF A1H1240
	B					GB A1G1240	GB A1H1240
1400 DISCHARGE FLANGE	CD	MS A4A1410	MS A4B1410	MS A4C1410	MS A4D1410	MS A4E1410	MS A4F1410
	SS	SS A4A1410	SS A4B1410	SS A4C1410	SS A4D1410	SS A4E1410	SS A4F1410
1500 STATOR ADAPTOR	CD	MS A4A1510	MS A4B1510	MS A4C1510	MS A4D1510	MS A4E1510	MS A4F1510
	SS	SS A4A1510	SS A4B1510	SS A4C1510	SS A4D1510	SS A4E1510	SS A4F1510
1700 STATOR SUPPORT	CD/SS	MS A4A1710	CC A4B1710	CC A4C1710	CC A4D1710	CC A4E1710	CC A4F1710
1800 CLAMP RING	CD/SS	MS A4A1810	MS A4B1810	MS A4C1810	MS A4D1810	MS A4E1810	MS A4F1810
	CD/SS	MS A4A1811	MS A4B1811	MS A4C1811	MS A4D1811	MS A4E1811	MS A4F1811
1900 INSPECTION PLATE	CD					MS A1G1900	MS A1H1900
	SS					SS A1G1900	SS A1H1900
2000 SHAFT PIN	CD/SS	4220487017	4220488017	4220488017	4220489017	4220489017	4220492017
2100 INTERMEDIATE SHAFT	CD	4250393001	4250394001	4250395001	4250396001	4250397001	4250398001
	SS	4250393017	4250394017	4250395017	4250396017	4250397017	4250398017
2200 SEAL RING	Q/R	3207902206	3207902210	3207902210	3207902216	3207902216	3207902223
	F	3207905206	3207905210	3207905210	3207905216	3207905216	3207905223
	B	3207904206	3207904210	3207904210	3207904216	3207904216	3207904223
5000 ROTOR	CD	TS A4A5000	TS A4B5000	TS A4C5000	TS A4D5000	TS A4E5000	TS A4F5000
	SS	SS A4A5000	SS A4B5000	SS A4C5000	SS A4D5000	SS A4E5000	SS A4F5000
6000 DRIVE SHAFT	CD	AS A1C6000	AS A1D6000	AS A1E6000	AS A1F6000	AS A1G6000	AS A1H6000
	SS	SS A1C6000	SS A1D6000	SS A1E6000	SS A1F6000	SS A1G6000	SS A1H6000
6000 DRIVE SHAFT (2-PIECE OPTION)	CD	4250378001	4250379001	4250379001	4250380001	4250380001	4250381001
	SS	4250378015	4250379015	4250379015	4250380015	4250380015	4250381015
6100 RETAINING RING	CD/SS	ST A1C6100	ST A1D6100	ST A1E6100	ST A1F6100	ST A1G6100	ST A1H6100
6200 CONNECTING ROD	CD	AS A1C6200	AS A1D6200	AS A1E6200	AS A1F6200	AS A1G6200	AS A1H6200
	SS	AS A1C6200	AS A1D6200	AS A1E6200	AS A1F6200	AS A1G6200	AS A1H6200
6300 DRIVE PIN	CD/SS	TR A1C6300	TR A1D6300	TR A1E6300	TR A1F6300	TR A1G6300	TR A1H6300
6400 JOINT SEAL	Q/R	RD A1C6400	RD A1D6400	RD A1E6400	RD A1F6400	RD A1G6400	RD A1H6400
	F/R	RF A1C6400	RF A1D6400	RF A1E6400	RF A1F6400	RF A1G6400	RF A1H6400
6500 STATOR	Q	RQ A4A6510	RQ A4B6510	RQ A4C6510	RQ A4D6510	RQ A4E6510	RQ A4F6510
	R	RR A4A6510	RR A4B6510	RR A4C6510	RR A4D6510	RR A4E6510	RR A4F6510
	F	RF A4A6510	RF A4B6510	RF A4C6510	RF A4D6510	RF A4E6510	RF A4F6510
	B	RB A4A6510	RB A4B6510	RB A4C6510	RB A4D6510	RB A4E6510	RB A4F6510
6700 BEARING SHIMS	CD/SS		GP A1D6700	GP A1D6700	GP A1F6700	GP A1F6700	GP A1H6700
6800 SLINGER RING	CD/SS	RZ A2A6800	RZ A1D6800	RZ A1D6800	RZ A1F6800	RZ A1F6800	RZ A1H6800
6800 SLINGER/PIN RETNR (2-PIECE OPTION)	CD/SS	4230528000	4230529000	4230529000	4230530000	4230530000	4230531000
6850 LANTERN RING	CD/SS	GR A2A6850	GR A1D6850	GR A1D6850	GR A1F6850	GR A1F6850	GR A1H6850
6900 PACKING	STD.	PC A2A6901	PC A1D6901	PC A1D6901	PC A1F6901	PC A1F6901	PC A1H6901
	PTFE	3403655001	3403655002	3403655002	3403655003	3403655003	3403655004
7000 NAME PLATE	CD/SS	GA A2A7000	GA A2A7000	GA A2A7000	GA A1F7000	GA A1F7000	GA A1F7000
K O-RING	Q/R	CA141	CA145	CA175	CA188	CA197	CA228
	F/B	CF141	CF145	CF175	CF188	CF197	CF228
J SNAP RING	CD	EB015	EB020	EB027	EB036	EB045	EB049
	SS	EB515	EB520	EB527	EB536	EB545	EB549
R STATOR RING	CD/SS	EC022	EC028	EC034	EC039	EC046	EC055

*FRICTION DRIVE SEAL, TYPE 43 OR EQUAL
**POSITIVE DRIVE SEAL, TYPE 680 OR EQUAL

NOTE: CONTACT FACTORY FOR REPLACMENT MECHANICAL SEAL PART NUMBERS

PARTS LIST (Cont.)

REF. DESCRIPTION	TYPE	A1D PART NO.	A2D PART NO.	A1E PART NO.	A2E PART NO.	A1F/A1FE PART NO.	A2F/A2FE PART NO.
0100 BEARING HOUSING	CD/SS	CC A1D0110	CC A1D0110	CC A1D0110	CC A1D0110	CC A1F0110	CC A1F0110
0300 BEARING COVER	CD/SS	CC A1D0300	CC A1D0300	CC A1D0300	CC A1D0300	CC A1F0300	CC A1F0300
0500 GREASE RETAINER	CD/SS	MS A1D0500	MS A1D0500	MS A1D0500	MS A1D0500	MS A1F0500	MS A1F0500
0700 GREASE SEAL HSG.	CD/SS	MS A1D0700	MS A1D0700	MS A1D0700	MS A1D0700	MS A1F0700	MS A1F0700
0900 PACKING GLAND	CD/SS	SC A1D0910	SC A1D0910	SC A1D0910	SC A1D0910	SC A1F0910	SC A1F0910
1000 STUFFING BOX	CD	CC A1D1010	CC A1D1010	CC A1D1010	CC A1D1010	CC A1F1010	CC A1F1010
	SS	SC A1D1010	SC A1D1010	SC A1D1010	SC A1D1010	SC A1F1010	SC A1F1010
1000 SEAL *	CD/SS	SC A1D1011	SC A1D1011	SC A1D1011	SC A1D1011	SC A1F1011	SC A1F1011
HOUSING **	CD/SS	4240834007	4240834007	4240834007	4240834007	4240835007	4240835007
1100 SUCTION CHAMBER	CD	CC A1D1110	CC A1D1110	CC A1E1110	CC A1E1110	CC A1F1110	CC A1F1110
	SS	SC A1D1110	SC A1D1110	SC A1E1110	SC A1E1110	SC A1F1110	SC A1F1110
1200 STATOR GASKET	Q/R	GG A1D1200	GG A1D1200	GG A1E1200	GG A1E1200	GG A1F1200	GG A1F1200
	F	GF A1D1200	GF A1D1200	GF A1E1200	GF A1E1200	GF A1F1200	GF A1F1200
	B	GB A1D1200	GB A1D1200	GB A1E1200	GB A1E1200	GB A1F1200	GB A1F1200
1220 SUCTION CHAM. GASKET	Q/R	GG A1D1220	GG A1D1220	GG A1D1220	GG A1D1220	GG A1F1220	GG A1F1220
	F	GF A1D1220	GF A1D1220	GF A1D1220	GF A1D1220	GF A1F1220	GF A1F1220
	B	GB A1D1220	GB A1D1220	GB A1D1220	GB A1D1220	GB A1F1220	GB A1F1220
1240 INSP. PLATE GSKT.	Q/R			GG A1E1240	GG A1E1240	GG A1F1240	GG A1F1240
	F			GF A1E1240	GF A1E1240	GF A1F1240	GF A1F1240
	B			GB A1E1240	GB A1E1240	GB A1F1240	GB A1F1240
1400 DISCHARGE FLANGE	CD	MS A1D1410	MS A1D1410	MS A1E1410	MS A1E1410	MS A1F1410	MS A1F1410
	SS	SS A1D1410	SS A1D1410	SS A1E1410	SS A1E1410	SS A1F1410	SS A1F1410
1700 STATOR SUPPORT	CD/SS	CC A1D1710	CC A1D1710	CC A1E1710	CC A1E1710	CC A1F1710	CC A1F1710
1800 CLAMP RING	CD/SS	MS A1D1810	MS A1D1810	MS A1E1810	MS A1E1810	MS A1F1810	MS A1F1810
1900 INSPECTION PLATE	CD			MS A1E1900	MS A1E1900	MS A1F1900	MS A1F1900
	SS			SS A1E1900	SS A1E1900	SS A1F1900	SS A1F1900
2000 SHAFT PIN	CD/SS	4220488017	4220488017	4220488017	4220488017	4220489017	4220489017
2100 INTERMEDIATE SHAFT	CD	4250394001	4250394001	4250395001	4250395001	4250396001	4250396001
	SS	4250394017	4250394017	4250395017	4250395017	4250396017	4250396017
2200 SEAL O-RING	Q/R	3207902210	3207902210	3207902210	3207902210	3207902216	3207902216
	F	3207905210	3207905210	3207905210	3207905210	3207905216	3207905216
	B	3207904210	3207904210	327904210	3207904210	3207904216	3207904216
5000 ROTOR	CD	TS A1D5000	TS A2D5000	TS A1E5000	TS A2E5000	TS A1F5000	TS A2F5000
	CD					TS A1FE5000	TS A2FE5000
	SS	SS A1D5000	SS A2D5000	SS A1E5000	SS A2E5000	SS A1F5000	SS A2F5000
	SS					SS A1FE5000	SS A2FE5000
6000 DRIVE SHAFT	CD	AS A1D6000	AS A1D6000	AS A1E6000	AS A1E6000	AS A1F6000	AS A1F6000
	SS	SS A1D6000	SS A1D6000	SS A1E6000	SS A1E6000	SS A1F6000	SS A1F6000
6000 DRIVE SHAFT (2-PIECE OPTION)	CD	4250379001	4250379001	4250379001	4250379001	4250380001	4250380001
	SS	4250379015	4250379015	4250379015	4250379015	4250380015	4250380015
6100 RETAINING RING	CD/SS	ST A1D6100	ST A1D6100	ST A1E6100	ST A1E6100	ST A1F6100	ST A1F6100
6200 CONNECTING ROD	CD	AS A1D6200	AS A1D6200	AS A1E6200	AS A1E6200	AS A1F6200	AS A1F6200
	SS	AS A1D6200	AS A1D6200	AS A1E6200	AS A1E6200	AS A1F6200	AS A1F6200
6300 DRIVE PIN	CD/SS	TR A1D6300	TR A1D6300	TR A1E6300	TR A1E6300	TR A1F6300	TR A1F6300
6400 JOINT SEAL	Q/R	RD A1D6400	RD A1D6400	RD A1E6400	RD A1E6400	RD A1F6400	RD A1F6400
	F/B	RF A1D6400	RF A1D6400	RF A1E6400	RF A1E6400	RF A1F6400	RF A1F6400
6500 STATOR	Q	RQ A1D6510	RQ A2D6510	RQ A1E6510	RQ A2E6510	RQ A1F6510	RQ A2F6510
	Q					RQ A1FE6510	RQ A2FE6510
	R	RR A1D6510	RR A2D6510	RR A1E6510	RR A2E6510	RR A1F6510	RR A2F6510
	R					RR A1FE6510	RR A2FE6510
	F	RF A1D6510	RF A2D6510	RF A1E6510	RF A2E6510	RF A1F6510	RF A2F6510
	B	RB A1D6510	RB A2D6510	RB A1E6510	RB A2E6510	RB A1F6510	RB A2F6510
	B					RB A1FE6510	RB A2FE6510
6700 BEARING SHIMS	CD/SS	GP A1D6700	GP A1D6700	GP A1D6700	GP A1D6700	GP A1F6700	GP A1F6700
6800 SLINGER RING	CD/SS	RZ A1D6800	RZ A1D6800	RZ A1D6800	RZ A1D6800	RZ A1F6800	RZ A1F6800
6800 SLINGER/PIN RETNR (2-PIECE OPTION)	CD/SS	4230529000	4230529000	4230529000	4230529000	4230530000	4230530000
6850 LANTERN RING	CD/SS	GR A1D6850	GR A1D6850	GR A1D6850	GR A1D6850	GR A1F6850	GR A1F6850
6900 PACKING	STD.	PC A1D6901	PC A1D6901	PC A1D6901	PC A1D6901	PC A1F6901	PC A1F6901
	PTFE	3403655002	3403655002	3403655002	3403655002	3403655003	3403655003
7000 NAME PLATE	CD/SS	GA A2A7000	GA A2A7000	GA A2A7000	GA A2A7000	GA A1F7000	GA A1F7000
K O-RING	Q/R	CA145	CA145	CA175	CA175	CA188	CA188
	F/B	CF145	CF145	CF175	CF175	CF188	CF188
J SNAP RING	CD	EB020	EB020	EB027	EB027	EB036	EB036
	SS	EB520	EB520	EB527	EB527	EB536	EB536
R STATOR RING	CD/SS	EC039	EC039	EC046	EC046	EC055	EC055

*FRICTION DRIVE SEAL, TYPE 43 OR EQUAL

**POSITIVE DRIVE SEAL, TYPE 680 OR EQUAL

NOTE: CONTACT FACTORY FOR REPLACMENT MECHANICAL SEAL PART NUMBERS

PARTS LIST (Cont.)

REF. DESCRIPTION	TYPE	A1G A1GE PART NO.	A2G A2GE PART NO.	A1H A1HE PART NO.	A2H A2HE PART NO.	A1J A1JE PART NO.	A2J A2JE PART NO.	A1K A1KE PART NO.
0100 BEARING HOUSING	CD/SS	CC A1F0110	CC A1F0110	CC A1H0110	CC A1H0110	CC A1H0110	CC A1H0110	CC A1H0110
0300 BEARING COVER	CD/SS	CC A1F0300	CC A1F0300	CC A1H0300	CC A1H0300	CC A1H0300	CC A1H0300	CC A1H0300
0500 GREASE RETAINER	CD/SS	MS A1F0500	MS A1F0500	MS A1H0500	MS A1H0500	MS A1H0500	MS A1H0500	MS A1H0500
0700 GREASE SEAL HSG.	CD/SS	MS A1F0700	MS A1F0700	MS A1H0700	MS A1H0700	MS A1H0700	MS A1H0700	MS A1H0700
0900 PACKING GLAND	CD/SS	SC A1F0910	SC A1F0910	SC A1H0910	SC A1H0910	SC A1H0910	SC A1H0910	SC A1H0910
1000 STUFFING BOX	CD	CC A1F1010	CC A1F1010	CC A1H1010	CC A1H1010	CC A1H1010	CC A1H1010	CC A1H1010
	SS	SC A1F1010	SC A1F1010	SC A1H1010	SC A1H1010	SC A1H1010	SC A1H1010	SC A1H1010
1000 SEAL *	CD/SS	SC A1F1011	SC A1F1011	SC A1H1011	SC A1H1011	SC A1H1011	SC A1H1011	SC A1H1011
HOUSING **	CD/SS	4240835007	4240835007	4240836007	4240836007	4240836007	4240836007	4240836007
1100 SUCTION CHAMBER	CD	CC A1G1110	CC A1G1110	CC A1H1110	CC A1H1110	CC A1H1110	CC A1H1110	CC A1H1110
	SS	SC A1G1110	SC A1G1110	SC A1H1110	SC A1H1110	SC A1H1110	SC A1H1110	SC A1H1110
1200 STATOR GASKET	Q/R	GG A1G1200	GG A1G1200	GG A1H1200	GG A1H1200	GG A1H1200	GG A1H1200	GG A1H1200
	F	GF A1G1200	GF A1G1200	GF A1H1200	GF A1H1200	GF A1H1200	GF A1H1200	GF A1H1200
	B	GB A1G1200	GB A1G1200	GB A1H1200	GB A1H1200	GB A1H1200	GB A1H1200	GB A1H1200
1220 SUCTION CHAM. GASKET	Q/R	GG A1F1220	GG A1F1220	GG A1H1220	GG A1H1220	GG A1H1220	GG A1H1220	GG A1H1220
	F	GF A1F1220	GF A1F1220	GF A1H1220	GF A1H1220	GF A1H1220	GF A1H1220	GF A1H1220
	B	GB A1F1220	GB A1F1220	GB A1H1220	GB A1H1220	GB A1H1220	GB A1H1220	GB A1H1220
1240 INSP. PLATE GSKT.	Q/R	GG A1G1240	GG A1G1240	GG A1H1240	GG A1H1240	GG A1H1240	GG A1H1240	GG A1H1240
	F	GF A1G1240	GF A1G1240	GF A1H1240	GF A1H1240	GF A1H1240	GF A1H1240	GF A1H1240
	B	GB A1G1240	GB A1G1240	GB A1H1240	GB A1H1240	GB A1H1240	GB A1H1240	GB A1H1240
1400 DISCHARGE FLANGE	CD	MS A1G1410	MS A1G1410	MS A1H1410	MS A1H1410	MS A1H1410	MS A1H1410	MS A1H1410
	SS	SS A1G1410	SS A1G1410	SS A1H1410	SS A1H1410	SS A1H1410	SS A1H1410	SS A1H1410
1700 STATOR SUPPORT	CD/SS	CC A1G1710	CC A1G1710	CC A1H1710	CC A1H1710	CC A1H1710	CC A1H1710	CC A1H1710
1800 CLAMP RING	CD/SS	MS A1G1810	MS A1G1810	MS A1H1810	MS A1H1810	MS A1H1810	MS A1H1810	MS A1H1810
1900 INSPECTION PLATE	CD	MS A1G1900	MS A1G1900	MS A1H1900	MS A1H1900	MS A1H1900	MS A1H1900	MS A1H1900
	SS	SS A1G1900	SS A1G1900	SS A1H1900	SS A1H1900	SS A1H1900	SS A1H1900	SS A1H1900
2000 SHAFT PIN	CD/SS	4220489017	4220489017	4220492017	4220492017	4220492017	4220492017	4220492017
2100 INTERMEDIATE SHAFT	CD	4250397001	4250397001	4250398001	4250398001	4250398001	4250398001	4250398001
	SS	425397017	4250397017	4250398017	4250398017	4250398017	4250398017	4250398017
2200 SEAL O-RING	Q/R	3207902216	3207902216	3207902223	3207902223	3207902223	3207902223	3207902223
	F	3207905216	3207905216	3207905223	3207905223	3207905223	3207905223	3207905223
	B	3207904216	3207904216	3207904223	3207904223	3207904223	3207904223	3207904223
5000 ROTOR	CD	TS A1G5000	TS A2G5000	TS A1H5000	TS A2H5000	TS A1J5000	TS A2J5000	TS A1K5000
	CD	TS A1GE5000	TS A2GE5000	TS A1HE5000	TS A2HE5000	TS A1JE5000	TS A2JE5000	TS A1KE5000
	SS	SS A1G5000	SS A2G5000	SS A1H5000	SS A2H5000	SS A1J5000	SS A2J5000	SS A1K5000
	SS	SS A1GE5000	SS A2GE5000	SS A1HE5000	SS A2HE5000	SS A1JE5000	SS A2JE5000	SS A1KE5000
6000 DRIVE SHAFT	CD	AS A1G6000	AS A1G6000	AS A1H6000	AS A1H6000	AS A1H6000	AS A1H6000	AS A1H6000
	SS	SS A1G6000	SS A1G6000	SS A1H6000	SS A1H6000	SS A1H6000	SS A1H6000	SS A1H6000
6000 DRIVE SHAFT (2-PIECE OPTION)	CD	4250380001	4250380001	4250381001	4250381001	4250381001	4250381001	4250381001
	SS	4250380015	4250380015	4250381001	4250381001	4250381001	4250381001	4250381001
6100 RETAINING RING	CD/SS	ST A1G6100	ST A1G6100	ST A1H6100	ST A1H6100	ST A1H6100	ST A1H6100	ST A1H6100
6200 CONNECTING ROD	CD	AS A1G6200	AS A1G6200	AS A1H6200	AS A1H6200	AS A1H6200	AS A1H6200	AS A1H6200
	SS	AS A1G6200	AS A1G6200	AS A1H6200	AS A1H6200	AS A1H6200	AS A1H6200	AS A1H6200
6300 DRIVE PIN	CD/SS	TR A1G6300	TR A1G6300	TR A1H6300	TR A1H6300	TR A1H6300	TR A1H6300	TR A1H6300
6400 JOINT SEAL	Q/R	RD A1G6400	RD A1G6400	RD A1H6400	RD A1H6400	RD A1H6400	RD A1H6400	RD A1H6400
	F/B	RF A1G6400	RF A1G6400	RF A1H6400	RF A1H6400	RF A1H6400	RF A1H6400	RF A1H6400
6500 STATOR	Q	RQ A1G6510	RQ A2G6510	RQ A1H6510	RQ A2H6510	RQ A1J6510	RQ A2J6510	RQ A1K6510
	Q	RQ A1GE6510	RQ A2GE6510	RQ A1HE6510	RQ A2HE6510	RQ A1JE6510	RQ A2JE6510	RQ A1KE6510
	R	RR A1G6510	RR A2G6510	RR A1H6510	RR A2H6510	RR A1J6510	RR A2J6510	RR A1K6510
	R	RR A1GE6510	RR A2GE6510	RR A1HE6510	RR A2HE6510	RR A1JE6510	RR A2JE6510	RR A1KE6510
	F	RF A1G6510	RF A2G6510	RF A1H6510	RF A2H6510	RF A1J6510	RF A2J6510	RF A1K6510
	B	RB A1G6510	RB A2G6510	RB A1H6510	RB A2H6510	RB A1J6510	RB A2J6510	RB A1K6510
	B	RB A1GE6510	RB A2GE6510	RB A1HE6510	RB A2HE6510	RB A1JE6510	RB A2JE6510	RB A1KE6510
6700 BEARING SHIMS	CD/SS	GP A1F6700	GP A1F6700	GP A1H6700	GP A1H6700	GP A1H6700	GP A1H6700	GP A1H6700
6800 SLINGER RING	CD/SS	RZ A1F6800	RZ A1F6800	RZ A1H6800	RZ A1H6800	RZ A1H6800	RZ A1H6800	RZ A1H6800
6800 SLINGER/PIN RETNR (2-PIECE OPTION)	CD/SS	4230530000	4230530000	4230531000	4230531000	4230531000	4230531000	4230531000
6850 LANTERN RING	CD/SS	GR A1F6850	GR A1F6850	GR A1H6850	GR A1H6850	GR A1H6850	GR A1H6850	GR A1H6850
6900 PACKING	STD.	PC A1F6901	PC A1F6901	PC A1H6901	PC A1H6901	PC A1H6901	PC A1H6901	PC A1H6901
	PTFE	3403655003	3403655003	3403655004	3403655004	3403655004	3403655004	3403655004
7000 NAME PLATE	CD/SS	GA A1F7000	GA A1F7000	GA A1F7000	GA A1F7000	GA A1F7000	GA A1F7000	GA A1F7000
K O-RING	Q/R	CA197	CA197	CA228	CA228	CA228	CA228	CA228
	F/B	CF197	CF197	CF228	CF228	CF228	CF228	CF228
J SNAP RING	CD	EB045	EB045	EB049	EB049	EB049	EB049	EB049
	SS	EB545	EB545	EB549	EB549	EB549	EB549	EB549
R STATOR RING	CD/SS	EC060	EC060	EC065	EC065	EC065	EC065	EC065

*FRICTION DRIVE SEAL, TYPE 43 OR EQUAL
**POSITIVE DRIVE SEAL, TYPE 680 OR EQUAL

NOTE: CONTACT FACTORY FOR REPLACMENT MECHANICAL SEAL PART NUMBERS

PARTS LIST (Cont.)

REF. DESCRIPTION	TYPE	B2A PART NO.	B4A PART NO.	B1B PART NO.	B2B PART NO.	B4B PART NO.	B1C PART NO.
0100 DRIVE ADAPTOR	CD/SS	CC B2A0100	CC B2A0100	CC B2A0100	CC B2A0100	CC B1D0100	CC B2A0100
0900 PACKING GLAND	CD/SS	SC A2A0910	SC A2A0910	SC A2A0910	SC A2A0910	SC A1D0910	SC A2A0910
1000 STUFFING BOX	CD	CC A2A1010	CC A2A1010	CC A2A1010	CC A2A1010	CC A1D1010	CC A2A1010
	SS	SC A2A1010	SC A2A1010	SC A2A1010	SC A2A1010	SC A1D1010	SC A2A1010
1000 SEAL *	CD/SS	SC A2A1011	SC A2A1011	SC A2A1011	SC A2A1011	SC A2A1011	SC A2A1011
HOUSING **	CD/SS	4240833007	4240833007	4240833007	4240833007	4240833007	4240833007
1100 SUCTION CHAMBER	CD	CC A2A1110	CC A1C1110	CC A1B1110	CC A1B1110	CC A1D1110	CC A1C1110
	SS	SC A2A1110	SC A1C1110	SC A1B1110	SC A1B1110	SC A1D1110	SC A1C1110
1200 STATOR GASKET	Q/R	GG A2A1200	GG A2A1200	GG A1B1200	GG A1B1200	GG A1B1200	GG A1C1200
	F	GF A2A1200	GF A2A1200	GF A1B1200	GF A1B1200	GF A1B1200	GF A1C1200
	B	GB A2A1200	GB A2A1200	GB A1B1200	GB A1B1200	GB A1B1200	GB A1C1200
1210 STATOR ADAPTOR GASKET	Q/R		GG A1C1200			GG A1D1200	
	F		GF A1C1200			GF A1D1200	
	B		GB A1C1200			GB A1D1200	
1220 SUCTION CHAM. GASKET	Q/R	GG A2A1220	GG A2A1220	GG A2A1220	GG A2A1220	GG A1D1220	GG A2A1220
	F	GF A2A1220	GF A2A1220	GF A2A1220	GF A2A1220	GF A1D1220	GF A2A1220
	B	GB A2A1220	GB A2A1220	GB A2A1220	GB A2A1220	GB A1D1220	GB A2A1220
1400 DISCHARGE FLANGE	CD	MS A2A1410	MS A4A1410	MS A1B1410	MS A1B1410	MS A4B1410	MS A1C1410
	SS	SS A2A1410	SS A4A1410	SS A1B1410	SS A1B1410	SS A4B1410	SS A1C1410
1500 STATOR ADAPTOR	CD		MS A4A1510			MS A4B1510	
	SS		SS A4A1510			SS A4B1510	
1600 LOCKING RING	CD/SS	MT B2A1600	MT B2A1600	MT B2A1600	MT B2A1600	MT B1D1600	MT B2A1600
1700 STATOR SUPPORT	CD/SS	MS A2A1710	MS A4A1710	MS A2A1710	MS A2A1710	CCA4B1710	MD A1C1710
1800 CLAMP RING	CD/SS	MS A2A1810	MS A4A1810	MS A1B1810	MS A1B1810	MS A4B1810	MS A1C1810
	CD/SS		MS A4A1811			MS A4B1811	
2000 SHAFT PIN	CD/SS	4220487017	4220487017	4220487017	4220487017	4220488017	4220487017
5000 ROTOR	CD	TS A2A5000	TS A4A5000	TS A1B5000	TS A2B5000	TS A4B5000	TS A1C5000
	SS	SS A2A5000	SS A4A5000	SS A1B5000	SS A2B5000	SS A4B5000	SS A1C5000
6000 DRIVE SHAFT - KEY	CD	AS B2A6000	AS B1C6000	AS B2A6000	AS B2A6000	AS B1D6000	AS B1C6000
	SS	SS B2A6000	SS B1C6000	SS B2A6000	SS B2A6000	SS B1D6000	SS B1C6000
6000 CC DRIVE SHAFT - PIN	CD	4250778001	4250779001	4250778001	4250778001	4250780001	4250779001
	SS	4250778015	4250779015	4250778015	4250778015	4250780015	4250779015
6100 RETAINING RING	CD/SS	ST A2A6100	ST A1C6100	ST A2A6100	ST A2A6100	ST A1D6100	ST A1C6100
6200 CONNECTING ROD	CD	AS A2A6200	AS A1C6200	AS A2A6200	AS A2A6200	AS A1D6200	AS A1C6200
	SS	AS A2A6200	AS A1C6200	AS A2A6200	AS A2A6200	AS A1D6200	AS A1C6200
6300 DRIVE PIN	CD/SS	TR A2A6300	TR A1C6300	TR A2A6300	TR A2A6300	TR A1D6300	TR A1C6300
6400 JOINT SEAL	Q/R	RD A2A6400	RD A1C6400	RD A2A6400	RD A2A6400	RD A1D6400	RD A1C6400
	F/B	RF A2A6400	RF A1C6400	RF A2A6400	RF A2A6400	RF A1D6400	RF A1C6400
6500 STATOR	Q	RQ A2A6510	RQ A4A6510	RQ A1B6510	RQ A2B6510	RQ A4B6510	RQ A1C6510
	R	RR A2A6510	RR A4A6510	RR A1B6510	RR A2B6510	RR A4B6510	RR A1C6510
	F	RF A2A6510	RF A4A6510	RF A1B6510	RF A2B6510	RF A4B6510	RF A1C6510
	B	RB A2A6510	RB A4A6510	RB A1B6510	RB A2B6510	RB A4B6510	RB A1C6510
6800 SLINGER RING - KEY	CD/SS	RZ A2A6800	RZ A2A6800	RZ A2A6800	RZ A2A6800	RZ A1D6800	RZ A2A6800
6800 SLINGER/PIN RETNR - 2 PC/CC	CD/SS	4230528000	4230528000	4230528000	4230528000	4230529000	4230528000
6850 LANTERN RING	CD/SS	GR A2A6850	GR A2A6850	GR A2A6850	GR A2A6850	GR A1D6850	GR A2A6850
6900 PACKING	STD.	PC A2A6901	PC A2A6901	PC A2A6901	PC A2A6901	PC A1D6901	PC A2A6901
	PTFE	3403655001	3403655001	3403655001	3403655001	3403655002	3403655001
7000 NAME PLATE	CD/SS	GA B2A7000	GA B2A7000	GA B2A7000	GA B2A7000	GA B2A7000	GA B2A7000
K O-RING	Q/R	CA137	CA141	CA137	CA137	CA145	CA141
	F/B	CF137	CF141	CF137	CF137	CF145	CF141
J SNAP RING	CD	EB007	EB015	EB007	EB007	EB020	EB015
	SS	EB507	EB515	EB507	EB507	EB520	EB515
R STATOR RING	CD/SS	EC022	EC022	EC028	EC028	EC028	EC034

*FRICTION DRIVE SEAL, TYPE 43 OR EQUAL

**POSITIVE DRIVE SEAL, TYPE 680 OR EQUAL

NOTE: CONTACT FACTORY FOR REPLACMENT MECHANICAL SEAL PART NUMBERS

PARTS LIST (Cont.)

REF. DESCRIPTION	TYPE	B2C PART NO.	B4C PART NO.	B1D PART NO.	B2D PART NO.	B4D PART NO.	B1E PART NO.
0100 DRIVE ADAPTOR	CD/SS	CC B2A0100	CC B1D0100	CC B1D0100	CC B1D0100	CC B1F0100	CC B1D0100
0900 PACKING GLAND	CD/SS	SC A2A0910	SC A1D0910	SC A1D0910	SC A1D0910	SC A1F0910	SC A1D0910
1000 STUFFING BOX	CD	CC A2A1010	CC A1D1010	CC A1D1010	CC A1D1010	CC A1F1010	CC A1D1010
	SS	SC A2A1010	SC A1D1010	SC A1D1010	SC A1D1010	SC A1F1010	SC A1D1010
1000 SEAL *	CD/SS	SC A2A1011	SC A1D1011	SC A1D1011	SC A1D1011	SC A1F1011	SC A1D1011
HOUSING **	CD/SS	4240833007	4240834007	4240834007	4240834007	4240835007	4240834007
1100 SUCTION CHAMBER	CD	CC A1C1110	CC A1E1110	CC A1D1110	CC A1D1110	CC A1F1110	CC A1E1110
	SS	SC A1C1110	SC A1E1000	SC A1D1110	SC A1D1110	SC A1F1110	SC A1E1110
1200 STATOR GASKET	Q/R	GG A1C1200	GG A1C1200	GG A1D1200	GG A1D1200	GG A1D1200	GG A1E1200
	F	GF A1C1200	GF A1C1200	GF A1D1200	GF A1D1200	GF A1D1200	GF A1E1200
	B	GB A1C1200	GB A1C1200	GB A1D1200	GB A1D1200	GB A1D1200	GB A1E1200
1210 STATOR ADAPTOR GASKET	Q/R		GG A1E1200			GG A1F1200	
	F		GF A1E1200			GF A1F1200	
	B		GB A1E1200			GB A1F1200	
1220 SUCTION CHAM. GASKET	Q/R	GG A2A1220	GG A1D1220	GG A1D1220	GG A1D1220	GG A1F1220	GG A1D1220
	F	GF A2A1220	GF A1D1220	GF A1D1220	GF A1D1220	GF A1F1220	GF A1D1220
	B	GB A2A1220	GB A1D1220	GB A1D1220	GB A1D1220	GB A1F1220	GB A1D1220
1240 INSPT. PLATE GSKT.	Q/R						GG A1E1240
	F						GF A1E1240
	B						GB A1E1240
1400 DISCHARGE FLANGE	CD	MS A1C1410	MS A4C1410	MS A1D1410	MS A1D1410	MS A1D1410	MS A1E1410
	SS	SS A1C1410	SS A4C1410	SS A1D1410	SS A1D1410	SS A1D1410	SS A1E1410
1500 STATOR ADAPTOR	CD		MS A4C1510			MS A4D1510	
	SS		SS A4C1510			SS A4D1510	
1600 LOCKING RING	CD/SS	MT B2A1600	MT B1D1600	MT B1D1600	MT B1D1600	MT B1F1600	MT B1D1600
1700 STATOR SUPPORT	CD/SS	MS A1C1710	MS A4C1710	MS A1D1710	CC A1D1710	CC A4D1710	CC A1E1710
1800 CLAMP RING	CD/SS	MS A1C1810	MS A4C1810	MS A1D1810	MS A1D1810	MS A4D1810	MS A1E1810
	CD/SS		MS A4C1811			MS A4D1811	
1900 INSPECTION PLATE	CD						MS A1E1900
	SS						SS A1E1900
2000 SHAFT PIN	CD/SS	4220487017	4220488017	4220488017	4220488017	4220489017	4220488017
5000 ROTOR	CD	TS A2C5000	TS A4C5000	TS A1D5000	TS A2D5000	TS A4D5000	TS A1E5000
	SS	SS A2C5000	SS A4C5000	SS A1D5000	SS A2D5000	SS A4D5000	SS A1E5000
6000 CC DRIVE SHAFT – KEY	CD	AS B1C6000	AS B1E6000	AS B1D6000	AS B1D6000	AS B1F6000	AS B1E6000
	SS	SS B1C6000	SS B1E6000	SS B1D6000	SS B1D6000	SS B1F6000	SS B1E6000
6000 CC DRIVE SHAFT – PIN	CD	4250779001	4250760001	4250780001	4250780001	4250745001	425076001
	SS	4250779015	4250760015	4250780015	4250780015	4250745015	4250760015
6100 RETAINING RING	CD/SS	ST A1C6100	ST A1E6100	ST A1D6100	ST A1D6100	ST A1F6100	ST A1E6100
6200 CONNECTING ROD	CD	AS A1C6200	AS A1E6200	AS A1D6200	AS A1D6200	AS A1F6200	AS A1E6200
	SS	AS A1C6200	SS A1E6200	AS A1D6200	AS A1D6200	SS A1F6200	SS A1E6200
6300 DRIVE PIN	CD/SS	TR A1C6300	TR A1E6300	TR A1D6300	TR A1D6300	TR A1F6300	TR A1E6300
6400 JOINT SEAL	Q/R	RD A1C6400	RD A1E6400	RD A1D6400	RD A1D6400	RD A1F6400	RD A1E6400
	F/B	RF A1C6400	RF A1E6400	RF A1D6400	RF A1D6400	RF A1F6400	RF A1E6400
6500 STATOR	Q	RQ A2C6510	RQ A4C6510	RQ A1D6510	RQ A2D6510	RQ A4D6510	RQ A1E6510
	R	RR A2C6510	RR A4C6510	RR A1D6510	RR A2D6510	RR A4D6510	RR A1E6510
	F	RF A2C6510	RF A4C6510	RF A1D6510	RF A2D6510	RF A4D6510	RF A1E6510
	B	RB A2C6510	RB A4C6510	RB A1D6510	RB A2D6510	RB A4D6510	RB A1E6510
6800 SLINGER RING - KEY	CD/SS	RZ A2A6800	RZ A1D6800	RZ A1D6800	RZ A1D6800	RZ A1F6800	RZ A1D6800
6800 SLINGER/PIN RETNR - 2 PC/CC	CD/SS	4230528000	4230529000	4230529000	4230529000	4230530000	4230529000
6850 LANTERN RING	CD/SS	GR A2A6850	GR A1D6850	GR A1D6850	GR A1D6850	GR A1F6850	GR A1D6850
6900 PACKING	STD.	PC A2A6901	PC A1D6901	PC A1D6901	PC A1D6901	PC A1F6901	PC A1F6901
	PTFE	3403655001	3403655002	3403655002	3403655002	3403655003	3403655002
7000 NAME PLATE	CD/SS	GA B2A7000	GA B2A7000	GA B2A7000	GA B2A7000	GA B2A7000	GA B2A7000
K O-RING	Q/R	CA141	CA175	CA145	CA145	CA188	CA175
	F/B	CF141	CF175	CF145	CF145	CF188	CF175
J SNAP RING	CD	EB015	EB027	EB020	EB020	EB036	EB027
	SS	EB515	EB527	EB520	EB520	EB536	EB527
R STATOR RING	CD/SS	EC034	EC034	EC039	EC039	EC039	EC046

*FRICTION DRIVE SEAL, TYPE 43 OR EQUAL

**POSITIVE DRIVE SEAL, TYPE 680 OR EQUAL

NOTE: CONTACT FACTORY FOR REPLACMENT MECHANICAL SEAL PART NUMBERS

PARTS LIST (Cont.)

REF. DESCRIPTION	TYPE	B2E PART NO.	B4E PART NO.	B1F/B1FE PART NO.	B2F/B2FE PART NO.	B4F PART NO.	B1G/B1GE PART NO.
0100 DRIVE ADAPTOR	CD/SS	CC B1D0100	CC B1F0100	CC B1F0100	CC B1F0100	CC B1H0100	CC B1F0100
0900 PACKING GLAND	CD/SS	SC A1D0910	SC A1F0910	SC A1F0910	SC A1F0910	SC A1H0910	SC A1F0910
1000 STUFFING BOX	CD	CC A1D1010	CC A1F1010	CC A1F1010	CC A1F1010	CC A1H1010	CC A1F1010
	SS	SC A1D1010	SC A1F1010	SC A1F1010	SC A1F1010	SC A1H1010	SC A1F1010
1000 SEAL *	CD/SS	SC A1D1011	SC A1F1011	SC A1F1011	SC A1F1011	SC A1H1011	SC A1F1011
HOUSING **	CD/SS	4240834007	4240835007	4240835007	4240835007	4240836007	4240835007
1100 SUCTION CHAMBER	CD	CC A1E1110	CC A1G1110	CC A1F1110	CC A1F1110	CC A1H1110	CC A1G1110
	SS	SC A1E1110	SC A1G1110	SC A1F1110	SC A1F1110	SC A1H1110	SC A1G1110
1200 STATOR GASKET	Q/R	GG A1E1200	GG A1E1200	GG A1F1200	GG A1F1200	GG A1F1200	GG A1G1200
	F	GF A1E1200	GF A1E1200	GF A1F1200	GF A1F1200	GF A1F1200	GF A1G1200
	B	GB A1E1200	GB A1E1200	GB A1F1200	GB A1F1200	GB A1F1200	GB A1G1200
1210 STATOR ADAPTOR GASKET	Q/R		GG A1G1200			GG A1H1200	
	F		GF A1G1200			GF A1H1200	
	B		GB A1G1200			GB A1H1200	
1220 SUCTION CHAM. GASKET	Q/R	GG A1D1220	GG A1F1220	GG A1F1220	GG A1F1220	GG A1H1220	GG A1F1220
	F	GF A1D1220	GF A1F1220	GF A1F1220	GF A1F1220	GF A1H1220	GF A1F1220
	B	GB A1D1220	GB A1F1220	GB A1F1220	GB A1F1220	GB A1H1220	GB A1F1220
1240 INSPT. PLATE GSKT.	Q/R	GG A1E1240	GG A1G1240	GG A1F1240	GG A1F1240	GG A1H1240	GG A1G1240
	F	GF A1E1240	GF A1G1240	GF A1F1240	GF A1F1240	GF A1H1240	GF A1G1240
	BA	GB A1E1240	GB A1G1240	GB A1F1240	GB A1F1240	GB A1H1240	GB A1G1240
1400 DISCHARGE FLANGE	CD	MS A1E1410	MS A4E1410	MS A1F1410	MS A1F1410	MS A4F1410	MS A1G1410
	SS	SS A1E1410	SS A4E1410	SS A1F1410	SS A1F1410	SS A4F1410	SS A1G1410
1500 STATOR ADAPTOR	CD		MS A4E1510			MS A4F1510	
	SS		SS A4E1510			SS A4F1510	
1600 LOCKING RING	CD/SS	MT B1D1600	MT B1F1600	MT B1F1600	MT B1F1600		MT B1F1600
1700 STATOR SUPPORT	CD/SS	CC A1E1710	CC A1E1710	CC A1F1710	CC A1F1710	CC A4F1710	CC A1G1710
1800 CLAMP RING	CD/SS	MS A1E1810	MS A4E1810	MS A1F1810	MS A1F1810	MS A4F1810	MS A1G1810
	CD/SS		MS A4E1811			MS A4F1811	
1900 INSPECTION PLATE	CD	MS A1E1900	MS A1G1900	MS A1F1900	MS A1F1900	MS A1H1900	MS A1G1900
	SS	SS A1E1900	SS A1G1900	SS A1F1900	SS A1F1900	SS A1H1900	SS A1G1900
2000 SHAFT PIN	CD/SS	4220488017	4220489017	4220489017	4220489017	4220492017	4220489017
5000 ROTOR	CD	TS A2E5000	TS A4E5000	TS A1F5000	TS A2F5000	TS A4F5000	TS A1G5000
	CD			TS A1FE5000	TS A2FE5000		TS A1GE5000
	SS	SS A2E5000	SS A4E5000	SS A1F5000	SS A2F5000	SS A4F5000	SS A1G5000
	SS			SS A1FE5000	SS A2FE5000		SS A1GE5000
6000 DRIVE SHAFT - KEY	CD	AS B1E6000	AS B1G6000	AS B1F6000	AS B1F6000		AS B1G6000
	SS	SS B1E6000	SS B1G6000	SS B1F6000	SS B1F6000		SS B1G6000
6000 CC DRIVE SHAFT – PIN	CD	4250760001	4250744001	4250745001	4250745001	AS B1H6000	4250744001
	SS	4250760015	4250744015	4250745015	4250745015	SS B1H6000	4250744015
6100 RETAINING RING	CD/SS	ST A1E6100	ST A1G6100	ST A1F6100	ST A1F6100	ST A1H6100	ST A1G6100
6200 CONNECTING ROD	CD	AS A1E6200	AS A1G6200	AS A1F6200	AS A1F6200	AS A1H6200	AS A1G6200
	SS	SS A1E6200	SS A1G6200	SS A1F6200	SS A1F6200	SS A1H6200	SS A1G6200
6300 DRIVE PIN	CD/SS	TR A1E6300	TR A1G6300	TR A1F6300	TR A1F6300	TR A1H6300	TR A1G6300
6400 JOINT SEAL	Q/R	RD A1E6400	RD A1G6400	RD A1F6400	RD A1F6400	RD A1H6400	RD A1G6400
	F/B	RF A1E6400	RF A1G6400	RF A1F6400	RF A1F6400	RF A1H6400	RF A1G6400
6500 STATOR	Q	RQ A2E6510	RQ A4E6510	RQ A1F6510	RQ A2F6510	RQ A4F6510	RQ A1G6510
	Q			RQ A1FE6510	RQ A2FE6510		RQ A1GE6510
	R	RR A2E6510	RR A4E6510	RR A1F6510	RR A2F6510	RR A4F6510	RR A1G6510
	R			RR A1FE6510	RR A2FE6510		RR A1GE6510
	F	RF A2E6510	RF A4E6510	RF A1F6510	RF A2F6510	RF A4F6510	RF A1G6510
	B	RB A2E6510	RB A4E6510	RB A1F6510	RB A2F6510	RB A4F6510	RB A1G6510
	B			RB A1FE6510	RB A2FE6510		RB A1GE6510
6800 SLINGER RING - KEY	CD/SS	RZ A1D6800	RZ A1F6800	RZ A1F6800	RZ A1F6800		RZ A1F6800
6800 SLINGER/PIN RETNR - 2 PC/CC	CD/SS	4230529000	4230530000	4230530000	4230530000	4230531000	4230530000
6850 LANTERN RING	CD/SS	GR A1D6850	GR A1F6850	GR A1F6850	GR A1F6850	GR A1H6850	GR A1F6850
6900 PACKING	STD.	PC A1D6901	PC A1F6901	PC A1F6901	PC A1F6901	PC A1H6901	PC A1F6901
	PTFE	3403655002	3403655003	3403655003	3403655003	3403655004	3403655003
7000 NAME PLATE	CD/SS	GA B2A7000	GA B2A7000	GA B2A7000	GA B2A7000	GA B2A7000	GA B2A7000
K O-RING	Q/R	CA175	CA197	CA188	CA188	CA228	CA197
	F/B	CF175	CF197	CF188	CF188	CF228	CF197
J SNAP RING	CD	EB027	EB045	EB036	EB036	EB049	EB045
	SS	EB527	EB545	EB536	EB536	EB549	EB545
R STATOR RING	CD/SS	EC046	EC046	EC055	EC055	EC065	EC060

*FRICTION DRIVE SEAL, TYPE 43 OR EQUAL

**POSITIVE DRIVE SEAL, TYPE 680 OR EQUAL

NOTE: CONTACT FACTORY FOR REPLACMENT MECHANICAL SEAL PART NUMBERS

PARTS LIST (Cont.)

REF. DESCRIPTION	TYPE	B2G/B2GE PART NO.	B4G PART NO.	B1H/B1HE PART NO.	B2H/B2HE PART NO.	B1J/B1JE PART NO.	B2J/B2JE PART NO.
0100 DRIVE ADAPTOR	CD/SS	CC B1F0100	CC B1H0100	CC B1H0100	CC B1H0100	CC B1H0100	CC B1H0100
0900 PACKING GLAND	CD/SS	SC A1F0910	SC A1H0910	SC A1H0910	SC A1H0910	SC A1H0910	SC A1H0910
1000 STUFFING BOX	CD	CC A1F1010	CC A1H1010	CC A1H1010	CC A1H1010	CC A1H1010	CC A1H1010
	SS	SC A1F1010	SC A1H1010	SC A1H1010	SC A1H1010	SC A1H1010	SC A1H1010
1000 SEAL *	CD/SS	SC A1F1011	SC A1H1011	SC A1H1011	SC A1H1011	SC A1H1011	SC A1F1011
HOUSING **	CD/SS	4240835007	4240836007	4240836007	4240836007	4240836007	4240835007
1100 SUCTION CHAMBER	CD	CC A1G1110	CC A1H1110	CC A1H1110	CC A1H1110	CC A1H1110	CC A1H1110
	SS	SC A1G1110	SC A1H1110	SC A1H1110	SC A1H1110	SC A1H1110	SC A1H1110
1200 STATOR GASKET	Q/R	GG A1G1200	GG A1G1200	GG A1H1200	GG A1H1200	GG A1H1200	GG A1H1200
	F	GF A1G1200	GF A1G1200	GF A1H1200	GF A1H1200	GF A1H1200	GF A1H1200
	B	GB A1G1200	GB A1G1200	GB A1H1200	GB A1H1200	GB A1H1200	GB A1H1200
1210 STATOR ADAPTOR GASKET	Q/R		GG A1H1200				
	F		GF A1H1200				
	B		GB A1H1200				
1220 SUCTION CHAM. GASKET	Q/R	GG A1F1220	GG A1H1220	GG A1H1220	GG A1H1220	GG A1H1220	GG A1H1220
	F	GF A1F1220	GF A1H1220	GF A1H1220	GF A1H1220	GF A1H1220	GF A1H1220
	B	GB A1F1220	GB A1H1220	GB A1H1220	GB A1H1220	GB A1H1220	GB A1H1220
1240 INSPT. PLATE GSKT.	Q/R	GG A1G1240	GG A1H1240	GG A1H1240	GG A1H1240	GG A1H1240	GG A1H1240
	F	GF A1G1240	GF A1H1240	GF A1H1240	GF A1H1240	GF A1H1240	GF A1H1240
	BA	GB A1G1240	GB A1H1240	GB A1H1240	GB A1H1240	GB A1H1240	GB A1H1240
1400 DISCHARGE FLANGE	CD	MS A1G1410	MS A4G1410	MS A1H1410	MS A1H1410	MS A1H1410	MS A1H1410
	SS	SS A1G1410	SS A4G1410	SS A1H1410	SS A1H1410	SS A1H1410	SS A1H1410
1500 STATOR ADAPTOR	CD		MS A4G1510				
	SS		SS A4G1510				
1600 LOCKING RING	CD/SS	MT B1F1600					
1700 STATOR SUPPORT	CD/SS	CC A1G1710	CC A4G1710	CC A1H1710	CC A1H1710	CC A1H1710	CC A1H1710
1800 CLAMP RING	CD/SS	MS A1G1810	MS A4G1810	MS A1H1810	MS A1H1810	MS A1H1810	MS A1H1810
	CD/SS		MS A4G1811				
1900 INSPECTION PLATE	CD	MS A1G1900	MS A1H1900	MS A1H1900	MS A1H1900	MS A1H1900	MS A1H1900
	SS	SS A1G1900	SS A1H1900	SS A1H1900	SS A1H1900	SS A1H1900	SS A1H1900
2000 SHAFT PIN	CD/SS	4220489017	4220492017	4220492017	4220492017	4220492017	4220492017
5000 ROTOR	CD	TS A2G5000	TS A4G5000	TS A1H5000	TS A2H5000	TS A1J5000	TS A2J5000
	CD	TS A2GE5000		TS A1H5000	TS A2HE5000	TS A1JE5000	TS A2JE5000
	SS	SS A2G5000	SS A4G5000	SS A1H5000	SS A2H5000	SS A1J5000	SS A2J5000
	SS	SS A2GE5000		SS A1HE5000	SS A2H5000	SS A1JE5000	SS A2JE5000
6000 DRIVE SHAFT - KEY	CD	AS B1G6000					
	SS	SS B1G6000					
6000 CC DRIVE SHAFT – PIN	CD	4250744001	AS B1H6000	AS B1H6000	AS B1H6000	AS B1H6000	AS B1H6000
	SS	4250744015	SS B1H6000	SS B1H6000	SS B1H6000	SS B1H6000	SS B1H6000
6100 RETAINING RING	CD/SS	ST A1G6100	ST A1H6100	ST A1H6100	ST A1H6100	ST A1H6100	ST A1H6100
6200 CONNECTING ROD	CD	AS A1G6200	AS A1H6200	AS A1H6200	AS A1H6200	AS A1H6200	AS A1H6200
	SS	SS A1G6200	SS A1H6200	SS A1H6200	SS A1H6200	SS A1H6200	SS A1H6200
6300 DRIVE PIN	CD/SS	TR A1G6300	TR A1H6300	TR A1H6300	TR A1H6300	TR A1H6300	TR A1H6300
6400 JOINT SEAL	Q/R	RD A1G6400	RD A1H6400	RD A1H6400	RD A1H6400	RD A1H6400	RD A1H6400
	F/B	RF A1G6400	RF A1H6400	RF A1H6400	RF A1H6400	RF A1H6400	RF A1H6400
6500 STATOR	Q	RQ A2G6510	RQ A4G6510	RQ A1H6510	RQ A2H6510	RQ A1J6510	RQ A2J6510
	Q	RQ A2GE6510		RQ A1HE6510	RQ A2HE6510	RQ A1JE6510	RQ A2JE6510
	R	RR A2G6510	RR A4G6510	RR A1H6510	RR A2H6510	RR A1J6510	RR A2J6510
	R	RR A2GE6510		RR A1HE6510	RR A2HE6510	RR A1JE6510	RR A2JE6510
	F	RF A2G6510	RF A4G6510	RF A1H6510	RF A2H6510	RF A1J6510	RF A2J6510
	B	RB A2G6510	RB A4G6510	RB A1H6510	RB A2H6510	RB A1J6510	RB A2J6510
	B	RB A2GE6510		RB A1HE6510	RB A2HE6510	RB A1JE6510	RB A2JE6510
6800 SLINGER RING - KEY	CD/SS	RZ A1F6800					
6800 SLINGER/PIN RETNR - 2 PC/CC	CD/SS	4230530000	4230531000	4230531000	4230531000	4230531000	4230531000
6850 LANTERN RING	CD/SS	GR A1F6850	GR A1H6850	GR A1H6850	GR A1H6850	GR A1H6850	GR A1H6850
6900 PACKING	STD.	PC A1F6901	PC A1H6901	PC A1H6901	PC A1H6901	PC A1H6901	PC A1H6901
	PTFE	3403655003	3403655004	3403655004	3403655004	3403655004	3403655004
7000 NAME PLATE	CD/SS	GA B2A7000	GA B2A7000	GA B2A7000	GA B2A7000	GA B2A7000	GA B2A7000
K O-RING	Q/R	CA197	CA228	CA228	CA228	CA228	CA228
	F/B	CF197	CF228	CF228	CF228	CF228	CF228
J SNAP RING	CD	EB045	EB049	EB049	EB049	EB049	EB049
	SS	EB545	EB549	EB549	EB549	EB549	EB549
R STATOR RING	CD/SS	EC060	EC065	EC065	EC065	EC065	EC065

*FRICTION DRIVE SEAL, TYPE 43 OR EQUAL

**POSITIVE DRIVE SEAL, TYPE 680 OR EQUAL

NOTE: CONTACT FACTORY FOR REPLACMENT MECHANICAL SEAL PART NUMBERS

REF. DESCRIPTION		TYPE	B1K/B1KE PART NO.	B2K/B2KE PART NO.	B1L PART NO.
0100 DRIVE ADAPTOR		CD/SS	CC B1H0100	CC B1H0100	CC B1H0100
0900 PACKING GLAND		CD/SS	SC A1H0910	SC A1H0910	SC A1H0910
1000 STUFFING BOX		CD	CC A1H1010	CC A1H1010	CC A1H1010
		SS	SC A1H1010	SC A1H1010	SC A1H1010
1000 SEAL	*	CD/SS	SC A1H1011	SC A1H1011	SC A1H1011
HOUSING	**	CD/SS	4240836007	4240836007	4240836007
1100 SUCTION		CD	CC A1H1110	CC A1H1110	CC A1H1110
CHAMBER		SS	SC A1H1110	SC A1H1110	SC A1H1110
1200 STATOR GASKET		Q/R	GG A1H1200	GG A1H1200	GG A1H1200
		F	GF A1H1200	GF A1H1200	GF A1H1200
		B	GB A1H1200	GB A1H1200	GB A1H1200
1210 STATOR		Q/R			
ADAPTOR GASKET		F			
		B			
1220 SUCTION		Q/R	GG A1H1220	GG A1H1220	GG A1H1220
CHAM. GASKET		F	GF A1H1220	GF A1H1220	GF A1H1220
		B	GB A1H1220	GB A1H1220	GB A1H1220
1240 INSPT. PLATE GSKT.		Q/R	GG A1H1240	GG A1H1240	GG A1H1240
		F	GF A1H1240	GF A1H1240	GF A1H1240
		BA	GB A1H1240	GB A1H1240	GB A1H1240
1400 DISCHARGE		CD	MS A1H1410	MS A1H1410	MS A1H1410
FLANGE		SS	SS A1H1410	SS A1H1410	SS A1H1410
1500 STATOR ADAPTOR		CD			
		SS			
1600 LOCKING RING		CD/SS			
1700 STATOR SUPPORT		CD/SS	CC A1H1710	CC A1H1710	CC A1H1710
1800 CLAMP RING		CD/SS	MS A1H1810	MS A1H1810	MS A1H1810
		CD/SS			
1900 INSPECTION PLATE		CD	MS A1H1900	MS A1H1900	MS A1H1900
		SS	SS A1H1900	SS A1H1900	SS A1H1900
2000 SHAFT PIN		CD/SS	4220492017	4220492017	4220492017
5000 ROTOR		CD	TS A1K5000	TS A2K5000	TS A1L5000
		CD	TS A1K5000	TS A2KE5000	TS A1LE5000
		SS	SS A1K5000	SS A2K5000	SS A1L5000
		SS	SS A1KE5000	SS A2K5000	SS A1LE5000
6000 DRIVE SHAFT - KEY		CD			
		SS			
6000 CC DRIVE SHAFT –		CD	AS B1H6000	AS B1H6000	AS B1H6000
PIN		SS	SS B1H6000	SS B1H6000	SS B1H6000
6100 RETAINING RING		CD/SS	ST A1H6100	ST A1H6100	ST A1H6100
6200 CONNECTING ROD		CD	AS A1H6200	AS A1H6200	AS A1H6200
		SS	SS A1H6200	SS A1H6200	SS A1H6200
6300 DRIVE PIN		CD/SS	TR A1H6300	TR A1H6300	TR A1H6300
6400 JOINT SEAL		Q/R	RD A1H6400	RD A1H6400	RD A1H6400
		F/B	RF A1H6400	RF A1H6400	RF A1H6400
6500 STATOR		Q	RQ A1K6510	RQ A2K6510	RQ A1L6510
		Q	RQ A1KE6510	RQ A2KE6510	
		R	RR A1K6510	RR A2K6510	RR A1L6510
		R	RR A1KE6510	RR A2KE6510	
		F	RF A1K6510	RF A2K6510	RF A1L6510
		B	RB A1K6510	RB A2K6510	RB A1L6510
		B	RB A1KE6510	RB A2KE6510	
6800 SLINGER RING - KEY		CD/SS			
6800 SLINGER/PIN RETNR		CD/SS	4230531000	4230531000	4230531000
- 2 PC/CC					
6850 LANTERN RING		CD/SS	GR A1H6850	GR A1H6850	GR A1H6850
6900 PACKING		STD.	PC A1H6901	PC A1H6901	PC A1H6901
		PTFE	3403655004	3403655004	3403655004
7000 NAME PLATE		CD/SS	GA B2A7000	GA B2A7000	GA B2A7000
K O-RING		Q/R	CA228	CA228	CA228
		F/B	CF228	CF228	CF228
J SNAP RING		CD	EB049	EB049	EB049
		SS	EB549	EB549	EB549
R STATOR RING		CD/SS	EC065	EC065	EC065

*FRICTION DRIVE SEAL, TYPE 43 OR EQUAL

**POSITIVE DRIVE SEAL, TYPE 680 OR EQUAL

NOTE: CONTACT FACTORY FOR REPLACMENT MECHANICAL SEAL PART NUMBERS

PARTS LIST (Cont.)

REF. DESCRIPTION	TYPE	C1E PART NO.	C2E PART NO.	C1F/C1FE PART NO.	C2F/C2FE PART NO.	C1G/C1GE PART NO.
0100 BEARING HOUSING	CD/SS	CC A1D0110	CC A1D0110	CC A1F0110	CC A1F0110	CC A1F0110
0300 BEARING COVER	CD/SS	CC A1D0300	CC A1D0300	CC A1F0300	CC A1F0300	CC A1F0300
0500 GREASE RETAINER	CD/SS	MA A1D0500	MS A1D0500	MS A1F0500	MS A1F0500	MS A1F0500
0700 GREASE SEAL HSG.	CD/SS	MS A1D0700	MS A1D0700	MS A1F0700	MS A1F0700	MS A1F0700
0900 PACKING GLAND	CD/SS	SC A1D0910	SC A1D0910	SC A1F0910	SC A1F0910	SC A1F0910
1000 STUFFING BOX	CD	CC A1D1010	CC A1D1010	CC A1F1010	CC A1F1010	CC A1F1010
	SS	SC A1D1010	SC A1D1010	SC A1F1010	SC A1F1010	SC A1F1010
1000 SEAL *	CD/SS	SC A1D1011	SC A1D1011	SC A1F1011	SC A1F1011	SC A1F1011
HOUSING **	CD/SS	4240834007	4240834007	4240835007	4240835007	4240835007
1100 SUCTION CHAMBER	CD	CC C1E1110	CC C1E1110	CC C1F1110	CC C1F1110	CC C1G1110
	SS	SC C1E1110	SC C1E1110	SC C1F1110	SC C1F1110	SC C1G1110
1200 STATOR GASKET	Q/R	GG A1E1200	GG A1E1200	GG A1F1200	GG A1F1200	GG A1G1200
	F	GF A1E1200	GF A1E1200	GF A1F1200	GF A1F1200	GF A1G1200
	B	GB A1E1200	GB A1E1200	GB A1F1200	GB A1F1200	GB A1G1200
1220 SUCTION CHAM. GASKET	Q/R	GG A1D1220	GG A1D1220	GG A1F1220	GG A1F1220	GG A1F1220
	F	GF A1D1220	GF A1D1220	GF A1F1220	GF A1F1220	GF A1F1220
	B	GB A1D1220	GB A1D1220	GB A1F1220	GB A1F1220	GB A1F1220
1400 DISCHARGE FLANGE	CD	MS A1E1410	MS A1E1410	MS A1F1410	MS A1F1410	MS A1G1410
	SS	SS A1E1410	SS A1E1410	SS A1F1410	SS A1F1410	SS A1G1410
1700 STATOR SUPPORT	CD/SS	CC A1E1710	CC A1E1710	CC A1F1710	CC A1F1710	CC A1G1710
1800 CLAMP RING	CD/SS	MS A1E1810	MS A1E1810	MS A1F1810	MS A1F1810	MS A1G1810
2000 SHAFT PIN	CD/SS	4220488017	4220488017	4220489017	4220489017	4220489017
2100 INTERMEDIATE SHAFT	CD	4250395001	4250395001	4250396001	4250396001	4250397001
	SS	4250395017	4250395017	4250396017	4250396017	4250397017
2200 SEAL O-RING	Q/R	3207902210	3207902210	3207902216	3207902216	3207902216
	F	3207905210	3207905210	3207905216	3207905216	3207905216
	B	3207904210	3207904210	3207904216	3207904216	3207904216
5000 ROTOR	CD	TS A1E5000	TS A2E5000	TS A1F5000	TS A2F5000	TS A1G5000
	CD			TS A1FE5000	TS A2FE5000	TS A1GE5000
	SS	SS A1E5000	SS A2E5000	SS A1F5000	SS A2F5000	SS A1G5000
	SS			SS A1FE5000	SS A2FE5000	SS A1GE5000
6000 DRIVE SHAFT	CD	AS A1E6000	AS A1E6000	AS A1F6000	AS A1F6000	AS A1G6000
	SS	SS A1E6000	SS A1E6000	SS A1F6000	SS A1F6000	SS A1G6000
6000 DRIVE SHAFT (2-PIECE OPTION)	CD	4250379001	4250379001	4250380001	4250380001	4250380001
	SS	4250379015	4250379015	4250380015	4250380015	4250380015
6100 RETAINING RING	CD/SS	ST A1E6100	ST A1E6100	ST A1F6100	ST A1F6100	ST A1G6100
6200 CONNECTING ROD	CD	AS C1E6200	AS C1E6200	AS C1F6200	AS C1F6200	AS C1G6200
	SS	SS C1E6200	SS C1E6200	SS C1F6200	SS C1F6200	SS C1G6200
6300 DRIVE PIN	CD/SS	TR A1E6300	TR A1E6300	TR A1F6300	TR A1F6300	TR A1G6300
6400 JOINT SEAL	Q/R	RD A1E6400	RD A1E6400	RD A1F6400	RD A1F6400	RD A1G6400
	F/B	RF A1E6400	RF A1E6400	RF A1F6400	RF A1F6400	RF A1G6400
6500 STATOR	Q	RQ A1E6510	RQ A2E6510	RQ A1F6510	RQ A2F6510	RQ A1G6510
	Q			RQ A1FE6510	RQ A2FE6510	RQ A1GE6510
	R	RR A1E6510	RR A2E6510	RR A1F6510	RR A2F6510	RR A1G6510
	R			RR A1FE6510	RR A2FE6510	RR A1GE6510
	F	RF A1E6510	RF A2E6510	RF A1F6510	RF A2F6510	RF A1G6510
	B	RB A1E6510	RB A2E6510	RB A1F6510	RB A2F6510	RB A1G6510
	B			RB A1FE6510	RB A2FE6510	RB A1GE6510
6700 BEARING SHIMS	CD/SS	GP A1D6700	GP A1D6700	GP A1F6700	GP A1F6700	GP A1F6700
6800 SLINGER RING	CD/SS	RZ A1D6800	RZ A1D6800	RZ A1F6800	RZ A1F6800	RZ A1F6800
6800 SLINGER/PIN RETNR. (2-PIECE OPTION)	CD/SS	4230529000	4230530000	4230530000	4230530000	4230530000
6850 LANTERN RING	CD/SS	GR A1D6850	GR A1D6850	GR A1F6850	GR A1F6850	GR A1F6850
6900 PACKING	STD.	PC A1D6901	PC A1D6901	PC A1F6901	PC A1F6901	PC A1F6901
	PTFE	3403655002	3403655002	3403655003	3403655003	3403655003
7000 NAME PLATE	CD/SS	GA A2A7000	GA A2A7000	GA A1F7000	GA A1F7000	GA A1F7000
K O-RING	Q/R	CA175	CA175	CA188	CA188	CA197
	F/B	CF175	CF175	CF188	CF188	CF197
J SNAP RING	CD	EB027	EB027	EB036	EB036	EB045
	SS	EB527	EB527	EB536	EB536	EB545
R STATOR RING	CD/SS	EC046	EC046	EC055	EC055	EC060

*FRICTION DRIVE SEAL, TYPE 43 OR EQUAL

**POSITIVE DRIVE SEAL, TYPE 680 OR EQUAL

NOTE: CONTACT FACTORY FOR REPLACMENT MECHANICAL SEAL PART NUMBERS

PARTS LIST (Cont.)

REF. DESCRIPTION	TYPE	C2G/C2GE PART NO.	C1H/C1HE PART NO.	C2H/C2HE PART NO.	C1J/C1JE PART NO.	C2J/C2JE PART NO.	C1K/C1KE PART NO.
0100 BEARING HOUSING	CD/SS	CC A1F0110	CC A1H0110	CC A1H0110	CC A1H0110	CC A1H0110	CC A1H0110
0300 BEARING COVER	CD/SS	CC A1F0300	CC A1H0300	CC A1H0300	CC A1H0300	CC A1H0300	CC A1H0300
0500 GREASE RETAINER	CD/SS	MS A1F0500	MS A1H0500	MS A1H0500	MS A1H0500	MS A1H0500	MS A1H0500
0700 GREASE SEAL HSG.	CD/SS	MS A1F0700	MS A1H0700	MS A1H0700	MS A1H0700	MS A1H0700	MS A1H0700
0900 PACKING GLAND	CD/SS	SC A1F0910	SC A1H0910	SC A1H0910	SC A1H0910	SC A1H0910	SC A1H0910
1000 STUFFING BOX	CD	CC A1F1010	CC A1H1010	CC A1H1010	CC A1H1010	CC A1H1010	CC A1H1010
	SS	SC A1F1010	SC A1H1010	SC A1H1010	SC A1H1010	SC A1H1010	SC A1H1010
1000 SEAL *	CD/SS	SC A1F1011	SC A1H1011	SC A1H1011	SC A1H1011	SC A1H1011	SC A1H1011
HOUSING **	CD/SS	4240835007	4240836007	4240836007	4240836007	4240836007	4240836007
1100 SUCTION CHAMBER	CD	CC C1G1110	CC C1H1110	CC C1H1110	CC C1H1110	CC C1H1110	CC C1H1110
	SS	SC C1G1110	SC C1H1110	SC C1H1110	SC C1H1110	SC C1H1110	SC C1H1110
1200 STATOR GASKET	Q/R	GG A1G1200	GG A1H1200	GG A1H1200	GG A1H1200	GG A1H1200	GG A1H1200
	F	GF A1G1200	GF A1H1200	GF A1H1200	GF A1H1200	GF A1H1200	GF A1H1200
	B	GB A1G1200	GB A1H1200	GB A1H1200	GB A1H1200	GB A1H1200	GB A1H1200
1220 SUCTION CHAM. GASKET	Q/R	GG A1F1220	GG A1H1220	GG A1H1220	GG A1H1220	GG A1H1220	GG A1H1220
	F	GF A1F1220	GF A1H1220	GF A1H1220	GF A1H1220	GF A1H1220	GF A1H1220
	B	GB A1F1220	GB A1H1220	GB A1H1220	GB A1H1220	GB A1H1220	GB A1H1220
1400 DISCHARGE FLANGE	CD	MS A1G1410	MS A1H1410	MS A1H1410	MS A1H1410	MS A1H1410	MS A1H1410
	SS	SS A1G1410	SS A1H1410	SS A1H1410	SS A1H1410	SS A1H1410	SS A1H1410
1700 STATOR SUPPORT	CD/SS	CC A1G1710	CC A1H1710	CC A1H1710	CC A1H1710	CC A1H1710	CC A1H1710
1800 CLAMP RING	CD/SS	MS A1G1810	MS A1H1810	MS A1H1810	MS A1H1810	MS A1H1810	MS A1H1810
2000 SHAFT PIN	CD/SS	4220489017	4220492017	4220492017	4220492017	4220492017	4220492017
2100 INTERMEDIATE SHAFT	CD	4250397001	4250398001	4250398001	4250398001	4250398001	4250398001
	SS	4250397017	4250398017	4250398017	4250398017	4250398017	4250398017
2200 SEAL RING	Q/R	3207902216	3207902223	3207902223	3207902223	3207902223	3207902223
	F	3207905216	3207905223	3207905223	3207905223	3207905223	3207905223
	B	3207904216	3207904223	3207904223	3207904223	3207904223	3207904223
5000 ROTOR	CD	TS A2G5000	TS A1H5000	TS A2H5000	TS A1J5000	TS A2J5000	TS A1K5000
	CD	TS A2GE5000	TS A1HE5000	TS A2HE5000	TS A1JE5000	TS A2JE5000	TS A1KE5000
	SS	SS A2G5000	SS A1H5000	SS A2H5000	SS A1J5000	SS A2J5000	SS A1K5000
	SS	SS A2GE5000	SS A1HE5000	SS A2HE5000	SS A1JE5000	SS A2JE5000	SS A1KE5000
6000 DRIVE SHAFT	CD	AS A1G6000	AS A1H6000	AS A1H6000	AS A1H6000	AS A1H6000	AS A1H6000
	SS	SS A1G6000	SS A1H6000	SS A1H6000	SS A1H6000	SS A1H6000	SS A1H6000
6000 DRIVE SHAFT (2-PIECE OPTION)	CD	4250380001	4250381001	4250381001	4250381001	4250381001	4250381001
	SS	4250380015	4250381015	4250381015	4250381015	4250381015	4250381015
6100 RETAINING RING	CD/SS	ST A1G6100	ST A1H6100	ST A1H6100	ST A1H6100	ST A1H6100	ST A1H6100
6200 CONNECTING ROD	CD	AS C1G6200	AS C1H6200	AS C1H6200	AS C1H6200	AS C1H6200	AS C1H6200
	SS	SS C1G6200	SS C1H6200	SS C1H6200	SS C1H6200	SS C1H6200	SS C1H6200
6300 DRIVE PIN	CD/SS	TR A1G6300	TR A1H6300	TR A1H6300	TR A1H6300	TR A1H6300	TR A1H6300
6400 JOINT SEAL	Q/R	RD A1G6400	RD A1H6400	RD A1H6400	RD A1H6400	RD A1H6400	RD A1H6400
	F/B	RF A1G6400	RF A1H6400	RF A1H6400	RF A1H6400	RF A1H6400	RF A1H6400
6500 STATOR	Q	RQ A2G6510	RQ A1H6510	RQ A2H6510	RQ A1J6510	RQ A2J6510	RQ A1K6510
	Q	RQ A2GE6510	RQ A1HE6510	RQ A2HE6510	RQ A1JE6510	RQ A2JE6510	RQ A1KE6510
	R	RR A2G6510	RR A1H6510	RR A2H6510	RR A1J6510	RR A2J6510	RR A1K6510
	R	RR A2GE6510	RR A1HE6510	RR A2HE6510	RR A1JE6510	RR A2JE6510	RR A1KE6510
	F	RF A2G6510	RF A1H6510	RF A2H6510	RF A1J6510	RF A2J6510	RF A1K6510
	B	RB A2G6510	RB A1H6510	RB A2H6510	RB A1J6510	RB A2J6510	RB A1K6510
	B	RB A2GE6510	RB A1HE6510	RB A2HE6510	RB A1JE6510	RB A2JE6510	RB A1KE6510
6700 BEARING SHIMS	CD/SS	GP A1F6700	GP A1H6700	GP A1H6700	GP A1H6700	GP A1H6700	GP A1H6700
6800 SLINGER RING	CD/SS	RZ A1F6800	RZ A1H6800	RZ A1H6800	RZ A1H6800	RZ A1H6800	RZ A1H6800
6800 SLINGER/PIN RETNR (2-PIECE OPTION)	CD/SS	4230530000	4230531000	4230531000	4230531000	4230531000	4230531000
6850 LANTERN RING	CD/SS	GR A1F6850	GR A1H6850	GR A1H6850	GR A1H6850	GR A1H6850	GR A1H6850
6900 PACKING	STD.	PC A1F6901	PC A1H6901	PC A1H6901	PC A1H6901	PC A1H6901	PC A1H6901
	PTFE	3403655003	3403655004	3403655004	3403655004	3403655004	3403655004
7000 NAME PLATE	CD/SS	GA A1F7000	GA A1F7000	GA A1F7000	GA A1F7000	GA A1F7000	GA A1F7000
K O-RING	Q/R	CA197	CA228	CA228	CA228	CA228	CA228
	F/B	CF197	CF228	CF228	CF228	CF228	CF228
J SNAP RING	CD	EB045	EB049	EB049	EB049	EB049	EB049
	SS	EB545	EB549	EB549	EB549	EB549	EB549
R STATOR RING	CD/SS	EC060	EC065	EC065	EC065	EC065	EC065

*FRICTION DRIVE SEAL, TYPE 43 OR EQUAL

**POSITIVE DRIVE SEAL, TYPE 680 OR EQUAL

NOTE: CONTACT FACTORY FOR REPLACMENT MECHANICAL SEAL PART NUMBERS

PARTS LIST (Cont.)

REF. DESCRIPTION	TYPE	D4B PART NO.	D4C PART NO.	D4D PART NO.	D4E PART NO.	D4F PART NO.	D4G PART NO.	D4H PART NO.
0100 BEARING HOUSING	CD/SS	CC A2A0110	CC A2A0110	CC A1D0110	CC A1D0110	CC A1F0110	CC A1F0110	CC A1H0110
0300 BEARING COVER	CD/SS			CC A1D0300	CC A1D0300	CC A1F0300	CC A1F0300	CC A1H0300
0500 GREASE RETAINER	CD/SS			MS A1D0500	MS A1D0500	MS A1F0500	MS A1F0500	MS A1H0500
0700 GREASE SEAL HSG.	CD/SS			MS A1D0700	MS A1D0700	MS A1F0700	MS A1F0700	MS A1H0700
0900 PACKING GLAND	CD/SS	SC A2A0910	SC A2A0910	SC A1D0910	SC A1D0910	SC A1F0910	SC A1F0910	SC A1H0910
1000 STUFFING BOX	CD	CC A2A1010	CC A1A1010	CC A1D1010	CC A1D1010	CC A1F1010	CC A1F1010	CC A1H1010
	SS	SC A2A1010	SC A1A1010	SC A1D1010	SC A1D1010	SC A1F1010	SC A1F1010	SC A1H1010
1000 SEAL *	CD/SS	SC A2A1011	SC A2A1011	SC A1D1011	SC A1D1011	SC A1F1011	SC A1F1011	SC A1H1011
HOUSING **	CD/SS	4240833007	4240833007	4240834007	4240834007	4240835007	4240835007	4240836007
1100 SUCTION	CD	CC A1B1110	CC A1C1110	CC A1D1110	CC A1E1110	CC A1F1110	CC A1G1110	CC A1H1110
CHAMBER	SS	SC A1B1110	SC A1C1110	SC A1D1110	SC A1E1110	SC A1F1110	SC A1G1110	SC A1H1110
1200 STATOR GASKET	Q/R	GG A1B1200	GG A1C1200	GG A1D1200	GG A1E1200	GG A1F1200	GG A1G1200	GG A1H1200
	F	GF A1B1200	GF A1C1200	GF A1D1200	GF A1E1200	GF A1F1200	GF A1G1200	GF A1H1200
	B	GB A1B1200	GB A1C1200	GB A1D1200	GB A1E1200	GB A1F1200	GB A1G1200	GB A1H1200
1220 SUCTION	Q/R	GG A2A1220	GG A2A1220	GG A1D1220	GG A1D1220	GG A1F1220	GG A1F1220	GG A1H1220
CHAM. GASKET	F	GF A2A1220	GF A2A1220	GF A1D1220	GF A1D1220	GF A1F1220	GF A1F1220	GF A1H1220
	B	GB A2A1220	GB A2A1220	GB A1D1220	GB A1D1220	GB A1F1220	GB A1F1220	GB A1H1220
1240 INSP. PLATE GSKT.	Q/R				GG A1E1240	GG A1F1240	GG A1G1240	GG A1H1240
	F				GF A1E1240	GF A1F1240	GF A1G1240	GF A1H1240
	B				GB A1E1240	GB A1F1240	GB A1G1240	GB A1H1240
1400 DISCHARGE	CD	MS A1B1410	MS A1C1410	MS A1D1410	MS A1E1410	MS A1F1410	MS A1G1410	MS A1H1410
FLANGE	SS	SS A1B1410	SS A1C1410	SS A1D1410	SS A1E1410	SS A1F1410	SS A1G1410	SS A1H1410
1700 STATOR SUPPORT	CD/SS	MS A2A1710	MS A1C1710	CC A1D1710	CC A1E1710	CC A1F1710	CC A1G1710	CC A1H1710
1800 CLAMP RING	CD/SS	MS A1B1810	MS A1C1810	MS A1D1810	MS A1E1810	MS A1F1810	MS A1G1810	MS A1H1810
1900 INSPECTION PLATE	CD				MS A1E1900	MS A1F1900	MS A1G1900	MS A1H1900
	SS				SS A1E1900	SS A1F1900	SS A1G1900	SS A1H1900
2000 SHAFT PIN	CD/SS	4220487017	4220487017	4220488017	4220488017	4220489017	4220489017	4220489017
2100 INTERMEDIATE	CD	4250392001	4250393001	4250394001	4250395001	4250396001	4250397001	4250398001
SHAFT	SS	4250392017	4250393017	4250394017	4250395017	4250396017	4250397017	4250398017
2200 SEAL	Q/R	3207902206	3207902206	3207902210	3207902210	3207902216	3207902216	3207902223
RING	F	3207905206	3207905206	3207905210	3207905210	3207905216	3207905216	3207905223
	B	3207904206	3207904206	3207904210	3207904210	3207904216	3207904216	3207904223
5000 ROTOR	CD	TS D4B5000	TS D4C5000	TS D4D5000	TS D4E5000	TS D4F5000	TS D4G5000	TS D4H5000
	SS	SS D4B5000	SS D4C5000	SS D4D5000	SS D4E5000	SS D4F5000	SS D4G5000	SS D4H5000
6000 DRIVE SHAFT	CD	AS A2A6000	AS A1C6000	AS A1D6000	AS A1E6000	AS A1F6000	AS A1G6000	AS A1H6000
	SS	SS A2A6000	SS A1C6000	SS A1D6000	SS A1E6000	SS A1F6000	SS A1G6000	SS A1H6000
6000 DRIVE SHAFT	CD	4250378001	4250378001	4250379001	4250379001	4250380001	4250380001	4250381001
(2-PIECE OPTION)	SS	4250378015	4250378015	4250379015	4250379015	4250380015	4250380015	4250381015
6100 RETAINING RING	CD/SS	ST A2A6100	ST A1C6100	ST A1D6100	ST A1E6100	ST A1F6100	ST A1G6100	ST A1H6100
6200 CONNECTING ROD	CD	AS A2A6200	AS A1C6200	AS A1D6200	AS A1E6200	AS A1F6200	AS A1G6200	AS A1H6200
	SS	AS A2A6200	AS A1C6200	AS A1D6200	SS A1E6200	SS A1F6200	SS A1G6200	SS A1H6200
6300 DRIVE PIN	CD/SS	TR A2A6300	TR A1C6300	TR A1D6300	TR A1E6300	TR A1F6300	TR A1G6300	TR A1H6300
6400 JOINT SEAL	Q/R	RD A2A6400	RD A1C6400	RD A1D6400	RD A1E6400	RD A1F6400	RD A1G6400	RD A1H6400
	F/B	RF A2A6400	RF A1C6400	RF A1D6400	RF A1E6400	RF A1F6400	RF A1G6400	RF A1H6400
6500 STATOR	Q	RQ A4B6510	RQ A4C6510	RQ A4D6510	RQ A4E6510	RQ A4F6510	RQ A4G6510	RQ A4H6510
	R	RR A4B6510	RR A4C6510	RR A4D6510	RR A4E6510	RR A4F6510	RR A4G6510	RR A4H6510
	F	RF A4B6510	RF A4C6510	RF A4D6510	RF A4E6510	RF A4F6510	RF A4G6510	RF A4H6510
	B	RB A4B6510	RB A4C6510	RB A4D6510	RB A4E6510	RB A4F6510	RB A4G6510	RB A4H6510
6700 BEARING SHIMS	CD/SS			GP A1D6700	GP A1D6700	GP A1F6700	GP A1F6700	GP A1H6700
6800 SLINGER RING	CD/SS	RZ A2A6800	RZ A2A6800	RZ A1D6800	RZ A1D6800	RZ A1F6800	RZ A1F6800	RZ A1H6800
6800 SLINGER/PIN RETNR	CD/SS	4230528000	4230528000	4230529000	4230529000	4230530000	4230530000	4230531000
(2-PIECE OPTION)								
6850 LANTERN RING	CD/SS	GR A2A6850	GR A2A6850	GR A1D6850	GR A1D6850	GR A1F6850	GR A1F6850	GR A1H6850
6900 PACKING	STD.	PC A2A6901	PC A2A6901	PC A1D6901	PC A1D6901	PC A1F6901	PC A1F6901	PC A1H6901
	PTFE	3403655001	3403655001	3403655002	3403655002	3403655003	3403655003	3403655004
7000 NAME PLATE	CD/SS	GA A2A7000	GA A2A7000	GA A2A7000	GA A2A7000	GA A1F7000	GA A1F7000	GA A1F7000
K O-RING	Q/R	CA137	CA141	CA145	CA175	CA188	CA197	CA228
	F/B	CF137	CF141	CF145	CF175	CF188	CF197	CF228
J SNAP RING	CD	EB007	EB015	EB020	EB027	EB036	EB045	EB049
	SS	EB507	EB515	EB520	EB527	EB536	EB545	EB549
R STATOR RING	CD/SS	EC028	EC034	EC039	EC046	EC055	EC060	EC065

*FRICTION DRIVE SEAL, TYPE 43 OR EQUAL

**POSITIVE DRIVE SEAL, TYPE 680 OR EQUAL

NOTE: CONTACT FACTORY FOR REPLACMENT MECHANICAL SEAL PART NUMBERS

PARTS LIST (Cont.)

REF. DESCRIPTION	TYPE	E4B PART NO.	E4C PART NO.	E4D PART NO.	E4E PART NO.
0100 DRIVE ADAPTOR	CD/SS	CC B2A0110	CC B2A0110	CC B1D0110	CC B1D0110
0900 PACKING GLAND	CD/SS	SC A2A0910	SC A2A0910	SC A1D0910	SC A1D0910
1000 STUFFING BOX	CD	CC A2A1010	CC A2A1010	CC A1D1010	CC A1D1010
	SS	SC A2A1010	SC A2A1010	SC A1D1010	SC A1D1010
1000 SEAL *	CD/SS	SC A2A1011	SC A2A1011	SC A1D1011	SC A1D1011
HOUSING **	CD/SS	4240833007	4240833007	4240834007	4240834007
1100 SUCTION	CD	CC A1B1110	CC A1C1110	CC A1D1110	CC A1E1110
CHAMBER STD.	SS	SC A1B1110	SC A1C1110	SC A1D1110	SC A1E1110
1200 STATOR GASKET	Q/R	GG A1B1200	GG A1C1200	GG A1D1200	GG A1E1200
	F	GF A1B1200	GF A1C1200	GF A1D1200	GF A1E1200
	B	GB 1B1200	GB A1C1200	GB A1D1200	GB A1E1200
1220 SUCTION	Q/R	GG A2A1220	GG A2A1220	GG A1D1220	GG A1D1220
CHAM. GASKET	F	GF A2A1220	GF A2A1220	GF A1D1220	GF A1D1220
	B	GB A2A1220	GB A2A1220	GB A1D1220	GB A1D1220
1240 INSPT. PLATE GSKT.	Q/R				GG A1E1240
	F				GF A1E1240
	B				GB A1E1240
1400 DISCHARGE	CD	MS A1B1410	MS A1C1410	MS A1D1410	MS A1E1410
FLANGE	SS	SS A1B1410	SS A1C1410	SS A1D1410	SS A1E1410
1600 LOCKING RING	CD/SS	MT B2A1600	MT B2A1600	MT B1D1600	MT B1D1600
1700 STATOR SUPPORT	CD/SS	MS A2A1710	MS A1C1710	CC A1D1710	CC A1E1710
1800 CLAMP RING	CD/SS	MS A1B1810	MS A1C1810	MS A1D1810	MS A1E1810
1900 INSPECTION PLATE	CD				MS A1E1900
	SS				SS A1E1900
2000 SHAFT PIN	CD/SS	4220487017	4220487017	4220488017	4220488017
5000 ROTOR	CD	TS D4B5000	TS D4C5000	TS D4D5000	TS D4E5000
	SS	SS D4B5000	SS D4C5000	SS D4D5000	SS D4E5000
6000 DRIVE SHAFT - KEY	CD	AS B2A6000	AS B1C6000	AS B1D6000	AS B1E6000
	SS	SS B2A6000	SS B1C6000	SS B1D6000	SS B1E6000
6000 CC DRIVE SHAFT –	CD	4250778001	4250779001	4250780001	4250760001
PIN	SS	4250778015	4250779015	4250780015	4250760015
6100 RETAINING RING	CD/SS	ST A2A6100	ST A1C6100	ST A1D6100	ST A1E6100
6200 CONNECTING ROD	CD	AS A2A6200	AS A1C6200	AS A1D6200	AS A1E6200
	SS	AS A2A6200	AS A1C6200	AS A1D6200	AS A1E6200
6300 DRIVE PIN	CD/SS	TR A2A6300	TR A1C6300	TR A1D6300	TR A1E6300
6400 JOINT SEAL	Q/R	RD A2A6400	RD A1C6400	RD A1D6400	RD A1E6400
	F/B	RF A2A6400	RF A1C6400	RF A1D6400	RF A1E6400
6500 STATOR	Q	RQ A4B6510	RQ A4C6510	RQ A4D6510	RQ A4E6510
	R	RR A4B6510	RR A4C6510	RR A4D6510	RR A4E6510
	F	RF A4B6510	RF A4C6510	RF A4D6510	RF A4E6510
	B	RB A4B6510	RB A4C6510	RB A4D6510	RB A4E6510
6800 SLINGER RING - KEY	CD/SS	RZ A2A6800	RZ A2A6800	RZ A1D6800	RZ A1D6800
6800 SLINGER/PIN RETNR	CD/SS	4230528000	4230528000	4230529000	4230529000
- 2 PC/CC					
6850 LANTERN RING	CD/SS	GR A2A6850	GR A2A6850	GR A1D6850	GR A1D6850
6900 PACKING	STD.	PC A2A6901	PC A2A6901	PC A1D6901	PC A1D6901
	PTFE	3403655001	3403655001	3403655002	3403655002
7000 NAME PLATE	CD/SS	GA B2A7000	GA B2A7000	GA B2A7000	GA B2A7000
K O-RING	Q/R	CA137	CA141	CA145	CA175
	F/B	CF137	CF141	CF145	CF175
J SNAP RING	CD	EB007	EB015	EB020	EB027
	SS	EB507	EB515	EB520	EB527
R STATOR RING	CD/SS	EC028	EC034	EC039	EC046

*FRICTION DRIVE SEAL, TYPE 43 OR EQUAL

**POSITIVE DRIVE SEAL, TYPE 680 OR EQUAL

NOTE: CONTACT FACTORY FOR REPLACMENT MECHANICAL SEAL PART NUMBERS

4-49. TROUBLESHOOTING CHART

PUMP PROBLEMS PROBABLE CAUSE AND REMEDY	Pump does not rotate.	Pump does not discharge.	Discharge output low.	Discharge output fluctuates.	Pump drive overloaded.	Pump noisy.	Shaft seal leaks.	Stator wears too fast.	Rotor wears too fast.
Incorrect power supply; drive not properly wired. *Check motor nameplate data; test voltage, phase, & frequency.*					●		●		●
Foreign matter in pump. *Remove foreign matter.*				●	●		●		●
If pump or stator is new, too much static friction. *Fill with liquid, and hand turn.*					●				●
Stator swells due to chemical attack. *Change stator material.*		●			●				●
Stator swells due to high liquid temp. *Reduce liquid temp. or use an undersized rotor.*	●	●			●				●
Blockage due to solids in liquid. *Decrease solids-to-liquid ratio.*		●		●	●	●	●		●
Liquid settles and hardens after pump shut down. *Clean and rinse pump after each use.*	●	●	●	●	●	●	●		●
Suction pipe not submerged. *Reposition suction pipe.*				●		●	●	●	
Air in suction pipe. *Tighten connections to stop leaks.*				●		●	●	●	
Pump speed too low. *Increase drive speed.*							●	●	
Suction lift too high (cavitation). *Reduce suction losses; move pump to lower elevation; increase pipe size.*	●	●		●		●	●	●	
Pump running dry; no prime. *Fill pump with liquid; relocate suction piping.*		●		●	●		●	●	
Stator worn excessively. *Replace stator & inspect rotor.*				●		●	●	●	
Rotor worn excessively. *Replace rotor.*		●		●		●	●	●	
Wrong direction of rotation. *Reverse drive motor polarity.*			●					●	
Discharge pressure too high. *Open discharge valve; reduce discharge pipe length; remove obstruction, increase pipe size.*	●	●			●		●	●	
Suction pipe leaks. *Tighten pipe connections.*				●		●	●		
Shaft packing leaks. *Tighten packing gland; replace packing; lubricate packing.*						●	●		
Stator material brittle. *Replace stator.*	●	●		●			●		
Pump speed too high. *Reduce drive speed.*	●	●		●	●	●			
Liquid viscosity or specific gravity too high. *Measure and compare with specification.*				●	●	●	●	●	●
Packing too tight. *Loosen gland nuts, & lubricate packing.*					●				●
Bent drive shaft. *Replace drive shaft.*				●	●				
Drive and pump misaligned. *Re-align drive and pump.*				●					●
Flexible drive coupling worn. *Repair or replace coupling.*				●					
Drive shaft bearings worn. *Replace bearings.*			●	●					
Incorrect packing. *Change packing material.*			●						
Packing too loose. *Tighten gland nuts, & lubricate.*			●						
Incorrect parts. *If pump has been rebuilt, check to verify original Moyno parts used.*	●	●		●	●	●	●	●	●

NOTE: If further troubleshooting procedural information is needed, please contact your authorized Moyno Pump distributor.

Your Authorized Service Distributor is:

Printed in U.S.A.

GOULDS PUMPS

Installation, Operation and Maintenance Instructions

Model 3296 EZMAG

ITT

IMPORTANT SAFETY NOTICE

To: Our Valued Customers

User safety is a major focus in the design of our products. Following the precautions outlined in this manual will minimize your risk of injury.

ITT Goulds pumps will provide safe, trouble-free service when properly installed, maintained, and operated.

Safe installation, operation, and maintenance of ITT Goulds Pumps equipment are an essential end user responsibility. This *Pump Safety Manual* identifies specific safety risks that must be considered at all times during product life. Understanding and adhering to these safety warnings is mandatory to ensure personnel, property, and/or the environment will not be harmed. Adherence to these warnings alone, however, is not sufficient — it is anticipated that the end user will also comply with industry and corporate safety standards. Identifying and eliminating unsafe installation, operating and maintenance practices is the responsibility of all individuals involved in the installation, operation, and maintenance of industrial equipment.

Please take the time to review and understand the safe installation, operation, and maintenance guidelines outlined in this Pump Safety Manual and the Instruction, Operation, and Maintenance (IOM) manual. Current manuals are available at www.gouldspumps.com/literature_ioms.html or by contacting your nearest Goulds Pumps sales representative.

These manuals must be read and understood before installation and start-up.

For additional information, contact your nearest Goulds Pumps sales representative or visit our Web site at www.gouldspumps.com.

SAFETY WARNINGS

Specific to pumping equipment, significant risks bear reinforcement above and beyond normal safety precautions.

⚠ WARNING

A pump is a pressure vessel with rotating parts that can be hazardous. Any pressure vessel can explode, rupture, or discharge its contents if sufficiently over pressurized causing death, personal injury, property damage, and/or damage to the environment. All necessary measures must be taken to ensure over pressurization does not occur.

⚠ WARNING

Operation of any pumping system with a blocked suction and discharge must be avoided in all cases. Operation, even for a brief period under these conditions, can cause superheating of enclosed pumpage and result in a violent explosion. All necessary measures must be taken by the end user to ensure this condition is avoided.

⚠ WARNING

The pump may handle hazardous and/or toxic fluids. Care must be taken to identify the contents of the pump and eliminate the possibility of exposure, particularly if hazardous and/or toxic. Potential hazards include, but are not limited to, high temperature, flammable, acidic, caustic, explosive, and other risks.

⚠ WARNING

Pumping equipment Instruction, Operation, and Maintenance manuals clearly identify accepted methods for disassembling pumping units. These methods must be adhered to. Specifically, applying heat to impellers and/or impeller retaining devices to aid in their removal is strictly forbidden. Trapped liquid can rapidly expand and result in a violent explosion and injury.

ITT Goulds Pumps will not accept responsibility for physical injury, damage, or delays caused by a failure to observe the instructions for installation, operation, and maintenance contained in this Pump Safety Manual or the current IOM available at www.gouldspumps.com/literature.

SAFETY

DEFINITIONS

Throughout this manual the words **WARNING**, **CAUTION**, **ELECTRICAL, and ATEX** are used to indicate where special operator attention is required.

Observe all Cautions and Warnings highlighted in this Pump Safety Manual and the IOM provided with your equipment.

⚠ **WARNING**
Indicates a hazardous situation which, if not avoided, could result in death or serious injury.

Example: Pump shall never be operated without coupling guard installed correctly.

⚠ **CAUTION**
Indicates a hazardous situation which, if not avoided, could result in minor or moderate injury.

Example: Throttling flow from the suction side may cause cavitation and pump damage.

ELECTRICAL HAZARD
Indicates the possibility of electrical risks if directions are not followed.

Example: Lock out driver power to prevent electric shock, accidental start-up, and physical injury.

When installed in potentially explosive atmospheres, the instructions that follow the Ex symbol must be followed. Personal injury and/or equipment damage may occur if these instructions are not followed. If there is any question regarding these requirements or if the equipment is to be modified, please contact an ITT Goulds Pumps representative before proceeding.

Example: Improper impeller adjustment could cause contact between the rotating and stationary parts, resulting in a spark and heat generation.

GENERAL PRECAUTIONS

⚠ WARNING

A pump is a pressure vessel with rotating parts that can be hazardous. Hazardous fluids may be contained by the pump including high temperature, flammable, acidic, caustic, explosive, and other risks. Operators and maintenance personnel must realize this and follow safety measures. Personal injuries will result if procedures outlined in this manual are not followed. ITT Goulds Pumps will not accept responsibility for physical injury, damage or delays caused by a failure to observe the instructions in this manual and the IOM provided with your equipment.

General Precautions		
WARNING		NEVER APPLY HEAT TO REMOVE IMPELLER. It may explode due to trapped liquid.
WARNING		NEVER use heat to disassemble pump due to risk of explosion from tapped liquid.
WARNING		NEVER operate pump without coupling guard correctly installed.
WARNING		NEVER run pump below recommended minimum flow when dry, or without prime.
WARNING		ALWAYS lock out power to the driver before performing pump maintenance.
WARNING		NEVER operate pump without safety devices installed.
WARNING		NEVER operate pump with discharge valve closed.
WARNING		NEVER operate pump with suction valve closed.
WARNING		DO NOT change service application without approval of an authorized ITT Goulds Pumps representative.
WARNING		**Safety Apparel:** • Insulated work gloves when handling hot bearings or using bearing heater • Heavy work gloves when handling parts with sharp edges, especially impellers • Safety glasses (with side shields) for eye protection • Steel-toed shoes for foot protection when handling parts, heavy tools, etc. • Other personal protective equipment to protect against hazardous/toxic fluids
WARNING		**Receiving:** Assembled pumping units and their components are heavy. Failure to properly lift and support equipment can result in serious physical injury and/or equipment damage. Lift equipment only at specifically identified lifting points or as instructed in the current IOM. Current manuals are available at www.gouldspumps.com/literature_ioms.html or from your local ITT Goulds Pumps sales representative. Note: Lifting devices (eyebolts, slings, spreaders, etc.) must be rated, selected, and used for the entire load being lifted.
WARNING		**Alignment:** Shaft alignment procedures must be followed to prevent catastrophic failure of drive components or unintended contact of rotating parts. Follow coupling manufacturer's coupling installation and operation procedures.

General Precautions		
WARNING		Before beginning any alignment procedure, make sure driver power is locked out. Failure to lock out driver power will result in serious physical injury.
CAUTION		**Piping:** Never draw piping into place by forcing at the flanged connections of the pump. This may impose dangerous strains on the unit and cause misalignment between pump and driver. Pipe strain will adversely effect the operation of the pump resulting in physical injury and damage to the equipment.
WARNING		**Flanged Connections:** Use only fasteners of the proper size and material.
WARNING		Replace all corroded fasteners.
WARNING		Ensure all fasteners are properly tightened and there are no missing fasteners.
WARNING		**Startup and Operation:** When installing in a potentially explosive environment, please ensure that the motor is properly certified.
WARNING		Operating pump in reverse rotation may result in contact of metal parts, heat generation, and breach of containment.
WARNING		Lock out driver power to prevent accidental start-up and physical injury.
WARNING		The impeller clearance setting procedure must be followed. Improperly setting the clearance or not following any of the proper procedures can result in sparks, unexpected heat generation and equipment damage.
WARNING		If using a cartridge mechanical seal, the centering clips must be installed and set screws loosened prior to setting impeller clearance. Failure to do so could result in sparks, heat generation, and mechanical seal damage.
WARNING		The coupling used in an ATEX classified environment must be properly certified and must be constructed from a non-sparking material.
WARNING		Never operate a pump without coupling guard properly installed. Personal injury will occur if pump is run without coupling guard.
WARNING		Make sure to properly lubricate the bearings. Failure to do so may result in excess heat generation, sparks, and / or premature failure.
CAUTION		The mechanical seal used in an ATEX classified environment must be properly certified. Prior to start up, ensure all points of potential leakage of process fluid to the work environment are closed.
CAUTION		Never operate the pump without liquid supplied to mechanical seal. Running a mechanical seal dry, even for a few seconds, can cause seal damage and must be avoided. Physical injury can occur if mechanical seal fails.
WARNING		Never attempt to replace packing until the driver is properly locked out and the coupling spacer is removed.
WARNING		Dynamic seals are not allowed in an ATEX classified environment.
WARNING		DO NOT operate pump below minimum rated flows or with suction and/or discharge valve closed. These conditions may create an explosive hazard due to vaporization of pumpage and can quickly lead to pump failure and physical injury.

General Precautions		
WARNING		Ensure pump is isolated from system and pressure is relieved before disassembling pump, removing plugs, opening vent or drain valves, or disconnecting piping.
WARNING		**Shutdown, Disassembly, and Reassembly:** Pump components can be heavy. Proper methods of lifting must be employed to avoid physical injury and/or equipment damage. Steel toed shoes must be worn at all times.
WARNING		The pump may handle hazardous and/or toxic fluids. Observe proper decontamination procedures. Proper personal protective equipment should be worn. Precautions must be taken to prevent physical injury. Pumpage must be handled and disposed of in conformance with applicable environmental regulations.
WARNING		Operator must be aware of pumpage and safety precautions to prevent physical injury.
WARNING		Lock out driver power to prevent accidental startup and physical injury.
CAUTION		Allow all system and pump components to cool before handling them to prevent physical injury.
CAUTION		If pump is a Model NM3171, NM3196, 3198, 3298, V3298, SP3298, 4150, 4550, or 3107, there may be a risk of static electric discharge from plastic parts that are not properly grounded. If pumped fluid is non-conductive, pump should be drained and flushed with a conductive fluid under conditions that will not allow for a spark to be released to the atmosphere.
WARNING		Never apply heat to remove an impeller. The use of heat may cause an explosion due to trapped fluid, resulting in severe physical injury and property damage.
CAUTION		Wear heavy work gloves when handling impellers as sharp edges may cause physical injury.
CAUTION		Wear insulated gloves when using a bearing heater. Bearings will get hot and can cause physical injury.

ATEX CONSIDERATIONS and INTENDED USE

Special care must be taken in potentially explosive environments to ensure that the equipment is properly maintained. This includes but is not limited to:

1. Monitoring the pump frame and liquid end temperature.
2. Maintaining proper bearing lubrication.
3. Ensuring that the pump is operated in the intended hydraulic range.

The ATEX conformance is only applicable when the pump unit is operated within its intended use. Operating, installing or maintaining the pump unit in any way that is not covered in the Instruction, Operation, and Maintenance manual (IOM) can cause serious personal injury or damage to the equipment. This includes any modification to the equipment or use of parts not provided by ITT Goulds Pumps. If there is any question regarding the intended use of the equipment, please contact an ITT Goulds representative before proceeding. Current IOMs are available at www.gouldspumps.com/literature_ioms.html or from your local ITT Goulds Pumps Sales representative.

All pumping unit (pump, seal, coupling, motor and pump accessories) certified for use in an ATEX classified environment, are identified by an ATEX tag secured to the pump or the baseplate on which it is mounted. A typical tag would look like this:

The CE and the Ex designate the ATEX compliance. The code directly below these symbols reads as follows:

II	=	Group 2
2	=	Category 2
G/D	=	Gas and Dust present
T4	=	Temperature class, can be T1 to T6 (see Table 1)

Table 1

Code	Max permissible surface temperature °F (°C)	Max permissible liquid temperature °F (°C)
T1	842 (450)	700 (372)
T2	572 (300)	530 (277)
T3	392 (200)	350 (177)
T4	275 (135)	235 (113)
T5	212 (100)	Option not available
T6	185 (85)	Option not available

The code classification marked on the equipment must be in accordance with the specified area where the equipment will be installed. If it is not, do not operate the equipment and contact your ITT Goulds Pumps sales representative before proceeding.

PARTS

The use of genuine Goulds parts will provide the safest and most reliable operation of your pump. ITT Goulds Pumps ISO certification and quality control procedures ensure the parts are manufactured to the highest quality and safety levels.

Please contact your local Goulds representative for details on genuine Goulds parts.

FOREWORD

This manual provides instructions for the Installation, Operation, and Maintenance of the Goulds Model 3296 EZMAG Magnetic Drive Process Pump. This manual must be read and understood before installation and start-up.

The design, materials, and workmanship incorporated in the manufacturing of Goulds pumps makes them capable of giving years of trouble-free service. The life and satisfactory service of any mechanical unit, however, is enhanced and extended by correct application, proper installation, periodic inspection, condition monitoring and careful maintenance. This instruction manual was prepared to assist operators in understanding the construction and the correct methods of installing, operating, and maintaining these pumps.

ITT - Goulds Pumps shall not be liable for physical injury, damage or delays caused by a failure to observe the instructions for Installation, Operation, and Maintenance contained in this manual.

Warranty is valid only when genuine ITT - Goulds Pumps parts are used.

Use of the equipment on a service other than stated in the order could nullify the warranty, unless written approval is obtained in advance from ITT - Goulds Pumps.

Supervision by an authorized ITT - Goulds Pumps representative is recommended to assure proper installation.

Additional manuals can be obtained by contacting your local ITT - Goulds Pumps representative or by calling 1-800-446-8537.

THIS MANUAL EXPLAINS

- **Proper Installation**
- **Start Up Procedures**
- **Operation Procedures**
- **Routine Maintenance**
- **Pump Overhaul**
- **Troubleshooting**
- **Ordering Spare or Repair Parts**

TABLE OF CONTENTS

SAFETY

DEFINITIONS

These pumps have been designed for safe and reliable operation when properly used and maintained in accordance with instructions contained in this manual. A pump is a pressure containing device with rotating parts that can be hazardous. Operators and maintenance personnel must realize this and follow safety measures. ITT Industries Goulds Pumps shall not be liable for physical injury, damage or delays caused by a failure to observe the instructions in this manual.

Throughout this manual the words **WARNING**, **CAUTION**, **ELECTRICAL, ATEX**, and **NOTE** are used to indicate procedures or situations which require special operator attention:

⚠ WARNING

Operating procedure, practice, etc. which, if not correctly followed, could result in personal injury or loss of life.

! CAUTION

Operating procedure, practice, etc. which, if not followed, could result in damage or destruction of equipment.

Particular care must be taken when electrical power source to the equipment is energized.

If equipment is to be installed in a potentially explosive atmosphere and these procedures are not followed, personal injury or equipment damage from an explosion may result.

NOTE: Operating procedure, condition, etc. which is essential to observe.

EXAMPLES

⚠ WARNING

Pump shall never be operated without coupling guard installed correctly.

! CAUTION

Throttling flow from the suction side may cause cavitation and pump damage.

Lock out driver power to prevent electric shock, accidental start-up, and physical injury.

Improper impeller adjustment could cause contact between the rotating and stationary parts, resulting in a spark and heat generation.

NOTE: Proper alignment is essential for long pump life.

MEDICAL PRECAUTIONS

WARNING

Magnetic drive pumps contain very strong magnets which may pose health risks. The following guidelines shall always be observed.

1. Individuals with artificial cardiac pacemakers, implanted defibrillators, metallic prosthetic heart valves, internal wound clips (from surgery), prosthetic joints, metallic wiring, or other metallic prosthetic devices shall avoid working with, being in proximity of, or handling the magnets contained in the pumps.
2. Individuals who have had previous surgeries (especially chest or head surgery) and who do not know if they have metallic clips internally should avoid work on this unit unless it can be firmly established by his or her physician that no metallic devices exist.

GENERAL PRECAUTIONS

WARNING

Personal injuries will result if procedures outlined in this manual are not followed.

	⚠	NEVER apply heat to remove impeller. It may explode due to trapped liquid.
	⚠	NEVER use heat to disassemble pump due to risk of explosion from trapped liquid.
Ex	⚠	NEVER operate pump without coupling guard correctly installed.
Ex	●	NEVER operate pump beyond the rated conditions to which the pump was sold.
Ex	●	NEVER start pump without proper prime (all models), or proper liquid level in self-priming pumps (Model 3796).
Ex	⚠	NEVER run pump below recommended minimum flow or when dry.
⚠	⚠	ALWAYS lock out power to the driver before performing pump maintenance.
	⚠	NEVER operate pump without safety devices installed.
Ex	⚠	NEVER operate pump with discharge valve closed.
Ex	●	NEVER operate pump with suction valve closed.
Ex	⚠	DO NOT change conditions of service without approval of an authorized Goulds representative.

EXPLOSION PREVENTION

Ex ***In order to reduce the possibility of accidental explosions in atmospheres containing explosive gases and/or dust, the instructions under the ATEX symbol must be closely followed. ATEX certification is a specification enforced in Europe for non-electrical and electrical equipment installed in Europe. The usefulness of the ATEX requirements is not limited to Europe. They are useful guidelines for equipment installed in any potentially explosive environment.***

Ex ***NOTE: When pumping unit is installed in a potentially explosive atmosphere, the instructions after the Ex symbol must be followed. Personal injury and/or equipment damage may occur if these instructions are not followed. If there is any question regarding these requirements or if the equipment is to be modified, please contact a Goulds representative before proceeding.***

SPECIAL ATEX CONSIDERATIONS

All installation and operation instructions in this manual must be strictly adhered to. In addition, care must be taken to ensure that the equipment is properly maintained. This includes but is not limited to:

1. Monitoring the pump frame and liquid end temperature.
2. Maintaining proper bearing lubrication.
3. Ensuring that the pump is operated in the intended hydraulic range.

ATEX IDENTIFICATION

For a pumping unit (pump, seal, coupling, motor and pump accessories) to be certified for use in an ATEX classified environment, the proper ATEX identification must be present.

The ATEX tag would be secured to the pump or the baseplate on which it is mounted. A typical tag would look like this:

The CE and the Ex designate the ATEX compliance. The code directly below these symbols reads as follows:

II	=	Group 2
2	=	Category 2
G/D	=	Gas and Dust present
Tx	=	Temperature class, can be T1 to Tx (see *Table 1*)

Table 1

Code	Max permissible surface temperature °F (°C)	Max permissible liquid temperature °F (°C)
T1	842 (450)	700 (372)
T2	572 (300)	530 (277)
T3	392 (200)	350 (177)
T4	275 (135)	235 (113)
T5	212 (100)	Option not available
T6	185 (85)	Option not available

The code classification marked on the equipment should be in accordance with the specified area where the equipment will be installed. If it is not, please contact your ITT/Goulds representative before proceeding.

INTENDED USE

The ATEX conformance is only applicable when the pump unit is operated within its intended use. All instructions within this manual must be followed at all times. Operating, installing or maintaining the pump unit in any way that is not covered in this manual can cause serious personal injury or damage to the equipment. This includes any modification to the equipment or use of parts not provided by ITT/Goulds. If there is any question regarding the intended use of the equipment, please contact an ITT/Goulds representative before proceeding.

CONDITION MONITORING

For additional safety precautions, and where noted in this manual, condition monitoring devices should be used. This includes, but is not limited to:

- Pressure gauges
- Flow meters
- Level indicators
- Motor load readings
- Temperature detectors
- Bearing monitors
- Leak detectors
- PumpSmart® control system

For assistance in selecting the proper instrumentation and its use, please contact your ITT/Goulds representative.

GENERAL INFORMATION

PUMP DESCRIPTION

The Model 3296 EZMAG is a sealless centrifugal pump with an enclosed impeller that is driven by a synchronous magnetic coupling. It meets the dimensional standards outlined in ANSI B73.1.

Magnetic Coupling - is a coaxial synchronous type using rare earth magnets. This concept results in a compact design and allows the impeller to turn at the same speed as the motor, (i.e.) there is no slip between the drive and driven magnets.

Magnets - Two types of rare earth magnets are available. Neodymium Iron (NdFe), which is used when pumpage temperatures are less than 365°F (180°C). For liquid pumpage between 365°F (180°C) and 536°F (280°C) Samarium Cobalt (SmCo) magnets are used as part of the high temperature option.

Containment Shell - isolates the pumped liquid from the atmosphere. Standard material is Hastelloy-C which provides excellent corrosion and erosion resistance.

Sleeve Bearings and Thrust Bearings - Goulds standard bearing material is Pure Sintered Alpha Grade Silicon Carbide. Dryguard™ bearings are available for dry-run protection.

Impeller - Model 3296 EZMAG utilizes an enclosed impeller, hydraulically balanced and keyed to the shaft.

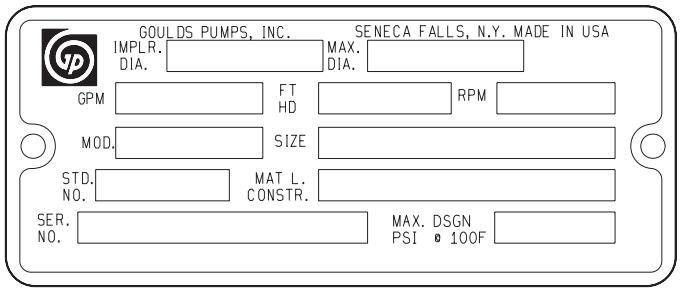
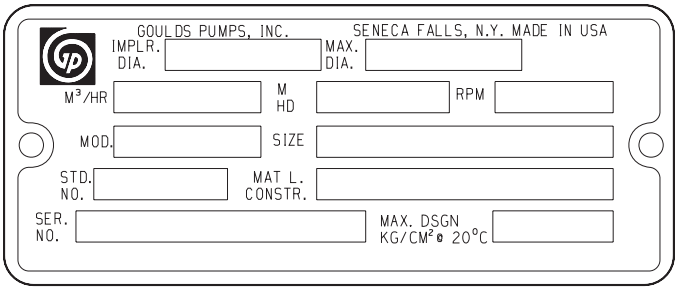
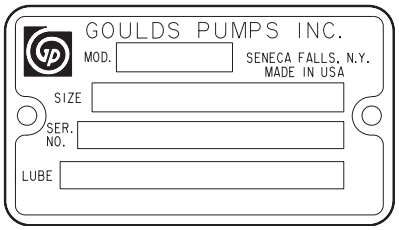
Bearing Frame - the standard configuration is cast iron with flood oil lubricated ball bearings. Greased-for-life bearings systems are available as an option. For protection and reliability of the bearings and the lubricant, bronze bearing isolators are provided as standard.

Casing - is top centerline discharge, self venting type incorporates a fully confined gasket. 150 lb. ANSI serrated raised face flanges are standard. The 3296 EZMAG has been designed such that there is a metal-to-metal fit between the casing and backplate.

NAMEPLATE INFORMATION

Every pump has two Goulds nameplates that provide information about the pump. The tags are located on the casing and bearing frame.

When ordering spare parts, you will need to identify pump model, size, serial number, and the item number of required parts. Information can be taken from the pump casing tag. Item numbers can be found in this manual.

Description	Fig. No.	Example
Pump Casing Tag - provides information about the pump's hydraulic characteristics. Note the format of the pump size: Discharge x Suction - Nominal maximum Impeller Diameter in inches (Example: 2x3-8) (Figs. 1 & 2).	**Fig. 1 English**	
	Fig. 2 Metric	
Bearing Frame Tag - provides information on the lubrication system used (Fig. 3).	**Fig. 3**	
ATEX Tag - If applicable, your pump unit may have the following ATEX tag affixed to the pump and/or baseplate. See the *Safety* section for a description of the symbols and codes (Fig. 4).	**Fig. 4**	

RECEIVING THE PUMP

Inspect the pump as soon as it is received. Make notes of damaged or missing items on the receipt and freight bill. File any claims with the transportation company immediately.

STORAGE REQUIREMENTS

Short Term - (Less than 3 months) Goulds normal packaging procedure is designed to protect the pump during shipping. Upon receipt store in a covered and dry location.

Long Term - (More than 6 months) Preservative treatment of bearings and machined surfaces is required (can be purchased from Goulds). Rotate shaft several times every 3 months. Refer to driver and coupling manuals for their long term storage procedures. Store in a dry covered location.

HANDLING

⚠ WARNING

Failure to properly lift and support equipment could result in serious injury or damage to pumps.

Use care when moving pumps. Lifting equipment must be able to adequately support the entire assembly. Hoist bare pumps, using a sling under the suction flange and bearing housing.

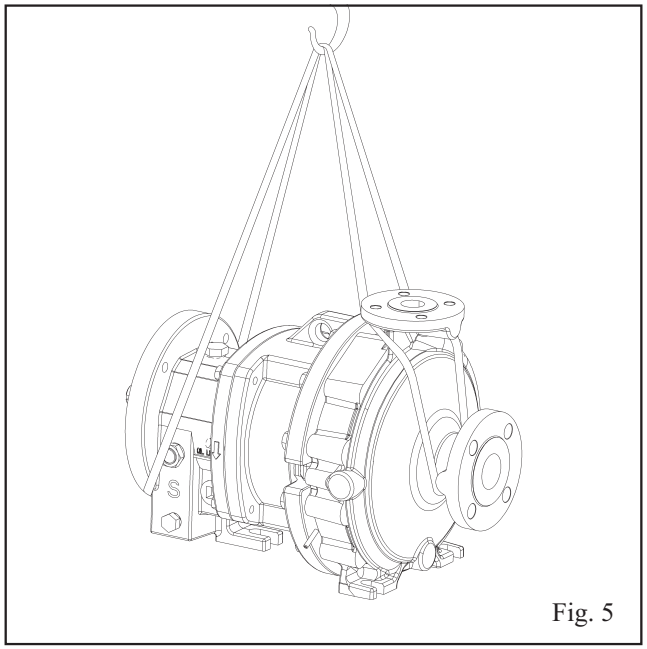


Fig. 5

Baseplate mounted units are moved with slings under the pump and driver.

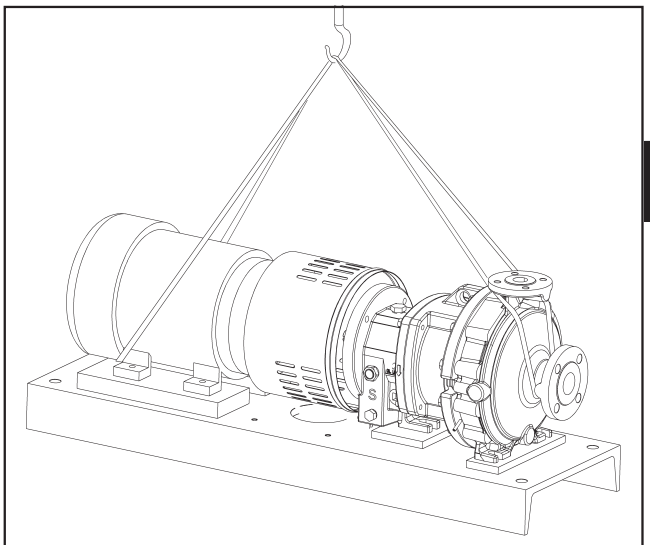


Fig. 6

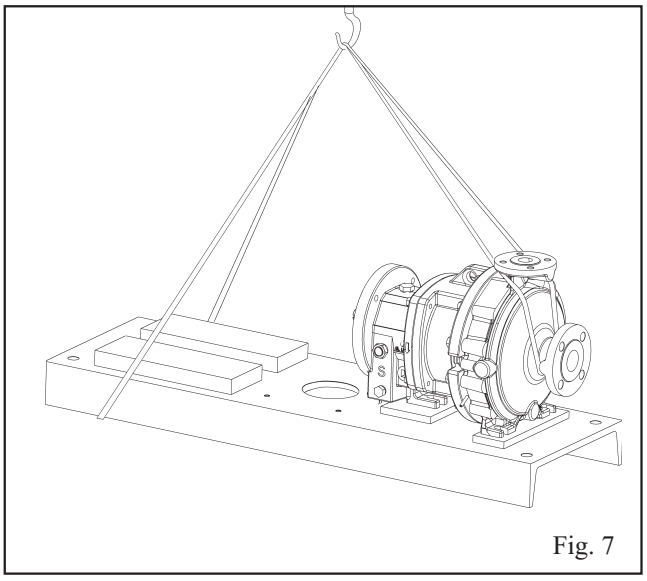


Fig. 7

INSTALLATION

BASEPLATE INSPECTION

Equipment that will operate in a potentially explosive environment must be installed in accordance with the following instructions.

1. Remove all equipment.
2. Completely clean the underside of baseplate. It is sometimes necessary to coat the underside of the baseplate with an epoxy primer. This may have been purchased as an option.
3. Remove the rust preventative solution from the machined pads with an appropriate solution.

SITE/FOUNDATION

A pump should be located near the supply of liquid and have adequate space for operation, maintenance, and inspection.

Baseplate mounted pumps are normally grouted to a concrete foundation, which has been poured on a solid footing. The foundation must be able to absorb any vibration and to form a permanent, rigid support for the pumping unit.

The location and size of the foundation bolts are shown on the outline assembly drawing, provided with the pump data package.

All equipment being installed must be properly grounded to prevent unexpected static electric discharge.

Foundation bolts commonly used are sleeve type (Fig. 8) and J type (Fig. 9). Both designs permit movement for final bolt adjustment.

1. Inspect foundation for dust, oil, chips, water, etc. and remove any contaminants. Do not use oil-based cleaners as grout will not bond to it
2. Prepare the foundation in accordance with the grout manufacturer's recommendations.

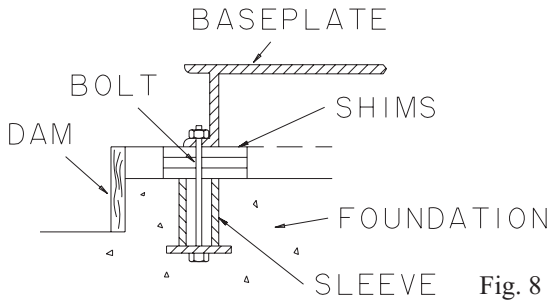


Fig. 8

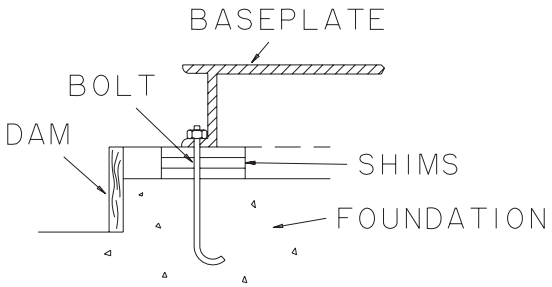


Fig. 9

LEVEL BASEPLATE

CAST IRON/CHEMBASE/FAB. STEEL

1. Place 2 sets of wedges or shims on the foundation, one set on each side of every foundation bolt. The wedges should extend .75 in. (20 mm) to 1.5 in. (40 mm) above the foundation, to allow for adequate grouting. This will provide even support for the baseplate once it is grouted.
2. Remove water and/or debris from anchor bolt holes/ sleeves prior to grouting. If the sleeve type bolts are being used, fill the sleeves with rags to prevent grout from entering.
3. Carefully lower baseplate onto foundation bolts.
4. Level baseplate to within .125 in.(3mm) over the length of the base and .062 in. (1.5 mm) over the width of the base by adjusting shims or wedges.
5. Hand tighten bolts.

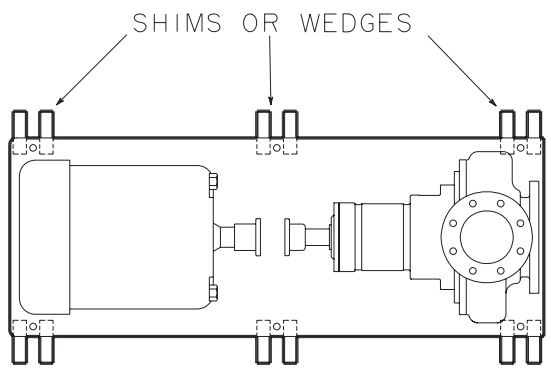


Fig. 10

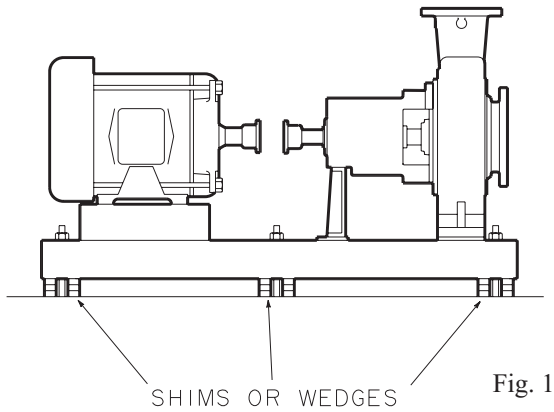


Fig. 11

FEATURE FAB. STEEL/API STYLE

(Baseplates provided w/vertical leveling adjustors)

1. Coat the jack screws with an anti seizing compound to allow for easy removal after the grout has been cured.
2. Cut round circular plates from bar stock to set the jack screws on. The edges of the plates should be chamfered to reduce stress concentrations.
3. Set the baseplate on the foundation and use the four corner jackscrews to raise the baseplate off the foundation 0.75" to 15". The center jack screws should not be touching the foundation.

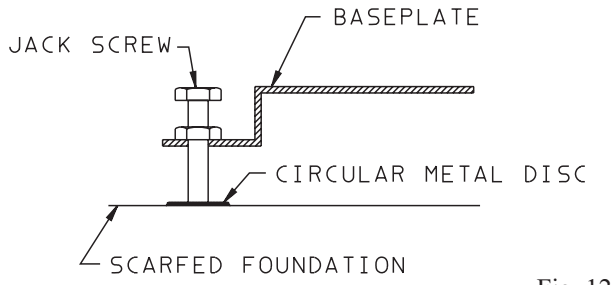


Fig. 12

4. Place two machinist levels on the motor pads, one lengthwise on a single motor pad, and another across the ends of both motor pads. See diagram below.

NOTE: When using a machinist level, it is important that the surface being leveled is free of all contaminants, such as dust, to ensure an accurate reading.

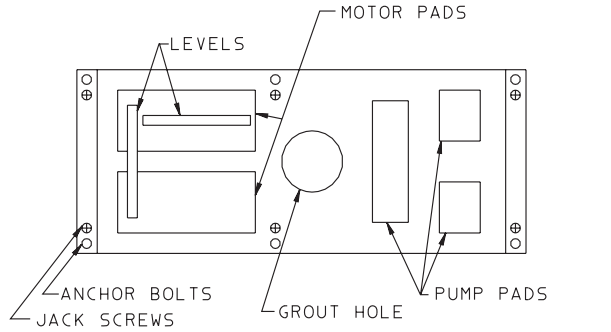


Fig. 13

5. Level the motor pads to zero, in both directions, by adjusting the four jack screws.
6. Next, turn down the center jack-screws so that they are resting on their metal discs on the foundation.
7. Place the two levels on the pump pads, one lengthwise on a single pump pad, and another across the middle of both pump pads. See diagram below.

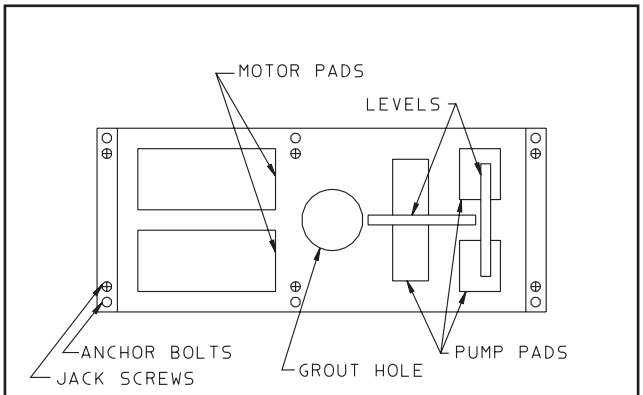
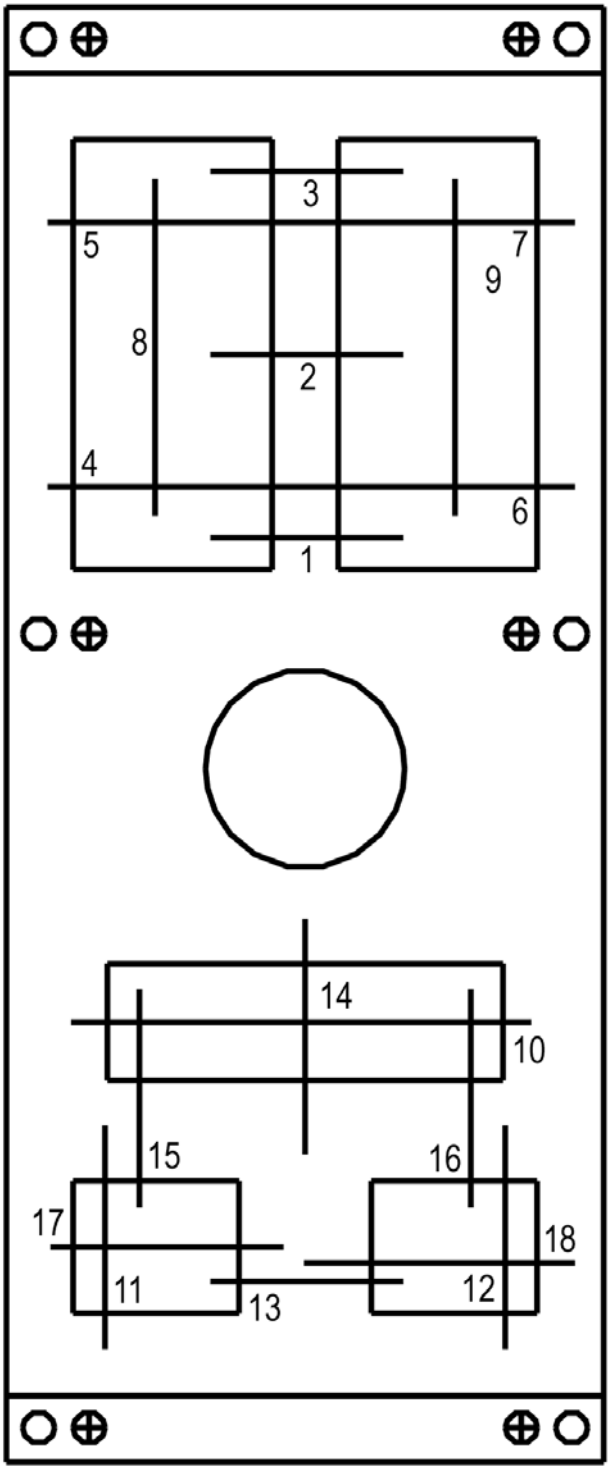


Fig. 14

8. Level the pump pads to zero, in both directions, by adjusting the jack screws.
9. Install the anchor bolts until they are hand tight.
10. Return the levels to the motor pads and check the level measurements.
11. Adjust the jack screws and anchor bolts, if necessary, until all level measurements are within the design requirements of 0.002 in/ft.
12. When taking readings, center the level over the pad being measured.

NOTE: The Baseplate Leveling Worksheet provided may be used when taking readings.

BASEPLATE LEVELING WORKSHEET



LEVEL MEASUREMENTS

1) _____

2) _____

3) _____

4) _____

5) _____

6) _____

7) _____

8) _____

9) _____

10) _____

11) _____

12) _____

13) _____

14) _____

15) _____

16) _____

17) _____

18) _____

PUMP EQUIPMENT #: _____ PUMP S/N: _____

LEVELED BY: _____ DATE: _____

ALIGNMENT AND ALIGNMENT PROCEDURE

WARNING

Before beginning any alignment procedure make sure driver power is locked out. Failure to lock out driver power can result in serious personal injury.

Alignment procedures must be followed to prevent unintended contact of rotating parts. Follow coupling manufacturer's installation and operation procedures.

The points at which alignment is checked and adjusted are:

- **Initial Alignment** is done prior to operation when the pump and the driver are at ambient temperature.
- **Final Alignment** is done after operation when the pump and driver are at operating temperature.

Alignment is achieved by adding or removing shims from under the feet of the driver and shifting equipment horizontally as needed.

NOTE: Proper alignment is the responsibility of the installer of the unit.

Accurate alignment of the equipment must be attained. Trouble-free operation can be accomplished by following these procedures:

ALIGNMENT CHECKS

Initial Alignment (Cold Alignment)

- Before Grouting Baseplate - To ensure alignment can be obtained.
- After Grouting Baseplate - To ensure no changes to alignment have occurred during grouting process.
- After Connecting Piping -To ensure that pipe strains haven't altered alignment. If changes have occurred, alter piping to remove pipe strains on pump flanges.

Final Alignment (Hot Alignment)

- After First Run -To obtain correct alignment when both pump and driver are at operating temperature. Thereafter, alignment should be checked periodically in accordance with plant operating and maintenance procedures.

ALIGNMENT CRITERIA

Good alignment is achieved when dial indicator readings as specified in the alignment procedure are .002 in. (.05 mm) Total Indicated Reading (T.I.R.) or less when the pump and driver are at operating temperature (Final Alignment).

During the installation phase, however, it is necessary to set the parallel alignment in the vertical direction to a different criteria due to differences in expansion rates of the pump and driver. *Table 2* below shows recommended cold settings for electric motor driven pumps based on different pumpage temperatures. Driver manufacturers should be consulted for recommended cold settings for other types of drivers (steam turbines, engines, etc.)

Table 2
Cold Settings of
Parallel Vertical Alignment

Pumpage Temperature	Set Driver Shaft
50°F (10°C)	.002in. (.05mm) LOW
150°F (65°C)	.001in. (.03mm) HIGH
250°F (120°C)	.005in. (.12mm) HIGH
350°F (175°C)	.009in. (.23mm) HIGH
425°F (218°C)	.013in. (.33mm) HIGH

SET UP

1. Mount two dial indicators on one of the coupling halves (X) so they contact the other coupling half (Y) (Fig. 15).
2. Check setting of indicators by rotating coupling half X to ensure indicators stay in contact with coupling half Y but do not bottom out. Adjust indicators accordingly.

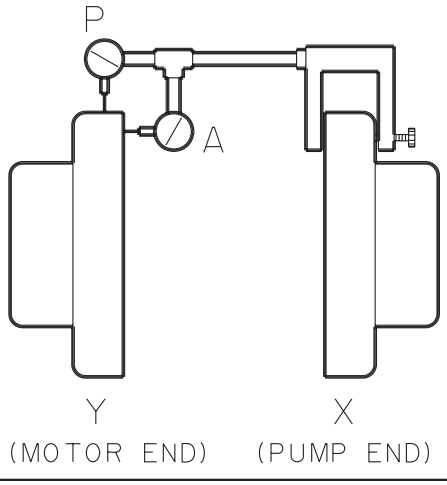


Fig. 15

MEASUREMENT

1. To ensure accuracy of indicator readings, always rotate both coupling halves together so indicators contact the same point on coupling half Y. This will eliminate any measurement problems due to runout on coupling half Y.
2. Take indicator measurements with driver feet hold down bolts tightened. Loosen hold down bolts prior to making alignment corrections.
3. Take care not to damage indicators when moving driver during alignment corrections.

ANGULAR ALIGNMENT

A unit is in angular alignment when indicator A (Angular indicator) does not vary by more that .002 in. (.05 mm) as measured at four locations 90° apart.

Vertical Correction (Top to Bottom)

1. Zero indicator A at top dead center (12 o'clock) of coupling half Y.
2. Rotate indicators to bottom dead center (6 o'clock). Observe needle and record reading.
3. **Negative Reading** - The coupling halves are further apart at the bottom than at the top. Correct by either raising the driver feet at the shaft end (add shims) or lowering the driver feet at the other end (remove shims) (Fig. 16).

   Positive Reading - The coupling halves are closer at the bottom than at the top. Correct by either lowering the driver feet at the shaft end (remove shims) or raising the driver feet at the other end (add shims).

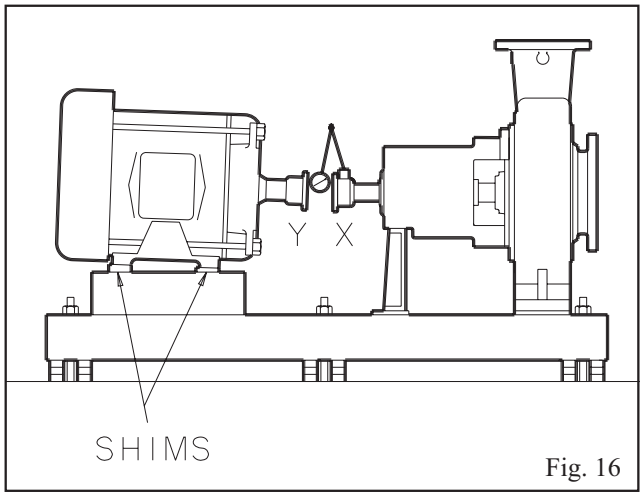


Fig. 16

4. Repeat steps 1-3 until indicator A reads .002 in (.05 mm) or less.

Horizontal Correction (Side to Side)

1. Zero indicator A on left side of coupling half Y, 90° from top dead center (9 o'clock).
2. Rotate indicators through top dead center to the right side, 180° from the start (3 o'clock). Observe needle and record reading.
3. **Negative Reading** - The coupling halves are further apart on the right side than the left. Correct by either sliding the shaft end of the driver to the left or the other end to the right.

   Positive Reading - The coupling halves are closer together on the right side than the left. Correct by either sliding the shaft end of the driver to the right or the other end to the left (Fig. 11).

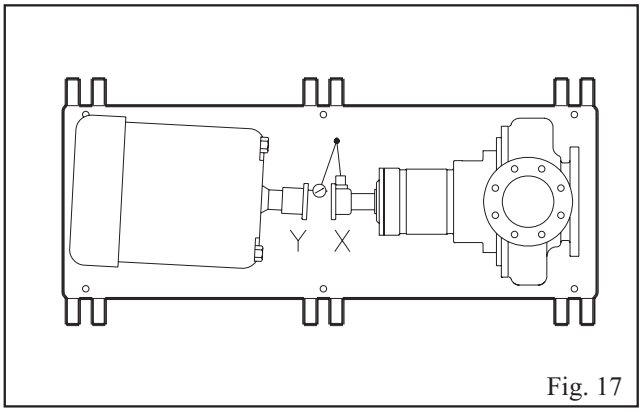


Fig. 17

4. Repeat steps 1 through 3 until indicator A reads .002 in. (.05 mm) or less.
5. Re-check both horizontal and vertical readings to ensure adjustment of one did not disturb the other. Correct as necessary.

PARALLEL ALIGNMENT

A unit is in parallel alignment when indicator P (parallel indicator) does not vary by more than .002 in. (.05 mm) as measured at four points 90° apart at operating temperature. Note the preliminary cold setting criteria, See *Table 2*.

Vertical Correction (Top to Bottom)

1. Zero indicator P at top dead center of coupling (12 o'clock) half Y (Fig. 15).
2. Rotate indicator to bottom dead center (6 o'clock). Observe needle and record reading.
3. **Negative Reading** - Coupling half X is lower than coupling half Y. Correct by removing shims of thickness equal to half of the indicator reading under each driver foot.

Positive Reading - Coupling half X is higher than coupling half Y. Correct by adding shims of thickness equal to half of the indicator reading from each driver foot (Fig. 18).

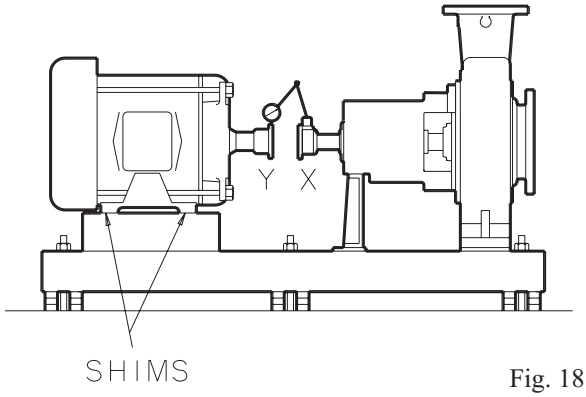


Fig. 18

NOTE: Equal amounts of shims must be added to or removed from each driver foot. Otherwise the vertical angular alignment will be affected.

4. Repeat steps 1 through 3 until indicator P reads within .002 in. (.05 mm) or less when hot, or per *Table 2* when cold.

Horizontal Correction (Side to Side)

1. Zero indicator P on the left side of coupling half Y, 90° from top dead center (9 o'clock).
2. Rotate indicators through top dead center to the right side, 180° from the start. Observe needle and record reading (3 o'clock).
3. **Negative Reading** - Coupling half Y is to the left of coupling half X. Correct by sliding driver evenly in the appropriate direction (Fig. 19).

   Positive Reading - Coupling half Y is to the right of coupling half X. Correct by sliding driver evenly in the appropriate direction.

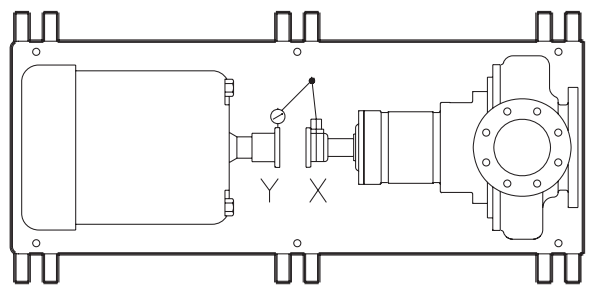


Fig. 19

NOTE: Failure to slide motor evenly will affect horizontal angular correction.

4. Repeat steps 1 through 3 until indicator P reads .002 in. (.05 mm) or less.
5. Re-check both horizontal and vertical readings to ensure adjustment of one did not disturb the other. Correct as necessary.

COMPLETE ALIGNMENT

A unit is in complete alignment when both indicators A (angular) and P (parallel) do not vary by more than .002 in. (.05 mm) as measured at four points 90° apart.

Vertical Correction (Top to Bottom)

1. Zero indicators A and P at top dead center (12 o'clock) of coupling half Y.
2. Rotate indicator to bottom dead center (6 o'clock). Observe the needles and record the readings.
3. Make corrections as outlined previously.

Horizontal Correction (Side to Side)

1. Zero indicators A and P on the left side of coupling half Y, 90° from top dead center (9 o'clock).
2. Rotate indicators through top dead center to the right side, 180° from the start (3 o'clock). Observe the needle, measure and record the reading.
3. Make corrections as outlined previously.
4. Recheck both vertical and horizontal readings to ensure adjustment of one did not disturb the other. Correct as necessary.

NOTE: With experience, the installer will understand the interaction between angular and parallel and will make corrections appropriately.

ALIGNMENT TROUBLESHOOTING

Table 3
Alignment Troubleshooting

PROBLEM	PROBABLE CAUSE	REMEDY
Cannot obtain horizontal (Side-to-Side) alignment, angular or parallel	Driver feet bolt bound	Loosen pump hold down bolts and slide pump and driver until horizontal alignment is achieved.
	Baseplate not leveled properly, probably twisted.	Determine which corner(s) of the baseplate are high or low and remove or add shims at the appropriate corner(s) and realign.
Cannot obtain vertical (Top to Bottom) alignment, angular or parallel	Baseplate not leveled properly, probably bowed.	Determine if center of baseplate should be raised or lowered and correct by evenly adding or removing shims at the center of the baseplate.

GROUT BASEPLATE

1. Clean areas of baseplate that will contact grout. Do not use an oil-based cleaner because grout will not bond to it.
2. Build a dam around foundation (Fig. 20). Thoroughly wet foundation.
3. Pour grout slowly through grout holes in baseplate, until level with the top of the dam. The use of non-shrink epoxy grout is recommended, follow manufacturers recommendations. If cementitious grout is used, remove air by puddling or with a vibrator (Fig. 20).

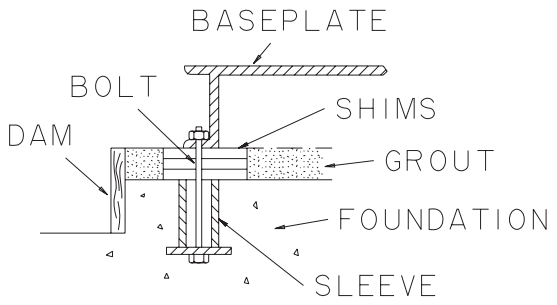


Fig. 20

4. Allow grout to set.
5. Fill remainder of baseplate with grout. Remove air as before (Fig. 21).

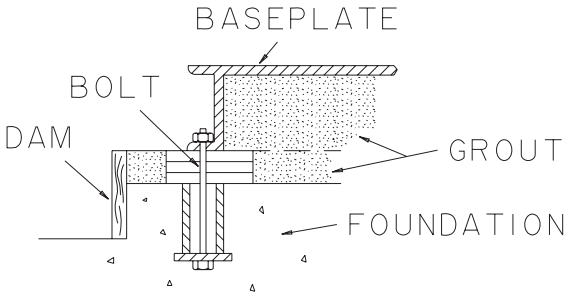


Fig. 21

6. Allow grout to set at least 48 hours.
7. Tighten foundation bolts.

Alignment Check
Re-check alignment before continuing, using methods previously described.

PIPING

Guidelines for piping are given in the "Hydraulic Institute Standards" (Edition 14, Centrifugal Pump section) and should be reviewed prior to pump installation.

WARNING

Never draw piping into place by forcing at the flanged connections of the pump. This will impose dangerous strains on the unit and cause misalignment between pump and driver. Pipe strain can adversely effect the operation of the pump. That could result in serious personal injury and damage to equipment.

Flange loads from the piping system, including those from thermal expansion of the piping, must not exceed the limits of the pump. Casing deformation can result in contact with rotating parts and result in excess heat generation, sparks and premature failure.

Ensure that pump and systems are free of foreign objects before operating and that objects cannot enter the pump during operation. Foreign objects in the pumpage or piping system can cause blockage of flow which can result in excess heat generation, sparks and premature failure

1. All piping must be supported independently and must line up naturally with the pump flanges.
2. Piping runs shall be designed to minimize friction losses.
3. DO NOT make final connection of piping to pump until grout has hardened and pump and driver hold-down bolts have been tightened.

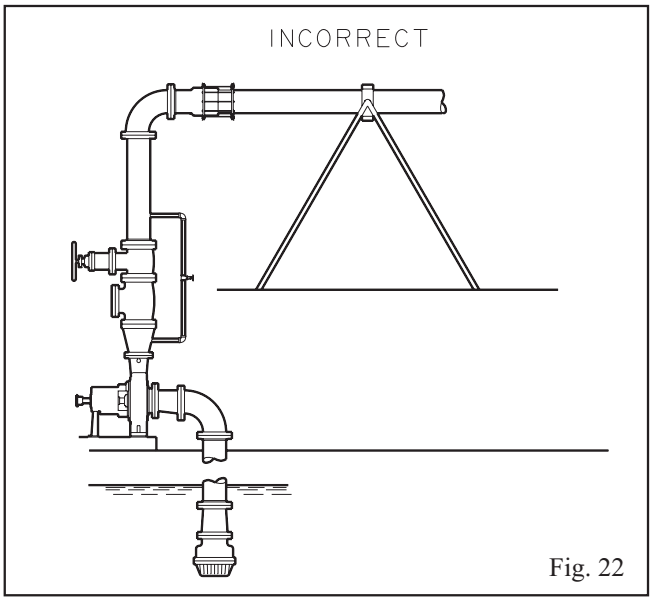


Fig. 22

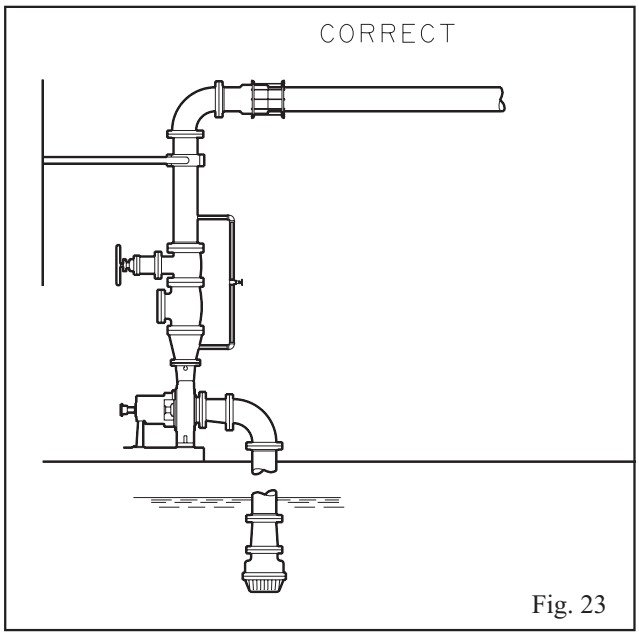


Fig. 23

4. It is suggested that expansion loops or joints, if used, be properly installed in suction and/or discharge lines when handling liquids at elevated temperatures, so linear expansion of piping will not draw pump out of alignment (Fig. 22 & 23).
5. The piping should be arranged to allow pump flushing prior to removal of the unit on services handling corrosive liquids.
6. System should be thoroughly cleaned prior to installation.

SUCTION PIPING

WARNING

$NPSH_A$ must always exceed $NPSH_R$ as shown on Goulds performance curves received with order. Reference Hydraulic Institute for NPSH and pipe friction values needed to evaluate suction piping.

Properly installed suction piping is a necessity for trouble free pump operation. Suction piping should be flushed **BEFORE** connection to the pump.

1. Use of elbows close to the pump suction flange should be avoided. There should be a minimum of 2 pipe diameters of straight pipe between the elbow and suction inlet. Where used, elbows should be long radius (Fig. 24).

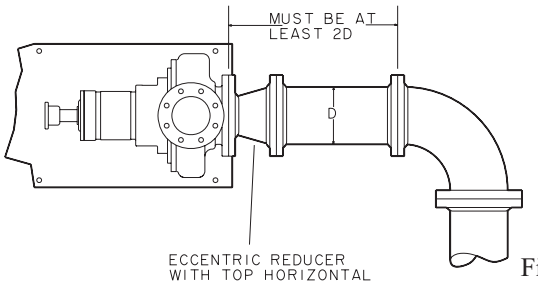


Fig. 24

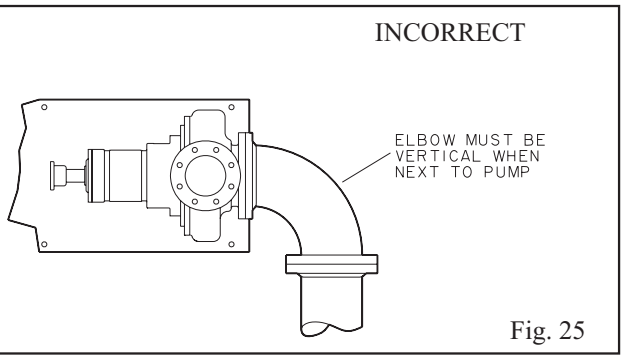


Fig. 25

2. Use suction pipe one or two sizes larger than the pump suction, with a reducer at the suction flange. **Suction piping should never be of smaller diameter than the pump suction.**
3. Reducers should be eccentric at the pump suction flange with sloping side down and horizontal side at the top (Figs. 26, 27, 28).

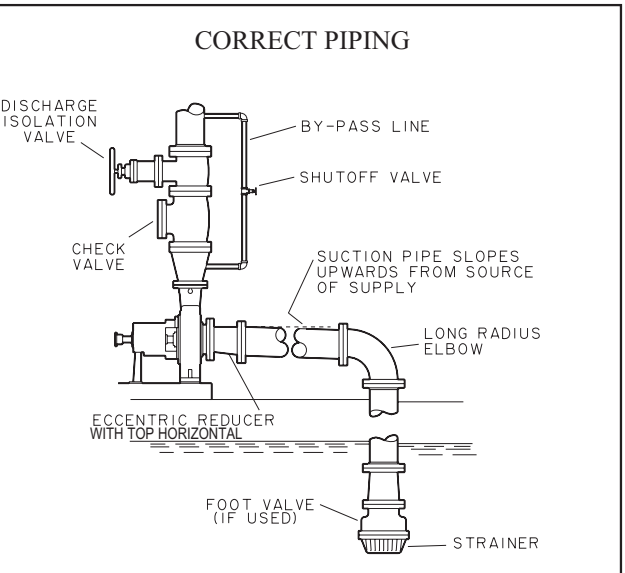


Fig. 26

CAUTION

Pump must never be throttled on suction side.

4. Suction strainers, when used, must have a net "free area" of at least three times the suction pipe area.
5. Separate suction lines are recommended when more than one pump is operating from the same source of supply.

Suction Lift Conditions

1. Suction pipe must be free from air pockets
2. Suction piping must slope upwards to pump.
3. All joints must be air tight.
4. A means of priming the pump must be provided, such as a foot valve.

Suction Head/Flooded Suction Conditions

1. An isolation valve should be installed in the suction line at least two pipe diameters from the suction to permit closing of the line for pump inspection and maintenance.

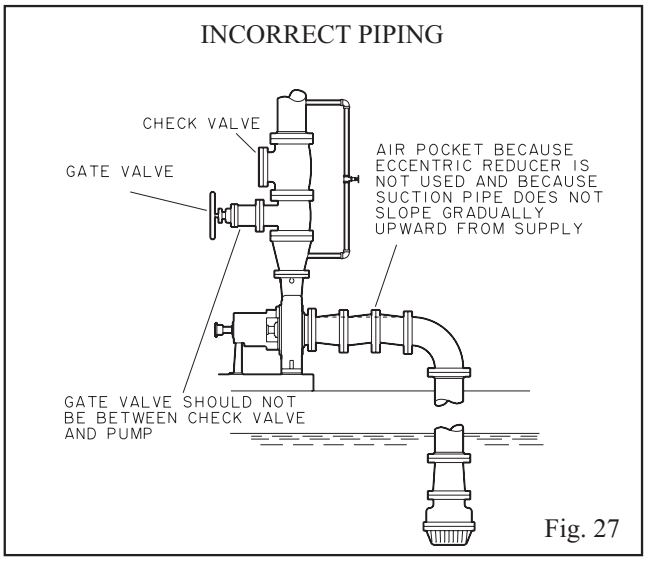


Fig. 27

CORRECT PIPING

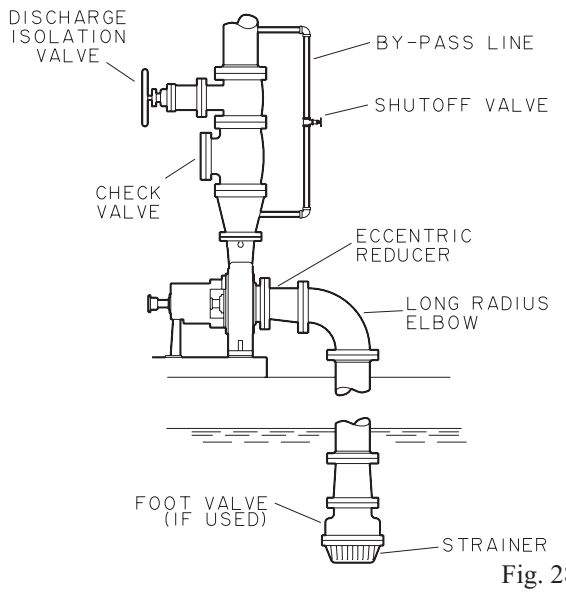


Fig. 28

2. Keep suction pipe free from air pockets.
3. Piping should be level or slope gradually downward from the source of supply
4. No portion of the piping should extend below pump suction flnge.
5. The size of entrance from supply should be one or two sizes larger than the suction pipe.
6. The suction pipe must be adequately submerged below the liquid surface to prevent vortices and air entrainment at the supply..

DISCHARGE PIPING

1. Isolation and check valves should be installed in discharge line. Locate the check valve between isolation valve and pump, this will permit inspection of the check valve. The isolation valve is required for priming, regulation of flow, and for inspection and maintenance of pump. The check valve prevents pump or seal damage due to reverse flow through the pump when the driver is turned off.
2. Increasers, if used, should be placed between pump and check valves.
3. Cushioning devices should be used to protect the pump from surges and water hammer, if quick-closing valves are installed in system.

FINAL PIPING CHECK

After Connecting Piping to the Pump

Rotate shaft by hand to ensure it rotates smoothly and there is no rubbing which could lead to excess heat generation and or sparks.

1. Rotate shaft several times by hand to be sure that there is no binding and all parts are free.
2. Check alignment, per the alignment procedure outlined previously to determine absence of pipe strain. If pipe strain exists, correct piping.

A build up of gases within the pump, sealing system and or process piping system may result in an explosive environment within the pump or process piping system. Ensure process piping system, pump and sealing system are properly vented prior to operation.

OPERATION

PREPARATION FOR START-UP

EZMAG

WARNING

Failure to properly lift and support equipment could result in serious injury or damage to pumps.

When installing in a potentially explosive environment, ensure that the motor is properly certified.

CHECKING ROTATION

CAUTION

Serious damage may result if pump is run in the wrong rotation.

1. Lock out power to driver.

WARNING

Lock out driver power to prevent accidental start-up that could result in serious personal injury.

2. Make sure coupling spacer is removed and coupling hubs are fastened tightly to the shafts and are not loose.

NOTE: Pump is shipped with coupling spacer removed.

3. Unlock driver power.
4. Make sure everyone is clear. Jog driver just long enough to determine direction of rotation. Rotation must correspond to arrow on bearing frame.
5. Lock out power to driver.

CHECKING ROTATION - Close Coupled

CAUTION

Serious damage may result if pump is run in the wrong rotation.

1. Unlock driver power.
2. Make sure everyone is clear. Jog driver just long enough to determine direction of rotation. Rotation of motor fan must correspond to arrow on close coupled frame.

COUPLE PUMP AND DRIVER

WARNING

Lock out driver power to prevent accidental start-up that could result in serious personal injury.

Lubricate coupling per manufacturer's instructions and install coupling spacer.

INSTALL COUPLING GUARD

Install coupling guard as defined in the appendix.

WARNING

Never operate a pump without a coupling guard properly installed. Operating pump without a properly installed coupling guard can result in serious personal injury.

LUBRICATING BEARINGS

WARNING

Operation of the unit without proper lubrication will cause bearing failure and pump seizure.

Flood Oil Lubrication - Pumps are shipped without oil. Fill bearing frame with oil, through filler connection until oil level reaches center of sight-glass (Fig 29). A high quality turbine type oil, with rust and oxidation inhibitors should be used as specified in *Table 6*.

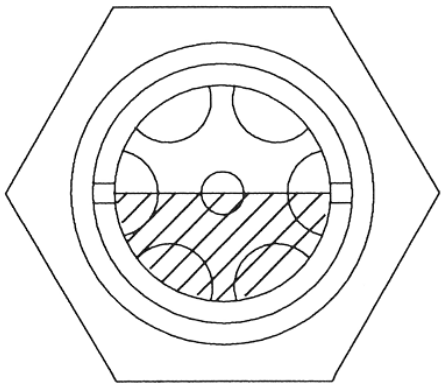


Fig. 29

CONNECT CONDITION MONITORING DEVICES

When installing in a potentially explosive environment, ensure that the condition monitoring devices are properly certified.

Always connect thermocouple to control panel and/or temperature switch in driver starter. If unit is also equipped with a power monitor, leak detector, or vibration monitoring system, these must also be connected.

PRIMING PUMP

Bearings must be lubricated properly in order to prevent excess heat generation, sparks and premature failure.

Never start pump until properly primed (pump casing and suction piping are full of liquid). Components such as internal sleeve bearings depend on liquid for lubrication and will quickly fail if run dry.

Your particular system conditions will dictate method used to prime pump.

STARTING PUMP

1. Make sure suction valve and any recirculation or cooling lines are open.
2. Fully close or partially open discharge valve as dictated by system conditions.
3. Start driver.

CAUTION

Immediately observe pressure gauges. If discharge pressure is not quickly attained - stop driver, reprime and attempt to restart.

4. Slowly open discharge valve until the desired flow is obtained.

CAUTION

Continuous operation against closed discharge valve will cause pump to overheat. Overheating the magnetic drive assembly will weaken or ruin the magnets.

WARNING

Continuous operation against closed discharge valve may vaporize liquid creating an explosive hazard due to confined vapor under high pressure and temperature.

OPERATION

4

GENERAL CONSIDERATIONS

Always vary capacity with valve in discharge line. ***NEVER*** throttle flow from suction side.

Driver may overload or magnets de-couple if pumpage specific gravity (density) is greater than originally assumed, or rated flow rate is exceeded.

Always operate the pump at or near the rated conditions to prevent damage resulting from cavitation or recirculation.

CAUTION

Do not operate above rated temperature range of magnets as this will weaken or ruin the magnets.

OPERATING AT REDUCED CAPACITY

WARNING

Do NOT operate pump below minimum rated flows or with discharge valve closed. These conditions may vaporize liquid creating an explosive hazard due to confined vapor under high pressure and temperature.

Service temperature in an ATEX classified environment is limited to the area classification specified on the ATEX tag affixed to the pump (reference* Table 1 *in the* Safety *section for ATEX classifications).

OPERATING UNDER FREEZING CONDITIONS

Exposure to freezing conditions, while pump is idle, could cause liquid to freeze and damage the pump. Liquid inside pump should be drained. Liquid inside cooling coils, if supplied, should also be drained.

Table 4
Temperature Ratings

Magnetic Types	Drive Designation	Rated Temperature
Neodymium Iron NdFe	A,B,C,D,E,F,G,H,I,J,K	356°F (180°C)
Samarium Cobalt SmCo	AA,BB,CC,DD,EE, FF,GG,HH,II,JJ,KK	536°F (280°C)

Table 5
Minimum Flow GPM (m^3/hr) at Speed (rpm)

Group	Pump Size	3500	2900	1750	1450
S	1x1 1/2 -6	23 (5)	15 (4)	11 (3)	8 (2)
	1 1/2 x 3 -6	30 (7)	25 (6)	15 (4)	13 (3)
	2 x 3 -6	56 (12.5)	47 (11)	28 (6)	23 (5)
	1 x 1 1/2-8	10 (2.5)	7 (2)	3 (1)	2 (1)
	1 1/2x 3 -8	34 (8)	29 (7)	17 (4)	14 (3)
S/M	2 x 3 -8	74 (17)	61 (14)	37 (9)	20 (5)
8" M	3 x 4 -7	157 (36)	127 (29)	78 (18)	64 (15)
	3 x 4 -8G	159 (36)	129 (30)	79 (18)	65 (15)
10" M	1 x 2 -10	21 (5)	13 (3)	9 (2)	7 (2)
	2 x 3 -10	78 (18)	65 (15)	38 (9)	31 (7)
	3 x 4 -10	173 (40)	144 (33)	86 (20)	72 (16)

* Based on water with a specific Gravity of 1.0 and Specific Heat of 1.0

SHUTDOWN

1. Slowly close discharge valve.
2. Shut down and lock out driver to prevent accidental rotation.

WARNING

When handling hazardous and/or toxic fluids, skin, eye and respiratory protection are required. If pump is being drained, precautions must be taken to prevent injury or environmental contamination. Pumpage must be handled and disposed of in conformance with applicable environmental regulations.

FINAL ALIGNMENT

Alignment procedures must be followed to prevent unintended contact of rotating parts. Follow coupling manufacturer's installation and operation procedures.

1. Run the unit under actual operating conditions for a sufficient length of time to bring the pump and driver up to operating temperature.
2. Check and reset alignment per alignment procedure outlined earlier.
3. Reinstall coupling guard per instruction in appendix.

PREVENTIVE MAINTENANCE

GENERAL COMMENTS

A routine maintenance program can extend the life of your pump. Well maintained equipment will last longer and require fewer repairs. You should keep maintenance records, this will help pinpoint potential causes of problems. A sample form is in the appendix that can be copied and used for this purpose.

The* Preventive Maintenance *section must be adhered to in order to keep the applicable ATEX classification of the equipment. Failure to follow these procedures will void the ATEX classification for the equipment.

MAINTENANCE SCHEDULE

ROUTINE MAINTENANCE

- Bearing lubrication
- Temperature monitoring
- Vibration analysis
- Discharge pressure

ROUTINE INSPECTIONS

- Check level and condition of oil through sight glass on bearing frame.
- Check for unusual noise, vibration, and bearing temperatures.
- Inspect pump and piping for leaks.

3 MONTH MAINTENANCE

- Check foundation hold down bolts of motor and pump for tightness.
- Change oil per section 5.
- Check alignment per section 3.

YEARLY INSPECTIONS

- Check pump capacity, pressure, and power. If the pump performance does not satisfy your process requirements, the pump should be disassembled and inspected. Worn parts should be replaced.

INSPECTION INTERVALS

Inspection intervals should be shortened appropriately if the pumpage is abrasive and/or corrosive,

or if the environment is classified as potentially explosive.

MAINTENANCE OF BEARINGS

OIL LUBRICATED BEARINGS

Oil level is measured through the sight glass. Oil level must not fall below center of site glass. An increase in level may be noted after start up due to oil circulation within the bearing frame. Change oil after 200 hours for new bearings, thereafter every 4000 operating hours or 6 months, whichever period is shorter. Change oil every 2000 operating hours under severe operating conditions, such as high temperature services [pumpage temperatures in excess of 325°F (160°C)].

We recommend using *Table 5* to help determine your lubricating oil needs.

Throughout this section on bearing lubrication, different pumpage temperatures are listed. If the equipment is ATEX certified and the listed temperature exceeds the applicable value shown in* Table 1 *under* Safety, *then that temperature is not valid. When this situation occurs, please consult with your ITT/Goulds representative.

STANDARD CLOSE-COUPLED PUMP

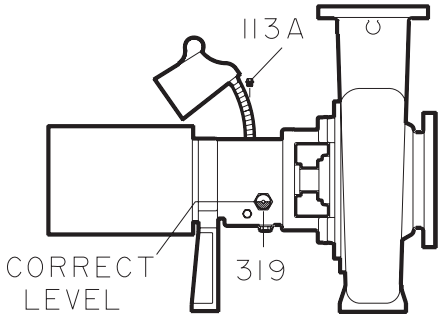
Some 3296s ARE CLOSE-COUPLED MOUNTED AS STANDARD. Close-coupled pumps do not have bearings which require lubrication.

OIL LUBRICATED BEARINGS

WARNING

Pumps are shipped without oil. Oil lubricated bearings must be lubricated at the job site.

Remove fill plug (113A) and add oil until level is at the center of the sight glass (319). Replace fill plug (Fig. 30) (*See Table 6).*



OIL LUBRICATED BEARINGS

Fig. 30

Table 6
Bearing Frame Lubrication Requirement

Oil Grades	Pumpage Temperature of less than 356°F (180°C)		Pumpage Temperature of greater than 356°F (180°C) with bearing frame cooling
ISO Grade	VG 68		Synthetic VG 68
Approximate SSU 100°F (38°C)	300		Synthetic 300
DIN 51517	C 68		Synthetic C68
Kenematic Viscosity at 40°C (105°F) (mm^2/sec)	68		Synthetic 68
Acceptable	Exxon Chevron Mobil Gulf Shell Phillips Phillips Phillips	Teresstic EP 68 GTS Oil 68 DTE 26 Harmony 68 Tellus Oil 68 Mangus Oil 315 MM SAE 20-20W HDS SAE 20-20W	Royal Purple Synfilm 68

Note: This is a list of oils that meet the lubrication requirements of this pump. It is not intended to be an endorsement of products listed nor exclude other oils that meet these requirements.

TROUBLESHOOTING PUMP

Table 7 Troubleshooting Pump		
No liquid delivered	Pump not primed	Reprime pump, check that pump and suction line are full of liquid.
	Suction line clogged	Check suction line presssure. If low, locate and remove obstructions.
	Impeller clogged with foreign material	Disassemble and remove blockage.
	Magnet de-coupling	Shut down. Check temperature and viscosity of pumpage. Check magnets with breakaway torque test.
Pump not producing rated flow or head	Air leak in suction line	Check for leakage and correct.
	Impeller partly clogged	Back flush pump to clean impeller.
	Worn impeller rings	Replace defective part as required.
	Insufficient suction head	Ensure that suction line shutoff valve is fully open and line is unobstructed. Check suction pressure.
	Worn or broken impeller	Inspect and replace if necessary.
Pump starts then stops pumping	Improperly primed pump	Reprime pump.
	Air leak in suction line	Check for leakage and correct.
	Magnet de-coupling	Shut down. Check temperature and viscosity of pumpage. Check magnets with breakaway torque test.
	Air or vapor pockets in suction line	Rearrange piping as necessary to eliminate air pockets.
Bearings run hot	Improper lubrication	Check lubricant for suitability and level.
	Lube cooling	Check cooling system.
	Improper alignment	Check pump alignment.
Pump is noisy or vibrates	Improper pump/driver alignment	Align shafts.
	Partly clogged impeller causing imbalance	Disassemble and remove blockage.
	Broken or bent impeller or shaft	Replace as required.
	Base not rigid enough	Tighten hold down bolts of pump and motor or adjust stilts. Check grout.
	Worn bearings	Replace.
	Suction or discharge piping not anchored or properly supported	Anchor per Hydraulic Institute Standards recommendations (Edition 14, Centrigual pump section).
	Pump is cavitating	Increase NPSH available.
Motor requires excessive power	Head lower than rating. Pumps too much liquid	Install throttle valve.
	Liquid heavier than expected.	Check specific gravity and viscosity.
	Head higher than rating, capacity at rating	Check impeller diameter.
	Rotating parts binding or severly worn	Check internal wearing parts for proper clearances.

TROUBLESHOOTING PUMP, con't

Table 7 Pump Troubleshooting		
Condition monitoring device shuts down pump	Damaged sleeve & thrust bearings	Replace as required.
	Plugged recirculation circuit	Disassemble and remove blockage. Determine and correct cause of blockage.
	Recirculation liquid vaporization	Check actual liquid temperature versus design temperature. Check actual NPSH available versus design. Check minimum flow requirement for pump size. Check recirculation circuit and flush screen for blockage. Correct all as necessary.
	Damaged containment shell	Replace as required.
	Magnets decoupled	Check temperature and viscosity of pumpage. Check magnets with breakaway torque test.
	Pump run dry	Check control device for proper operation. Check suction line for blockage. Reprime pump.
	Excessive motor power	System head lower than rating. Pumps too much liquid. Check rotating parts for binding and wear. Liquid heavier than expected.

DISASSEMBLY & REASSEMBLY

REQUIRED TOOLS

⚠ WARNING

This pump contains extremely strong magnets. Keep magnetic tools away from magnets.

- Assorted metric open end or socket sizes 13, 17, 18, 19 and 24 mm.
- Assorted Allen Head wrenches, sizes 2.5, 3, 5, 6mm with 4.75" minimum reach and 8mm with 6" minimum reach.
- Torque Wrench
- Strap Wrench
- 3/8" Eye-Bolt

PREPARATION FOR DISASSEMBLY

A build up of gases within the pump, sealing system and or process piping system may result in an explosive environment within the pump or process piping system. Ensure process piping system, pump, and sealing system are properly vented prior to operation.

WARNING

The 3296 pump often handles hazardous and/or toxic fluids. Skin, eye and respiratory protection required. Precautions must be taken to prevent injury or environmental contamination. Drain and decontaminate pump in accordance with all federal, state, local, and company regulations.

Leakage of process liquid may result in creating an explosive atmosphere. Follow all pump and seal assembly procedures.

1. Lock out power to driver.
2. Shut off all valves controlling flow to and from pump.
3. The pump should be drained and flushed before it is removed from the piping. After isolating the pump from the system, flush the pump using a compatible liquid.

WARNING

Failure to properly lift and support equipment could result in serious injury or damage to pumps.

WARNING

Ensure pump is isolated from system and pressure is relieved before any plugs are removed or piping disconnected.

4. Disconnect all piping and auxiliary equipment.
5. Remove coupling guard (For frame mounted version).
6. Remove coupling (For frame mounted version).
7. Remove casing foot and frame / C-face motor support foot bolts.
8. Remove pump from baseplate.
9. Drain Oil from frame mounted pump.

Decontamination Procedure

10. Connect clean flush liquid supply to discharge nozzle.
11. Devise a means of collecting flush liquid as it drains out the drain connection.
12. Flush to remove residue.

DISASSEMBLY (Frame Mounted)

WARNING

Each component must be individually decontaminated using procedures in accordance with all federal, state, local and company environmental regulations.

WARNING

The magnets contained in this unit are extremely powerful. Keep magnetic drive components and magnetic tools apart from each other by a minimum of six (6) feet [two (2) meters]. Serious injury to fingers and hands will result.

NOTE: When working on pump, use a bench with a non-magnetic work surface such as wood with a brass surface.

CAUTION

The shop area must be clean and free of any substances that would contaminate the magnets, ex. ferrous metals.

1. Secure pump on a workbench or worktop with the suction nozzle facing down.
2. Screw “eye-bolt,” size 3/8 inch into the drive shaft.

CAUTION

MAGNETIC FORCES: Strong axial forces are produced when pulling the bearing frame out of the frame adapter. These forces diminish abruptly once the assembly has been removed.

3. Remove screws holding the bearing frame to the frame adapter (370B) (Fig. 31).

4. If required, jacking screws can be used to separate the parts using the two threaded holes in the bearing frame. S-Group: M12; M-Group: M14.
5. Lift the bearing frame assembly off the adapter using a crane if available.
6. Remove the frame-to-adapter gasket (360W) (Fig. 31).
7. Secure the drive shaft so it is not able to rotate.

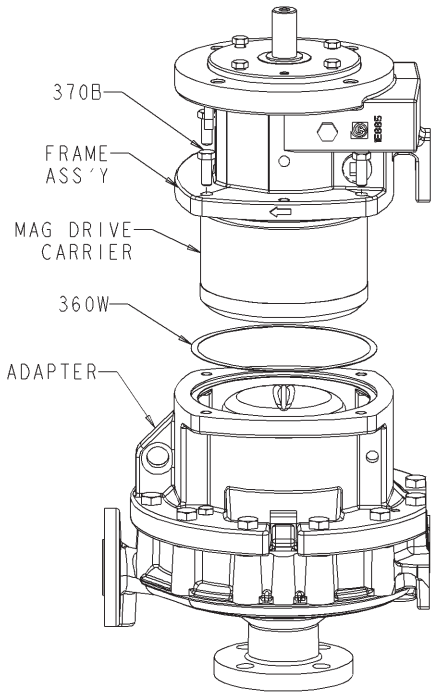


Fig. 31

8. Remove hex screw (791D) and lockwasher (382) from the drive shaft (Fig. 32).
9. Remove drive magnet assembly (740B) (Fig. 32).

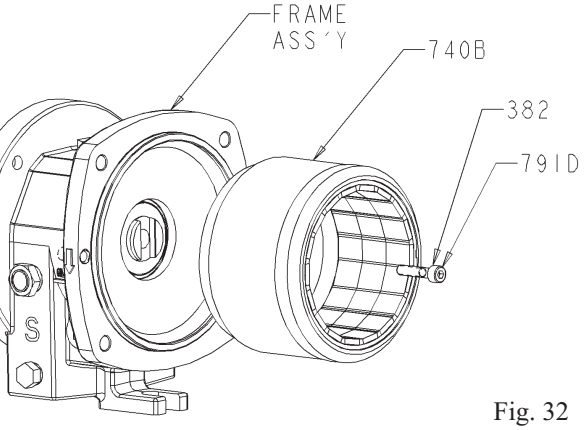


Fig. 32

10. Remove bearing end cover screws (370C) and bearing end cover (109A) (Fig. 33).
11. Remove wavy spring washer (529) and end cover gasket (360A) (Fig. 33).

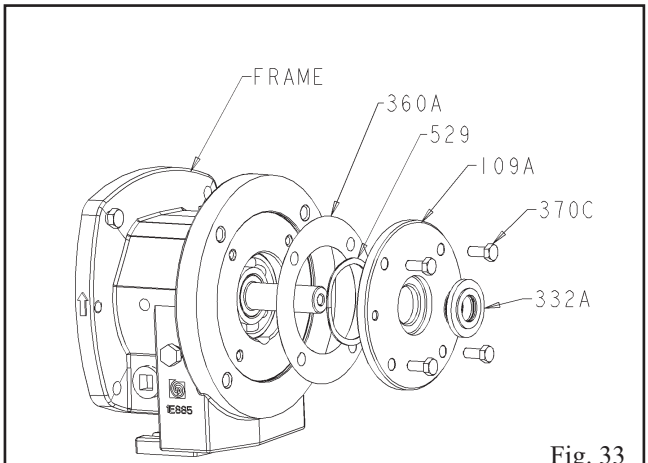


Fig. 33

12. Remove drive shaft with both bearings attached(Fig. 34).
13. Remove labyrinth oil seals (332A and 333D) (Fig. 33 and 34).

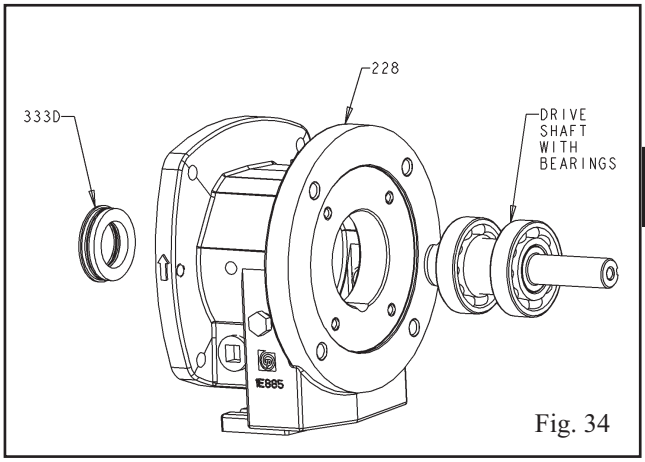


Fig. 34

14. Both radial ball bearings lie against the shaft collar so remove them using a press (Fig. 34).

6

15. Remove hex cap screws, adapter to case (370) (Fig. 35).

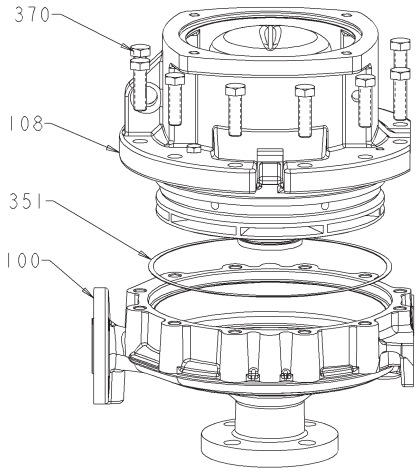


Fig. 35

16. Do not remove the three set screws (222E on the 6" S-Group) or two hex screws (370V on all other sizes). They hold the adapter (108), backplate (444) and containment shell (750) (Fig. 36).

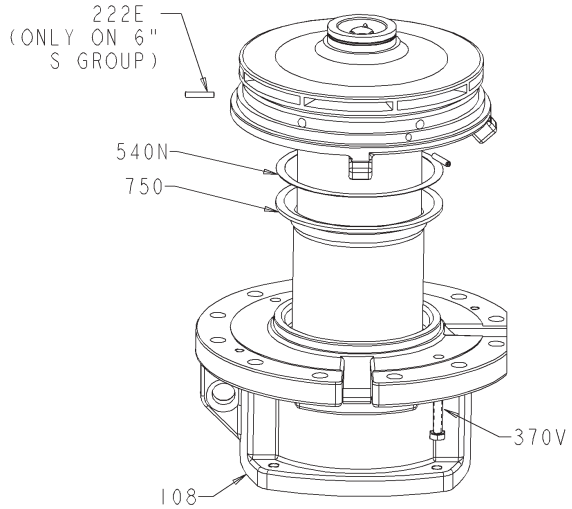


Fig. 36

17. Remove this entire assembly from the casing (100) (Fig. 35).
18. If required to remove this assembly from the casing, jacking screws can be used through the threaded holes in the adapter (108). S and M Groups: M8.
19. Place adapter, backplate, impeller assembly on the bench with the impeller facing upwards (Fig. 36).
20. Remove the three set screws, (222E) (6" S-Group) or two hex screws, 370V (all other sizes) (Fig. 36).
21. Remove adapter (108) and containment shell (750) (Fig. 36).
22. Place remaining unit on the workbench with the driven magnet facing downwards (Fig 37).
23. Place strap-wrench on the impeller and remove the impeller nut (304) and o-ring (412A) (Fig. 37).
24. Slide the impeller (101) off the shaft (Fig. 37).
25. Remove the impeller key (178), distance washer (199) and gasket (351X) (Fig. 37).

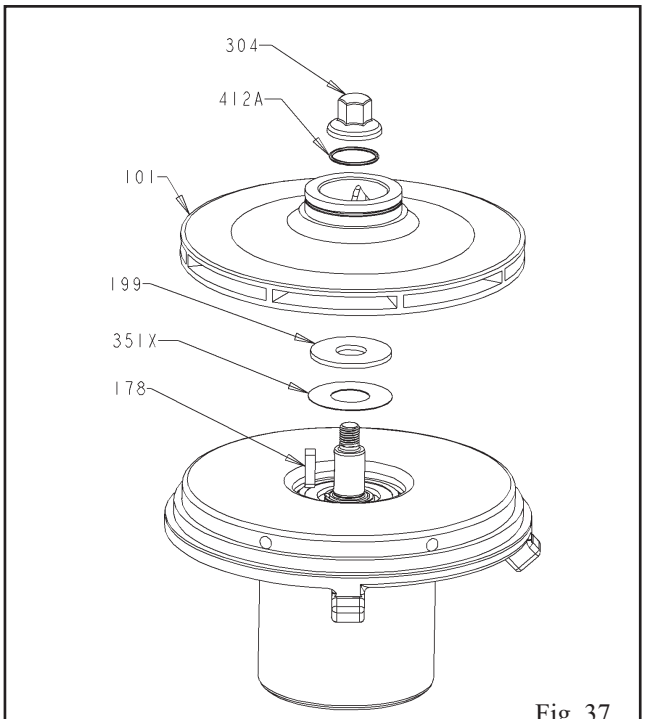


Fig. 37

26. Pull the backplate (444) and bearing cartridge (849) from the shaft (Fig. 38).
27. Remove second gasket (351X) (Fig. 38).
28. Undo screws (791E) and remove the bearing cartridge (849) from the backplate (444) (Fig. 38).

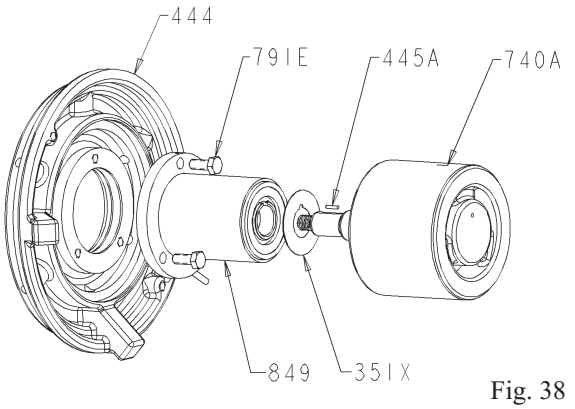


Fig. 38

29. The driven magnet assembly (740A) is a two-piece assembly on S-group pumps held with a drive-key. Item 740A is a single-piece component on M-Group pumps, with a parallel pin (445A) to drive the bearing cartridge.
30. The bearing cartridge (849) is one unit which, if necessary, is replaced completely.

DISASSEMBLY (Close-Coupled)

CAUTION

MAGNETIC FORCES: Strong axial forces are produced when pulling the drive section out of the frame adapter. These forces diminish abruptly once the assembly has been removed.

1. Secure the complete pump on a workbench in either horizontal or vertical position.
2. Remove hex screws (370B) holding the motor adapter (503) to the frame adapter (108) (Fig. 39).
3. If necessary, remove the motor adapter (503) from the frame adapter (108) using two levers or pry-bars. There are also two threaded holes (size M12) in the close-coupled motor adapter (503) for the use of jacking screws.
4. Lift the motor and motor adapter assembly off the frame adapter with a crane if necessary (Fig. 39).
5. Remove gasket (360W) (Fig. 39).
6. Remove hex screw (791D) and lockwasher (382) from the drive magnet (740B) (Fig. 39).

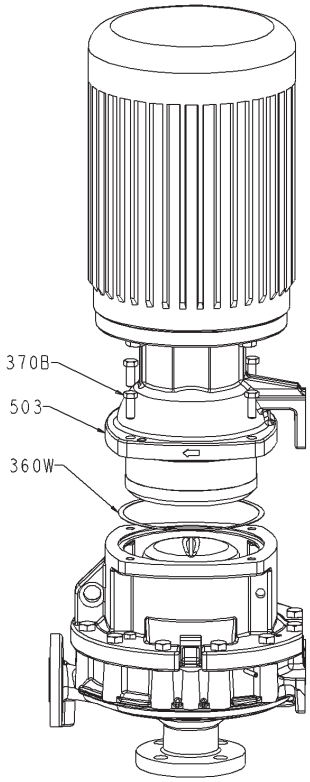


Fig. 39

7. Remove the drive magnet (740B) from the stub shaft (122A) (Fig. 40).
8. Remove the hex screws (371) holding the motor to the motor adapter (503) (Fig. 40).
9. Pull the motor adapter (503) off the motor flange (Fig. 40).
10. Undo the set-screw (222L) that holds the stub shaft onto the motor shaft (Fig. 40).
11. Remove the stub shaft (122A) from the motor (Fig. 40).

Refer to Step 15 from above for disassembly of the remaining pump.

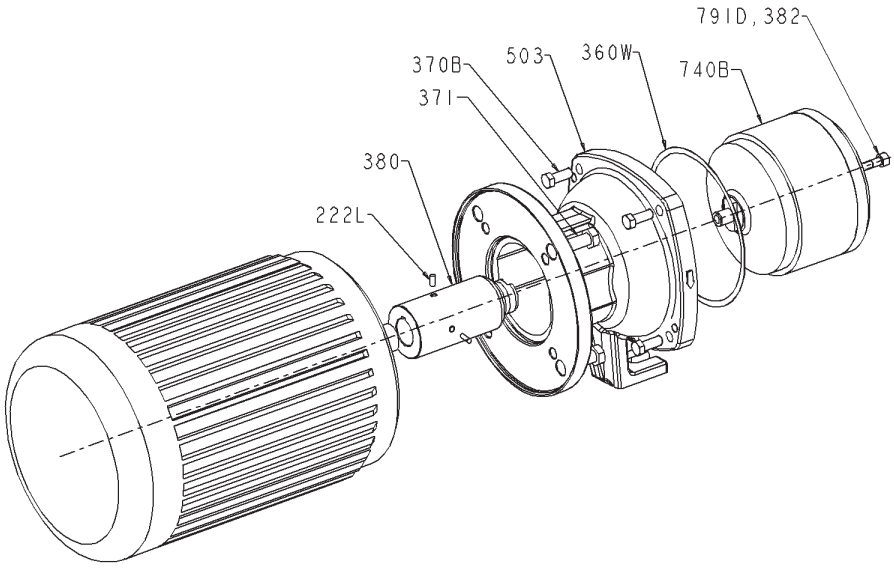


Fig. 40

INSPECTIONS

The pump parts must be inspected to the following criteria before they are reassembled to insure the pump will run properly. Any part not meeting the required criteria should be replaced.

NOTE: Clean parts in solvent to remove oil, grease or dirt. Protect machined surfaces against damage during cleaning.

Casing (100)

The casing (100) should be inspected for cracks and excessive wear or pitting. It should be repaired or replaced if it exceeds the following criteria (Fig. 41).

1. Localized wear or grooving greater than 1/8 in. (3.2 mm) deep.
2. Pitting greater than 1/8 in. (3.2 mm) deep.
3. Inspect case gasket seat surface for irregularities.

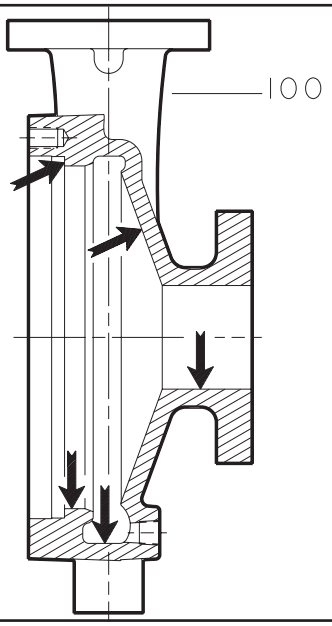


Fig. 41

4. Refer to *Table 8* to check wear ring clearances.

Table 8

Radial Wear Ring Clearance Impeller to Casing in (mm)

Group	Size	New	Replace
S	1x1.5-6	.010—.013 (.25—.32)	0.018 (0.44)
	1.5x3-6		
	2x3-6		
	1x1.5-8		
	1.5x3-8		
	2x3-8		
M	3x4-7	.014—.016 (.35—.42)	0.022 (0.59)
	4x4-8G		
	3x4-10		
	1x2-10		
	2x3-10		

Impeller (101)

1. Inspect wear ring surface for signs of pitting.
2. Inspect front wear ring clearance per wear ring clearance *Table 8*.
3. Inspect leading and trailing edges of vanes for pitting, and erosion or corrosion damage.

Frame Adapter (108)

1. Check frame adapter (108) for cracks or excessive corrosion damage. Replace if any of these conditions exist.
2. Make sure gasket surface is clean.

Silicon Carbide Bearings (849)

(Bearing Cartridge)

1. Inspect bearings for cracks , chips, or excessive wear.
2. Replace cartridge if any of these conditions exist.

Containment Shell (750)

1. Wall thickness 0.039 in. minimum.
2. Must be free from pitting or cracks.
3. Grooves in excess of .005 in. require containment shell replacement.

Magnets (740A & 740B)

Driven Magnet Assembly (740A)

WARNING

The magnets contained in this unit are extremely powerful. Keep magnetic drive components and magnetic tools apart from each other by a minimum of six (6) feet [two (2) meters]. Serious injury to fingers and hands will result otherwise.

1. Must be free from bulges.
2. Must be free of pits and scratches exceeding .005 in. deep.
3. Must be free of erosion or corrosion exceeding .005 in. deep.
4. Check pump-out vanes for cracks or corrosion.
5. Ensure circulation holes are open.

Drive Magnet Assembly (740B)

WARNING

The magnets contained in this unit are extremely powerful. Keep magnetic drive components and magnetic tools apart from each other by a minimum of six (6) feet [two (2) meters]. Serious injury to fingers and hands will result otherwise.

NOTE: The magnets are extremely brittle. It is normal to have chips (up to 10% of the magnet surface) per MMPA Standard No. 0100-90.

1. Magnets must be free of major cracks (extending over 50% of surface) and also free of imperfections that create loose particles.
2. If magnets and drive magnet carrier were exposed to product, they should be replaced.
3. Inspect drive magnet carrier for cracks and replace if any are found.
4. Drive magnet carrier hub O.D. must be free from grooves and scratches greater than .005 in.
5. Inspect magnets for proper bonding to metal carrier.

Bearing Frame (228)

1. Visually inspect frame and frame foot for cracks.
2. Inspect for corrosion or pitting if frame has been exposed to pumpage.
3. Inspect frame bearing bores. The maximum acceptable bore is 2.836 in. (S-Group) and 3.544 in. (M-Group).
4. Inspect ball bearings for containment and damage.
5. Make sure gasket surfaces are clean.
6. Visually inspect bearing end cover (109A) for cracks and pits. Gasket surface must be clean.
7. Inspect labyrinth seal O-rings (332A, 333D) for cuts and cracks.
8. Replace labyrinth seals if needed.

ASSEMBLY (Frame Mounted)

1. Press both radial ball bearings (112) onto the drive shaft (122B) (Fig. 42).

NOTE: There are several methods used to install bearings. The preferred method is to use an induction heater that heats as well as de-magnetizes the bearings.

2. Install the pre-assembled drive shaft into the bearing frame (228) from the motor side (Fig. 42).

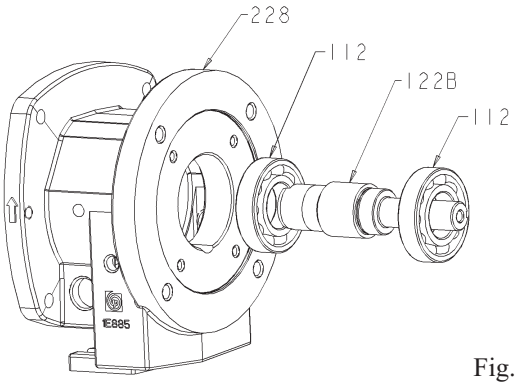


Fig. 42

3. Insert wavy washer (529) (Fig. 43).
4. Insert end cover gasket (360A) into the bearing frame (Fig. 43).
5. Mount bearing end cover (109A) using the hex screws (370C) (Fig. 43).

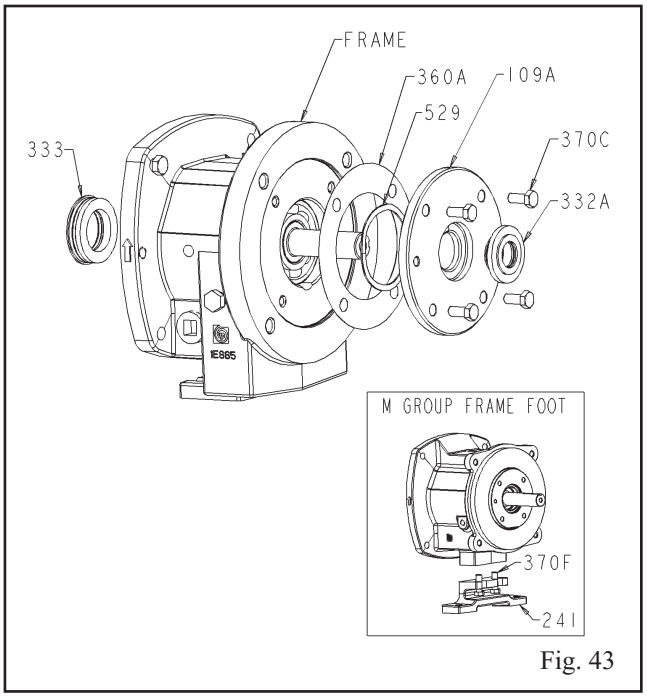


Fig. 43

6. Press inboard labyrinth seal (333D) and outboard labyrinth seal (332A) into the corresponding bores on the bearing frame. Press them until they are fully seated in the bore. Make sure to keep the oil return slot on the inside of the seal at the 6 o'clock position (Fig. 43).
7. Mount bearing frame foot (241) with hex screws (370F) and lock-washers (M-Group only) (Fig. 43).

8. Clamp the pre-assembled bearing frame assembly in a vice with the motor end of the shaft facing downwards (Fig. 44).

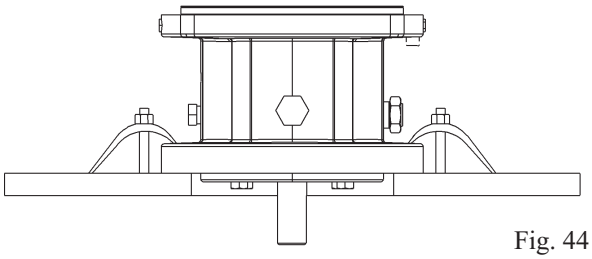


Fig. 44

9. Mount the drive magnet assembly (740B) onto the drive shaft so that the driver cams engage. Secure the drive magnet assembly using the lock- washer (382) and hex screw (791D) (Fig. 45).

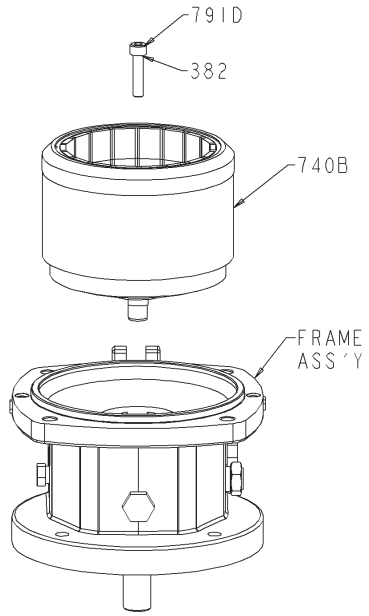


Fig. 45

10. Insert the bearing cartridge (849) into the backplate (444) (Fig. 46).

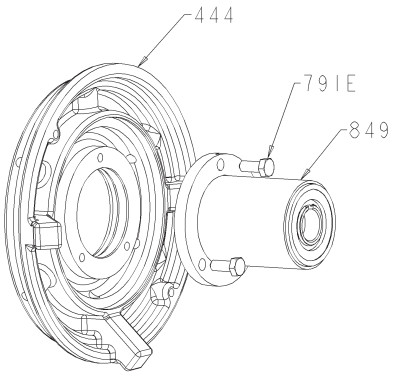


Fig. 46

11. Rotate the bearing cartridge until all three holes line up and secure with hex screws (791E) (Fig.46). Recommended torque values are shown in *Table 9*.

Table 9
***Bolt Torque Table**
Ft-Lbs (Nm)

Description	Group	Ft-lbs (Nm) Dry	Ft-lbs (Nm) Std. Lube
Adapter to casing screws (Item 370)	S/M (M12	65 (88)	49 (66)
	10" M (M16)	161 (219)	120 (164)
Impeller Nut (Item 304)	S	47 (64)	26 (35)
	M	116 (158)	52 (70)
Cartridge to Backplate Screws (Item 791E)	S/M	12 (16)	9 (12)
*Tighten in diametrically opposite sequence.			

12. If the driven carrier pin (445A) in the driven magnet assembly (740A) has to be replaced, drive it in carefully until it protrudes approximately 0.12" towards the impeller (M-Group only) (Fig. 47). The S-Group has a drive key, which is automatically positioned when installed.

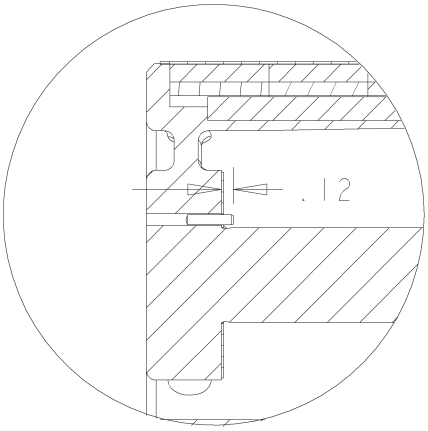


Fig. 47

13. Cut out a small notch on the inside diameter of the intermediate ring gasket (351X) so that a recess for the driven carrier pin (445A) or key (depending on size) is produced (Fig. 48)

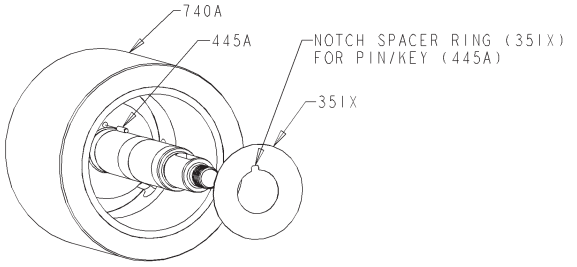


Fig. 48

14. Apply some anti-seizing compound to the shaft and shaft threads.
15. Mount the intermediate ring gasket (351X) on to the shaft of the driven magnet assembly (740A) (Fig. 49).
16. Place the driven magnet assembly (740A) on the workbench and mount the pre-assembled bearing cartridge (849) and backplate (444) assembly onto the driven magnet assembly from above. Make sure the driven carrier pin or key (445A) engages the carrier groove in the bearing cartridge (849). The backplate can be turned slightly to help with alignment (Fig. 49).

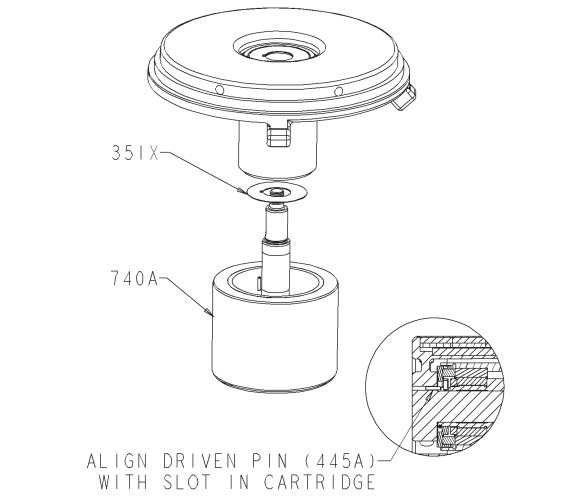


Fig. 49

17. Mount the second intermediate ring gasket (351X) and distance washer (199) onto the drive shaft as shown (Fig. 50).
18. Insert impeller key (178) into the key-slot on the shaft (Fig. 50).
19. Mount the impeller (101) onto the shaft (Fig. 50).
20. Insert the impeller nut o-ring (412A) into the groove on the backside of the impeller nut (304) (Fig. 50).

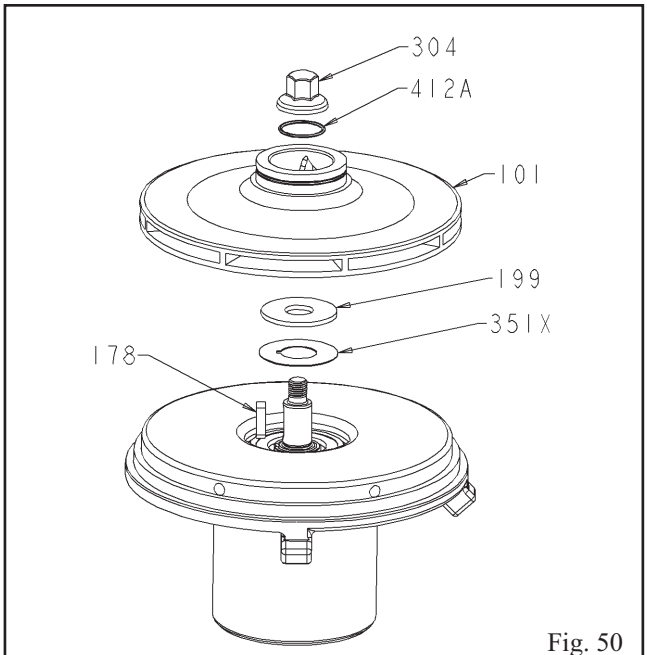


Fig. 50

21. Secure the driven magnet assembly with a strap wrench and tighten the impeller nut to the torque rating specified in *Table 9*.

NOTE: At this point, the backplate (444) should rotate freely and easily by hand. When raising the backplate, a slight amount of axial play (up to approximately 0.040in) must be felt to ensure proper assembly. The axial play of the plain bearings is automatically set during assembly.

22. Place the containment shell gasket (540N) and containment shell (750) onto the backplate (444) (Fig. 51).
23. Screw either the three set screws (222E for the S-Group) or two connection screws (370V for the M-Group) of the adapter (108) into the backplate and tighten (Fig. 51). This holds the containment shell in place.
24. Secure the casing (100) to the workbench with the suction flange facing downwards (Fig. 52).

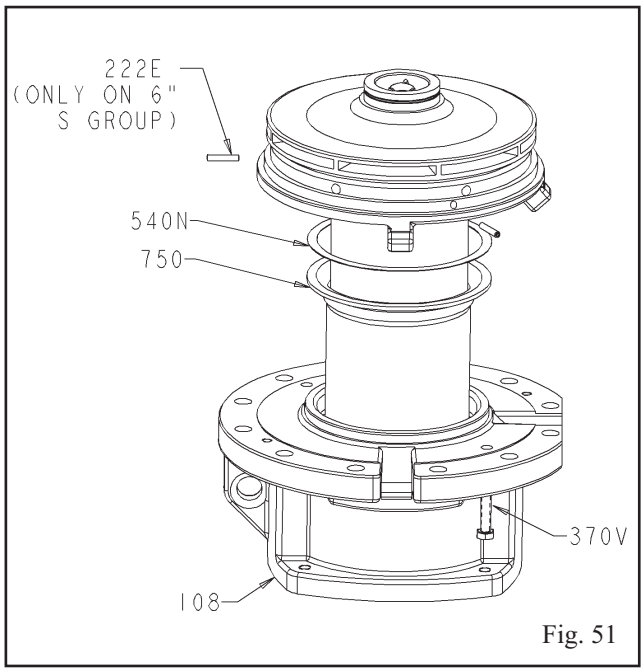


Fig. 51

25. Insert the casing gasket (351) into the casing (Fig. 52).
26. Insert the pre-assembled unit described above into the casing so that the crane hook of the adapter (108) faces the center of the discharge nozzle (Fig. 52).
27. Secure the adapter to the casing using hex cap screws (370) to the recommend torque rating specified in *Table 9* (Fig. 52).

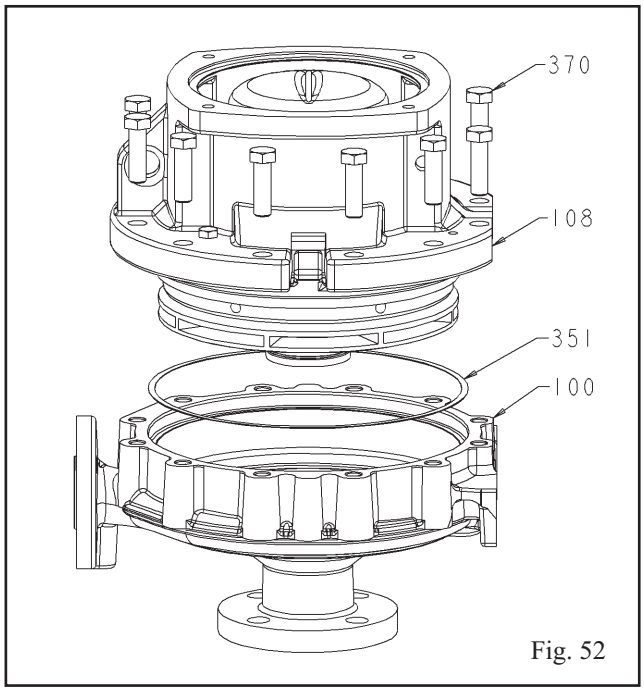


Fig. 52

28. Insert the gasket (360W) into the adapter (Fig. 53).
29. Place the bearing frame – drive magnet assembly already assembed on the workbench with the drive magnet facing downwards.
30. Screw a commercially available eye-bolt into the end of the drive shaft, size 3/8 inch.
31. Place the bearing frame assembly onto the adapter using a crane (Fig. 53).
32. Screw in hex cap screws (370B) to secure the bearing frame to the adapter (Fig. 53).
33. Turn the drive shaft by hand to ensure free rotation. Check by looking into the suction nozzle to make sure the impeller is rotating.

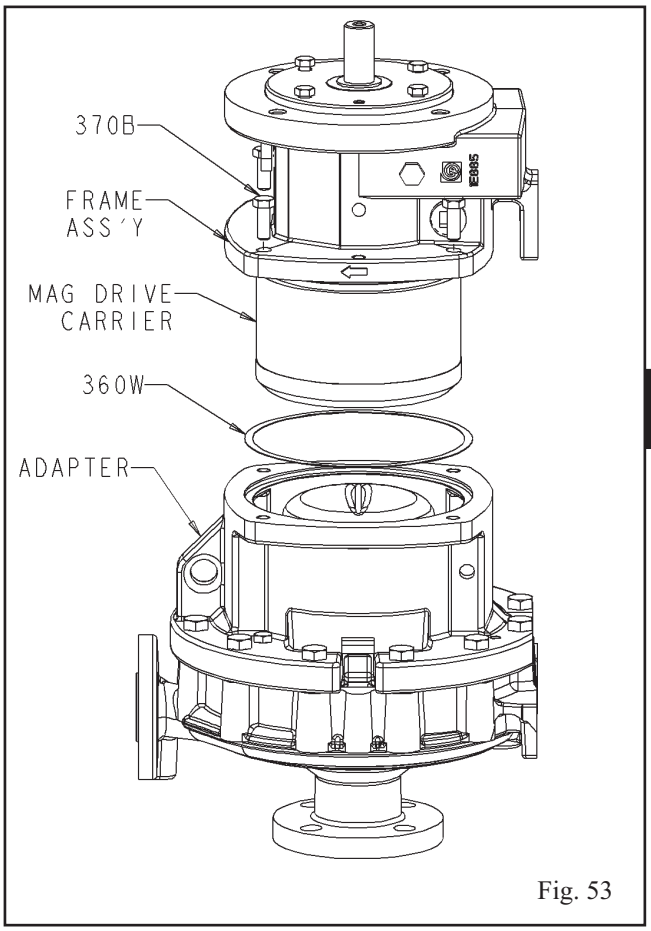


Fig. 53

ASSEMBLY (Close-Coupled)

1. Slide the stub shaft (122A) onto the motor shaft and secure it with the set screw (222L) (Fig. 54).
2. Mount the motor adapter (503) onto the motor using hex screws (371) (Fig. 54).
3. Mount the drive magnet (740B) onto the stub shaft (122A) so that the driver cams are fully engaged (Fig. 55).
4. Insert the lock-washer (382) and cap screw (791D) to secure the magnet to the shaft (Fig. 55).

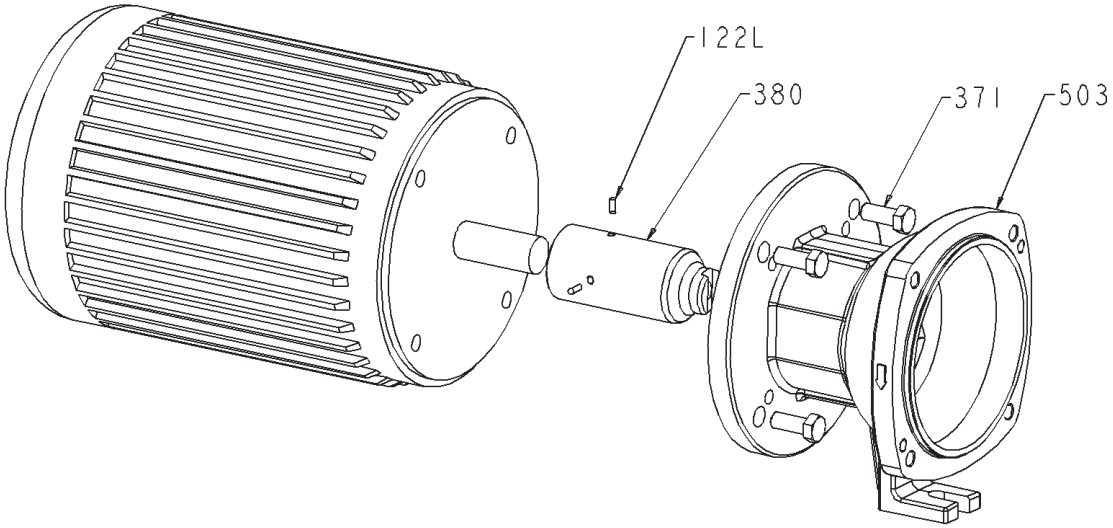


Fig. 54

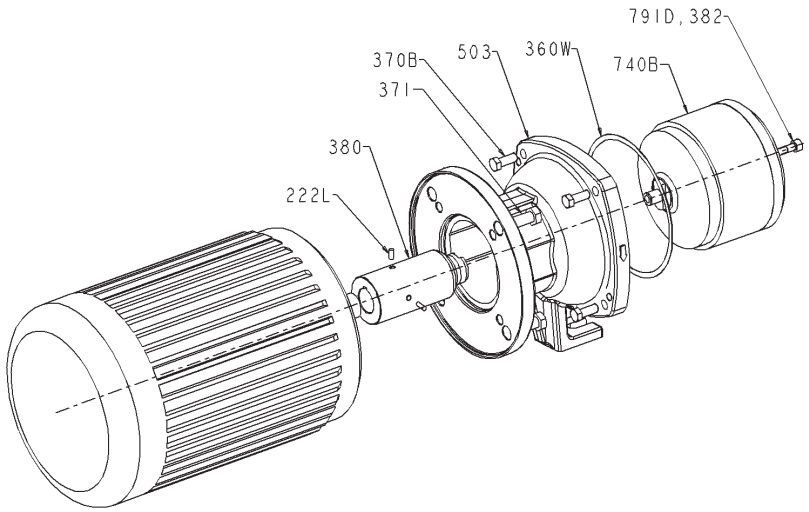


Fig. 55

Refer to Step 10 from above for assembly of the remaining pump.

Fig. 56 shows the final close-coupled pump assembly.

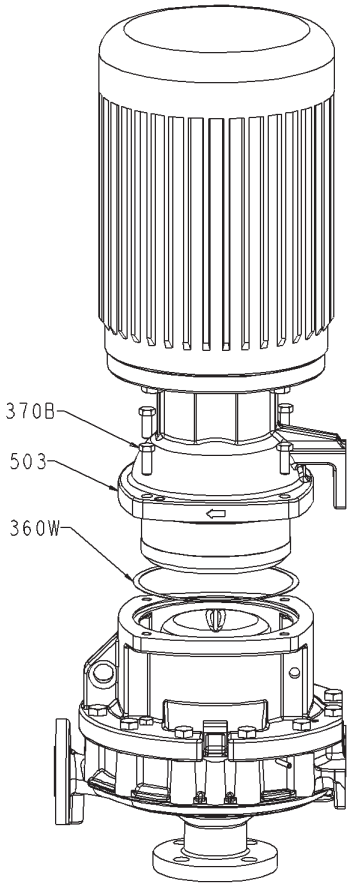
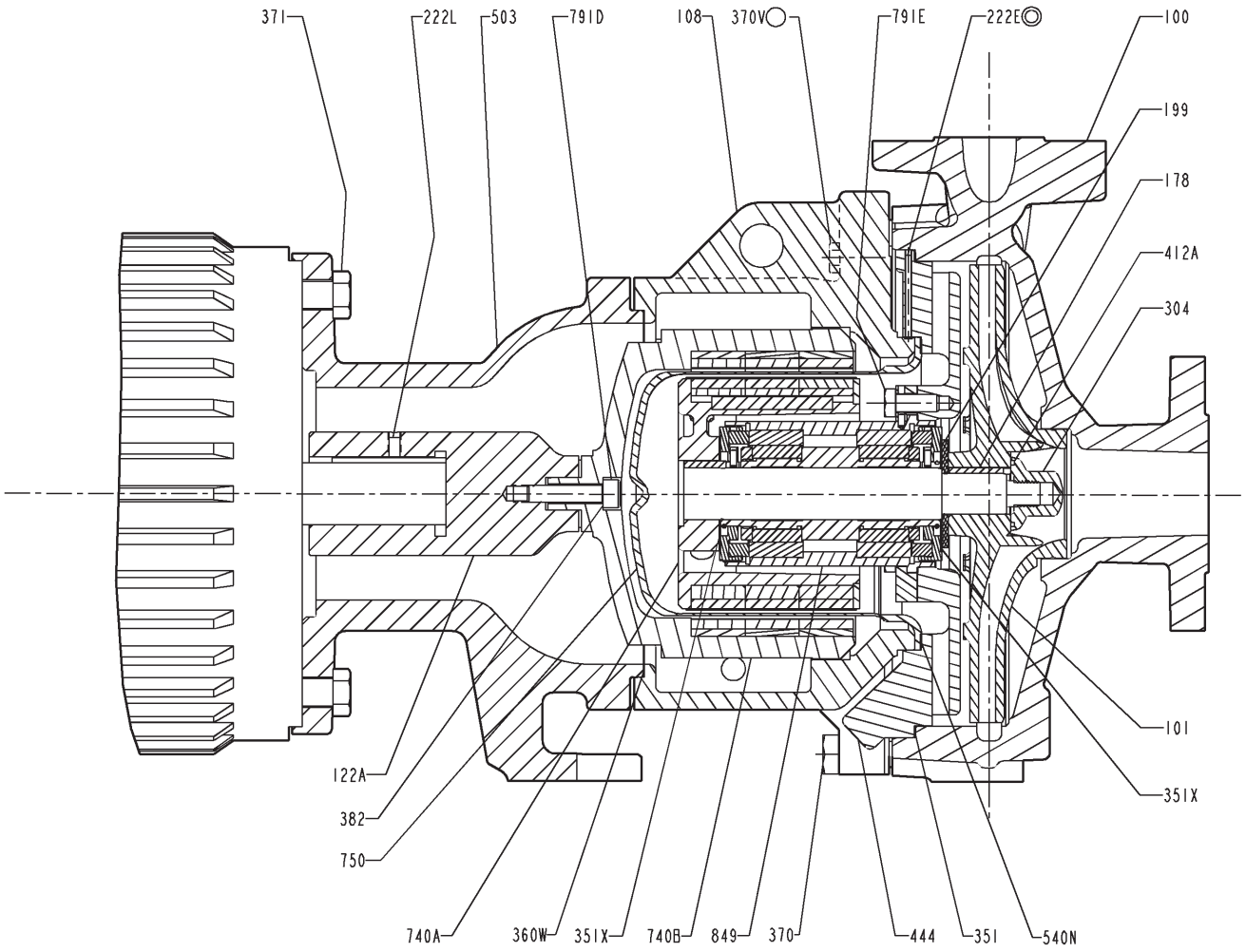


Fig. 56

Cross Sectional Assembly Model 3296 EZMAG
Close Coupled S Group & 2x3-8 M Group
Stainless Steel Construction

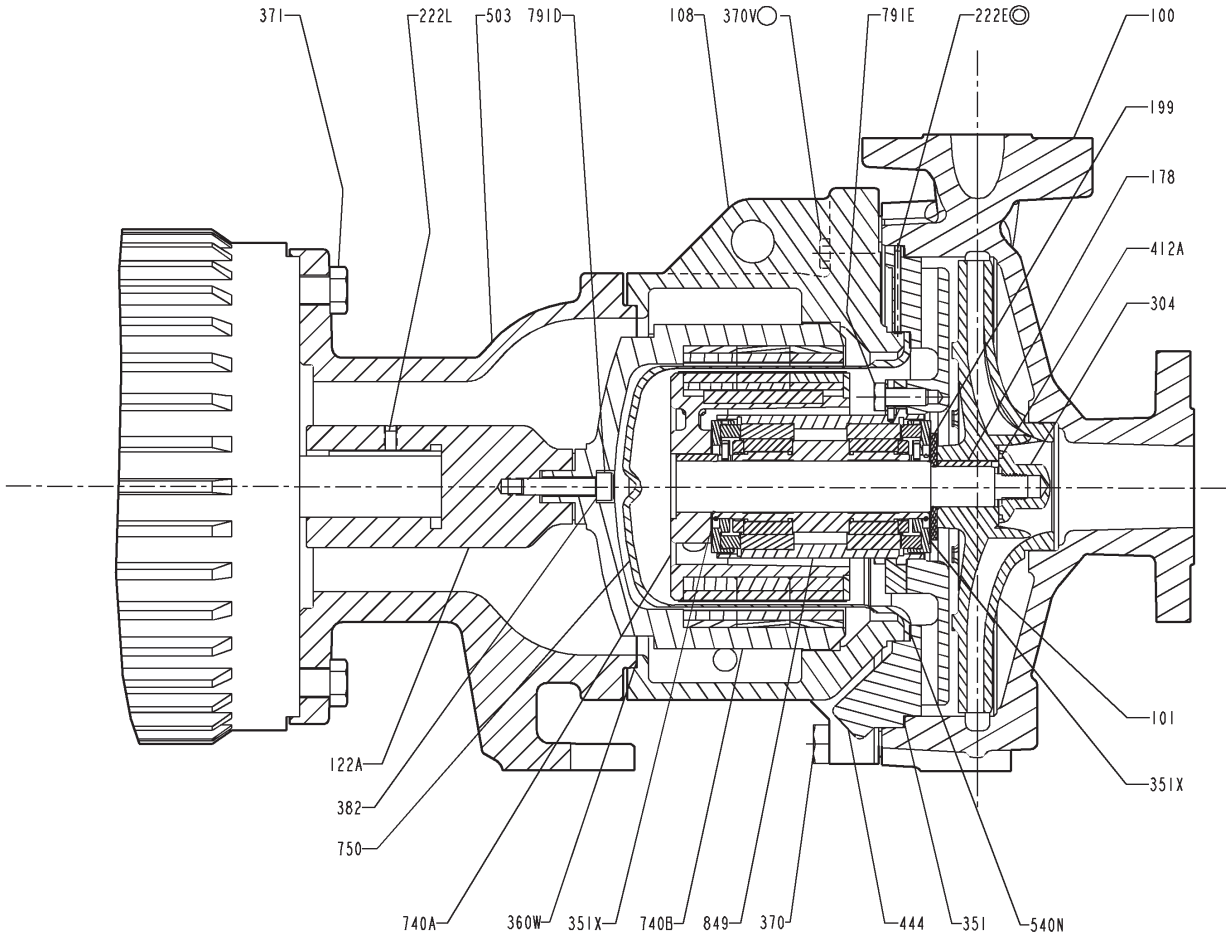


Parts List With Materials Of Construction
Model 3296 EZMAG Close Coupled S Group & 2x3-8 M Group
Stainless Steel Construction

Item No.	Qty	Part Name	Material
100	1	Casing	Stainless Steel (1.4408)
101	1	Impeller	Stainless Steel (1.4408)
108	1	Adapter, Frame	Ductile Iron
122A	1	Shaft, Stub	Steel 420 (1.4021)
178	1	Key, Impeller	303SS
199	1	Washer, Distance	Stainless Steel (1.4571)
222E	3	Screw, Set - Adapt to Backplate ◎	Stainless Steel
222L	2	Screw, Set - Stub Shaft to Motor	Steel
304	1	Impeller Nut	Stainless Steel (1.4417)
351	1⑤	Gasket, Backplate to Case	▲
351X	2⑤	Spacer, Intermediate ring	Viton
360W	1⑤	Gasket - Frame to Adapter	Aramid Fiber/EPDM
370	●	Hex Cap Scr - Adapt. to Case	Stainless Steel
370B	4	Hex Cap Scr - Frame to Adapt ⊕	Steel
370V	2	Hex Cap Scr - Adapt to Backplate O	Stainless Steel
371	4	Hex Cap Scr - Motor to Adapter	Steel
382	1	Lockwasher, Internal Tooth	Steel
412A	1	O-ring. Impeller Nut	Teflon
444	1	Backplate	Stainless Steel (1.4408)
503	1	Adapter, Close Coupled	Cast Iron
540N	1	Gasket, Containment Shell	▲
740A	1⑤	Carrier Assy, Driven	Duplex SS/NdFeB (1.4517)
740B	1⑤	Carrier Assy., Drive	Ductile Iron/NdFeB (0.7043)
750	1⑤	Shell, Containment	Hastelloy C (2.4610)
791D	1	Soc Hd. Cap Scr - Drive to Shaft	Steel
791E	3	Hex Cap Scr - Cart. To Backplate	Stainless Steel
849	1⑤	Bearing Cartridge Assembly ☐ (Standard) Silicon Carbide ☐(Optional) Dryguard Silicon Carbide	Duplex SS/SSiC
High Temperature Option Components			
740A	1 ⑤	Carrier Assembly, Driven	Duplex SS/SmCo (1.4517)
740B	1 ⑤	Carrier Assembly, Drive	Ductile Iron/SmCo (0.7043)
849	1 ⑤	High Temp Bearing Cartridge Assembly ☐(Standard) Silicon Carbide ☐(Optional) Dryguard Silicon Carbide	Duplex SS/SSiC

◎ Only on 6" Size Pumps
O Only on 8" Size Pumps
⊕ Items Not Illustrated
● Qty. 8 for 6" Pumps
● Qty. 12 for 8" Pumps
▲ Optional Gasket Material
☐ Aramid Fiber / EPDM (Standard)
☐ White Gylon (Optional)
☐ Fawn Gylon (Optional)
⑤ Recommended Spare Parts

Cross Sectional Assembly Model 3296 EZMAG
Close Coupled S Group & 2x3-8 M Group
Hastelloy-C

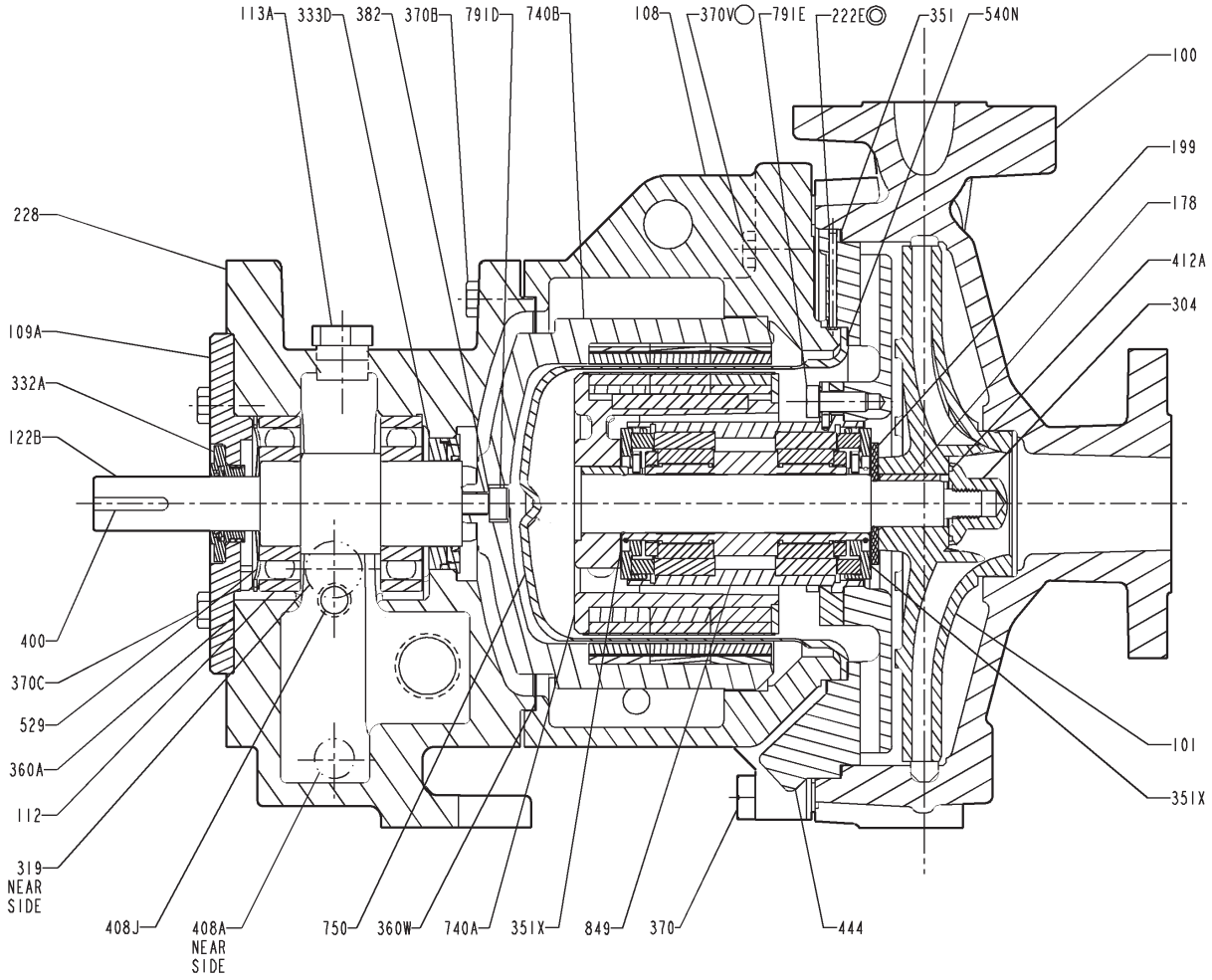


Parts List With Materials Of Construction
Model 3296 EZMAG Close Coupled S Group & 2x3-8 M Group
Hastelloy-C

Item No.	Qty	Part Name	Material
100	1	Casing	Hastelloy-C(2.4686))
101	1	Impeller	Hastelloy-C (2.4686)
108	1	Adapter, Frame	Ductile Iron
122A	1	Shaft, Stub	Steel 420 (1.4021)
178	1	Key, Impeller	Hastelloy-C
199	1	Washer, Distance	Hastelloy-C (2.4610)
222E	3	Screw, Set - Adapt to Backplate ◎	Stainless Steel
222L	2	Screw, Set - Stub Shaft to Motor	Steel
304	1	Impeller Nut	Hastelloy-C (2.4610)
351	1⑤	Gasket, Backplate to Case	▲
351X	2⑤	Spacer, Intermediate ring	Viton
360W	1⑤	Gasket - Frame to Adapter	Aramid Fiber/EPDM
370	●	Hex Cap Scr - Adapt. to Case	Stainless Steel
370B	4	Hex Cap Scr - Frame to Adapt ⊕	Steel
370V	2	Hex Cap Scr - Adapt to Backplate O	Stainless Steel
371	4	Hex Cap Scr - Motor to Adapter	Steel
382	1	Lockwasher, Internal Tooth	Steel
412A	1	O-ring. Impeller Nut	Teflon
444	1	Backplate	Hastelloy-C (2.4686)
503	1	Adapter, Close Coupled	Cast Iron
540N	1	Gasket, Containment Shell	▲
740A	1⑤	Carrier Assy, Driven	Hast-C/NdFeB (2.4686))
740B	1⑤	Carrier Assy., Drive	Ductile Iron/NdFeB (0.7043)
750	1⑤	Shell, Containment	Hastelloy C (2.4610)
791D	1	Soc Hd. Cap Scr - Drive to Shaft	Steel
791E	3	Hex Cap Scr - Cart. To Backplate	Hastelloy-C
849	1⑤	Bearing Cartridge Assy ☐(Standard) Silicon Carbide ☐(Optional) Dryguard Silicon Carbide	Hast-C/SSiC
High Temperature Option Components			
740A	1 ⑤	Carrier Assembly, Driven	Hast-C/SmCo (2.4686)
740B	1 ⑤	Carrier Assembly, Drive	Ductile Iron/SmCo (0.7043)
849	1 ⑤	High Temp Bearing Cartridge Assembly ☐(Standard) Silicon Carbide ☐(Optional) Dryguard Silicon Carbide	Hast-C/SSiC

◎ Only on 6" Size Pumps
O Only on 8" Size Pumps
⊕ Items Not Illustrated
● Qty. 8 for 6" Pumps
● Qty. 12 for 8" Pumps
▲ Optional Gasket Material
☐ Aramid Fiber / EPDM (Standard)
☐ White Gylon (Optional)
☐ Fawn Gylon (Optional)
⑤ Recommended Spare Parts

Cross Sectional Assembly Model 3296 EZMAG
S Group w/ Bearing Frame — Stainless Steel



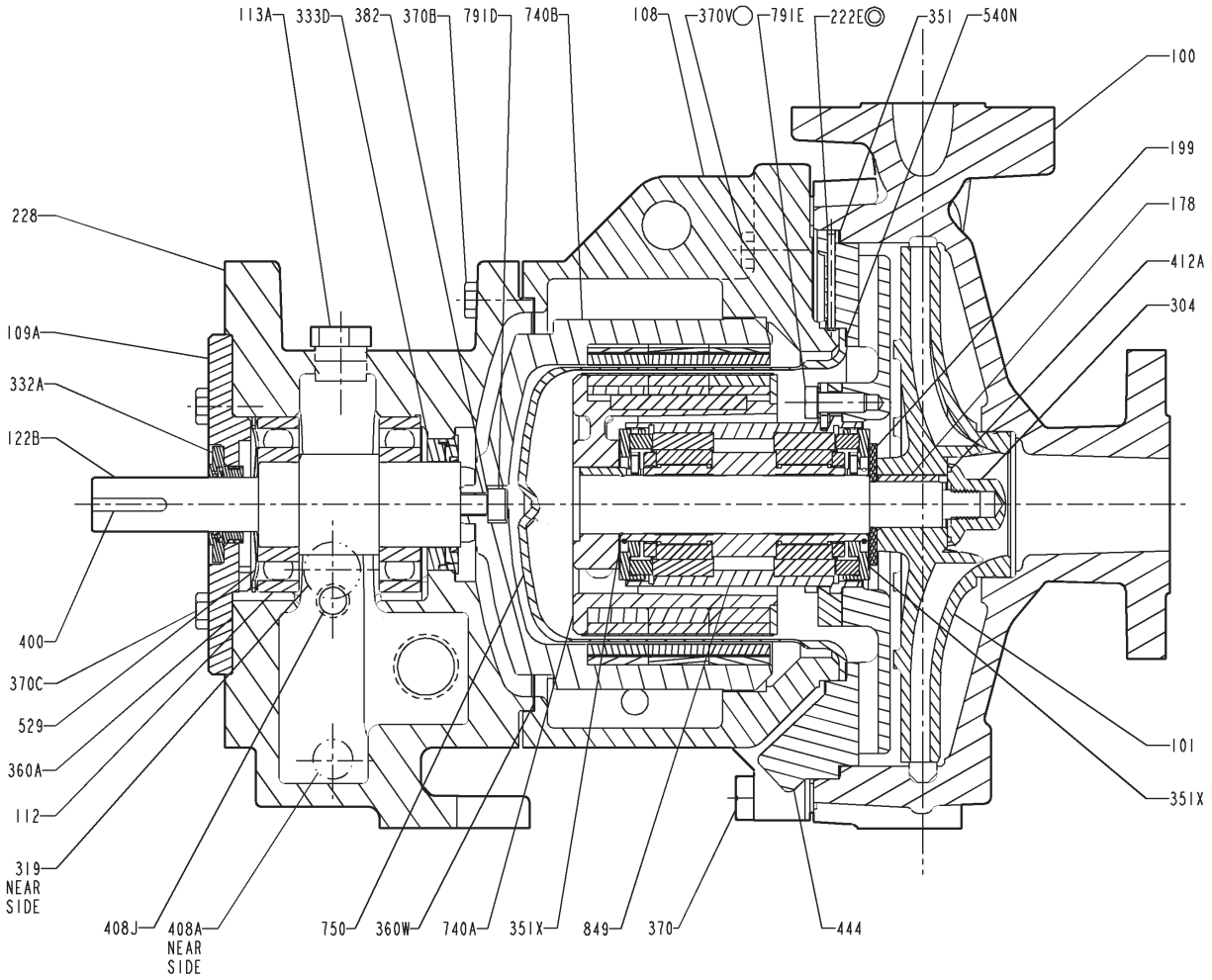
Parts List With Materials Of Construction
Model 3296 EZMAG S Group w/ Bearing Frame — Stainless Steel

Item Number	Qty	Part Name	Material
100	1	Casing	Stainless Steel (1.4408)
101	1	Impeller	Stainless Steel (1.4408)
108	1	Adapter, Frame	Ductile Iron
109A	1	End Cover, Frame	Ductile Iron
112	2⑤	Ball Bearing	Steel
113A	1	Plug, Fill	Steel
122B	1	Shaft, Drive	Steel 4140
178	1	Key Impeller	303SS
199	1	Washer, Distance	Stainless Steel (1.4571)
228	1	Frame, Bearing	Cast Iron
222E	3	Screw, Set - Adapt to Backplate ◎	Steel
304	1	Impeller Nut	Stainless Steel (1.4417)
319	1	Sight Window	Steel/Glass
332A	1	Seal, Labyrinth Oil - Coupling end	Bronze
333D	1	Seal, Labyrinth Oil - Radial end	Bronze
351	1⑤	Gasket, Backplate to Case	▲
351X	2⑤	Spacer, Intermediate ring	Viton
360A	1⑤	Gasket End Cover	Vellumiod
360W	1⑤	Gasket - Frame to Adapter	Aramid Fiber/EPDM
370	●	Hex Cap Scr - Adapt. to Case	Steel
370B	4	Hex Cap Scr - Frame to Adapt	Steel
390C	4	Hex Cap Scr - End Cover	Steel
370V	2	Hex Cap Scr - Adapt to Backplate O	Steel
382	1	Lockwasher, Internal Tooth	Steel
400	1	Key Coupling	Steel
408A	1	Plug, Drain	Steel
408J	1	Plug, Oiler	Steel
412A	1	O-ring. Impeller Nut	Teflon
444	1	Backplate	Stainless Steel (1.4408)
529	1	Washer, Wave	Steel
540N	1	Gasket, Containment Shell	▲
740A	1⑤	Carrier Assy, Driven	Duplex SS/NdFeB (1.4517)
740B	1⑤	Carrier Assy., Drive	Ductile Iron/NdFeB (0.7043)
750	1⑤	Shell, Containment	Hastelloy C (2.4610)
791D	1	Soc Hd. Cap Scr - Drive to Shaft	Steel
791E	3	Hex Cap scr - Cart. To Backplate	Stainless Steel
849	1⑤	Bearing Cartridge Assy	Duplex SS/SSiC
		☐(Standard) Silicon Carbide	
		☐(Optional) Dryguard Silicon Carbide	

High Temp Components			
740A	1⑤	Carrier Assy., Driven	Duplex SS/SmCo (1.4517)
740B	1⑤	Carrier Assy., Drive	Ductile Iron/SmCo (0.7043)
849	1⑤	High Temp Bearing Cartridge Assy	Duplex SS/SSic
		☐(Standard) Silicon Carbide	
		☐(Optional) Dryguard Silicon Carbide	

◎	Only on 6" Size Pumps	▲	Optional Gasket Material
O	Only on 8" Size Pumps		☐ Aramid Fiber / EPDM (Standard)
●	Qty. 8 for 6" Pumps		☐ White Gylon (Optional)
●	Qty. 12 for 8" Pumps		☐ Fawn Gylon (Optional)
		⑤	Recommended Spare Parts

Cross Sectional Assembly Model 3296 EZMAG
S Group w/ Bearing Frame — Hastelloy-C

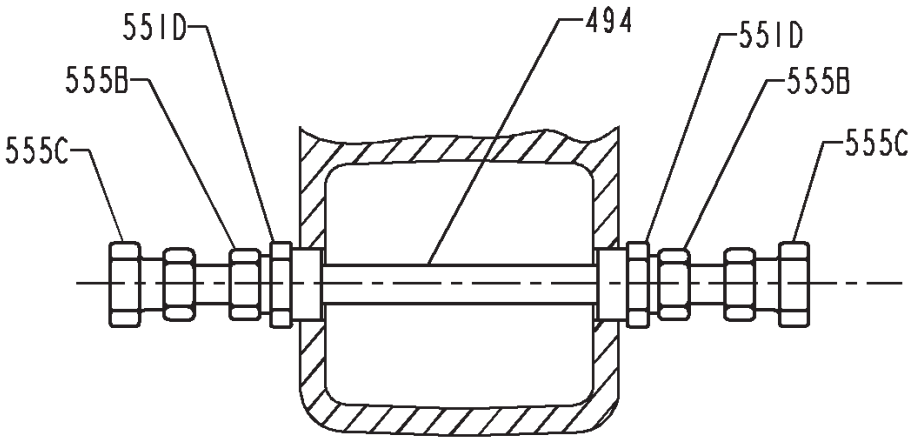


Parts List with Materials of Construction
Cross Sectional Assembly Model 3296 EZMAG
S Group w/ Bearing Frame — Hastelloy-C

Item Number	Qty	Part Name	Material
100	1	Casing	Hastelloy-C(2.4686))
101	1	Impeller	Hastelloy-C (2.4686)
108	1	Adapter, Frame	Ductile Iron
109A	1	End Cover, Frame	Ductile Iron
112	2⑤	Ball Bearing	Steel
113A	1	Plug, Fill	Steel
122B	1	Shaft, Drive	Steel 4140
178	1	Key, Impeller	Hastelloy-C
199	1	Washer, Distance	Hastelloy-C (2.4610)
228	1	Frame, Bearing	Cast Iron
222E	3	Screw, Set - Adapt to Backplate ¥	Steel
304	1	Impeller Nut	Hastelloy-C (2.4610)
319	1	Sight Window	Steel/Glass
332A	1	Seal, Labyrinth Oil - Coupling end	Bronze
333D	1	Seal Labyrinth Oil - Radial end	Bronze
351	1⑤	Gasket, Backplate to Case	▲
351X	2⑤	Spacer, Intermediate ring	Viton
360A	1⑤	Gasket - End Cover	Vellumiod
360W	1⑤	Gasket - Frame to Adapter	Aramid Fiber/EPDM
370	●	Hex Cap Scr - Adapt. to Case	Steel
370B	4	Hex Cap Scr - Frame to Adapt	Steel
370C	4	Hex Cap Scr - End Cover	Steel
370V	2	Hex Cap Scr - Adapt to Backplate ¢	Steel
382	1	Lockwasher, Internal Tooth	Steel
400	1	Key, Coupling	Steel
408A	1	Plug, Drain	Steel
408J	1	Plug, Oiler	Steel
412A	1	O-ring. Impeller Nut	Teflon
444	1	Backplate	Hastelloy-C (2.4686)
529	1	Washer Wave	Steel
540N	1	Gasket, Containment Shell	▲
740A	1⑤	Carrier Assy, Driven	Hast-C/NdFeB (2.4686))
740B	1⑤	Carrier Assy., Drive	Ductile Iron/NdFeB (0.7043)
750	1⑤	Shell, Containment	Hastelloy C (2.4610)
791D	1	Soc Hd. Cap Scr - Drive to Shaft	Steel
791E	3	Hex Cap Scr - Cart. To Backplate	Hastelloy-C
849	1⑤	Bearing Cartridge Assy ☐(Standard) Silicon Carbide ☐(Optional) Dryguard Silicon Carbide	Hast-C/SSiC
High Temperature Option Components			
740A	1⑤	Carrier Assembly, Driven	Hast-C/SmCo (1.4517)
740B	1⑤	Carrier Assembly, Drive	Ductile Iron/SmCo (0.7043)
849	1⑤	High Temp Bearing Cartridge Assembly ☐(Standard) Silicon Carbide ☐(Optional) Dryguard Silicon Carbide	Hast-C/SSiC

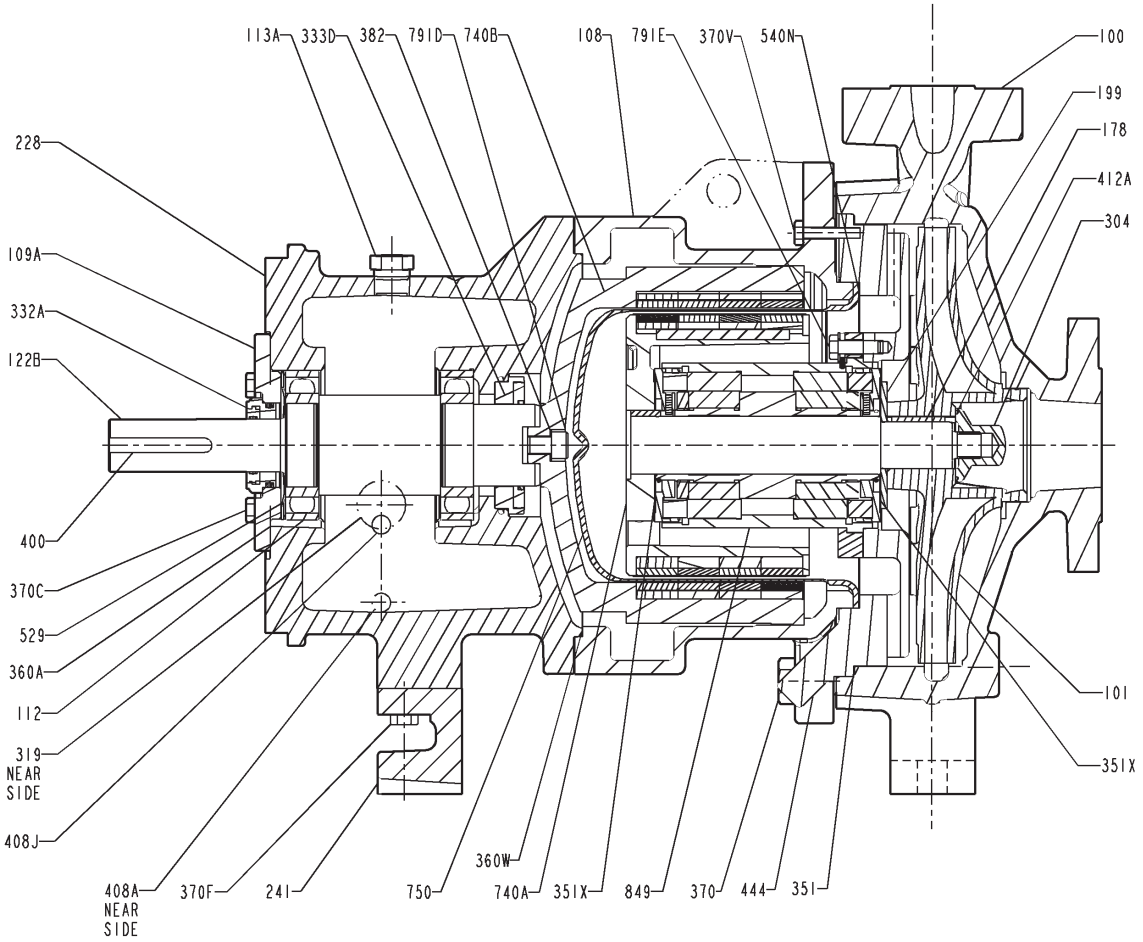
◎ Only on 6" Size Pumps
O Only on 8" Size Pumps
● Qty. 8 for 6" Pumps
● Qty. 12 for 8" Pumps
▲ Optional Gasket Material
☐ Aramid Fiber / EPDM (Standard)
☐ White (Optional)
☐ Fawn Gylon (Optional)
⑤ Recommended Spare Parts

Cross Sectional Model 3296 EZMAG
Frame Cooling Options



Item	Qty	Part Name	Material
494	2	Tube, Cooling	Stainless Steel
555B	2	Tube, Thermocouple Fitting	Stainless Steel
555C	2	Tube Fitting Str	Brass
551D	1	Hex Bushing	Iron

Cross Sectional Assembly Model 3296 EZMAG
M Group, w/ Bearing Frame
Stainless Steel



Parts List with Materials Of Construction
Model 3296 EZMAG M Group w/ Bearing Frame — Stainless Steel

Item No.	Qty	Part Name	Material
100	1	Casing	Stainless Steel (1.4408)
101	1	Impeller	Stainless Steel (1.4408)
108	1	Adapter, Frame	Ductile Iron
109A	1	End Cover, Frame	Ductile Iron
112	2⑤	Ball Bearing	Steel
113A	1	Plug, Fill	Steel
122B	1	Shaft, Drive	Steel 4140
178	1	Key, Impeller	303SS
199	1	Washer, Distance	Stainless Steel (1.4571)
228	1	Frame, Bearing	Cast Iron
241	1	Foot, Frame	Cast Iron
304	1	Impeller Nut	Stainless Steel (1.4417)
319	1	Sight Window	Steel/Glass
332A	1	Seal, Labyrinth Oil - Coupling end	Bronze
333D	1	Seal, Labyrinth Oil - Radial end	Bronze
351	1⑤	Gasket, Backplate to Case	▲
351X	2⑤	Spacer, Intermediate ring	Viton
360A	1⑤	Gasket End Cover	Vellumiod
360W	1⑤	Gasket - Frame to Adapter	Aramid Fiber/EPDM
370	●	Hex Cap Scr - Adapt. to Case	Steel
370B	4	Hex Cap Scr - Frame to Adapt	Steel
370C	4	Hex Cap Scr - End Cover	Steel
370F	2	Hex Cap Scr - Foot to Frame	Steel
370V	2	Hex Cap Scr - Adapt to Backplate	Steel
382	1	Lockwasher, Internal Tooth	Steel
400	1	Key Coupling	Steel
408A	1	Plug, Drain	Steel
408J	1	Plug, Oiler	Steel
412A	1	O-ring. Impeller Nut	Teflon
444	1	Backplate	Stainless Steel (1.4408)
529	1	Washer, Wave	Steel
540N	1	Gasket, Containment Shell	▲
740A	1⑤	Carrier Assy, Driven	Duplex SS/NdFeB (1.4517)
740B	1⑤	Carrier Assy., Drive	Ductile Iron/NdFeB (0.7043)
750	1⑤	Shell, Containment	Hastelloy C (2.4610)
791D	1	Soc Hd. Cap Scr - Drive to Shaft	Steel
791E	3	Hex Cap Scr - Cart. To Backplate	Stainless Steel
849	1⑤	Bearing Cartridge Assy	Duplex SS/SSiC
		☐ Silicon Carbide (Standard)	
		☐Dryguard Silicon Carbide (Optional)	
High Temperature Option Components			
740A	1 ⑤	Carrier Assembly, Driven	Duplex SS/SmCo (1.4517)
740B	1 ⑤	Carrier Assembly, Drive	Ductile Iron/SmCo (0.7043)
849	1 ⑤	High Temp Bearing Cartridge Assembly	Duplex SS/SSiC
		☐Silicon Carbide (Standard)	
		☐Dryguard Silicon Carbide (Optional)	

● Qty. 12 for 2x3-8, 3x4-7 & 3x4-8G
● Qty. 8 for 1x2-10, 2x3-10 & 3x4-10
▲ Optional Gasket Material
☐ Aramid Fiber / EPDM (Standard)
☐ White Gylon (Optional)
☐ Fawn Gylon (Optional)
⑤ Recommended Spare Parts

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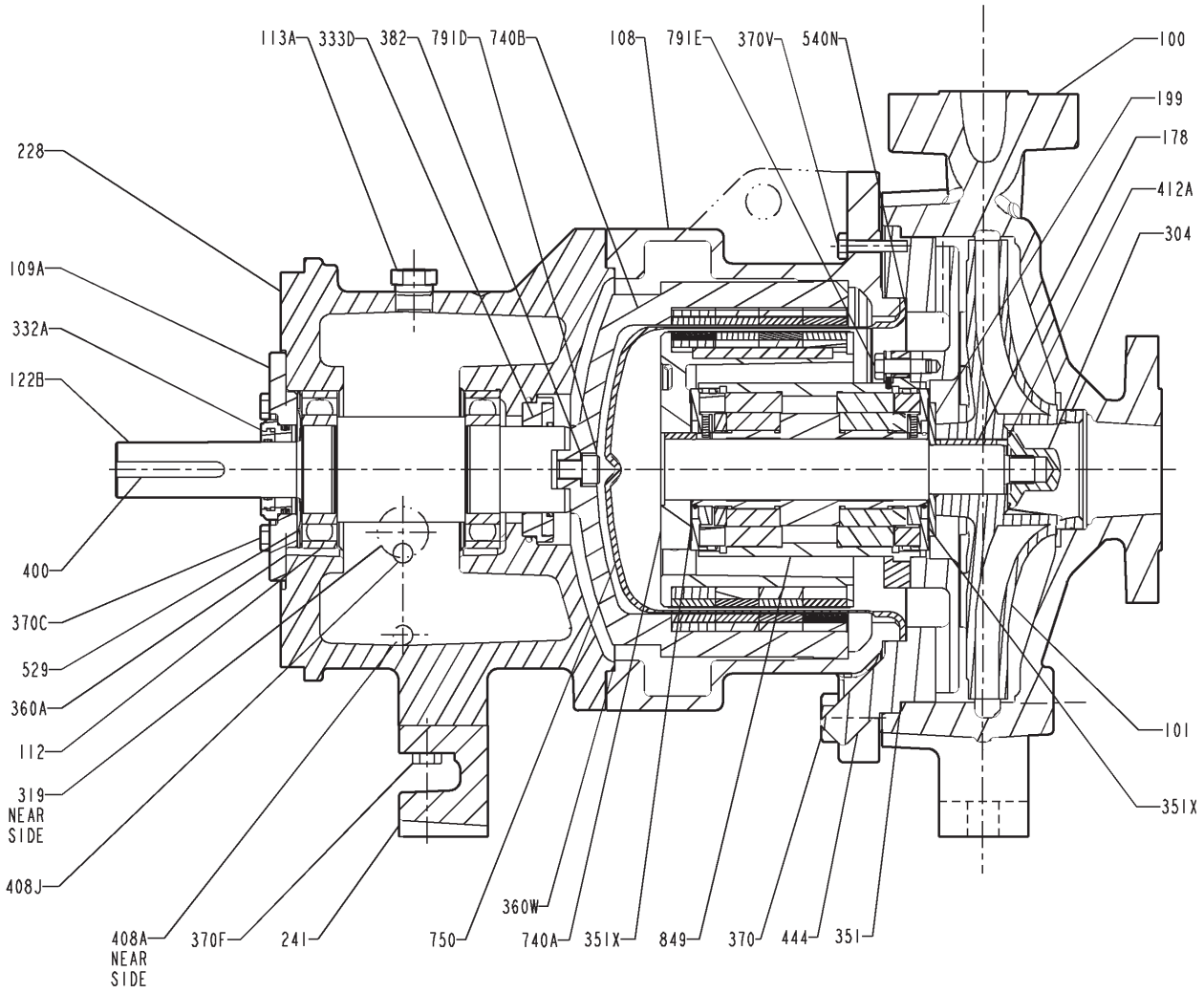
Cross Sectional Assembly Model 3296 EZMAG
M Group, w/Bearing Frame
Hastelloy-C



Parts List with Materials Of Construction
3296 EZMAG M Group, w/ Bearing Frame — Hastelloy-C

Item No.	Qty	Part Name	Material
100	1	Casing	Hastelloy-C(2.4686))
101	1	Impeller	Hastelloy-C (2.4686)
108	1	Adapter, Frame	Ductile Iron
109A	1	End Cover, Frame	Ductile Iron
112	2⑤	Ball Bearing	Steel
113A	1	Plug, Fill	Steel
122B	1	Shaft, Drive	Steel 4140
178	1	Key Impeller	Hastelloy-C
199	1	Washer, Distance	Hastelloy-C (2.4610)
228	1	Frame, Bearing	Cast Iron
241	1	Foot Frame	Cast Iron
304	1	Impeller Nut	Hastelloy-C (2.4610)
319	1	Sight Window	Steel/Glass
332A	1	Seal, Labyrinth Oil - Coupling end	Bronze
333D	1	Seal Labyrinth Oil - Radial end	Bronze
351	1⑤	Gasket, Backplate to Case	▲
351X	2⑤	Spacer, Intermediate ring	Viton
360A	1⑤	Gasket - End Cover	Vellumiod
360W	1⑤	Gasket - Frame to Adapter	Aramid Fiber/EPDM
370	●	Hex Cap Scr - Adapt. to Case	Stainless Steel
370B	4	Hex Cap Scr - Frame to Adapt	Steel
370C	4	Hex Cap Scr - End Cover	Steel
370F	2	Hex Cap Scr - Foot to Frame	Steel
370V	2	Hex Cap Scr - Adapt to Backplate	Stainless Steel
382	1	Lockwasher, Internal Tooth	Steel
400	1	Key, Coupling	Steel
408A	1	Plug, Drain	Steel
408J	1	Plug, Oiler	Steel
412A	1	O-ring. Impeller Nut	Teflon
444	1	Backplate	Hastelloy-C (2.4686)
529	1	Washer Wave	Steel
540N	1	Gasket, Containment Shell	▲
740A	1⑤	Carrier Assy, Driven	Hast-C/NdFeB (2.4686))
740B	1⑤	Carrier Assy., Drive	Ductile Iron/NdFeB (0.7043)
750	1⑤	Shell, Containment	Hastelloy C (2.4610)
791D	1	Soc Hd. Cap Scr - Drive to Shaft	Steel
791E	3	Hex Cap scr - Cart. To Backplate	Hastelloy-C
849	1⑤	Bearing Cartridge Assy ☐ (Standard) Silicon Carbide ☐ (Optional) Dryguard Silicon Carbide	Hast-C/SSiC
High Temperature Option Components			
740A	1 ⑤	Carrier Assembly, Driven	Hast-C/SmCo (2.4686)
740B	1 ⑤	Carrier Assembly, Drive	Ductile Iron/SmCo (0.7043)
849	1 ⑤	High Temp Bearing Cartridge Assembly ☐ (Standard) Silicon Carbide ☐ (Optional) Dryguard Silicon Carbide	Hast-C/SSiC

● Qty. 12 for 2x3-8, 3x4-7 & 3x4-8G
● Qty. 8 for 1x2-10, 2x3-10 & 3x4-10
▲ Optional Gasket Material
☐ Aramid Fiber / EPDM (Standard)
☐ White Gylon (Optional)
☐ Fawn Gylon (Optional)
⑤ Recommended Spare Parts

SPARE AND REPAIR PARTS

When ordering parts, always refer to part name, Goulds Serial No., and indicate Item No. from the sectional drawing.

RECOMMENDED SPARES

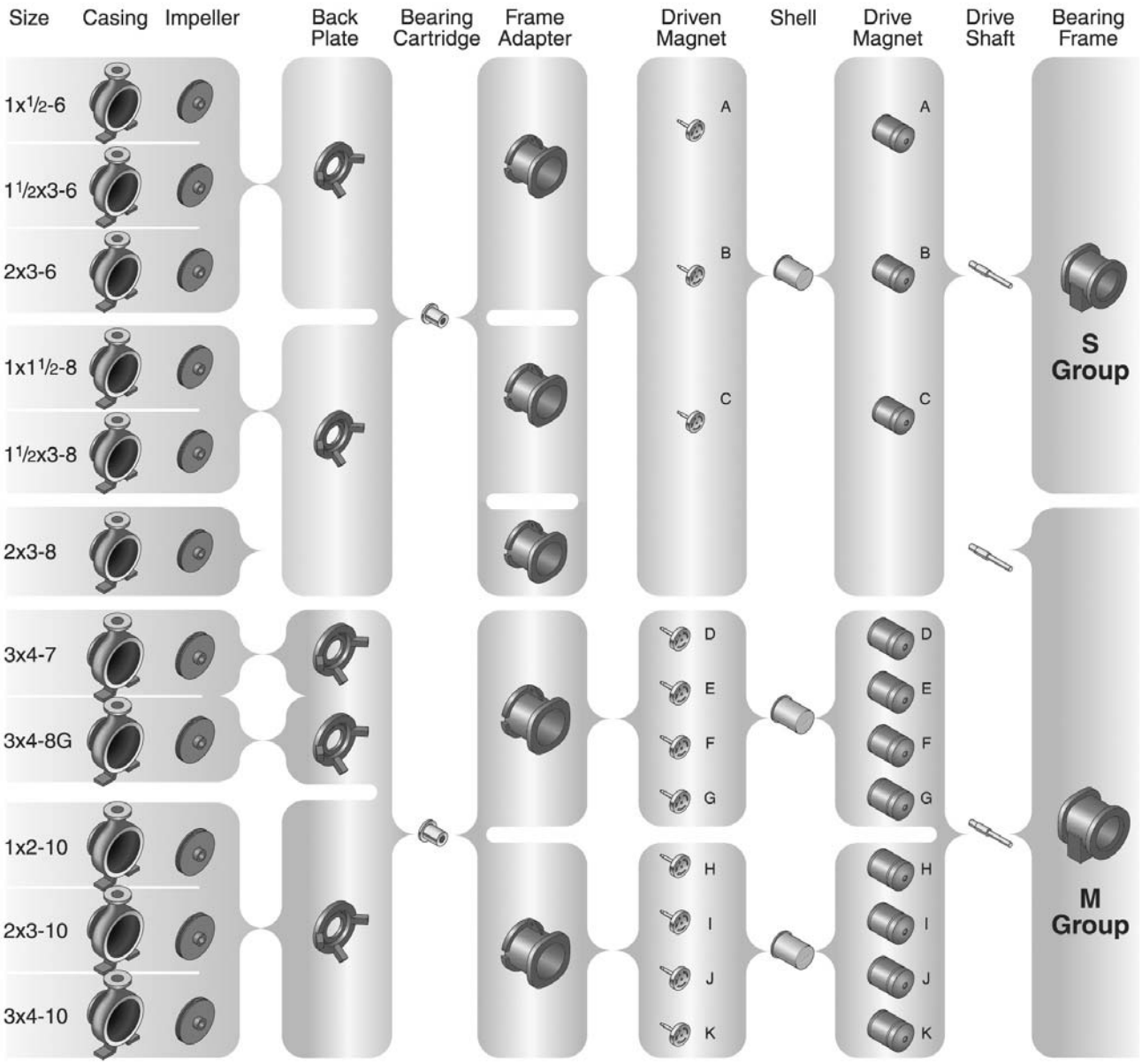
Part	Material	Item	Qty.
Gasket, Backplate to Case	Aramid Fiber with EPDM Rubber	351	1
Ball Bearing	Steel	112	2
Spacer, Intermediate Ring	Grafoil	351X	2
Gasket, End Cover	Vellumoid	360A	1
Gasket, Frame to Adapter	Aramid Fiber with EPDM Rubber	360W	1
Carrier Assembly, Driven	Duplex SS/NdFeB	740A	1
Carrier Assembly, Drive	Ductile Iron/NdFeB	740B	1
Containment Shell	Hast-C	750	1
Bearing Cartridge Assembly	Duplex SS/SSiC	849	1

PARTS

- Impeller (101)
- Impeller Nut and O-Ring (304, 412A)
- Impeller Key (178)
- Driven Magnet Assembly (740A)
- Drive Magnet Assembly (740B)
- Containment Shell (750)
- Driven Shaft (122B)
- Labyrinth Oil Seals (332A, 333D)

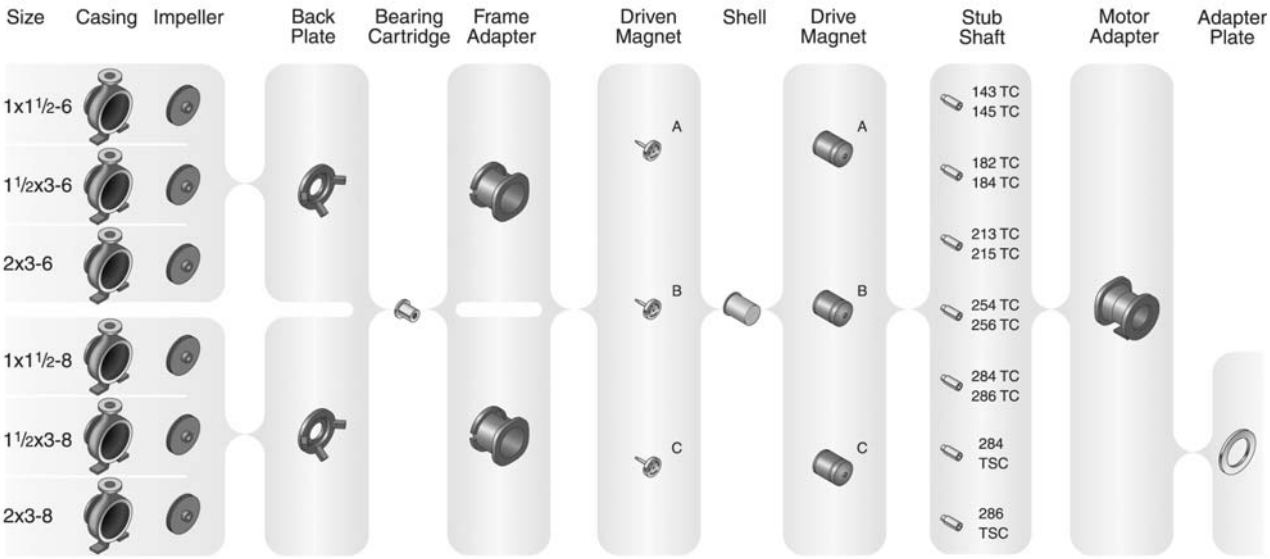
INTERCHANGEABILITY - Frame Mounted

3296 EZMAG



INTERCHANGEABILITY - Close Coupled

3296 EZMAG



8

RETURN OF MATERIALS

If it is necessary to return the pump to a Goulds factory or repair facility for service, certain procedures must be followed.

Do not return any parts without authorization from the warranty engineer, a warranty claim number, and the preprinted shipping label supplied by Goulds. In rare instances because of the pumpage, ITT Goulds Pumps Warranty Services may, at their option, decide not to have the parts returned.

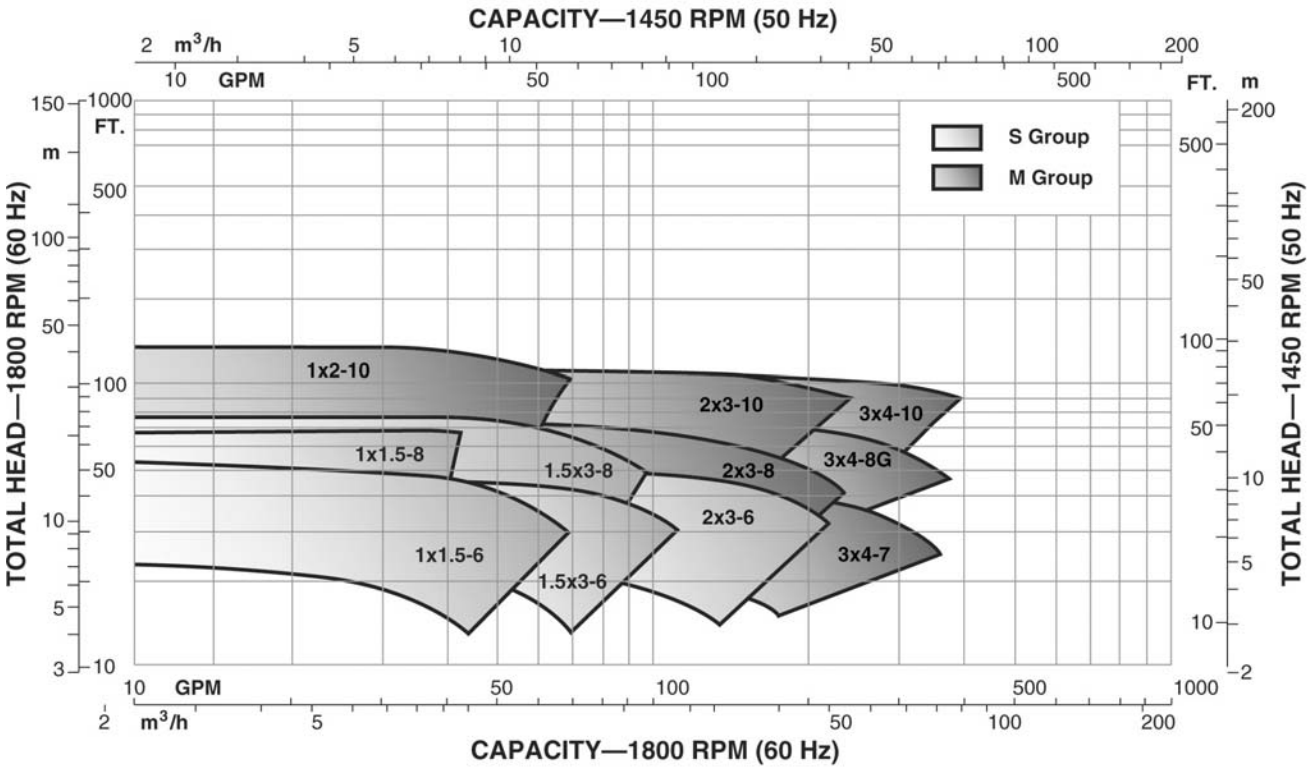
The parts being returned must be decontaminated prior to shipment. The decontamination must be verified in writing. The correct Material Safety Data Sheet (MSDS) must accompany the parts along with decontamination "sign off." This information is stated within the ITT Goulds Pumps "decontamination procedure." The warranty service engineer will send a copy of this procedure to the customer. Please remember that inspection of the parts cannot be started until we receive the proper documentation. This is a safety and legal issue and consequently strict adherence to procedure is mandatory — there will be no exceptions.

Before shipping check with your carrier for special procedures that may be required when shipping highly magnetic materials.

All pumps must be decontaminated prior to return. Reference *Operation Section's Preparation for Disassembly* and *Decontamination Procedure*.

APPENDIX I

HYDRAULIC COVERAGE CHARTS



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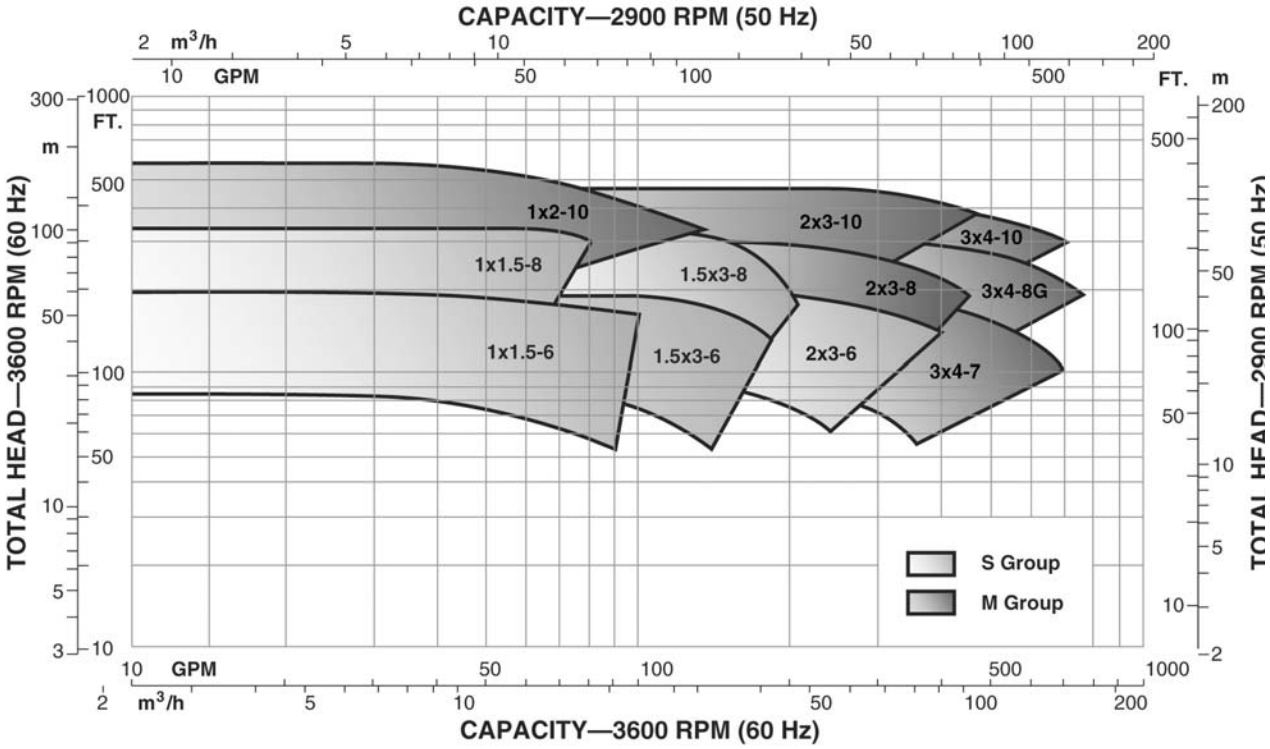
HYDRAULIC COVERAGE CHARTS



APPENDIX II

COUPLING GUARD INSTALLATION - Frame-Mounted Only

WARNING

Before assembly or disassembly of the coupling guard is performed, the motor must be de-energized, the motor controller/starter put in a locked-out position, and a caution tag placed at the starter indicating the disconnect. Replace coupling guard before resuming normal operation of the pump. Goulds Pumps assumes no liability for avoiding this practice.

The coupling guard used in an ATEX classified environment must be constructed from a non-sparking material.

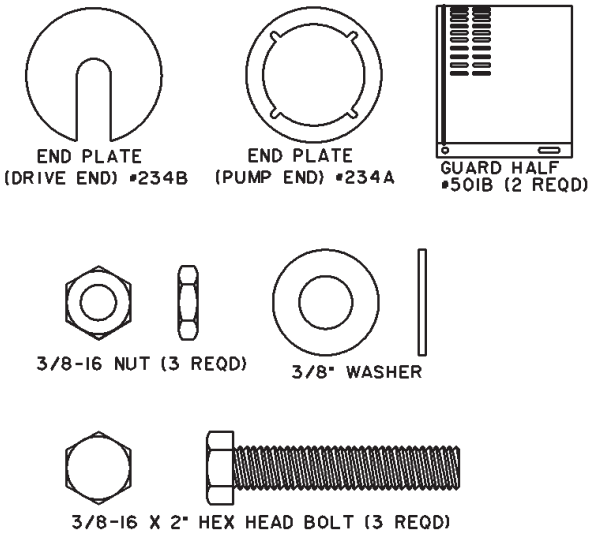


Fig. II-1

Simplicity of design allows complete assembly of the coupling guard, including the end plate (pump end), in about fifteen minutes. If the end plate is already in place, assembly can be accomplished in about five minutes.

Assembly:

NOTE: If end plate (pump end) is already installed, make any necessary coupling adjustments and then proceed to Step 2.

1. **STX, MTX, LTX** - Align end plate (pump end) to the Bearing Frame. (No impeller adjustment required.)

   XLT-X Align the end plate (pump end) to the pump bearing housing so that the large slots on the end plate clear the bearing housing tap bolts and the small slots are aligned to the impeller adjusting bolts. Attach the end plate to the bearing housing using the jam nuts on the impeller adjusting bolts as shown in Fig. II-3.

   After the end plate is attached to the bearing housing, the impeller clearance must be checked and reset as explained in *Preventive Maintenance* Section.

NOTE: Coupling adjustments should be completed before proceeding with coupling guard assembly.

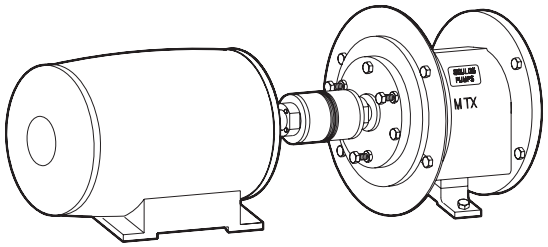


Fig. II-2

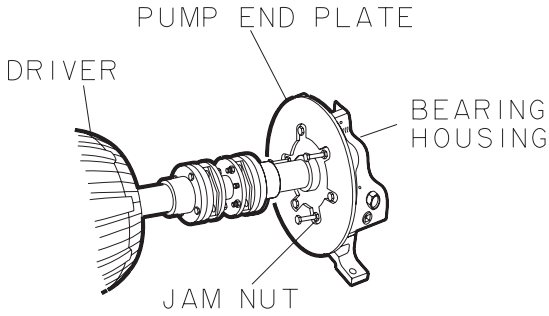


Fig. II-3

2. Spread bottom of coupling guard half (pump end) slightly and place over pump end plate as shown in Fig. II-4. The annular groove in the guard half is located around the end plate (Fig. II-5).

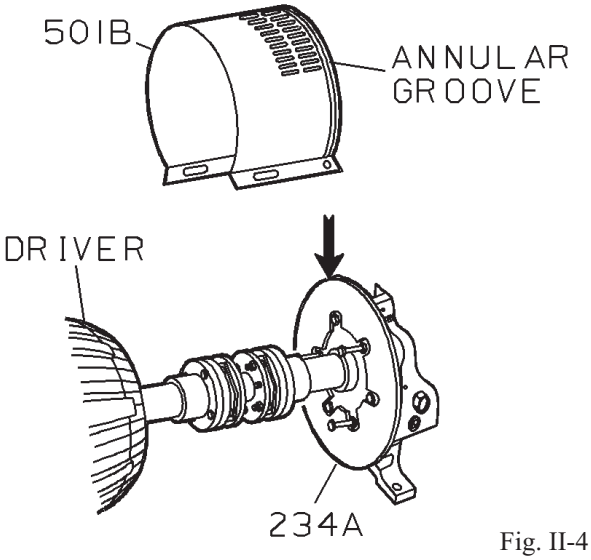


Fig. II-4

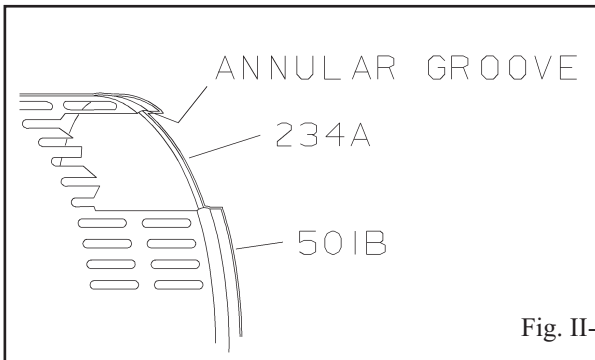


Fig. II-5

3. After the coupling guard half (pump end) is located around the end plate, secure it with a bolt, nut and two (2) washers through the round hole at the front end of the guard half as shown in Fig. II-6. Tighten securely (Fig. II-7).

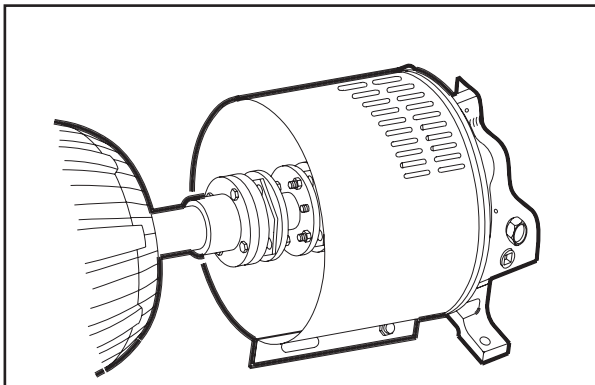


Fig. II-6

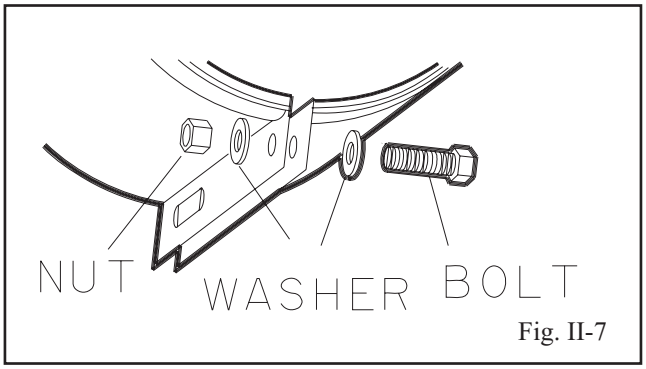


Fig. II-7

4. Spread bottom of coupling guard half (driver end) slightly and place over coupling guard half (pump end) so that annular groove in coupling guard half (driver end) faces the motor as shown in Fig. II-8.

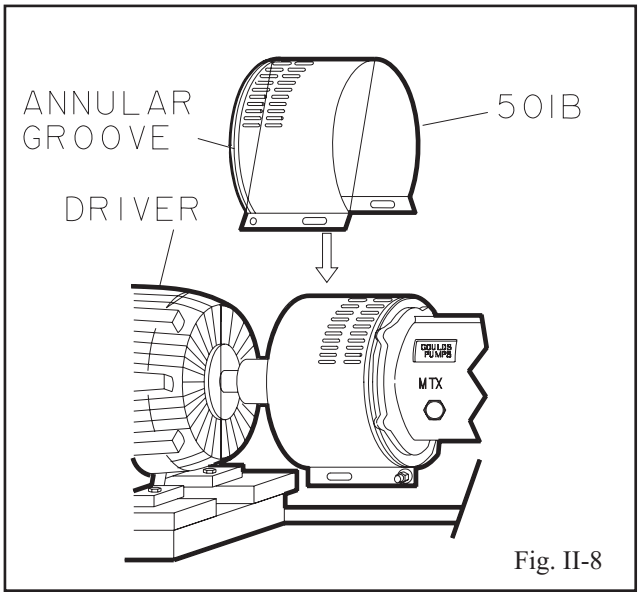


Fig. II-8

5. Place end plate (driver end) over motor shaft as shown in Fig. II-9. Locate the end plate in the annular groove at the rear of the coupling guard half (driver end) and secure with a bolt, nut, and two (2) washers through the round hole at the rear of the guard half. Finger tighten only.

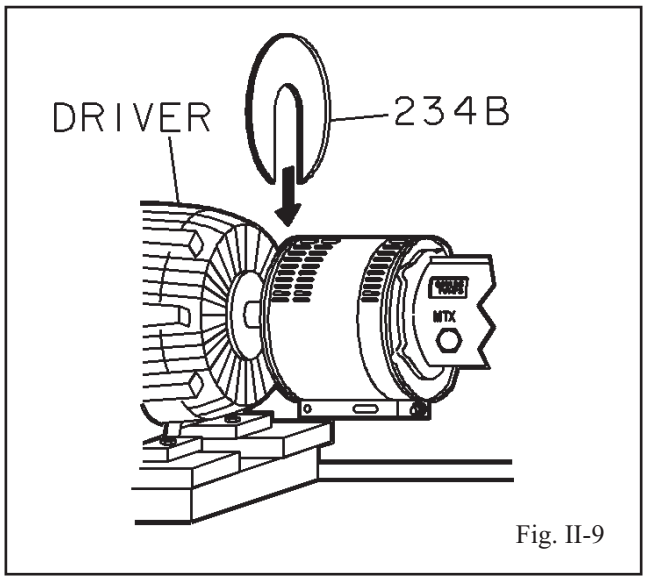


Fig. II-9

6. Adjust length of coupling guard to completely cover shafts and coupling as shown in Fig. II-10 by sliding coupling guard half (driver end) towards motor. After adjusting guard length, secure with bolt, nut and two (2) washers through the slotted holes at the center of the guard and tighten. Check all nuts on the guard assembly for tightness.

⚠ WARNING

Before assembly or disassembly of the coupling guard is performed, the motor must be de-energized, the motor controller/starter put in a locked-out position, and a caution tag placed at the starter indicating the disconnect. Replace coupling guard before resuming normal operation if the pump. Goulds Pumps assumes no liability for avoiding this practice.

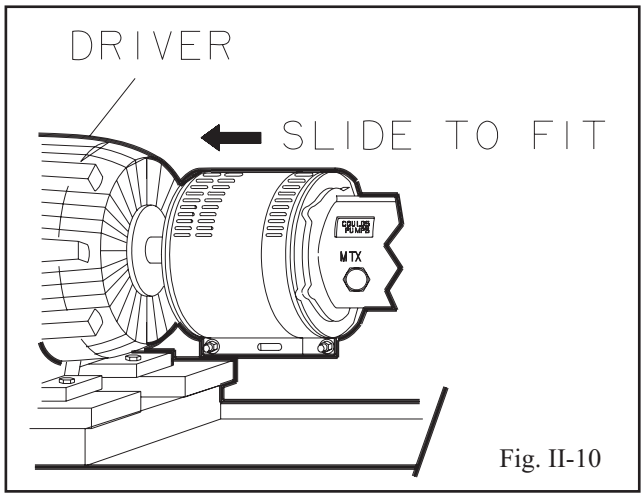


Fig. II-10

Disassembly

The coupling guard must be removed for certain maintenance and adjustments to the pump, such as adjustment of the coupling, impeller clearance adjustment, etc. The coupling guard should be replaced after maintenance is completed.

DO NOT resume normal pump operation with the coupling guard removed.

NOTE: Refer to illustrations for assembly in reverse order.

1. Remove nut, bolt, and washers from center slotted hole in the coupling guard. Slide motor end coupling guard half towards pump. Fig. II-10.
2. Remove nut, bolt, and washers from coupling guard half (driver end), and remove end plate. Fig. II-9.
3. Spread bottom of coupling guard half slightly and lift off. Fig. II-8.
4. Remove remaining nut, bolt, and washers from coupling guard half (pump end). Spread bottom of coupling guard half slightly and lift off. Fig. II-4.

This completes disassembly of the coupling guard.

***NOTE: It is not necessary to remove the end plate (pump end) from the pump bearing housing. The bearing housing tap bolts are accessible without removing the end plate in case maintenance of internal pump parts is necessary. Before removing the pump bearing housing, refer to* Disassembly & Reassembly.**

8

APPENDIX III

POWER MONITORS

When installing in a potentially explosive environment, ensure that condition monitoring devices are properly certified.

CONDENSED POWER MONITOR USER GUIDE

ITT Goulds Pumps offers various power monitoring devices which have been designed to protect pumps from dry-running, running against a closed discharge valve, and running outside of the recommended operating region for specific pump sizes, speeds, and impeller diameters. Power monitors were designed to detect power relative to load which is a linear function as opposed to measuring amperage relative to load which is a parabolic function. The linear characteristic of measuring power vs. load enhances sensitivity at low power conditions where increments in power are critical. *See Fig. IV-1* for comparison of power measurements vs. amperage measurements. Quite simply, a properly adjusted and installed power monitor is an insurance policy for securing extended pump life.

The following summarized concepts will assist in effectively selecting and properly calibrating power monitors used to protect pumps.

1. Read your power monitor installation instructions and wiring diagram before attempting to calibrate the unit.
2. Understand your pump's recommended operating envelope as provided by ITT Goulds Pumps.
3. Understand your systems requirements and limitations.

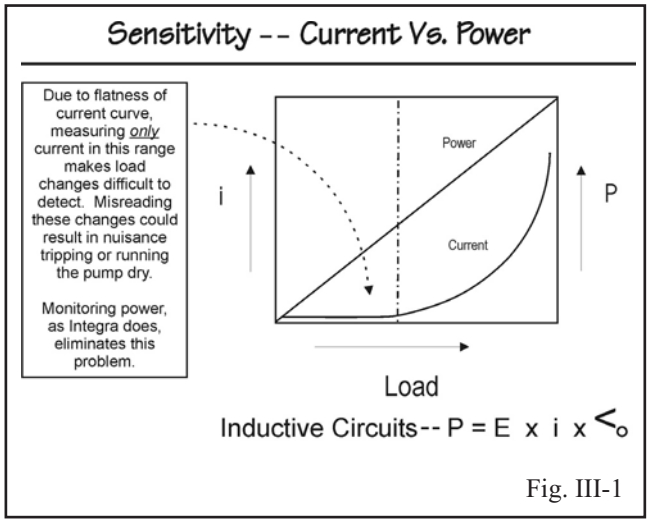


Fig. III-1

4. Understand the full range of your operating duty, including power requirements at the rated, minimum and maximum flow conditions.
5. Understand the relationship and limitations of item numbers 2, 3 , and 4 as a an integrated functional system.
6. Identify potential failures that would most likely be characteristic of your specific process and the pump type selected.
7. Understand the power scope and torque scope of the selected electric motor.
8. A complete analysis of the above concepts will define a safe operating range for your specific system.
9. When feasible, further define your operating range to run as close to BEP (best efficiency point) as practical.
10. Select single trip unit or dual trip unit as practical for your specific system. A dual trip unit is recommended for the 3296 EZMAG. A low setting should always be used to protect against dry run. A high setting will detect if an upset condition has occurred resulting in bearing damage.
11. Set **low power trip point** at the power draw required when operating at the manufacturer's recommended minimum flow or higher, yet less than the normal operating point.
12. Set the **high trip point** at the power draw required when operating at the manufacturer's recommended maximum flow or lower, or at flow rate that will prevent cavitation.
13. When using dual trip power monitors, select high and low trip points designed to protect your specific system within the recommended pump operating region defined by ITT Goulds Pumps. This method will create boundaries for a safe pump operating envelope.

POWER MONITORS, (cont'd)

14. Set nuisance trip feature for each power trip. The nuisance trip device should be set at an appropriate interval that will allow the system to experience momentary fluctuations in power draw. However, the nuisance trip device should also be set at an appropriate time frame that will prevent the pump from experiencing excessive heat or dangerous operating conditions.
15. Set the delay timer for start-up conditions that will appropriately allow the system to come to normal operating power within a time frame that will maximize protection of your pump.
16. For variable speed operation, consult ITT Goulds Pumps or the power monitor manufacturer for appropriate auxiliary devices designed for operating at multiple speeds or fluctuating frequencies.
17. Select electrical enclosure that is suitable for the operating environment or install the unit in an appropriate electrical panel.
18. Do not activate the power monitor's manual override until a thorough examination of the source of the problem is defined and corrected.
19. Investigate and select power monitor features that are most suitable for your particular application and plant safety.

Power monitors can be strategically calibrated to protect your pump from any of the following conditions by appropriately determining the power draw at any of these individual conditions.

- Dry Running
- Closed Discharge Valve
- No Prime
- Inadequate Suction Conditions/Plugged Suction
- Cavitation
- Air Lock
- Decoupled Magnets
- Solidified, Plugged, or Frozen Discharge Line
- Fluctuating Viscosities, Precipitation, or Coagulation
- Broken or Damaged Shaft
- Broken or Damaged Coupling
- Jammed Impeller
- Bad Bearings
- Rapid Cycling

The power draw at each of these conditions can be simulated in a plant test or estimated through calculations or interpolations from the pump performance curve. By defining these dangerous power fluctuations, appropriate calibration of the power monitoring unit will prevent avoidable pump failures. *See Fig. IV-2* for a typical power evaluation recorded from a common centrifugal pump electric motor.

Contact ITT Goulds Pumps or your regional distributor for assistance and power analysis for your specific system.

For further details and evaluation of power monitoring units, a comprehensive power monitor user guide is available from ITT Goulds Pumps distribution network.

MONITORING PUMP HORSEPOWER TELLS YOU WHAT'S HAPPENING TO THE FLUID

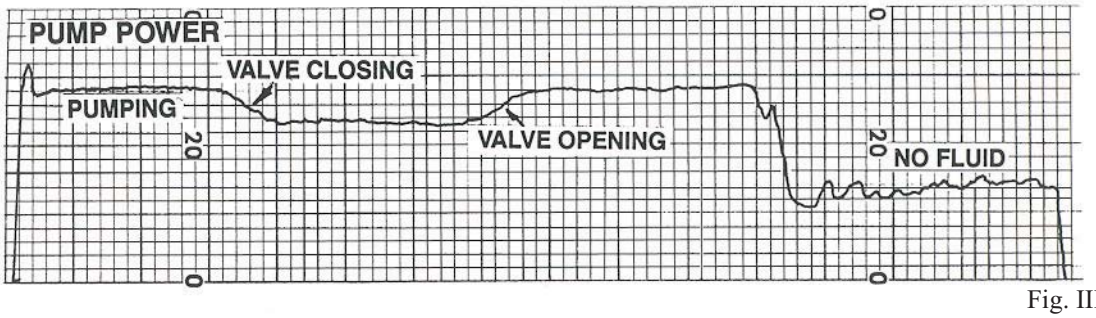


Fig. III-2

APPENDIX IV

Reliability Tips for Operating Magnetically-Driven Sealless Pumps

QUICK REFERENCE SUMMARY

These summarized reliability guidelines outline recommendations for creating an optimal environment for operating mag-drive pumps. For further details on the subject of operating a mag drive pump with superior reliability, please contact your ITT Goulds Pumps' representative.

- Do not operate mag-drive pumps under no flow conditions.
- Do not operate mag-drive pumps against a closed discharge valve.
- Do not operate mag-drive pumps with solids that exceed the manufacturer's maximum limits in particle size or concentration.
- Before operating mag-drive pumps, confirm chemical compatibility of process liquid with all wetted pump components in an effort to reduce corrosion, permeation, or erosion.
- Do not operate mag-drive pumps with process liquids that may exceed the maximum temperature limits or fall below the minimum temperature limits defined by the pump manufacturer.
- Do not operate mag-drive pumps outside of the manufacturer's recommended operating range. Otherwise, recirculate adequate flow through bypass lines when operating near or below the manufacturer's recommended mechanical and thermal operating flow to prevent excessive temperature rise or recirculation cavitation.
- Consider the inner and outer magnet assemblies' material temperature limits and recoverable flux density losses due to increased temperatures.
- Do not operate mag-drive pump without considering the process liquid's vapor pressure characteristics over the temperature range of the application. Adequate NPSHa as well as vapor pressure is mandatory to prevent cavitation or vaporization in localized low pressure regions within the pump.
- Use power monitoring devices when potentially operating near or outside of the manufacturer's recommended operating envelope.
- Consider temperature controlling devices such as heat jackets or steam tracing for pumps that are subject to process fluids that transform characteristics such as viscosity, specific gravity, crystallization, coagulation, solidification, or vaporization with variable process temperatures.
- Use temperature monitoring devices such as thermocouples, RTDs, temperature controllers, or thermometers when process fluid is susceptible to critical variations in temperature.
- Use leak detectors such as fiber optic sensing devices or pressure monitoring devices when fluid is prohibited from entering the atmosphere.
- Ensure selected motor torque at maximum power and start-up conditions for corrected hydraulic and magnetic loss power at worst case fluid specific gravity and viscosity is less than the magnet break-away torque at maximum temperature.
- Keep process liquid in liquid form. Prevent the liquid from flashing.
- Do not exceed the manufacturer's maximum viscosity limits as internal fluid circulation velocities will be inadequate to properly cool and lubricate sleeve bearings.

HOW TO ORDER

When ordering parts call
1-800-446-8537
or your local Goulds Representative

EMERGENCY SERVICE

Emergency parts service is available
24 hours/day, 365 days/year . . .
Call 1-800-446-8537

Visit our website at gouldspumps.com

ITT

Form I3296EZMAG Rev. 06/08

Appendix F

Investigation-Derived Waste (IDW) Plan

APPENDIX F
Investigation-Derived Waste Management Plan

Former Charlotte Naval Ammunition Depot
Charlotte, North Carolina

December 2011

Revision 2 – May 2012

DERP-FUDS Project No.: I04NC080301
Contract No.:W912DY-10-D0025
Delivery Order No.: 0007

PREPARED FOR:

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U.S. Army Engineering and Support Center
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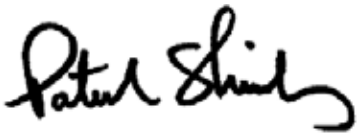

Patrick Shirley, PG
PIKA-PIRNIE JV Team Certified Project Manager

Investigation-Derived Waste Management Plan

Former Charlotte Naval Ammunition Depot
Charlotte, North Carolina

Prepared for:
U.S. Army Corps of Engineers,
Huntsville Center

Prepared by:
PIKA-PIRNIE JV, LLC
12723 Capricorn Drive
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Stafford, Texas 77477

Our Reference:
DERP-FUDS Project No.: I04NC080301
Contract No.: W912DY-10-D0025
Delivery Order No.: 0007

Date:
December 2011
Revision 2 – May 2012

Table of Contents

Acronyms and Abbreviations

CFR	Code of Federal Regulations
CNAD	Charlotte Naval Ammunition Depot
CVOC	Chlorinated Volatile Organic Compound
DO	Delivery Order
DOT	Department of Transportation
GAC	Granular Activated Carbon
IDW	Investigation-Derived Waste
JV	Joint Venture
PIKA	PIKA International, Inc.
PIKA-PIRNIE JV Team	PIKA International, Inc. (PIKA)/Malcolm Pirnie, Inc. (Pirnie) Joint Venture (JV), LLC
Pirnie	Malcolm Pirnie, Inc.
PPE	Personal Protective Equipment
SAP	Sampling and Analysis Plan
QAPP	Quality Assurance Project Plan
RAWP	Remedial Action Work Plan
TCLP	Toxicity Characteristic Leaching Procedure
U.S.	United States
USACE	United States Army Corps of Engineers, Huntsville Center
VOC	Volatile Organic Compound

1. Introduction

This document has been prepared on behalf of the United States (U.S.) Army Corps of Engineers, Huntsville Center (USACE) and presents the Investigation-Derived Waste (IDW) Management Plan for the remedial activities being implemented to address the presence of chlorinated volatile organic compounds (CVOCs) in groundwater originating from the Former Charlotte Naval Ammunition Depot (CNAD) property located in Charlotte, North Carolina. For the purposes of this Plan, the contamination source area, located at 2820 Nevada Boulevard (currently occupied by Norfolk Southern operation), will be referred to as the Site; while the Former CNAD Site will be used to represent all contiguous property affected by the release (Site).

Pursuant to the Delivery Order (DO) 0007 for Remedial Action, this IDW Management Plan identifies the types of waste generated and procedures to characterize, transport, and dispose of hazardous and non-hazardous waste associated with investigation and remediation activities at the Site.

2. Types of IDW

2.1 Water

Potential sources of waste water include drilling fluids, rinse water from equipment decontamination, monitoring well development and purge water, produced during Site activities. Water will be containerized in US Department of Transportation (DOT)-approved 55-gallon closed top drums or frac tanks, with secondary containment. All containers will be clearly labeled as "***This Container is On Hold Pending Analysis***" and include the following information:

- Generator name and address;
- Source and contents; and
- Generation date.

2.2 Soil

Drill cuttings from monitoring and injection well installation activities will be generated as part of the project. Cuttings will be placed in DOT-approved 55-gallon drums or covered roll off boxes until profiled. All containers will be labeled in the manner consistent with that specified above for water.

2.3 Disposable Sampling and Personal Protective Equipment (PPE)

PPE (*e.g.,* nitrile gloves, Tyvek coveralls), paper towels, acetate soil liners, low density polyethylene tubing, and other disposable materials generated during project activities will be collected in plastic garbage bags during the field activities. Disposal of these materials will include the following options:

- Placement in a 55-gallon drum and disposed of at non-hazardous waste landfill;
- Placement in a general refuse dumpster; or
- Placement in the roll off to be disposed of with the soil IDW.

3. Containment and Storage of IDW

Wastes generated at the Site will be placed in labeled DOT-approved containers. Miscellaneous disposable equipment and PPE will be contained in plastic garbage bags, and placed in 55-gallon drums, as described above. Generated waste will be segregated to the extent possible so that potentially clean media are not comingled with potentially impacted media that may be characterized as hazardous waste.

During the initial field effort including the installation of monitoring and injection wells, generated wastes will tentatively be stored in three secure areas at the Site. Accumulation Area #1 is proposed for the grassed area located on the southeast portion of the Norfolk Southern property, Both IDW liquids and solids are anticipated to be stored in this area. Waste generated from the installation of monitor wells NADMW69, NADMW70, NADMW72, NADMW73, NADMW74, NADMW75, NADMW76, and injection wells IW-31 through IW-53 for a total of 30 wells are anticipated to be stored here.

A second accumulation area (Accumulation Area #2) will be located on the north side of the Norfolk Southern property, north of the rail lines and east of the storm water pond. This area will contain solid and liquid wastes generated as part of the installation of monitoring wells NADMW68 and NADMW71 and injection wells IW-20 through IW-30 and BIW-16 through BIW-25 for a total of 23 wells. This area may be field modified to an area south of the existing fenceline, based on the final locations of the wells.

A third accumulation area will be on the Arrowood Southern property, located on the north side of Cordage Street. This area will contain solid and liquid wastes generated as part of the installation of monitor well NADMW-66 and 67, injection wells IW-1 through IW-19 and BIW-1 through BIW-15 for a total of 36 wells.

Each storage area will be temporary and will be maintained in good condition. All containers utilized will be sealed and or covered when not being actively loaded or unloaded. A waste inventory will be maintained to record and track IDW materials generated at the Site to ensure their proper management and off-site disposal. Periodic inspections of the storage areas will be performed and documented.

The three accumulation areas will be utilized during the installation of the monitoring and injection wells as part of the remedial system construction. Following completion of the remedial system construction, the generated waste will include purge water from the routine monitoring events. This water will be containerized in 55-gallon drums and stored in an enclosed secondary containment structure adjacent to one of the injection buildings.

4. Characterization of IDW

Historic site descriptions, provided in remedial reports, identify a trichloroethylene (TCE) vapor degreasing operation formerly conducted at the facility. While this confirms that TCE was used as a solvent on site, there is no confirmed evidence that the TCE in the groundwater was a direct result of this operation *(i.e.,* no source identified), no evidence as to the condition of the material, and no records identifying the concentration of the TCE, prior to use, exceeding the criteria that would make waste associated with the remedial actions "F" listed waste.

Based on these conditions, PIKA-PIRNIE proposes to handle wastes generated as part of this investigation in accordance with appropriate procedures, based on the toxicity characteristics of the material. IDW, generated during investigations will be screened for toxicity. Material containing concentrations of TCE exceeding toxicity criteria will be handled as hazardous waste, while concentrations below this criteria will be treated as non-hazardous.

4.1 Water

All containerized water will be sampled in accordance with the procedures identified in the Field Sampling Plan (SAP) (Appendix C-1 of the Remedial Action Work Plan [RAWP]). Subsequent analysis for characterization will be performed according to the Quality Assurance Project Plan (QAPP) (Appendix C-2 of RAWP), to determine if the groundwater is hazardous or non-hazardous.

Groundwater will be sampled for Site-related VOCs and the results will be compared to toxicity characteristic levels specified in 40 Code of Federal Regulations (CFR) 261.24, in order to determine the appropriate waste classification (hazardous or non-hazardous). Based on the analytical data, the groundwater will be properly maintained in the respective accumulation areas and disposed off site. Under no circumstances will water that has contacted potentially contaminated materials be discharged to any water body.

If laboratory analytical results of IDW water show compounds present at concentrations exceeding toxicity characteristic levels specified in 40 Code of Federal Regulations (CFR) 261.24, on-site treatment of water, including use of an air bubbler line, air stripping, granular activated carbon (GAC) filtration, or a combination of these, may be utilized in order to reduce concentrations of compounds to non-hazardous levels. Treatment will be provided by pumping generated waters from one frac tank, treating waters via air stripping or carbon absorption technologies, then to a second frac tank. Treated water would then

be re-sampled to verify the concentrations of compounds present. This procedure may be repeated multiple times in order to achieve the concentrations desired. If necessary, permits will be obtained for these on-Site treatment options.

4.2 Soil

Soils will be sampled in accordance with procedures outlined in the FSP. Subsequent analyses will be performed according to the QAPP, to profile the soil as either hazardous or non-hazardous. Composite soil samples will be collected and initially analyzed for toxicity characteristic leaching procedure (TCLP) metals and TCLP VOCs in accordance with the method specified in the QAPP. If contaminants are identified, the concentrations will be evaluated with respect to the promulgated toxicity characteristic levels specified in 40 CFR 261.24. Contaminated soil with the TCLP concentrations above the maximum levels in 40 CFR 261.24 will be classified as a characteristic hazardous waste. Contaminated soil with TCLP VOCs concentrations below the promulgated regulatory levels will be classified and disposed of as non-hazardous waste.

4.3 Disposable Equipment and PPE

It is presumed that all disposable equipment (*i.e.,* paper towels, plastic sampling sleeves, and plastic tubing) and PPE (*e.g.,* nitrile gloves, Tyvek coveralls), will be classified as non-hazardous solid waste. Therefore, no samples of these materials will be collected for waste characterization purposes. (see Section 2.3)

5. Waste Management

5.1 On Site

No wastes generated will be disposed of on Site. Wastes will be temporarily stored in designated accumulation areas until proper waste characterization, transportation and disposal can be completed. Proper notification will be made to the appropriate agencies in the event of a spill of investigative derived waste material. Property owners will also be notified of any spills on their respective properties.

5.2 Off Site

Any hazardous waste generated during Site activities will be labeled, marked, placarded, and transported in accordance with DOT Hazardous Materials Regulations (49 CFR Parts 171 through 180), 40 CFR Part 263, and applicable State regulations. Individuals who participate in hazardous materials shipping will have the necessary DOT training stipulated in the regulations cited above. Site personnel shipping hazardous materials will complete a Hazardous Materials Shipment Form included with the field forms in the SSHP (Appendix B-2 of the RAWP). All hazardous waste will be transported to and disposed of at a permitted Hazardous Waste Treatment, Storage, and Disposal Facility. All hazardous waste shipping manifests will be completed and signed by USACE personnel, identifying the USACE as the Waste Generator. PIKA International, Inc. (PIKA)/Malcolm Pirnie, Inc. (Pirnie) Joint Venture (JV), LLC (the PIKA-PIRNIE JV Team) members will assist in the preparation and coordination of the waste removal.

Non-hazardous waste, including solids and liquids, will be transported off site to a non-hazardous waste disposal facility. All non-hazardous waste shipping manifests will be completed and signed by USACE personnel, identifying the USACE as the Waste Generator. PIKA-PIRNIE JV Team members will assist in the preparation and coordination of the waste removal.

Appendix G

Construction Quality Control Plan
(CQCP)

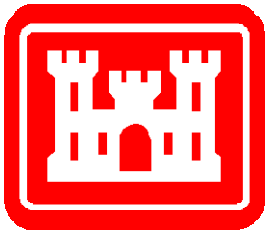
Appendix G
Construction Quality Control Plan

Former Charlotte Naval Ammunition Depot
Charlotte, North Carolina

December 2011

DERP-FUDS Project No. I04NC080301
Contract No.:W912DY-10-D0025
Delivery Order No.: 0007

PREPARED FOR:



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Construction Quality Control Plan

Former Charlotte Naval Ammunition Depot
Charlotte, North Carolina

Prepared for:
U.S. Army Corps of Engineers,
Huntsville Center

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Acronyms and Abbreviations

BMP	Best Management Practice
CNAD	Charlotte Naval Ammunition Depot
CO	Change Order
CQC	Construction Quality Control
CQCP	Construction Quality Control Plan
ERD	Enhanced Reductive Dechlorination
H&S	Health and Safety
IDW	Investigation-Derived Waste
JV	Joint Venture
MNA	Monitored Natural Attenuation
NAD	North American Datum
NCDENR	North Carolina Dept. of Environment and Natural Resources
PC	Performing Contractor
PIKA	PIKA International, Inc.
PIKA-PIRNIE JV Team	PIKA International, Inc./Malcolm Pirnie, Inc. Joint Venture LLC Team
Pirnie	Malcolm Pirnie, Inc.
PM	Project Manager
PMP	Project Management Plan
QA/QC	Quality Assurance/Quality Control
QC	Quality Control
RA	Remedial Action
RAR	Remedial Action Report
RAWP	Remedial Action Work Plan
T&M	Time and Materials
US	United States
USACE	United States Army Corps of Engineers, Huntsville Center

1. Introduction

This Construction Quality Control Plan (CQCP) establishes the organization, guidelines and uniform procedures to be followed by the Project Team associated with the completion of remedial actions at the former Charlotte Naval Ammunition Depot (CNAD) located in Charlotte, North Carolina. The CQCP provides guidance on what is expected regarding the range of construction management functions and the roles and responsibilities of the organizational structure to provide a comprehensive management approach. The CQCP will describe the Construction Quality Control (CQC) functions performed by the performing contractor (PC), PIKA International, Inc. (PIKA)/Malcolm Pirnie, Inc. (Pirnie) Joint Venture (JV), LLC (the PIKA-PIRNIE JV Team JV Team). Quality Control (QC) is defined as the totality of features, attributes, and characteristics of a facility, product, process, component, service, or workmanship that bear on its ability to satisfy a given need: fitness for purpose. It is usually referenced to and measured by the degree of conformance to a predetermined standard of performance. In simple terms, quality is meeting the United States (US) Army Corps of Engineers', Huntsville Center (USACE's) requirements and striving to meet the underlying goal of perfection. The primary objective of the CQCP is to establish the framework required to facilitate meeting the USACEs project goals of implementing remedial actions at the Former CNAD facility.

1.1 Project Background

A summary of the project background and history can be seen in Section 2 of the Remedial Action Work Plan (RAWP).

1.2 Definitions

Whenever the terms listed below are used, the intent and meaning shall be interpreted as indicated.

CQC: Those actions which provide a means to measure and regulate the characteristics of an item or service to comply with the requirements of the contract documents. QC will be performed by the PIKA-PIRNIE JV Team, except where designated in the Specifications.

CQC Manager: Authorized representative of the CQC organization responsible for managing the CQC program.

CQC Monitors: Authorized representative of the CQC organization, responsible for observing and documenting activities related to CQC during construction.

CQC Officer: Authorized representative of the CQC organization and professional engineer licensed in North Carolina where the project is located, responsible for certifying that construction was performed in accordance with the intent of the contract documents and design.

CQC Team: Authorized representatives of the PIKA-PIRNIE JV Team CQC organization which include the Project Manager (PM), CQC Manager, CQC Monitor, and CQC Officer.

CQCP: This plan, generated by the PIKA-PIRNIE JV Team will outline the procedures and processes that will be implemented by all parties throughout the duration of the project. This plan will serve as guidance to ensure that implementation of the remedial actions (RAs) at the Former CNAD is in accordance with the project documents.

Contract Drawings: The official plans, profiles, typical cross-sections, elevations, and details, as well as their amendments and supplemental drawings, which show the locations, character, dimensions, and details of the work to be performed. Contract drawings are also referred to as “plans”, “construction drawings”, or “permit drawings”.

Contract Specifications: The qualitative requirements for products, materials, and workmanship upon which the contract is based. Contract specifications are also referred to as “Project Specifications” or “Construction Specifications”.

Non-Conformance: A deficiency in characteristic, documentation, or procedure that renders the quality of an item or activity unacceptable or indeterminate. Examples of non-conformance include, but are not limited to, physical defects, test failures, and inadequate documentation.

Owner (USACE): The USACE is the Owner of the contract for the Former CNAD facility in Charlotte, North Carolina. The USACE staff dedicated to the project is responsible for the overall management and decision making for the project.

PC (PIKA-PIRNIE JV Team): The individual or firm responsible for administering the construction contract and providing overall construction services for the project including planning, organizing, and control of the design and construction activities. Responsibility includes scheduling, cost control, engineering, procurement, and

contracting functions. The PIKA-PIRNIE JV Team Field Engineer and CQC Monitor are the primary contacts on the project site representing the owner.

Procedure: A document that specifies or describes how an activity is to be performed.

Project Documents: Subcontractor submittals, construction drawing, record drawings, specifications, shop drawings, construction quality assurance/quality control (QA/QC) plans, health and safety (H&S) plan, and project schedule.

Record Drawings: Drawings recording the constructed dimensions, details, and coordinates of the project. Also referred to as "as-builts."

Subcontractor(s): The person or persons, firms, partnership, corporation, or any combination, private, who as an independent subcontractor, has entered into a contract with the PIKA-PIRNIE JV Team and is responsible for the implementation of certain construction activities.

Surveyor: The entity licensed in the state of North Carolina to make and document measurements as necessary to determine relative positions of the construction.

Testing: Verification that an item meets specified requirements by subjecting that item to a set of physical, chemical, environmental, or operating conditions.

2. CQCP Roles and Responsibilities

The roles and responsibilities of the PIKA-PIRNIE JV Team are detailed in Appendix D – Project Management Plan (PMP).

2.1 USACE (Owner)

As stated in Section 1.1, the USACE retains responsibility for environmental clean-up activities related to past releases at the Former CNAD facility, even though it no longer owns the property. The USACE Program Manager for this work is **Ms. Julie Hiscox**. The USACE PM dedicated to the project who is responsible for the overall management and decision making for the project is **Mr. Ray Livermore**.

2.2 PIKA-PIRNIE JV Team (PC)

The PIKA-PIRNIE JV Team's primary role on this project during the construction phase is to serve as the USACE's PC. Activities that will be carried out by the PIKA-PIRNIE JV Team will include providing appropriate means to deliver on time the agreed upon project goals. The PIKA-PIRNIE JV Team will also conduct communications with the USACE on a periodic basis to relay the progress of field activities, communications with other interested parties including the site occupant (Norfolk Southern and Arrowood-Southern Company), the North Carolina Department of Environment and Natural Resources (NCDENR), and other surrounding properties. Additional services include reviewing subcontractor(s) submittals, resolving technical issues related to construction, providing interpretation of the drawings and specifications and approving substantial design modifications and technical revisions. The PIKA-PIRNIE JV Team is ultimately responsible for the successful implementation of the remedial system on behalf of the USACE.

Listed below are the names and duties of the primary project members of the PIKA-PIRNIE JV Team:

- **Mr. Patrick Shirley, PG** – Mr. Shirley will serve as the principal PM for the Former CNAD project. He will be responsible for the management of the project, including maintaining cost and schedule, ensuring completion of the project in accordance with the contract documents, and maintaining a culture of H&S throughout the duration of the project.

- **Mr. Andrew Davis, PE** – Mr. Davis will serve as the project/task manager for the Former CNAD project. He will provide management of the project on a daily basis and communicate with the various team members while reporting back to the PIKA-PIRNIE JV Team and USACE PMs. Mr. Davis will also serve as the CQC Officer and is the authorized representative of the CQC Team and licensed professional engineer in North Carolina, and will be responsible for oversight, review, and approval of all work completed under this contract and certifying that the construction was performed in accordance with the construction documents for the Former CNAD project.

- **Mr. Tim Hays** – Mr. Hays will serve as the CQC Monitor/field engineer/H&S inspector during the Former CNAD project. He will be responsible for the oversight of daily activities on behalf of the PIKA-PIRNIE JV Team, and reporting back to the CQC Managers and PMs for the PIKA-PIRNIE JV Team.

3. Communications

In order to facilitate construction, and to clearly define construction goals and activities, close coordination between the USACE, the PIKA-PIRNIE JV Team, the NCDENR, property owners, and subcontractor(s) is essential. To meet this objective, pre-construction and progress meetings will be held.

3.1 Pre-Construction Meetings

In advance of construction activities, a pre-construction meeting will be held at the site. The purpose of this meeting, attended by the USACE (if applicable), the PIKA-PIRNIE JV Team, primary subcontractor, impacted property owners, will be to:

- Review the construction drawings, specifications, CQCP, work area security, H&S procedures, and related issues.
- Provide all parties with relevant project documents.
- Review responsibilities of each party.
- Define lines of communication and authority.
- Establish reporting and documenting procedures.
- Review procedures for handling submittals.
- Review testing equipment and procedures.
- Review procedures for field directives and Change Orders (COs).
- Establish testing protocols and procedures for correcting and documenting construction or non-conformance.
- Conduct a site inspection to discuss work areas, stockpile areas, lay down areas, access roads, haul roads, and related items.
- Review the project schedule and critical path items.

- The meeting will be documented by the CQC Manager or designated Field Manager. Copies of the minutes and relevant documents will be prepared and provided to all parties.

3.2 Daily Meeting

During construction activities, daily meetings will be held each morning prior the start of work. At a minimum, this meeting will be attended by the CQC Monitor, subcontractor(s), and any applicable personnel (property owners, operators). The purpose of this meeting is to:

- Discuss daily activities.
- Discuss the subcontractor's personnel and equipment assignments for the day.
- Review the previous day's activities and accomplishments.
- Resolve any outstanding problems or disputes.

3.3 Weekly Progress Meetings

Weekly progress meetings shall be held, when necessary. The CQC Manager, CQC Monitor, and subcontractor(s) will be present in person or by teleconference. The meetings will be held to discuss progress, problems, construction schedule, changes, test data, safety, environmental issues, and any other issues necessary. The CQC Manager will prepare the agenda for each meeting and prepare meeting minutes for distribution to all parties.

4. Project Schedule

The PIKA-PIRNIE JV Team will prepare and submit for approval a baseline schedule using the latest version of Microsoft Project (see Figure 6-1 of the RAWP). The schedule shall incorporate critical dates and milestones and will be reviewed and agreed upon by the PIKA-PIRNIE JV Team, USACE, and the applicable subcontractors prior to the start of construction. The schedule is used to establish the plan for the work, to monitor progress, and to plan and identify upcoming activities to allow the CQC Team to proactively address issues which may impede construction progress.

The project schedule shall be updated as needed to reflect adherence to the baseline and to provide for a minimum 2-week look ahead. The project schedule will be reviewed in the weekly project status meetings with the objective of identifying any corrective actions that may be required to meet project milestones.

5. Construction Quality Management

The PIKA-PIRNIE JV Team has prime responsibility for constructing the work in accordance with the Contract Documents, including the plans, specifications, construction contracts, permits, and other related documents. The Contract Documents specify the QC activities that the PIKA-PIRNIE JV Team must implement to assure that the construction is being performed to the desired level of quality. These activities are typically associated with the testing of the installed material or system (*i.e.,* compaction testing on engineered fills at regular intervals, concrete cylinder tests at specified quantities, pressure testing, etc.). The PIKA-PIRNIE JV Team has prepared the design documents which will specify the QC activities required.

5.1 Responsibilities of CQC Team

5.1.1 Communications with Subcontractors

Communications of an official nature must be clear, direct, and professional. When written communications are required, they must be documented on the appropriate forms. Copies of applicable communications will be provided to the USACE, the PIKA-PIRNIE JV Team, applicable property owners, and the NCDENR when applicable.

5.1.2 Communications with Owners

Only those individuals assigned to this project, as defined in this manual, will communicate with representatives of the USACE. All communications must be through proper channels as defined in the project organization chart. Communications of an official nature must be written, clear, direct, and professional.

5.1.3 Responsibilities of CQC Managers

The CQC Manager administers the CQC program. CQC procedures and reports must be reviewed by the CQC Manager for compliance with the project CQCP. The CQC Manager acts as an auditor to monitor and document the proper and complete implementation of the CQCP. The CQC Manager has authority to identify deficiencies and implement corrective action to the CQCP. The CQC Manager will ensure that field as-built/record drawings are being maintained and updated as required. The CQC Manager reports directly to the PM and CQC Officer.

5.1.4 Responsibilities of CQC Monitors

The CQC Monitors implement the CQCP under the direction of the CQC Manager. The CQC Monitors perform all construction monitoring and construction materials testing. The CQC Monitors maintain all documentation and test data summaries related to construction monitoring and construction material testing. The CQC Monitors report directly to the CQC Manager.

5.2 Control of Documents, Records, and Field Log Forms

Document control and records management includes the indexing, filing, distribution, control and retrieval of all documentation that is received or generated during the construction project. Complete, thorough documentation is the basis for claims avoidance and claims management. Thorough documentation of every aspect of the Contract is imperative.

Anticipated document types, include, but are not limited to, the following:

- Monthly schedule updates;
- Monthly Progress Reports;
- Remedial Action Report (RAR);
- Performance Monitoring Reports;
- Monitored Natural Attenuation (MNA) Monitoring Reports;
- QC Inspector Daily Reports;
- Meeting Minutes;
- Transmittals;
- Contracts;
- Proposals;

- Photos;
- Safety Records;
- Warranties;
- COs and Field Orders;
- E-mail Correspondence;
- Application for Payments;
- Drawings and Design Submittals;
- Permits (if required); and
- Quarterly Progress Reports.

The PIKA-PIRNIE JV Team Administrative Document Control Specialist (Ms. Heather Kirlin) is responsible for receiving, date stamping, distributing, logging, filing, indexing for retrieval, and archiving all project documents for project use and historical purposes as they pertain to the PIKA-PIRNIE JV Team's internal filing system.

5.2.1 Project Control of Contract Documents

Contract documents, including specifications, drawings, and COs, are controlled by the PM. The PM maintains one or more copies of the most current set of contract documents for use by the PIKA-PIRNIE JV Team. Upon issuance of new copies or revisions, it is the responsibility of the PIKA-PIRNIE JV Team to notify applicable personnel of the revisions, provide revised contract documents, and order the recall or replacement of all unrevised copies of the contract documents.

5.2.2 Project Design Modifications

Design and specification changes may be required during construction. Design and specification changes will only be made with written agreement of the CQC Manager/design engineer, PM, USACE, and NCDENR. These changes will be made

by CO to the contract. When COs are issued, they will be prepared by the PM. The PM will distribute COs for signature and execution to the required parties.

5.2.3 Project Control of Record Drawings

Record drawing(s) information generated by the PIKA-PIRNIE JV Team is controlled by the CQC Manager. During the progress of the work, the CQC Manager obtains as-built information provided from the CQC Monitors, subcontractor, surveyors, or others and compiles all as-built data onto one set of drawings. The record drawing set must be maintained and clearly marked as Record Drawings.

5.2.4 Project Control of Field Log Forms and Photographs

Daily report forms, test report forms, and other project forms (See **Appendix A** of this document) are controlled by the CQC Manager who maintains a master of each form for copies. Upon issuance of a new form, the CQC Manager must recall and remove all superseded copies along with the master, notify the CQC Monitors, and provide new copies for their use. Photographs will be taken periodically during all site activities to document progress using a digital camera. On a routine basis (weekly at a minimum) the photographs will be downloaded from the camera to the PIKA-PIRNIE JV Team server and organized by activity for which they represent (well installation, system installation, enhanced reductive dechlorination [ERD] operation) and date.

5.2.5 Processing Daily Reports

The CQC Monitors will write a daily and weekly record of work progress. The daily and weekly reports are reviewed by the CQC Manager for legibility, clarity, traceability, and completeness. The review must be evidenced by signature. The weekly summary construction report will be prepared by field personnel and submitted to the CQC Manager for review and approval.

5.2.6 Processing Testing Reports

A test report must be completed by the CQC Monitors whenever testing is performed. The test reports must be reviewed by the CQC Manager. The review includes a check for mathematical accuracy, conformance to test requirements, conformance to specifications, and for clarity, legibility, traceability, and completeness. The review must be evidenced by a signature of the reviewer. Test reports (or summaries) from

independent testing laboratories will also be transmitted to the CQC Manager for review.

5.3 Materials and Equipment Control

5.3.1 Submittal List

Material submittals may be used by the CQC Team to establish the acceptability of materials. When sample submittals are required, they will be made available to the CQC Team. Acceptance and proper review of submittals are the responsibility of the CQC Manager.

5.3.2 Shop Drawings and Submittal Approvals

All shop drawings and submittals are due during the pre-construction phase of the project. Only the major pieces of equipment and key materials are expected to be reviewed by the CQC Team. The shop drawings shall include sufficient information and detail to verify that the proposed equipment/materials are consistent with the approved Contract Documents. The CQC Team review of the submittals does not relieve the PIKA-PIRNIE JV Team from their responsibility of meeting the requirements of the Contract.

Shop drawings will be submitted in advance of construction activities in order to allow sufficient time for review, and for necessary revisions and re-submittals. Before submitting each shop drawing, the subcontractor is responsible to have determined and verified all field measurements, quantities, and performance criteria. After review, the CQC Team will give the USACE specific written notice of any variations that the shop drawing may have from the requirements of the Contract Documents.

5.3.3 Record "As-Built" Documents

As part of the project, record drawings will be prepared and maintained by the PIKA-PIRNIE JV Team, and will show the precise, as-built locations of all buried, imbedded or concealed piping or conduit, including piping or conduit fixtures, fittings and accessories, and other buried features installed by the PIKA-PIRNIE JV Team. Structural and mechanical features will also be shown on the record drawings. Upon Substantial Completion of the project, the PIKA-PIRNIE JV Team will submit to the USACE one (1) complete set of record drawings. USACE will review the record

drawings for accuracy to the extent possible and provide the PIKA-PIRNIE JV Team with comments for correction and re-submittal. USACE's review of the record drawings does not relieve the PIKA-PIRNIE JV Team from their responsibility to provide accurate drawings reflecting as-built conditions.

5.3.4 Testing

The PIKA-PIRNIE JV Team is required to perform materials and product testing to conform to the requirements of the Contract Documents. All materials provided and work performed shall be protected from damage before and after installation. The PIKA-PIRNIE JV Team shall be responsible for work, equipment, and materials until inspected, tested, and finally accepted in accordance with the technical specifications. The subcontractor will conduct verification testing in the following areas:

- All piping will need to be hydrostatic (water conveyance piping) or pneumatically (process air piping) tested as specified on the Design Drawings (Appendix A of the RAWP). If tests indicate the work does not meet specified requirements, remove the work, replace, and retest.

- Trenching backfill shall be field verified for proper compaction and accepted by the consultant/engineer. No fill shall be placed over a layer that has not been observed and accepted.

- Manufacturer's representatives shall perform functional testing of all significant process equipment before startup.

- Instrumentation, controls and complete system-integration shall be tested by the PIKA-PIRNIE JV Team and subcontractor prior to system startup. Tests shall be conducted to determine whether the equipment has been properly assembled, aligned, adjusted, wired, or connected. The demonstration test of each piece of equipment shall include check-out from the control panel. All alarm systems and safety lockout systems shall be demonstrated for proper function along with all process instrumentation and controls.

5.3.5 Delivery

Upon delivery of any construction related materials/equipment, the CQC Monitor will:

- Observe materials for damage during shipping and handling. Identify damaged materials and document that damaged materials are set aside.

- Observe that the materials are in accordance with the specifications and are protected from water, mechanical abrasions, excessive heat, direct sunlight, and other damage.

- Document that all manufacturing documentation required by the specifications has been received.

- Document the condition of the site, pre-arrival, in order to document that no damage occurred to the site as a result of the delivery.

5.3.6 Inspections

QC inspections will be conducted on a daily basis by the CQC Monitor to ensure that the provisions of this CQCP are being implemented. The inspections will be documented as follows:

Daily QC Inspection Reports

The CQC Monitor is required to record daily inspections and observations on a Daily CQC Inspection Report Form. Recording information in these daily reports establishes factual entries that may not otherwise be recalled and reconstructed at a later date. In addition, these reports provide recorded evidence that the PIKA-PIRNIE JV Team is in compliance with the CQCP requirements.

The primary components of the daily report include the following:

- QC Inspector's name and the date

- Weather conditions

- Resources (labor and equipment on each item of work inspected)

- Work being performed

- Observations of inspections and time of inspection, including out of sequence schedule activities observed
- Description of deficiencies or issues and how corrected
- Re-inspections of work resulting in closeout of non-conformances

The CQC Monitor must complete and submit a summary of the Daily Inspection Report to the CQC Manager each day. All entries shall be clear, concise, and factual; personal opinions are not to be recorded. Inspections shall be documented in detail recording how the work is performed, comparing the work to the Contract Documents and stating the extent of compliance with these documents.

5.3.7 Compliance and Conformance Control

The PIKA-PIRNIE JV Team is responsible for complying with all federal, state, and local rules, regulations, requirements established by the regulatory agencies associated with environmental protection. The CQC Team is responsible for monitoring environmental compliance and verifying that all regulatory and environmental requirements are met.

Environmental inspection and monitoring is a component of the construction management quality program presented in this CQCP. The PIKA-PIRNIE JV Team has overall responsibility for the implementation of the environmental monitoring aspect of the CQCP; the CQC Monitor will include environmental monitoring as part of their daily inspection activities. This monitoring will be performed to verify that all permit conditions are being achieved and that the subcontractor activities are in compliance. Documentation of these items will be provided in the Daily Inspection Report Forms.

5.4 Survey Control

Surveying of lines and grades shall be conducted during construction of system components. Surveying shall be performed to provide documentation for record drawings.

5.4.1 Survey Control

Existing benchmarks will be used for the site survey control to establish the horizontal location and vertical elevation (North American Datum [NAD], 1983) and an on-site benchmark, respectively. The vertical and horizontal controls for each site benchmark have been established within normal land surveying standards.

5.4.2 Surveying Personnel

Surveying will be performed under the direct supervision of a Registered Land Surveyor licensed in the State of North Carolina where the project is located. The survey crew should consist of the Senior Surveyor and as many Surveying Assistants as are required to satisfactorily undertake the work. Surveying personnel shall be experienced in the provision of these services in addition to preparing detailed and accurate documentation.

5.4.3 Documentation

The Surveyor shall retain original field survey notes. A copy of these notes will be given to the CQC Manager prior to the covering of the surveyed component. The results from the field surveys will be used as the basis for the preparation of the record drawings. At a minimum, these drawings shall show the final vertical and horizontal, and elevations for the items listed in the design documents.

5.4.4 Certification

Survey results shall be certified by a land surveyor or professional engineer and submitted to the CQC Manager for review.

5.5 CO Procedures

Any modification which will alter the Contract Documents or the nature, scope or quality of the work, regardless of whether or not an adjustment in contract price or time will result, will be initiated and accomplished in writing. The following procedures will be followed in processing COs:

- A Change Request Form shall be completed for all COs to the Contract Documents consistent with the requirements of the USACE. This form will be submitted to the USACE PM for review and approval.

- Minor Field Changes: Immediate, minor field changes (minor means that the changes does not impact functionality, safety parameters, structures, community, regulatory compliance, and/or go beyond the cost/schedule threshold established by the project management team for that baseline) that affect the work or site/system configuration can be approved at the CQC field lead with additional approval by the PIKA-PIRNIE JV Team management team. These changes shall still be documented on the Change Request Form with the proper approvals from the aforementioned Leads prior to implementation of the field change.

- In the case of a modification initiated by the USACE, the PIKA-PIRNIE JV Team will provide a proposal or estimate for the modification. This letter of request will include a description of the modification proposed, an explanation of why the modification is required and a statement as to the method of payment to be used.

- The PIKA-PIRNIE JV Team will submit in writing sufficient documentation showing the basis for the proposed modification amount. If lump sum, the modification will at a minimum show the estimated materials and labor breakdown so the USACE can properly evaluate the proposed work and costs. If "time and materials" (T&M), the modification will have the proper documentation attached, including estimates of T&M.

- Upon receipt of the subcontractor's proposal, the USACE will review to determine if the proposal is reasonable and acceptable.

- When the modification is determined to be acceptable to all parties, the USACE will prepare a CO. The CO will include a description of the proposed modification, an explanation of why the modification is required, the agreed upon price, and the method of payment. Any work performed prior to the CO being issued will be at the PIKA-PIRNIE JV Team's own risk.

6. Compliance Monitoring

The PIKA-PIRNIE JV Team is required to review and revise plans (as needed) associated with environmental protection during construction; the CQC Team will review and comment on these plans and will provide oversight of the implementation of these plans. The plans will include the following:

- ***Investigation Derived Waste (IDW) Management Plan***: An existing IDW Management Plan has been prepared (Appendix F of the RAWP) and will be referenced during the completion of the project. These include a description of measures taken regarding the management of soil, groundwater, and decon water generated during the implementation of remedial activities.

- ***Erosion Control and Sedimentation Plan:*** Due to the limited areas of disturbance planned for during all construction activities an Erosion Control and Sedimentation Plan is not required for submittal to the regulatory agency. However, industry standard Best Management Practices (BMPs) will be utilized to control impacts from erosion and sedimentation runoff during construction.

The subcontractor's adherence to the requirements of these plans and procedures will be documented in the Daily QC Inspection Reports by the CQC Monitors.

Appendix A

Example Field Forms

Daily Log

Page ____ of ____

Project # _____ Date _____

Site Location _____ Prepared By _____

Date/Time	Description of Activities

Drilling Safety Checklist

Rev: 0 - 6/9/08

Planning with Driller Prior to Drilling

1) Drilling location accessible for rig and has proper ground support? ☐
2) Utility clearance markings present and clearance checklist completed? ☐
3) Area clear of trip hazards and combustibles (dry, tall grass)? ☐
4) For high traffic areas, work area is secured by high visibility cones or tape? ☐
5) Staging and decon areas appropriate for rig and support vehicles? ☐
6) Communication systems set for emergencies (verify cell phone coverage)? ☐
7) Hand signals defined and understood by all workers? ☐
8) Emergency assembly areas identified? ☐

Inspections by ARCADIS

1) Rig and equipment in clean condition (free of oil leaks, grease, mud)? ☐
2) All contract-required equipment and supplies on site? ☐
3) Driller certified per State requirements?

Inspections by Driller and Observed by ARCADIS

1) Rig inspected for leaks prior to start of work and daily? ☐
2) Hydraulics hoses inspected daily? ☐
3) Cables, ropes, hoists inspected daily for damage, wear, fraying? ☐
4) Fire extinguishers present and in good condition? ☐
5) First aid kits clean and adequately supplied? ☐
6) Emergency stops clearly identified and functional? ☐
7) Back up alarms functional (rig and support vehicles, as appropriate)? ☐
8) General rig/support vehicle maintenance (tires, fluids, etc) ☐
9) Augers, rods, sampling devices in functional condition ☐

Drill Rig Operation

1) Rig stable with jacks deployed? ☐
2) Right tools available for the job (e.g., no "cheater" bars)? ☐
3) Adequate barricade or exclusion zone established (boom height minimum) ☐
4) Adequate swing clearance (hoists)? ☐
5) Hand shovel to bare soil before drilling (no subgrade hazards) ☐
6) Defined housekeeping area to prevent falls (behind rig, decon area)? ☐
7) Spotters used to back rig and support vehicles? ☐

IDW Management

1) Drums staged properly (bolts and labels facing out, adequate access) ☐
2) Drums, roll off boxes, tanks labeled and dated? ☐
3) Roll off boxes lined with plastic and have functional doors and tarp? ☐
4) Liquid frac tanks equipped with fall protection? ☐
5) Soil stockpiles sized to allow covering with sheeting and containment? ☐

Personal Protective Equipment

1) Workers wearing PPE specified by HASP/JSA/Work Order ☐
2) Client PPE requirements are followed? ☐
3) Fall protection used when working at height (on rig mast, etc.)? ☐

NOTE: This checklist is intended to address common safety issues associated with general environmental drilling operations. The checklist is not meant to be comprehensive and does not replace the site-specific Health and Safety Plan, client safety requirements, or the contractor's company safety requirements. Specific drilling methods and certain project sites will have additional inspection and safety requirements. Contact the project health and safety officer if you have questions about the safety of the drilling equipment or site conditions.

PIKA INTERNATIONAL, INC. A JOINT VENTURE MALCOLM PIRNIE

SOIL CORE / SAMPLING LOG

Boring/Well _____ Project/No. _____ Page _____ of _____

Site Location _____ Drilling Started _____ Drilling Completed _____

Drilling Contractor _____ Driller _____ Helper _____

Drilling Fluid Used _____ Drilling Method _____

Length and Diameter of Coring Device _____ Sampling Interval _____ feet

Land-Surface Elev. _____ feet ☐ Surveyed ☐ Estimated Datum _____

Total Depth Drilled _____ Feet Hole Diameter _____ Coring Device _____

Prepared By _____ Hammer Weight _____ Hammer Drop _____ ins.

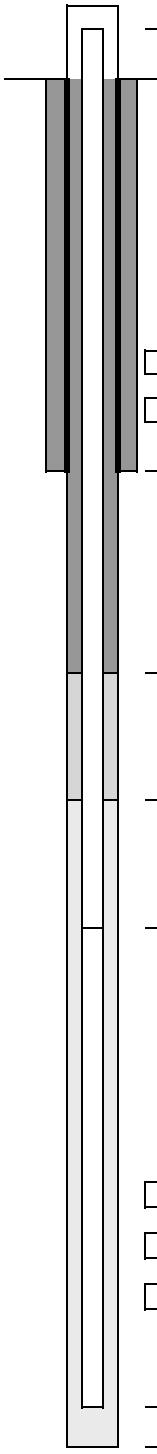
Sampling Data:

Depth	Grab/Composite	Time	Laboratory Analysis

Soil Characterization:

Sample/Core Depth (Feet bls) From	To	Core Recovery (Feet)	OVM Reading (ppm)	Blow Counts per 6 Inches	Sample/Core Description Soil type, %, Grain Size, Angularity, Grading, Consistency, Plasticity, Color, etc.

WELL CONSTRUCTION LOG- TELESCOPING



____ ft
LAND SURFACE

12 inch diameter drilled hole

Outer well casing,

6 inch diameter, PVC Surface Casing

☐ Backfill

☐ Grout _____

_____ ft*

Inner Well casing

____ inch diameter,

_____

_____ ft*

☐ slurry

Bentonite

_____ ft* ☐ pellets

_____ ft*

Well Screen.

_____ inch diameter

_____, _____ slot

☐ Gravel Pack

☐ Sand Pack

☐ Formation Collaspse

_____ ft*

_____ ft*

Measuring Point is
Top of Well Casing
Unless Otherwise Noted.

* Depth Below Land Surface

TBD = To be determined.

Project _____ Well _____

Town/City _____

County _____ State VA

Permit No. _____

Land-Surface Elevation and Datum:

_____ feet ☐ Surveyed

☐ Estimated

Installation Date(s) _____

Drilling Method _____

Drilling Contractor _____

Drilling Fluid _____

_____

Development Technique(s) and Date(s)

_____

_____

_____

Fluid Loss During Drilling _____ gallons

Water Removed During Development _____ gallons

Static Depth to Water _____ feet below M.P.

Pumping Depth to Water _____ feet below M.P.

Pumping Duration _____ hours

Yield _____ gpm Date _____

Specific Capacity _____ gpm/ft

Well Purpose _____

_____

_____

Remarks _____

_____

_____

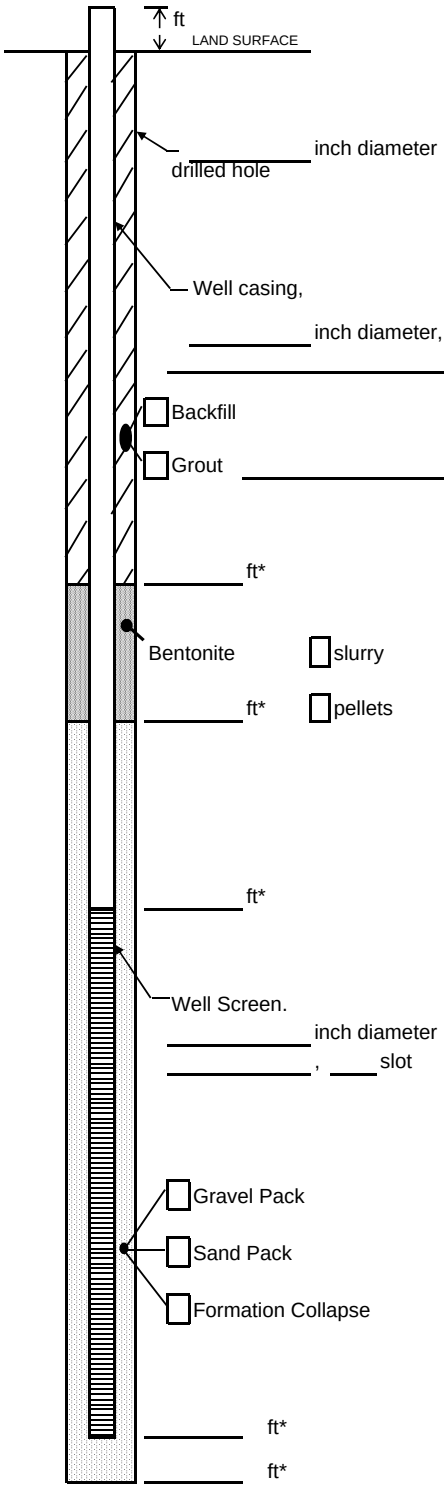
_____

_____

Prepared by _____

Well Construction Log

(Unconsolidated)



Measuring Point is
Top of Well Casing
Unless Otherwise Noted.

* Depth Below Land Surface

Project _____ Well _____

Town/City _____

County _____ State _____

Permit No. _____

Land-Surface Elevation and Datum:

_____ feet ☐ Surveyed ☐ Estimated

Installation Date(s) _____

Drilling Method _____

Drilling Contractor _____

Drilling Fluid _____

Development Technique(s) and Date(s)

Fluid Loss During Drilling _____ gallons

Water Removed During Development _____ gallons

Static Depth to Water _____ feet below M.P.

Pumping Depth to Water _____ feet below M.P.

Pumping Duration _____ hours

Yield _____ gpm Date _____

Specific Capacity _____ gpm/ft

Well Purpose Injection

Remarks _____

Prepared by _____

Monitoring Well Development Log

Page _____ of _____

Project/No. _____ Well _____ Date _____

Total Depth _____ Casing Diameter (inches) _____ Purge Method

Water Level _____ Well Volume (gal) _____ Centrifugal _____

Water Column _____ Total Volume Purged _____ Submersible _____

Pump On _____ Pump Off _____ Other _____

Developed By _____

Well Casing Volumes

gallon/foot	1-¼" = 0.06	2" = 0.16	3" = 0.37	4" = 0.65
	1-½" = 0.09	2-½" = 0.26	3-½" = 0.50	6" = 1.47

Time	Minutes Elapsed	Rate (gpm) (mL/min)	DTW (ft)	Gallons Purged	pH	Specific Conductance (mS/cm)	Temp. (C) (F)	REMARKS (PID readings, color, odor, etc.)

PIKA
International, Inc.
A Joint Venture
Malcolm Pirnie

Water Level Measurement Log

Project No.: Date:

Project Name/Location: Prepared By:

Well ID	Depth (feet)	Diameter (inches)	Well Stick-up (feet)	Screened Interval (ft bgs)	Depth to Water (feet)	Well Condition
Monitoring Wells						
				to		
				to		
				to		
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				to		

ft bgs = feet below ground surface
NA = Not Applicable

PIKA INTERNATIONAL, INC. A JOINT VENTURE MALCOLM PIRNIE

In-Situ Groundwater Parameter Log

Client: _____ Project # : _____ Date: _____
Location: _____ City: _____ State: _____
Location # : _____ Instument: _____
Technician: _____ Project Mngr: _____

WELL	TIME	DTW (ft bmp)	DO%	DO (mg/L)	DO (Charge*) (25-75)	REDOX (ORP) (mV)	pH	SC (µS/cm)	TEMP (^{0}C)

NOTES:

* DO CHARGE MUST BE 25 - 75 FOR PROPER OPERATION OF DO PROBE

PIKA INTERNATIONAL, INC. A JOINT VENTURE MALCOLM PIRNIE

Groundwater Sampling Form

Page ____ of ____

Project No. _____ Well ID _____ Date _____

Project Name/Location _____ Weather _____

Measuring Pt. Description _____ Screen Setting (ft-bmp) _____ Casing Diameter (in.) _____ Well Material ____ PVC ____ SS ____ Other

Total Depth (ft-bmp) _____ Static Water Level (ft-bmp) _____ Water Column in Well _____ Gallons in Well _____

Calc.Gallons Purged _____ Pump Intake (ft-bmp) _____ Purge Method: Centrifugal _____ Sample Method _____

Gallons Purged _____ MP Elevation _____ Submersible _____

Disp. Bailer _____ Pump On/Off _____

Sample Time: Label _____ Replicate/ Code No. _____ Other _____

Sampled by _____

Time	Minutes Elapsed	Rate (gpm) (mL/min)	Depth to Water (ft) TOC	Gallons Purged	pH	Cond. (µmhos) (mS/cm)	Turbidity (NTU)	Dissolved Oxygen (mg/L)	Temp. (°C) (°F)	Redox (mV)	Appearance	
											Color	Odor

Constituents Sampled	**Container**	**Number**	**Preservative**

Well Information

Well Location: _____ Well Locked at Arrival: Yes / No

Condition of Well: _____ Well Locked at Departure: Yes / No

Well Completion: Flush Mount / Stick Up Key Number To Well: _____

NOTES: _____

Well Casing Volumes

Gallons/Foot	1" = 0.04	1.5" = 0.09	2.5" = 0.26	3.5" = 0.50	6" = 1.47
	1.25" = 0.06	2" = 0.16	3" = 0.37	4" = 0.65	

Instrument Calibration Log

Project Name: _____ Date: _____

Project Number: _____

Calibrating Personnel: _____

Time of Calibration: _____

Weather Conditions: _____

Barometric Pressure: _____ inches H x 25.4 = _____ mm Hg

CALIBRANT	INSTRUMENT	INITIAL READING	VALUE ENTERED	FINAL READING	TIME	TEMP
pH 10.0						
pH 7.00						
pH 4.01						
Conductivity (_____)						
Turbidity (NTU)						
DO (mg/L)						
DO%						
ORP (mV)						
_____						
_____						
_____						

Notes: _____

Soil-Vapor Sample Log

Sample ID _____ Project/No. _____

Date _____ Sampling Personnel _____

Time _____

Weather _____

DESCRIPTION OF SAMPLE LOCATION:

_____ Outdoor

Location _____

Est. depth to water (ft): _____

Soil type: _____

_____

Odor: _____

Color: _____

_____ Indoor

Location _____

Basement: yes / no

Room size ft x ft: _____

Floor material: cement / wood / dirt

Slab Thickness (ft): _____

Visible cracks?: yes / no

Sub-slab material: dirt / gravel

PROBE INSTALLATION:

Date: _____

Method: _____

Diameter: _____

Depth: _____

Packing material: _____

Location Sketch:

PURGE:

Date: _____

Time: _____

Rate: _____

Volume: _____

SAMPLE COLLECTION:

Sample Time: _____

Sample Rate: _____

Sample Volume: _____

CONTAINER DESCRIPTION:

_____ L Summa Canister

Appendix H

Groundwater Monitoring Plan

Appendix H
Groundwater Monitoring Program
Charlotte Naval Ammunition Depot - Charlotte, North Carolina

Well ID	Aquifer Zone	Total Depth (feet bgs)	Screened Interval (feet bgs)	Well Diameter (inches)	Active Injection Period		Post-Injection Period
					Baseline (Once)	Performance Monitoring Events 1 - 8 (Quarterly)	MNA Monitoring (Quarterly for 2 Years)
					VOC, TOC, DG, Bio	VOC, TOC, DG	VOC, TOC, DG, Bio[1]
NADMW-25	T	19.5	9.0 - 19.0		*	*	*
NADMW-32	T	31.0	9.0 - 29.0		*	*	*
NADMW-38	T	26.0	14.5 - 24.5		*		*
NADMW-39	T	21.0	10.0 - 20.0		*		*
NADMW-42	T	31.0	20.5 - 30.5		*	*	*
NADMW-44	T	20.8	10.0 - 20.0		*		*
NADMW-49	T	31.0	19.0 - 29.0		*	*	*
NADMW-50	T	20.0	9.8 - 19.8		*	*	*
NADMW-52	T	33.0	19.5 - 29.5		*		*
NADMW-58	T	29.2	16.0 - 26.0		*	*	*
NADMW-66	T	25.0	15.0 - 25.0	2	*	*	*
NADMW-67	T	25.0	15.0 - 25.0	2	*	*	*
NADMW-68	T	25.0	15.0 - 25.0	2	*	*	*
NADMW-69	T	25.0	15.0 - 25.0	2	*	*	*
NADMW-70	T	25.0	15.0 - 25.0	2	*	*	*
SAIC-17	T	10.7	5.13 - 10.13		*	*	*
VERSAR-17	T	15.0	? - 15.0		*	*	*
NADMW-21	B	70.0	19.5 - 69.5		*	*	*
NADMW-22	B	75.0	24.5 - 74.5		*	*	*
NADMW-51	B	33.5	20.0 - 30.0		*	*	*
NADMW-71	B	250.0	230.0 - 250.0	2	*		*
NADMW-72	B	250.0	230.0 - 250.0	2	*		*
NADMW-73	B	250.0	230.0 - 250.0	2	*		*
NADMW-74	B	250.0	230.0 - 250.0	2	*		*
NADMW-75	B	250.0	230.0 - 250.0	2	*		*
NADMW-76	B	250.0	230.0 - 250.0	2	*		*
SAIC-12	B	36.5	25.5 - 35.0		*		*
SAIC-13	B	55.2	44.5 - 54.5		*		*
SAIC-14	B/MP	350.7	--		*	*	*
SAIC-15	B/MP	204.8	--		*	*	*
SAIC-21	B	105.7	93.88 - 103.88		*	*	*
VERSAR-20	B	33.8	23.8 - 33.8		*	*	*

Notes:
Shading indicates a proposed monitoring well.
events.
[1] Biogeochemical parameters will be included in the MNA monitoring program on an annual frequency.
bgs = below ground surface
Bio = biogeochemical parameters: nitrate, total and dissolved iron, sulfate and alkalinity
DG = dissolved gases: methane, ethane and ethene
MNA = monitored natural attenuation
MP = Mulit-port FLUTe™ well
TOC = total organic carbon
VOCs = volatile organic compounds
Aquifer Zones:
T = Transition Zone
B = Bedrock Zone