integral consulting inc.

PERFLUORINATED COMPOUNDS WORK PLAN

West Deptford, New Jersey, Plant

Prepared for
Solvay Specialty Polymers USA, LLC
10 Leonard Lane
West Deptford, NJ 08086

Prepared by
Integral Consulting Inc.
200 Harry S. Truman Parkway
Suite 330
Annapolis, MD 21401

November 15, 2013

PERFLUORINATED COMPOUNDS WORK PLAN

West Deptford, New Jersey, Plant

Prepared for
Solvay Specialty Polymers USA, LLC
10 Leonard Lane
West Deptford, NJ 08086

Prepared by

200 Harry S. Truman Pkwy.
Suite 330
Annapolis, MD 21401

November 15, 2013

CONTENTS

LIST OF FIGURES

LIST OF TABLES

ACRONYMS AND ABBREVIATIONS

AMP	air modeling plan
DQO	data quality objective
DRBC	Delaware River Basin Commission
DSA	dredged spoils area
EPA	U.S. Environmental Protection Agency
FSP	field sampling plan
ISRA	Industrial Site Recovery Act
LSRP	Licensed Site Remediation Professional
MUA	Municipal Utility Authority
NaPFO	sodium perfluorooctanoate
NJDEP	New Jersey Department of Environmental Protection
Pennwalt	Pennwalt Ltd
PFC	perfluorinated compound
PFNA	perfluorononanoic acid
PFOA	perfluorooctanoate acid
PFOS	perfluorooctanesulfonic acid
POTW	publicly owned treatment works
PVDF	polyvinylidene fluoride
PWS	public water system
QAPP	quality assurance project plan
QA/QC	quality assurance/quality control
Site	West Deptford, New Jersey, Plant
Solvay	Solvay Specialty Polymers USA, LLC
SOP	standard operating procedure
SSC	site-specific compound
USACE	U.S. Army Corps of Engineers

1 PROJECT BACKGROUND

This work plan outlines a voluntary program for investigation of perfluorinated compounds (PFCs) in the environment at and near the Solvay Specialty Polymers USA, LLC (Solvay) West Deptford, New Jersey, Plant (Site) located at 10 Leonard Lane in West Deptford Township Gloucester County, New Jersey. Certain PFCs were used historically in manufacturing operations at the facility and have been detected in some samples of municipal water supplies collected nearby as well as in the adjacent Delaware River. Additional potential PFC sources may exist and may be identified as a result of this work plan.

On September 6, 2013, Solvay met with the New Jersey Department of Environmental Protection (NJDEP) and the U.S. Environmental Protection Agency (EPA) to begin discussing these issues. At this meeting, Solvay, NJDEP, and EPA discussed and reached consensus on several general fields of follow up activities. Among other things, Solvay committed to working collaboratively with the agencies to investigate the concentrations of PFCs in groundwater, surface water, and sediment at and near the facility. This commitment was detailed in Solvay's follow up letter to NJDEP dated September 16, 2013 and is more fully detailed in this work plan. Solvay also agreed to voluntarily retain a Licensed Site Remediation Professional (LSRP), Tom Buggey of Roux Associates, to help facilitate completion of Solvay's PFC related environmental work.

Specifically, this work plan has been prepared on behalf of Solvay to conduct the following activities:

- Sampling identified Municipal Utility Authority (MUA) water systems
- Sampling appropriate existing onsite groundwater monitoring wells
- Sampling surface water and sediments at selected locations in the Delaware River
- Conducting air dispersion and deposition modeling of historic PFC emissions from the Site.

This work plan and associated documents detail the scope and methods of the data collection efforts for the described investigation of PFC presence at and around the Solvay Site.

1.1 SITE LOCATION

The facility is located in an agricultural and industrial area situated between a railroad line located north of Crown Point Road and the Delaware River at approximately River Mile 90, across from the Philadelphia Airport. A residential area is located to the south of Crown Point Road.

The Site encompasses 243 acres. Approximately 34 acres of the property are used for active manufacturing operations. This active portion of the Site is referred to as the Main Plant Area. The remaining acreage is either unused or leased for agriculture.

Three figures are included in this work plan to illustrate the Site location and proposed sampling at different spatial scales. Figure 1 shows the location of the Site along the Delaware River, the proximity to the airport, and historic surface water sampling locations from a study conducted by the Delaware River Basin Commission (DRBC) (MacGillivray 2012). Figure 2 shows the Site along with neighboring townships and proposed sample locations at wells maintained by seven nearby MUAs. Figure 3 shows the Main Plant Area along with proposed groundwater sample locations from existing onsite monitoring wells.

1.2 HISTORICAL OPERATIONS AND ACTIVITIES AT THE SITE

PFCs were first used at the Site in 1985 by Pennwalt Ltd. (Pennwalt) to produce polyvinylidene fluoride (PVDF) resin. The Site was purchased by Elf Atochem North America Inc. in 1989 and by Ausimont USA in 1990. Solvay acquired the Site with the acquisition of Ausimont USA, Inc. in May 2002, renamed to Solvay Solexis, Inc. in 2003, and continued the production of fluoropolymers, fluorocarbons, and fluoroelastomers. In October 2012, Solvay Solexis merged with Solvay Specialty Polymers USA, LLC.

The telomer-based fluorosurfactant, Surflon®, was used to produce PVDF at the Site from 1985 to 2010. Sodium perfluorooctanoate (NaPFO) was also used on the Site from 1995 to 2003. Solvay voluntarily joined the EPA 2010/2015 PFOA Stewardship Program (USEPA 2013) in 2006 and by 2010 proactively phased out use of perfluorooctanoate acid (PFOA) and all long carbon-chain PFCs at the Site.

As part of an ongoing but separate remedial investigation, NJDEP provides project oversight of this Site under the Industrial Site Recovery Act (ISRA) for corrective action/site remediation. Highlights of the separate ISRA-related activities that have occurred onsite over the past 10 years include:

- In 2004, a deed notice and NJDEP-approved engineered soil cover was installed at a dredged spoils area (DSA).
- In 2010, a groundwater treatment system was installed to remove volatile organic compounds and other compounds using air stripping. Treated groundwater is beneficially reused at the Site to reduce loading on the aquifer.
- In April 2012, NJDEP established interim groundwater quality standards for the site-specific compounds (SSC) 1-chloro-1,1-difluoroethane (142B), 1,1,1-trifluoroethane

(143A) and 1,1-dichloro-1-fluoroethane (141B). In November 2012, a Remedial Action Permit was issued for the DSA.

1.3 SUMMARY OF AVAILABLE DATA

Environmental monitoring for PFCs has been conducted in the Delaware River and in drinking water supplied by local municipalities. The studies and findings are summarized below.

1.3.1 DRBC Sampling

The DRBC conducted two pilot multi-year surveys of contaminants of emerging concern in the main stem Delaware River within the tidal wedge (MacGillivray 2012). The first study looked at finfish at sampling locations ranging from River Mile 58 to 289 from 2004 to 2006 and measured 10 PFCs in fish fillets from several species and different year classes. A subsequent DRBC study measured PFC concentrations in surface water from River Mile 50 to 131. Specifically, from 2007 to 2009, surface water from the following 6 locations in the Delaware River were sampled annually by the DRBC for unregulated constituents of interest including 13 PFCs (Table 1).

Table 1. Locations of Historical PFC Surface Water Sampling by DRBC

Site	River Mile	Latitude	Longitude	Description
E1	50	39.45500	-75.56000	Liston Point
E4	68.1	39.65472	-75.54667	Delaware Memorial Bridge
E7	80	39.81336	-75.39058	Marcus Hook Creek
E9	90	39.8835	-75.18616	Schuylkill River
E12	105.4	39.99478	-75.05978	Pennsauken Creek
E16	131.1	40.18156	-74.74505	Biles Channel

The concentration of perfluorononanoic acid (PFNA) in surface water exhibited a spatial gradient that increased in concentration from River Mile 131 (at Biles Channel) to a peak at River Mile 80 (at Marcus Hook Creek). PFNA concentrations decreased from River Mile 80 downstream to River Mile 50 (Liston Point). Similarly, the concentration of PFOA in surface water increased with distance downstream, peaking at River Mile 68 (Delaware Memorial Bridge), then decreased further downstream at River Mile 50. Overall, PFOA and PFNA maximum concentrations in surface water decreased substantially between 2007 and 2009 downstream of the Site.

1.3.2 Municipal Utility Authority Occurrence Studies

Within the past 10 years, several studies have provided measurements of PFCs in public drinking water supplies in New Jersey, including locations in the vicinity of the Site. These studies are briefly summarized below.

NJDEP conducted two statewide Occurrence Studies for detection of PFOA and other PFCs in New Jersey public drinking water (NJDEP 2007; Post et al. 2009, 2013). In the first study in 2006, 23 public water systems (PWSs) were sampled to measure concentrations of PFOA and perfluorooctanesulfonic acid (PFOS), both of which have eight carbons (or are "C8 PFCs"). PFOA was detected in 65 percent of the 23 tested systems at concentrations greater than the analytical reporting limit of 0.004 μg/L. Approximately 12 percent of PWSs had at least one sample with a PFOA concentration greater than NJDEP's preliminary health-based guidance value for drinking water (i.e., 0.04 μg/L).

In 2009, a second study was performed that involved sampling of 29 PWSs and measuring concentrations for PFOA, PFOS, PFNA, and 7 other PFCs in surface water and raw (unfinished) groundwater from mostly unconfined aquifers. PFOA was detected with concentrations greater than the reporting limit (0.005 μg/L) in 57 percent of all samples. In this study, PFNA levels exceeded the NJDEP preliminary guidance value for PFOA (0.04 μg/L) at three locations, with the maximum of 0.096 μg/L in raw groundwater Well #7 from Paulsboro, New Jersey (Post et al. 2013).

Additional samples of raw and treated water were collected by the Paulsboro Water Company in September 2013. Table 2 summarizes the concentrations of PFCs measured in three wells in both raw and finished water. Concentrations of PFNA ranged from 0.0098 μg/L to 0.14 μg/L in raw water to 0.016 μg/L to 0.15 μg/L in finished water across all three wells, with the maximum concentrations occurring in Well #7 (NJDEP 2013; Post et al. 2013). The wells in Paulsboro are among the wells that will be resampled in this current work plan, as noted in Figure 2.

Table 2. Concentrations of PFCs Measured in Wells at Paulsboro Water Authority in September 2013

			Concentration [a] (µg/L)					
			Well #7 [b]		Well #8 [b]		Well #9 [b]	
Analyte	Formula	CAS Number	Raw	Finished	Raw	Finished [c]	Raw	Finished [c]
Perfluoroheptanoic acid (PFHpA; C7)	$C_6F_{13}COOH$	375-85-9	0.0038	0.0040	0.0037	0.0040	0.0035	0.0040
Perfluorohexanesulfonic acid (PFHxS; C6)	$C_6F_{13}SO_3H$	355-46-4	0.0044	0.0047	0.0059	0.0061	0.0035	0.0061
Perfluorohexanoic acid (PFHxA; C6)	$C_5F_{11}COOH$	307-24-4	0.0049	0.0050	0.0068	0.0064	0.0085	0.0064
Perfluorononanoic acid (PFNA;C9)	$C_8F_{17}COOH$	375-95-1	0.14	0.15	0.015	0.016	0.0098	0.016
Perfluorooctanesulfonic acid (PFOS; C8)	$C_8F_{17}SO_3H$	1763-23-1	0.0060	0.0074	0.0084	0.0090	0.0040	0.0090
Perfluorooctanoic acid (PFOA;C8)	$C_7F_{15}COOH$	335-67-1	0.032	0.035	0.019	0.018	0.053	0.018

Notes:

CAS = Chemical Abstracts Service registry number

[a] Source file: Adobe Acrobat electronic copy of Eurofins Eaton Analytical - Laboratory Report for QC Laboratories. Samples Received September 18, 2013. Sample Group: Paulsboro PFC, Folder #449989. Analytical Protocol: USEPA Method #537.

[b] Sample Numbers (Raw, Finished): Well #7: 20130910296, 201309190304; Well #8: 201309190305, 201309190307; Well #9: 201309190306, 201309190307.

[c] Results for finished water for Well #8 and Well #9 are reported as a single result (i.e., "#8 + #9 WTP").

2 PROPOSED WORK PLAN

The following section provides a summary of the proposed work plan objectives and major elements of the plans for sampling, chemical analysis, and implementation of quality assurance/quality control (QA/QC) procedures. Additional details for implementation of the site investigation of the work plan are provided in the appendices to this document:

- Appendix A. Quality Assurance Project Plan (QAPP)
- Appendix B. Field Sampling Plan (FSP)
- Appendix C. Air Modeling Plan (AMP).

In addition, separate addenda to the FSP will be prepared for sampling at each of the seven identified MUAs in the vicinity of the Site. These documents will reflect specific information obtained through an interviews with MUA personnel, including which wells are available for sampling, procedures for handling purge water of inactive wells, and any site-specific health and safety considerations.

2.1 OBJECTIVES

This technical work plan describes the scope for evaluating the presence of long carbon-chain PFCs (i.e., C8 to C13) in the following environmental media at or near the Site:

- Water in the distribution systems of seven MUAs near the Site
- Groundwater from monitoring wells located onsite
- Surface water and sediments at select locations in the Delaware River.

In addition, air dispersion and deposition modeling is proposed to evaluate historical PFC emissions. This work plan summarizes the major elements of the proposed modeling, including key assumptions and proposed steps to assess and address potential influential data gaps. The air modeling results are expected to describe the potential extent and geographical distribution of historical deposition patterns. These results will inform a conceptual site model for PFC fate and transport.

Solvay is committed to expediting the field sampling events, data validation, and reporting of results to better understand PFC related facts and circumstances as quickly as possible. A proposed schedule for these activities is provided in both Appendices A and B (Figure A-2 and Figure B-1).

2.2 DATA QUALITY OBJECTIVES

Data quality objectives (DQOs) have been established to ensure that the data collected are sufficient and of adequate quality for their intended uses to achieve project objectives. The primary objectives of the investigation are as follows:

- Quantitatively assess the presence or absence of PFCs in municipal water supplies by collecting finished (treated) and/or raw water from accessible wells at seven identified MUAs in the vicinity of the Site
- Quantitatively assess the presence or absence of PFCs in groundwater obtained from onsite monitoring wells screened in shallow, intermediate, and deep zones of the unconfined aquifer and samples from monitoring wells in the confined aquifer
- Quantitatively assess the presence or absence of PFCs in the surface water and sediment offshore of the Site as well as locations upstream and downstream from the Site
- Determine if concentrations of PFCs in surface water and sediment in the shoreline proximal to the Site are elevated with respect to locations upstream and downstream from the Site
- Conduct air modeling to assess the potential aerial extent of historic emissions of PFCs from the Site.

The DQOs are consistent with USEPA *Guidance on Systematic Planning Using the Data Quality Objectives Process* (USEPA 2006). Table 3 summarizes each of the steps of the DQO process that are applicable to planning for this project.

Table 3. DQO Process for the Work Plan

Step		Description
1	State the Problem	Determine if the designated investigation areas for sampling of groundwater, municipal water supplies, surface water, and sediment contain PFCs listed in the QAPP and FSP (Appendices A and B). Also, use air modeling to assess the potential aerial extent of historical emissions of constituents from the Site as described in the AMP (Appendix C).
2	Identify the Decision	The results of the sampling described in this work plan and QAPP (Appendix A) will be assessed for precision, accuracy, completeness, sensitivity, representativeness, and comparability (PACSRC). The overall goal of the sampling is to determine the presence and range of concentrations of PFCs in each of the media sampled.
3	Identify Inputs to the Decision	Analytical methods are listed in Table A-3 of the QAPP. Quality assurance protocols and procedures for addressing data quality issues are described in the QAPP.
4	Define the Boundaries	The boundaries of the investigation areas are defined as follows for each field investigation: a) onsite groundwater wells are defined by the Site property; b) municipal water supplies are defined as accessible wells and water supplies at nearby MUA facilities; c) the surface water and sediment collection area is limited to the historical extent of surface water sampling within the Delaware River by the DRBC and includes additional locations adjacent to the Site.
5	Develop a Decision Rule	If the PACSRC results are satisfactory and the sampling results provide sufficient characterization to meet the project objectives in Section 2.1, no additional work will be performed in this investigation.
6	Specify Limits on Decision Errors	The data generated will be evaluated to determine if the quality control objectives in the QAPP are met.
7	Optimize the Design for Obtaining Data	Samples will be obtained as follows for each field investigation: a) onsite groundwater wells – locations will be selected to maximize the chance of detecting PFCs based on historical site knowledge of production processes and uses associated with PFCs and knowledge of hydrogeological properties of this area. Wells will be selected to characterize water from shallow, intermediate, and deep zones in the unconfined aquifer and the confined aquifer; b) municipal water supplies – all accessible wells from seven MUAs in the vicinity of the Site will be sampled; for active wells, both raw and finished water will be obtained; c) surface water and sediment – a stratified random sampling design will be used whereby strata will represent areas in the Delaware River that were previously sampled by DRBC in 2007–2009 (MacGillivray 2012), additional locations to provide greater spatial coverage, the confluence of the river and adjacent creeks, and nearby a publically owned treatment works outfall adjacent to the Site.

Notes:
AMP = air modeling plan
DQO = data quality objective
DRBC = Delaware River Basin Commission
FSP = field sampling plan
MUA = Municipal Utility Authority
PACSRC = precision, accuracy, completeness, sensitivity, representativeness, and comparability
PFC = perfluorinated compound
QAPP = quality assurance project plan

2.3 SAMPLE COLLECTION DESIGN AND RATIONALE

This section provides a summary of the sample collection design and rationale for collecting distribution water from MUA wells, groundwater from onsite wells, and surface water and sediment from the Delaware River. Additional details are provided in the QAPP (Appendix A) and FSP (Appendix B). Standard operating procedures (SOPs) included with this work plan are consistent with NJDEP's Field Sampling Procedures Manual (NJDEP 2005), N.J.A.C. 7:18-1 et seq., and 40 CFR 141.

2.3.1 MUA Sampling

Samples of raw and finished water will be collected from drinking water supplies maintained by the following seven MUAs:

- East Greenwich Township Water Department
- Greenwich Township Water Department
- National Park Water Department
- Paulsboro Water Department
- West Deptford Township Water Department
- Westville Water Department
- Woodbury City Water Department.

These MUAs were selected in consultation with NJDEP because of their relative proximity to the Site (NDEP 2013b). Figure 2 shows the approximate locations of the wells at these locations. Prior to initiating sampling, Solvay will interview each facility manager to confirm the status of wells and obtain information on well maintenance, purge water handling procedures, and historical monitoring data. Based on this information, a separate addendum to the FSP will be prepared for each MUA sampling event.

2.3.2 Groundwater Sampling

Groundwater samples will be collected from 31 existing monitoring wells located onsite as shown in Figure 3. These wells were chosen to provide a representative set of wells that capture the various aquifer units and subdivisions with a lateral spatial extent sufficient to determine the presence and extent of PFCs in groundwater beneath the site. The wells were selected from upgradient, within the axes, at the lateral bounds of the property, and a transect along the downgradient property boundary.

2.3.3 Surface Water and Sediment Sampling

Surface water samples will be collected from 26 stations at 12 locations in the Delaware River. Surface sediment will be collected from 15 stations, and subsurface sediment will be collected from an additional 11 stations. Surface water and sediment stations will be co-located as shown in Figures 1 and 4. These stations were selected based on a stratified random sampling design whereby strata (locations) represent the following areas in the Delaware River:

- Six locations that were previously sampled by DRBC in 2007–2009 (MacGillivray 2012) (see Section 1.3.1)
- Three additional locations in the Delaware River to provide greater spatial coverage
- Two locations at the confluence of the river and two creeks within the watershed containing the Site
- One location at nearby a publicly owned treatment works (POTW) outfall adjacent to the Site.

In each area, sediment will be collected from up to three discrete stations to provide a measure of variability at a localized scale. A surface sample (0 to 6 inches) will be collected from each of the 26 stations. In addition, deeper intervals will be obtained by collecting a sediment core from 10 of the 26 stations. Sediment cores will be subsampled to provide information on the vertical profile of PFCs in the following surface and subsurface intervals: 0–6 in. [0–15 cm] (matching the surface sample interval), 6–12 in. [15–30 cm], 12–24 in. [30–61 cm], 24–36 in. [61–91 cm], and >36 in. [>91 cm]).

Samples will be collected outside the main channel, near the shoreline for both health and safety considerations as well as to avoid areas impacted by dredging activity. The U.S. Army Corps of Engineers (USACE) has been widening the main channel of the Delaware River as part of the Delaware River Main Channel Deepening project (USACE 2013a,b,c). The goal of the project is to deepen and widen the existing Delaware River Federal Navigation Channel from 40 to 45 ft from Camden, New Jersey, to the mouth of Delaware Bay. There are three potential implications of this activity for the surface water and sediment sampling as proposed in this work plan:

- Disturbance and removal of sediment along the main channel, which will expose material at the subsurface
- Potential mixing and transport of sediment during the dredging process (although USACE's demonstration projects suggest that procedures are designed to limit mixing beyond the main channel)
- Potential leaching from dredge spoils deposited in federally owned confined upland disposal facilities within the watershed.

Two of the areas designated as USACE containment facilities, National Park and Fort Mifflin, are approximately 2 miles upstream of the Site. The National Park facility received up to 1.2 million cubic yards of material dredged in 2012 and 2013 from the channel widening occurring throughout approximately 11 river miles extending from Walt Whitman Bridge to southwest of the Philadelphia Airport (Contract #3). In 2014, Fort Mifflin is scheduled to receive material from the 45-ft deepening of the entire Delaware River Main Channel.

2.4 PARAMETERS TO BE TESTED AND FREQUENCY

Consistent with NJDEP's request for investigating concentrations of longer carbon-chain PFCs (i.e., C8 to C13), the PFCs listed below in Table 4 will be measured in each sample using the analytical protocols given in the QAPP.

Table 4. Long Carbon-Chain PFCs for Laboratory Sample Analysis

Chemical Name	Acronym	Number of Carbons
Perfluorooctanoic acid	PFOA	8
Perfluorononanoic acid	PFNA	9
Perfluorodecanoic acid	PFDA	10
Perfluoroundecanoic acid	PFUnDA	11
Perfluorododecanoic acid	PFDoDA	12
Perfluorotridecanoic acid	PFTrDA	13
Perfluorooctanesulfonic acid	PFOS	8

Notes:
PFC = perfluorinated compound

In addition, because variability in PFC concentrations may be correlated with variability in the physical and chemical properties of the sampled medium, conventional parameters will be measured in the laboratory and field. Conventional laboratory parameters will include total organic carbon, total suspended solids, pH, total hardness, and alkalinity for water samples and grain size and total organic carbon for sediment samples. Field water quality parameters will include water temperature, pH, dissolved oxygen, salinity, oxidation-reduction potential, turbidity, and conductivity.

2.5 INTENDED DATA USAGE

The data generated from this work plan will be used to determine the extent to which long carbon-chain PFCs (i.e., C8 to C13) are present in the environment at and near the Site.

3 REPORTING

A data summary report will be prepared that describes the methods and presents the results of the chemical analysis as well as the data evaluation in support of the work plan objectives. The report and corresponding electronic data deliverables will be delivered in electronic and hard copy format to NJDEP.

The validated results of the sampling at individual MUA wells will be communicated to NJDEP and each individual MUA along with a memorandum that provides a summary of the data.

4 REFERENCES

MacGillivray, A.R. 2012. Contaminants of emerging concern in the tidal Delaware River, pilot monitoring study 2007–2009. Delaware River Basin Commission, West Trenton, NJ. 87 pp. July.

NJDEP. 2005. Field sampling procedures manual. Available at: www.nj.gov/dep/srp/guidance/fspm/. New Jersey Department of Environmental Protection, Trenton, NJ. August.

NJDEP. 2007. Determination of perfluorooctanoic acid (PFOA) in aqueous samples. Final Report. New Jersey Department of Environmental Protection, Division of Water Supply, Bureau of Safe Drinking Water, Trenton, NJ. 17 pp. January.

NJDEP. 2012. Delaware River recruitment survey sampling area. Map. In: Studying the Delaware River – 2011 Report. Available at: www.njfishandwildlife.com/artdelstudy12.htm. New Jersey Department of Environmental Protection Agency, Division of Fish and Wildlife, Trenton, NJ. April.

NJDEP. 2013. Electronic mail communication from Erica Bergman, NJDEP Case Manager, to Mitch Gertz, Solvay, dated October 17, 2013 including Laboratory Report for QC Laboratories prepared by Eurofins Eaton Analytical, dated October 8, 2013.

Post, G.B., J.B. Louis, K.R. Cooper, B.J. Boros-Russo, and R.L. Lippincott. 2009. Occurrence and potential significance of perfluorooctanoic acid (PFOA) detected in New Jersey public drinking water systems. *Environ. Sci. Technol.* 43:4547-4554.

Post, G.B., J.B. Louis, R.L. Lippincott, and N.A. Procopio. 2013. Occurrence of perfluorinated compounds in raw water from New Jersey public drinking water systems. *Environ. Sci. Technol.* Accepted for publication November 4, 2013.

USACE. 2013a. Delaware River main channel deepening. Fact Sheet. www.nap.usace.army.mil/Missions/Factsheets/FactSheetArticleView/tabid/4694/Article/6559/delaware-river-main-channel-deepening.aspx. Accessed on November 5, 2013. U.S. Army Corps of Engineers, Philadelphia District. April.

USACE. 2013b. Contract #3 (in progress). www.nap.usace.army.mil/Portals/39/docs/Civil/Deepening/Contract3.pdf. Accessed on November 5, 2013. U.S. Army Corps of Engineers.

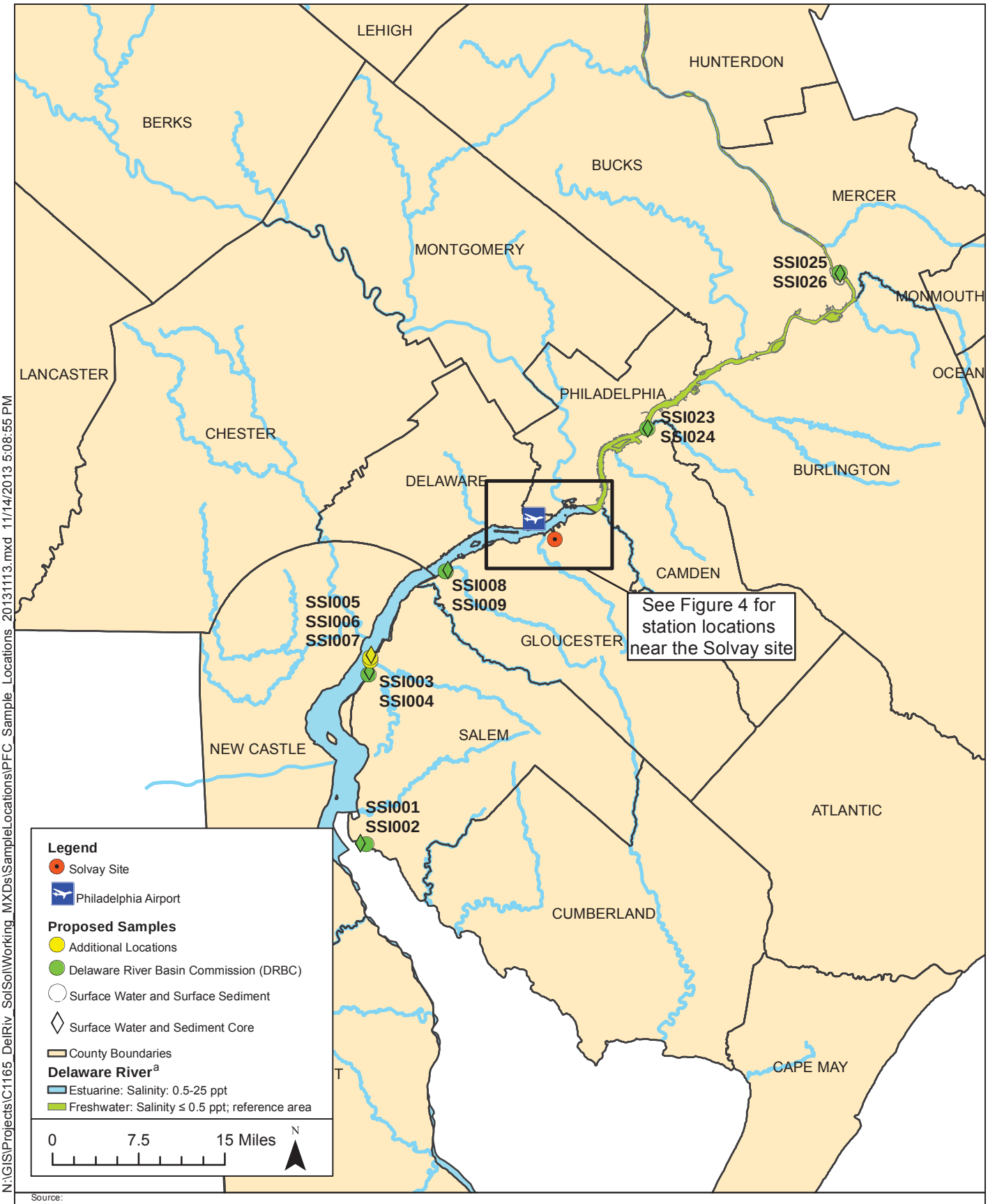
USACE. 2013c. Draft environmental assessment, Delaware River main channel deepening project. Delaware Bay economic loading, mechanical dredging, and placement of dredged

material at the Fort Mifflin confined disposal facility. www.nap.usace.army.mil/Portals/39/docs/Civil/Deepening/DRMCD%20-%20Draft%20Environmental%20Assessment%20-%20February%202013.pdf. Accessed on November 5, 2013. U.S. Army Corps of Engineers, Philadelphia District. 36 pp. February.

USEPA. 2006. Guidance on systematic planning using the data quality objectives process. EPA QA/G-4. EPA/240/B-06/001. Office of Environmental Information, Washington, DC. 121 pp. February.

USEPA. 2013. 2010/2015 PFOA Stewardship Program. www.epa.gov/oppt/pfoa/pubs/stewardship/. Accessed on November 5, 2013. U.S. Environmental Protection Agency, Office of Pollution Prevention and Toxics, Washington, DC.

FIGURES



Source:
a. NJDEP (2012)

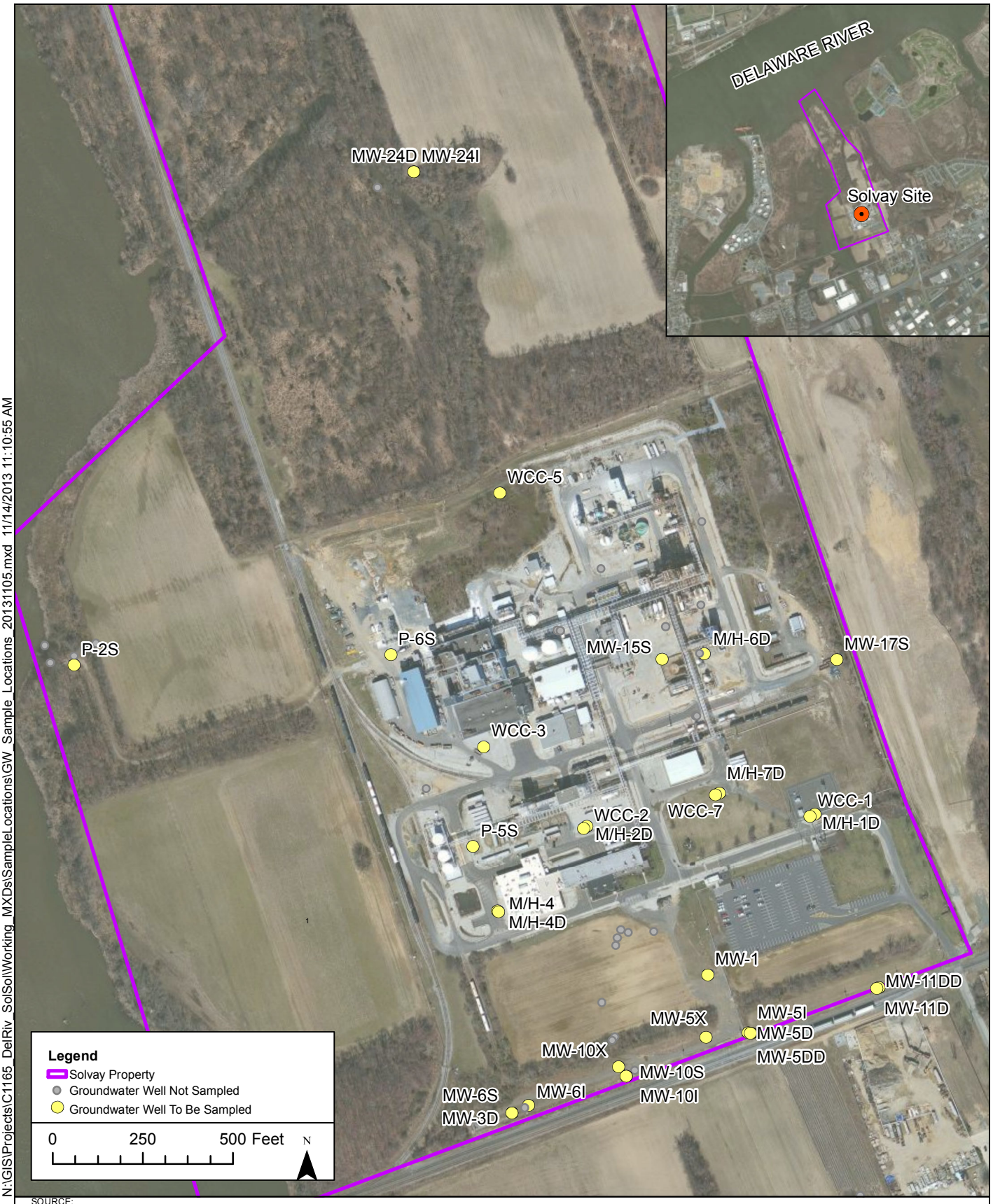
integral consulting inc.

Figure 1.
Proposed Co-Located Surface Water and Sediment Sampling Locations near Historic DRBC Monitoring Locations

N:\GIS\Projects\C1165_DelRiv_SolSol\Working_MXDs\SampleLocations\MUA_Sample_Locations_20131114.mxd 11/14/2013 2:08:18 PM

integral consulting inc.

Figure 2.
Proposed Sampling Locations of
Municipal Utility Authority (MUA) Wells

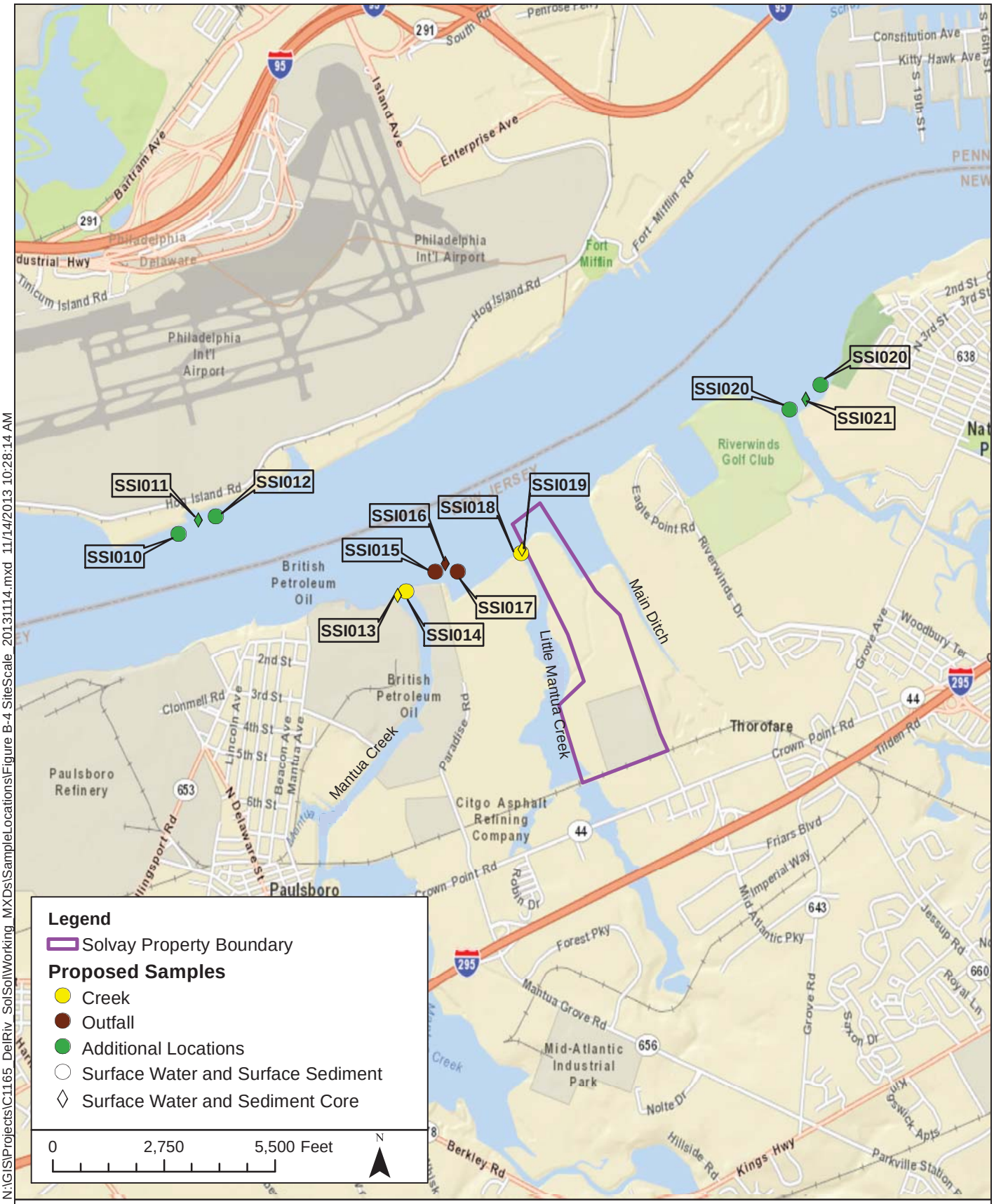


N:\GIS\Projects\C1165_DelRiv_SolSol\Working_MXDs\SampleLocations\GW_Sample_Locations_20131105.mxd 11/14/2013 11:10:55 AM

SOURCE:
Aerial Imagery: (c) 2010 Microsoft Corporation and its data suppliers.

integral consulting inc.

Figure 3.
Proposed Groundwater Sampling Locations



integral consulting inc.

Figure 4.
Proposed Co-Located Surface Water and Sediment Sampling Locations near the Site

APPENDIX A

QUALITY ASSURANCE PROJECT PLAN

APPENDIX A
QUALITY ASSURANCE PROJECT PLAN

Perfluorinated Compounds Work Plan

Prepared for
Solvay Specialty Polymers USA, LLC
10 Leonard Lane
West Deptford, NJ 08086

Prepared by

200 Harry S. Truman Pkwy.
Suite 330
Annapolis, MD 21401

November 15, 2013

CONTENTS

LIST OF FIGURES

LIST OF TABLES

ACRONYMS AND ABBREVIATIONS

COC	chain of custody
CLP	Contract Laboratory Program
EDD	electronic data deliverable
EPA	U.S. Environmental Protection Agency
FSP	field sampling plan
HAZWOPER	Hazardous Waste Operations and Emergency Response
HSP	health and safety plan
Integral	Integral Consulting Inc.
LC-MS/MS	liquid chromatography tandem mass spectrometry
LCS	laboratory control sample
LCSD	laboratory control sample duplicate
LDC	Laboratory Data Consultants, Inc.
LFB	laboratory fortified blank
MDL	method detection limit
MRL	method reporting limit
MS	matrix spike
MSD	matrix spike duplicate
MUA	Municipal Utility Authority
NJDEP	New Jersey Department of Environmental Protection
PFC	perfluorinated compound
QA	quality assurance
QAPP	quality assurance project plan
QA/QC	quality assurance and quality control
QC	quality control
RPD	relative percent difference
RSD	relative standard deviation
Solvay	Solvay Specialty Polymers USA, LLC
SOP	standard operating procedure

TOC	total organic carbon

1 PROJECT MANAGEMENT

1.1 DISTRIBUTION LIST

Title	Name
NJDEP Project Manager	Erica Bergmann
Roux Licensed Site Remediation Professional	Tom Buggey
Solvay Specialty Polymers USA, LLC Project Manager	Mitch Gertz
Integral Principal in Charge	Judi Durda
Integral Project Manager	Phil Goodrum
Integral Field Lead	Matt Behum
Integral Laboratory Coordinator	Craig Hutchings
Database Administrator	Jesse Sheffield
Eurofins Eaton Analytical Project Manager	Linda Geddes
TestAmerica Project Manager	Jannel Franklin
Laboratory Data Consultants Project Manager	Linda Rauto

1.2 INTRODUCTION AND TASK ORGANIZATION

This quality assurance project plan (QAPP) and associated attachments have been prepared on behalf of Solvay Specialty Polymers USA, LLC (Solvay), pursuant to the agreement with New Jersey Department of Environmental Protection (NJDEP) outlined in a meeting between the two parties on September 6, 2013, where it was agreed that Solvay would develop a plan designed to investigate potential pathway(s) of long-chain perfluorinated compounds (PFCs) from the Site to the environment. The following elements are included in the project scope:

- Sampling identified Municipal Utility Authority (MUA) water systems
- Sampling appropriate existing groundwater monitoring wells
- Sampling surface water and sediments at select locations in the Delaware River
- Conducting air dispersion and deposition modeling of historical PFC emissions from the Site.

This document is the QAPP, Appendix A to the overall work plan for the Perfluorinated Compounds Work Plan of the West Deptford, New Jersey, Plant. The companion document, Appendix B, consists of the field sampling plan (FSP).

This section reviews the organizational structure for activities associated with the work plan, including project management and oversight, field work, sample analysis, and data management. The organizational structure for this project is illustrated in Figure A-1. Contact information for key personnel is provided in Section 1.3.

1.3 PROJECT ORGANIZATION

Solvay has retained Integral Consulting Inc. (Integral) to study perfluorinated compounds at the West Deptford, New Jersey, Plant site (the Site). Tom Buggey of Roux Associates serves as the Licensed Site Remediation Professional. Figure A-1 illustrates the organization of personnel on the project. The primary contacts for NJDEP, Solvay, and Integral are provided in the following table. A description of the project organization and contacts pertaining to this QAPP are provided after the table.

1.3.1 General Project Management

NJDEP and Respondent Project Managers

Title	Name	Contact Information
NJDEP Project Manager	Erica Bergman	NJDEP - Bureau of Case Management 401 E. State Street - Mail Code 401-05 P.O. Box 420 Trenton, NJ 08625-0420 (609) 292-7406 erica.bergman@dep.state.nj.us
Solvay Project Manager	Mitch Gertz	Solvay Specialty Polymers USA, LLC 10 Leonard Lane West Deptford, NJ 08086 (856) 251-6630 mitchell.gertz@solvay.com
Licensed Site Remediation Professional	Thomas Buggey	Roux Associates (215) 962-3493 402 Heron Drive Logan Township, NJ 08085 tbuggey@rouxinc.com

The names and quality assurance (QA) responsibilities of key project personnel for Integral who will be involved in sampling and analysis activities are provided below.

Project Personnel Quality Assurance Responsibilities

Title	Responsibility	Name	Contact Information
Principal in Charge	Coordination of project information and related communications on behalf of Solvay	Judi Durda	Integral Consulting Inc. 200 Harry S. Truman Pkwy, Suite 330 Annapolis, MD 21401 (410) 573-1982 ext. 14 jdurda@integral-corp.com
Project Manager	Responsible for the successful completion of tasks described in this document, and coordination with NJDEP project manager and Solvay project manager to execute the study described in this document	Phil Goodrum	Integral Consulting Inc. 7030 E Genesee Street, Suite 110 Fayetteville, NY 13066 (310) 445-5090 pgoodrum@integral-corp.com
Corporate Health and Safety Manager	Oversight of health and safety program for field tasks	Eron Dodak	Integral Consulting Inc. 319 SW Washington Street, Suite 1150 Portland, OR 97201 (503) 943-3614 edodak@integral-corp.com
Field Lead	Field data collection and implementation of the health and safety plan	Matt Behum	Integral Consulting Inc. 200 Harry S. Truman Pkwy, Suite 330 Annapolis, MD 21401 (410) 573-1982 ext. 12 jdurda@integral-corp.com
Database Administrator	Database development and data management	Jesse Sheffield	Integral Consulting Inc. 285 Century Place, Suite 190 Louisville, CO 80027 (720)-465-3322 jsheffield@integral-corp.com
Laboratory Coordinator	Completeness of QA documentation and procedures; liaison between project personnel, chemical testing laboratories and data validators and for related QA communications with NJDEP	Craig Hutchings	Integral Consulting Inc. 1205 West Bay Dr. NW Olympia, WA 98502 (360) 705-3534 ext. 417 chutchings@integral-corp.com

1.3.2 Laboratories

The laboratories that will perform the chemical analysis on samples collected during this investigation are Eurofins Eaton Analytical of Monrovia, California, and TestAmerica laboratories located in Burlington, Vermont, Denver, Colorado, and Edison, New Jersey. Both laboratories are certified by NJDEP for the analyses they will be performing. Contact information and NJDEP certification numbers for the laboratories are provided below.

Laboratory Contract Information

Eurofins Eaton Analytical	Linda Geddes 750 Royal Oaks Drive Monrovia, CA 91016 (626) 386-1163 LindaGeddes@eurofinsUS.com	NJDEP Certification Number: CA016
TestAmerica	Jannel Franklin 777 New Durham Road Edison, NJ 08817 (732) 593-2551 Jannel.Franklin@testamericainc.com	NJDEP Certification Numbers: Burlington, VT: VT972 Denver, CO: CO004 Edison, NJ: 12028

The following responsibilities apply to the project managers and QA managers at the analytical laboratories used for this task.

The laboratory project managers are responsible for the successful and timely completion of sample analyses, and for performing the following tasks:

- Ensure that samples are received and logged in correctly, that the correct methods and modifications are used, and that data are reported within specified turnaround times.
- Review analytical data to ensure that procedures were followed as required in this QAPP, the cited methods, and laboratory standard operating procedures (SOPs).
- Keep the task laboratory coordinator apprised of the schedule and status of sample analyses and data package preparation.
- Notify the task laboratory coordinator if problems occur in sample receiving, analysis, or scheduling, or if control limits cannot be met.
- Take appropriate corrective action as necessary.
- Report data and supporting QA information as specified in this QAPP.

The laboratory QA manager is responsible for overseeing the QA activities in the laboratory and ensuring the quality of the data for this project. Specific responsibilities include the following:

- Oversee and implement the laboratory's QA program
- Maintain QA records for each laboratory production unit
- Ensure that QA and quality control (QC) procedures are implemented as required for each method and provide oversight of quality assurance and quality control (QA/QC) practices and procedures
- Review and address or approve nonconformity and corrective action reports.
- Coordinate response to any QC issues that affect this project with the laboratory project manager.

1.4 PROJECT SCHEDULE

Figure A-2 shows the key elements of the proposed schedule for sampling, analysis, data validation, and reporting. The schedule assumes that Solvay will submit the review drafts of the work plan and associated attachments to NJDEP by November 15, 2013, and that NJDEP will provide feedback or approval by December 2, 2013. As discussed with NJDEP's case manager, sampling has already occurred at the MUA in West Deptford. Sampling at six additional identified MUAs will occur after personnel are contacted to schedule sampling and obtain information on well construction, status of operation, procedures for handling purge water of inactive wells, and site-specific health and safety considerations. .

The following time periods are expected for each of the sampling efforts:

- Sampling drinking water supplies at MUAs: 1-2 days per MUA
- Sampling onsite groundwater wells: 2 weeks
- Sampling surface water and sediment in the Delaware River: 2 weeks.

Solvay has requested an expedited turn around time from laboratories. Samples will be shipped to the laboratories by overnight delivery service and pre-validated results are expected from the laboratories in about 10 days.

Third party (independent) validation, conducted by Laboratory Data Consultants , Inc. (LDC), will commence after the laboratories prepare electronic data deliverables (EDDs) (see Section 10.2.4). The time frame for generating validation reports is approximately three weeks and individual EDDs can be reviewed in parallel.

Final validated data reports are expected to be available as follows:

- MUAs – approximately weekly, beginning December 15 and ending January 31, 2014
- Groundwater – January 31, 2014

- Surface Water and Sediment – February 26, 2014.

2 CRITERIA FOR MEASUREMENT DATA

The overall QA objectives are to develop and implement procedures for field sampling, chain of custody (COC), laboratory analysis, field measurement, and reporting that will provide data that are scientifically valid, meet the data quality objectives, are to a degree of quality consistent with their intended use, and are defensible in a court of law. This section defines the goals for the QA/QC effort and the accuracy, precision, sensitivity, completeness, representativeness, and comparability of laboratory analyses. The chemicals of interests for each matrix are presented in Table A-1; Table A-2 presents sample preservation and holding time requirements; and Table A-3 presents the laboratory methods that will be used. The SOPs applied in this project will comply with the QA requirements of NJDEP's *Field Sampling Procedures Manual* (NJDEP 2005, Chapter 2).

2.1 DEFINITIONS

The data quality indicators and their use for assessment of data quality are as follows:

- **Precision** measures the reproducibility of measurements under a given set of conditions without consideration of the "true" or accurate value, i.e., variability between measurements of the same material for the same analyte. Precision is measured in terms of relative percent difference (RPD) between a sample and its duplicate or between a matrix spike (MS) and a matrix spike duplicate sample (MSD). The laboratory uses laboratory control samples/laboratory control sample duplicate (LCS/LCSD) pairs when MS/MSDs are not practical due to the nature of a sample or analytical method used, and they are prepared and analyzed with each batch of samples instead of MS/MSDs. An LCS/LCSD may also be prepared in place of an MS/MSD in the case that a sufficient sample volume was not obtained in the field to perform the MS/MSD analysis. For inorganic analyses, analytical precision is usually calculated based on the sample and spike duplicate results. Field sampling precision will be evaluated based on the RPD for field duplicate samples.
- **Accuracy** measures the bias of an analytical system by comparing the difference of a measurement with a reference value. The difference between the observed value and the reference value includes components of both systematic error (bias) and random error. Accuracy will vary from analysis to analysis because of individual sample and matrix effects. Control limits are defined as the mean recovery, plus or minus three standard deviations, of the 20 data points, with the warning limits set as the mean, plus or minus two standard deviations. Percent recoveries for MS, MSD, and LCS that are analyzed for every batch of up to 20 samples serve as a measure of analytical accuracy. Surrogate standards are added to all samples, blanks, MS, MSD, and LCS analyzed for

organic contaminants to evaluate accuracy of the method and help to determine matrix interferences. The spiking solutions used for accuracy determinations are not used for instrument calibrations.

- **Completeness** is defined as the percentage of measurements made that are judged to be valid measurements. For the data to be considered complete, they must meet all acceptance criteria including accuracy and precision and other criteria specified for an analytical method.
- **Representativeness** expresses the degree to which the data accurately and precisely represent a characteristic of a population, parameter variations at a sampling point, process condition, or an environmental condition. Representativeness is a qualitative parameter, which is dependent upon the proper design of the sampling program and the laboratory QC protocol.
- **Comparability** is a qualitative parameter expressing the confidence with which one data set can be compared with another. This goal is achieved through using standard techniques to collect and analyze representative samples and reporting analytical results in appropriate units.
- **Sensitivity** is evaluated by whether the laboratory met the practical quantitation limits established in the QAPP. The laboratory will determine the method detection limits (MDLs) for each method, instrument, analyte, and matrix by using the procedure described in Title 40 Code of Federal Regulations Part 136B. The MDL is defined as the minimum concentration of a substance that can be measured and reported with 99 percent confidence that the analyte concentration is greater than zero. The laboratory will select the practical quantitation limits for all analytes at concentration levels that exceed the calculated MDLs by a factor of two to ten. Reporting limits for the project are presented in Table A-4.

2.2 LEVEL OF QUALITY CONTROL EFFORTS

The level of QC effort for the field sampling efforts will include the following types of QC samples. The frequency of QC samples per sample or sampling event is specified in Section 6.2.

- Field "blind" duplicate samples will be obtained to provide an estimate of the reproducibility of measurements, sampling procedures, and analytical procedures.
- MS/MSD samples will be obtained to provide information about the effect of the sample matrix on digestion and measurement methodology.
- Equipment rinsate blanks will be collected to determine the effectiveness of the decontamination procedure if non-dedicated equipment is used during sampling.

The analytical laboratory will follow the QC requirements of the analytical methods in accordance with the requirements in the method.

2.3 ACCURACY, PRECISION, AND SENSITIVITY OF ANALYSES

Data generated in the field and the laboratory will be evaluated for accuracy, precision, and sensitivity as described below.

2.3.1 Accuracy

The following equation illustrates how accuracy is evaluated:

$$\text{Accuracy as percent recovery} = \frac{\text{Spiked Sample Result - Sample Result}}{\text{Spike True Value}} x100\% \quad (2\text{-}1)$$

Percent recoveries for MS, MSD, and LCS that are analyzed for every batch of up to 20 samples serve as a measure of analytical accuracy. Surrogate standards are added to all samples, blanks, MS, MSD, and LCS analyzed for organic contaminants to evaluate accuracy of the method and help to determine matrix interferences.

Laboratories will evaluate percent recovery data based on in-house statistically based control limits for each method of analysis and specific sample matrix. The laboratory will review the QC samples and surrogate standard recoveries for each analysis to ensure that internal QC data lie within the limits of acceptability. The laboratory will investigate any suspect trends and take appropriate corrective actions.

2.3.2 Precision

The following equation illustrates the method for calculating RPD to assess a method's precision:

$$\text{Precision as RPD} = \frac{2x\left|\text{ Result} - \text{Duplicate Result }\right|}{\text{Result} + \text{Duplicate Result}} x100\% \quad (2\text{-}2)$$

Precision is expressed in terms of the relative standard deviation (RSD) for three or more measurements and the RPD for two measurements. The RSD is the ratio of the standard deviation of three or more measurements to the average of the measurements, expressed as a percentage. The analytical laboratory will have statistically based acceptability limits for RPDs established for each method of analysis and sample matrix. Laboratory-evaluated duplicates may include spike duplicates (for inorganics), and MSDs or LCSDs (for organics). The

laboratory will review the QC samples to ensure that internal QC data lie within the limits of acceptability. Any suspect trends will be investigated and corrective actions taken.

Field precision will be monitored based on the field blind duplicate results for evaluation of the sampling techniques and sample handling procedures. Analytical data will not be qualified during the data validation process based on the field precision values. The analytical results of field blind duplicates will be evaluated as part of the data validation process described in Section 12.

Results for field and method blanks can reflect systematic bias that results from contamination of samples during collection or analysis. Any analytes detected in field or method blanks will also be evaluated as potential indicators of bias.

2.3.3 Sensitivity

The sensitivity of the analyses is reflected in the MDLs and method reporting limits (MRLs). Target MDLs and MRLs for this study are summarized in Table A-4. Nondetected results will be reported to the MDL unless no MDL is listed in Table A-4, in which case nondetected results will be reported to the MRL. Analytes detected at concentrations between the MRL and the MDL will be reported with a *J* qualifier to indicate that the value is an estimate (i.e., the analyte concentration is below the calibration range).

The achievement of reporting limits depends on the instrument's sensitivity and sample matrix effects. Therefore, it is important to monitor the instrument's sensitivity to ensure the data quality through appropriate instrument performance. The instrument's sensitivity will be monitored through the analysis of method blanks, calibration check samples, and laboratory control samples.

Additional laboratory QC results will be evaluated to provide supplementary information regarding overall quality of the data, performance of instruments and measurement systems, and sample-specific matrix effects.

2.4 DATA COMPLETENESS, REPRESENTATIVENESS, AND COMPARABILITY

Data generated in the field and the laboratory will be evaluated for completeness, representativeness, and comparability as described below

2.4.1 Completeness

The data will be reviewed and/or validated to keep invalid data from being processed through data collection. Completeness is evaluated using the following equation:

$$\text{Completeness} = \frac{\text{Acceptable Results}}{\text{Total Results}} x100\% \quad (2\text{-}3)$$

Acceptable results (usable data) are unqualified data and *U*- or *J*-qualified data. Completeness will be calculated for each suite of analytes for each sample type. The completeness goal for this project is 90 percent.

2.4.2 Representativeness

Representativeness is a qualitative QA/QC parameter. Representativeness is the degree to which data represent a characteristic of an environmental condition. Field personnel will be responsible for ensuring that samples are representative of field conditions by collecting and handling samples according to approved FSP and field SOPs. Errors in sample collection, packaging, preservation, or COC procedures may result in samples being judged as non-representative and may form a basis for rejecting the data.

Data generated by the laboratory must be representative of the laboratory database of accuracy and precision measurements for analytes in different matrices. Laboratory procedures for sample preparation will ensure that aliquots used for analysis are representative of the whole sample. Representativeness will be ensured by the proper handling and storage of samples and initiation of analysis within holding times.

2.4.3 Comparability

Comparability is the qualitative similarity of one data set to another (i.e., the extent to which different data sets can be combined for use). Comparability will be assessed by evaluating whether 1) sample collection and handling procedures adhered to the procedures specified in the QAPP and the work plan; and 2) laboratory procedures followed standard analytical protocols, used standard units and standardized report formats, followed the calculations as referenced in approved analytical methods, and used a standard statistical approach for QC measurements. Additionally, MUA well water samples will be analyzed at two laboratories to provide data on inter-laboratory variability.

3 DOCUMENTATION AND RECORDS

Records will be maintained documenting all activities and data related to sample collection and to laboratory analyses. Results of data verification and validation activities will also be documented. Procedures for documentation of these activities are described in this section.

This document, the FSP (Appendix B) and the health and safety plan (HSP; Attachment B1 to Appendix B) will be provided to every task participant listed in Section 1.3, with the exception that the FSP and HSP will not be provided to the laboratories. Any revisions or amendments to any of these documents will also be provided to these individuals.

3.1 FIELD RECORDS

Components of field documentation are discussed in Section 3 of the FSP. Integral's field lead will ensure that the field team receives the final, approved version of this document, the FSP, and HSP prior to the initiation of field activities. Field records that will be maintained include the following:

- Field logbooks
- Photo documentation
- Field data and sample collection information forms
- Field change request forms (as needed)
- Sample tracking/ COC forms.

Observations recorded in the field logbook will be used to provide context and aid in presentation and interpretation of analytical results. Additional details regarding the content and use of these documents are described in Section 3.1 of the FSP.

3.2 LABORATORY DATA REPORTS

All activities and results related to sample analysis will be documented at each laboratory. Internal laboratory documentation procedures are described in the laboratory QA manuals (Attachments A-1 and A-2).

Each laboratory will provide a data package for each sample delivery group or analysis batch that is comparable in content to a full contract laboratory program (CLP) package. The format of the data may differ from CLP requirements. Each data package will contain all information required for a complete QA review, including the following:

- A cover letter discussing analytical procedures and any difficulties that were encountered
- A case narrative referencing or describing the procedures used and discussing any analytical problems and deviations from SOPs and this document
- COCs and cooler receipt forms
- A summary of analyte concentrations (to two significant figures, unless otherwise justified), MRLs, and MDLs
- Laboratory data qualifier codes appended to analyte concentrations, as appropriate, and a summary of code definitions
- Sample preparation, digestion, extraction, dilution, and cleanup logs
- Instrument tuning data
- Initial and continuing calibration data, including instrument printouts and quantification summaries, for all analytes
- Results for method and instrument blanks
- Results for all QA/QC checks, including but not limited to surrogate spikes, internal standards, LCSs, MS samples, MSD samples, and laboratory duplicate samples provided on summary forms
- Instrument data quantification reports for all analyses and samples
- Copies of all laboratory worksheets and standards preparation logs.

Data will be delivered by the laboratories in both hard copy and electronic format to the task laboratory coordinator, who will be responsible for oversight of data verification and validation and for archiving the final data and data quality reports in the project file. EDDs will be compatible with the project database.

3.3 DATA QUALITY DOCUMENTATION

Data verification (i.e., confirming the accuracy and completeness of field and laboratory data) will be completed by Integral's technical team for data generated in the field, and by each laboratory for the data that it generates. Data validation reports for chemical analyses will be prepared as described in Section 12 and provided to Integral's Laboratory Coordinator. All changes to data stored in the database will be recorded in the database change log. Any data tables prepared from the database for data users will include all qualifiers that were applied by the laboratory and during data validation.

3.4 REPORTS AND DELIVERABLES

Data from samples collected from MUA wells will be provided to each respective MUA in a tabular format following receipt of the final validated results. Groundwater, surface water, and sediment sample data will be provided to NJDEP following receipt of all validated data. Laboratory and data validation reports will accompany all transmittals of data.

4 SPECIAL TRAINING REQUIREMENTS/CERTIFICATION

A technical team will be assembled with the requisite experience and technical skills to successfully complete the proposed study. All technical team personnel involved in sample collection will have extensive environmental sampling experience.

Sampling personnel will be required to have completed the 40-hour Hazardous Waste Operations and Emergency Response (HAZWOPER) standard training course and 8-hour refresher courses (see HSP for further explanation). Sampling personnel participating in the onsite groundwater sampling will be required to have completed additional Solvay site-specific safety training courses (see HSP for further details). The training provides employees with knowledge and skills that enable them to perform their jobs safely and with minimum risk to their personal health. Documentation of course completion will be maintained in personnel files.

The selected laboratories will hold certification through the NJDEP and National Environmental Laboratory Accreditation Program for the methods which that laboratory will perform, where applicable. Training and certification requirements for laboratory personnel are provided in the laboratory QA plans (Attachments A-1 and A-2).

5 ANALYTICAL METHOD REQUIREMENTS

Samples collected for this study will be analyzed for the chemicals of interest as indicated in Table A-1. The analytical methods that will be used are presented in the following sections and summarized in Table A-3. MRLs and MDLs are presented in Table A-4.

5.1 METHODS FOR MUA, GROUNDWATER, AND SURFACE WATER SAMPLES

All MUA samples will be analyzed for PFCs by Eurofins Eaton Analytical of Monrovia, California. Additionally, 10 to 20 percent of the MUA samples will be analyzed for PFCs by TestAmerica of Denver, Colorado, to provide information on inter-laboratory variability. All groundwater and surface water samples will be analyzed for PFCs by TestAmerica of Denver, Colorado.

Eurofins Eaton Analytical employs EPA Method 537 for the analysis of PFCs, and TestAmerica uses in-house method DV-LC-0012. Both methods use solid phase extraction with high performance liquid chromatography, tandem mass spectrometry (LC-MS/MS). The laboratories' SOPs for these methods are considered confidential and were not released; however, method performance information is provided with the laboratory QA manuals in Attachments A-1 and A-2.

Analyses for conventional parameters in surface water samples will be performed by TestAmerica of Edison, New Jersey, as indicated in Table A-3. Surface water samples will be analyzed for the following conventional parameters:

- Total organic carbon (TOC)
- Total suspended solids
- pH
- Total hardness
- Alkalinity.

The laboratories SOPs for these methods are provided with the laboratory QA manuals in Attachment A-2.

5.2 METHODS FOR SEDIMENT SAMPLES

Sediment samples will be analyzed for PFCs by TestAmerica of Denver, Colorado, and for conventional parameters by TestAmerica of Burlington, Vermont, and Edison, New Jersey, as indicated in Table A-3.

The analysis for PFCs will be performed by TestAmerica's in-house method DV-LC-0012. Samples are extracted by sonication and analysis is performed using LC-MS/MS.

Sediment samples will be analyzed for the following conventional parameters:

- Grain size
- TOC.

The laboratories' SOPs for these methods are provided with the laboratory QA manuals in Attachment A-2.

6 QUALITY ASSURANCE/QUALITY CONTROL REQUIREMENTS

QC samples will be prepared in the field and at the laboratory to monitor the precision and bias of the sample collection and analysis procedures.

6.1 FIELD SAMPLING

Field QC is discussed in Section 2.11 of the FSP.

6.2 LABORATORY ANALYSIS

The analytical method protocols include descriptions of QC procedures, control limits, and requirements for corrective actions. Laboratory control limits are provided in Tables A-5 and A-6. Control limits are updated periodically by the laboratories and control limits that are in effect at the laboratory at the time of analysis will be used for sample analysis and data validation. These may differ slightly from the control limits shown Tables A-5 and A-6. QC procedures will be completed by the laboratory, as required in the protocols and their SOPs. Laboratory QC procedures are summarized below.

6.2.1 Laboratory Control Samples

Laboratory control samples or laboratory fortified blanks (LFBs) will be performed for all parameters indicated in Table A-5 at a frequency of one every 20 samples, or one per extraction batch, whichever is more frequent. Laboratory control limits for recoveries of LCS are provided in Table A-5. If the LCS or LFB recoveries do not meet the laboratory control limits the samples should be reanalyzed.

6.2.2 Laboratory Duplicates

Laboratory duplicates will be performed for all parameters indicated in Table A-5 at a frequency of one every 20 samples, or one per extraction batch, whichever is more frequent. Laboratory control limits for laboratory duplicate RPDs are provided in Table A-5. If laboratory duplicate RPDs do not meet the laboratory control limits the data should be flagged by the laboratory to indicate this outlier.

6.2.3 Matrix Spikes

MSs and MSDs will be performed for all parameters indicated in Table A-5, at a frequency of one set every 20 samples, or one set per extraction batch, whichever is more frequent. An LCSD may be performed if insufficient sample volume is available for an MS/MSD. Laboratory control limits for recoveries of MSs and for RPD between MS/MSDs are provided in Table A-5. If the laboratory control limits for percent recovery or RPD are not met, the data should be flagged by the laboratory to indicate this outlier.

6.2.4 Surrogate Standards

Surrogates will be added to all samples for PFC analyses prior to extraction. Laboratory control limits for surrogate recoveries are provided in Table A-6. If the laboratory control limits for percent recovery are not met the samples should be reanalyzed.

6.2.5 Internal Standards

Internal standards are added to all sample extracts for PFCs. The area of the internal standards should be within the limits specified in the laboratory's SOP. If the internal standard area does not meet this criterion the sample should be reanalyzed.

6.2.6 Method Blanks

Method blanks will be analyzed at a frequency of one every 20 samples, or one per extraction batch, whichever is more frequent. Method blanks are not performed for grain size analyses. If target analytes are detected in the method blanks greater than the MRL the associated samples should reanalyzed.

6.2.7 Instrument Blanks

Instrument blanks will be performed with all analyses for TOC. Instrument banks will be analyzed following every calibration verification standard. If TOC is detected in the instrument blanks greater than the MRL the associated samples should reanalyzed.

7 INSTRUMENT/EQUIPMENT TESTING, INSPECTION, AND MAINTENANCE REQUIREMENTS

This section describes testing, inspection, and maintenance requirements for field and laboratory instruments.

7.1 FIELD INSTRUMENTS/EQUIPMENT

Maintenance of field instruments and equipment are discussed in Section 2.9.2 of the FSP.

7.2 LABORATORY INSTRUMENTS

Analytical instrument testing, inspection, maintenance, setup, and calibration will be conducted by the laboratories in accordance with the requirements identified in the laboratories' SOPs and manufacturer instructions. In addition, each of the specified analytical methods provides protocols for proper instrument setup and tuning and critical operating parameters. Instrument maintenance and repair will be documented in the maintenance log or record book.

8 INSTRUMENT CALIBRATION AND FREQUENCY

This section describes the frequency and requirements for the calibration of field and laboratory instruments.

8.1 FIELD INSTRUMENTS/EQUIPMENT

Calibration of field instruments and equipment are discussed in Section 2.9.2 of the FSP.

8.2 LABORATORY INSTRUMENTS

Laboratory instruments will be properly calibrated, and the calibration will be verified with appropriate check standards and instrument blanks for each parameter before beginning each analysis. Instrument calibration procedures and schedules will conform to analytical protocol requirements and descriptions provided in the laboratories' QA plans and SOPs.

All calibration standards will be obtained from a commercial vendor, and the laboratories will maintain traceability back to the National Institute of Standards and Technology. Stock standards will be used to make intermediate standards and calibration standards. Special attention will be given to expiration dating, proper labeling, proper refrigeration, and prevention of contamination. Documentation relating to the receipt, mixing, and use of standards will be recorded in a laboratory logbook. All calibration and spiking standards will be checked against standards from another source, as specified in the methods and the laboratory QA manual.

9 INSPECTION/ACCEPTANCE REQUIREMENTS FOR SUPPLIES AND CONSUMABLES

The quality of supplies and consumables used during sample collection and laboratory analysis can affect the quality of the project data. All equipment that comes into contact with the samples and extracts must be sufficiently clean to prevent detectable contamination, and the analyte concentrations must be accurate in all standards used for calibration and QC purposes.

During sample collection, the quality of laboratory water used for decontamination will be documented at the laboratory that provides that water. Precleaned sample jars (with documentation) will be provided by the laboratories. All containers will be visually inspected prior to use, and any suspect containers will be discarded.

Reagents of appropriate purity and suitably cleaned laboratory equipment will also be used for all stages of laboratory analyses. Details for acceptance requirements for supplies and consumables at the laboratories are provided in the laboratory SOPs and QA manuals (Attachments A-1 and A-2). All supplies will be obtained from reputable suppliers with appropriate documentation or certification. Supplies will be inspected to confirm that they meet use requirements, and certification records will be retained by Integral (i.e., for supplies used in the field) or the laboratories.

10 DATA MANAGEMENT

This section describes the data management procedures that will be used for incorporating data into the project database.

10.1 DATA MANAGEMENT PLAN

Data management processes will occur throughout all phases of this project, as part of, or underlying, all other activities. The data management processes that accompany each of these other activities differ, however. The principal data management processes are described in the following sections.

10.1.1 Implement the Project Database

A project database will be set up that is capable of accommodating all of the data that are planned to be collected. This step will be completed early in the process because analysis of existing data sets is necessary to help develop the work plan.

Activities carried out during implementation of the project database will include:

- Initiation of a project-specific instance of a general-purpose environmental database (database development is not required; Integral will use a model database that it has developed specifically for similar projects)
- Establishment of backup procedures for the database
- Initialization of the database with lists and definitions of valid values for the types of samples, analytes, and results that will be measured
- Establishment of access controls on the database so that only appropriately qualified staff can access or update the database.

10.1.2 Acquire Existing Site Data

Data that have been previously collected from the Site, and that are relevant to the analyses to be carried out under the work plan, will be obtained and loaded into the project database. Data management activities carried out during this process will include:

- Acquisition of data tables and documents
- Discussions with original investigators, as necessary, to compile detailed information on sampling locations, dates, and depths; methods; detection limits; measurement bases; and concentrations

- Acquisition of relevant spatial data to provide context for analytical measurements made at the Site and in the surrounding region
- Reformatting the data such that they conform to rules and constraints within the project database
- Loading those data into the project database
- Review of the data and execution of QA check to ensure that the entered data are correct and complete.

After existing data have been acquired, the quality of these data will be assessed for use in the investigation process.

10.1.3 Summarize Data for Project Planning

Data management activities carried out to support project planning will include:

- Recording planned sampling information in a way that allows later QA checks
- Ensuring that the types of data collected, the data structures used, and the data retrieval and summarization tools will all be able to support the planned data uses
- Organizing historical data as necessary so that it is in a consistent and well-documented format consistent with planned data uses
- Acquiring, organizing, transforming, and documenting the regional and site-specific spatial data needed to support data analysis and interpretation.

The overall project plan is documented in the work plan and this QAPP, which will be reviewed and approved by NJDEP.

10.1.4 Establish Electronic Data Deliverable Reporting Specifications

Analytical results will be provided by the laboratories in EDD formats that are specifically designed to facilitate loading of data into the project database. These specifications will be provided to the laboratories, and support will be provided to the laboratories in the production of appropriate deliverables. For this project, an EDD format will be used that facilitates upload of laboratory results into the project database, as well as the NJDEP EDD format described in the Technical Requirements for Site Remediation (N.J.A.C. 7:26E).

10.1.5 Integrate Field Sampling Results

Data management activities carried out to support field sampling will include (but not be limited to):

- Preparation of guidance and support materials, including maps and specifications for assignment of sample identifiers
- Receipt, entry, and QA checks of sampling information from field logbooks, sampling forms, and global positioning system devices
- Receipt, entry, and QA checks of methodological information and sample analysis results from analytical and testing laboratories
- Filing of hard copy materials.

10.1.6 Integrate Laboratory Analysis Results

Integration of laboratory results into the project database will include the following procedures:

- Review of EDDs provided by the laboratory to identify erroneous formats or missing or discrepant data; discrepancies will be resolved in cooperation with the laboratory
- Loading of data from EDDs into the project database
- Merging and linking of field-collected data and sample metadata not available to the laboratories with the appropriate lab-reported samples
- Production of summaries of field and laboratory QC measures to support data validation
- Following an independent validation according to specifications in the project QAPP, incorporation into the database of qualifiers and other results of data validation.

10.1.7 Summarize Data for Validation and Integrate Validation Results

Data management activities carried out to support data validation include:

- Preparation of summaries of sampling information, analytical results for field samples, and analytical results for laboratory QC samples, for use by data validators
- Updating of the database with data validation results, and QA checks of the updates
- Filing of hard copy materials
- Preparation of data tables for data reporting.

10.1.8 Maintain the Project Library

Data management activities carried out in support of document management will include:

- Document acquisition and transformation (e.g., scanning, text extraction) as necessary

- Entry of document citations, abstracts, keywords, reviewers' comments, and other information as necessary
- Linking of documents to relevant sampling or other Site data.

10.1.9 Summarize Data for Work Plan Analyses and Reporting

Data management activities carried out in support of analytical and reporting activities conducted under the work plan will include:

- Preparation of a wide variety of data summaries and maps
- Development of data summarization and reporting tools as necessary
- Acquisition and organization of ancillary data (e.g., guideline values, model parameters, model results) so that they can be efficiently used in conjunction with other data
- Preparation of report tables and figures.

10.1.10 Provide Access to Data

Data management activities carried out to allow respondents and regulators access to the project data include:

- Provision of tools, training, and support to respondent team members to allow querying of the database
- Setup of a website portal linked to the document and analytical chemistry (project) databases for the sharing of documents and data with regulators and reviewers
- Establishment of access controls on the website portal so that only appropriately qualified staff can access or update the website portal.
- Assistance to users to facilitate their understanding and use of the data.

10.1.11 Back Up and Maintain Database and Data Files

The project database, document library, and all geographic information system files will be stored on a networked server.

All data on the networked server will be stored on redundant, mirrored hard drives to protect against data loss due to hard drive failure. These drives will be mounted in a file server protected by an uninterruptible power supply. The data from these drives, as well as any other data stored elsewhere on the network, will be backed up daily after work hours to a separate, corporate backup server to protect against data loss due to user error. This backup enables access to the most recently changed versions of all files. Overall, components that make up the

data infrastructure (systems that store, backup, archive, etc.) and network systems will be monitored and maintained weekly to ensure that ongoing data storage and access needs are efficiently managed. Backup procedures will protect the data from system failure, accidental damage, catastrophic failures, and intrusion.

10.2 DATA SUMMARIZATION

Chemistry data will frequently be summarized for use in analyses or for presentation using tables or maps. Multiple concentration values for a given sample are often stored within a database as a result of field or laboratory replications, from field splits created for QC evaluations, or from sample reanalyses. Although field splits and laboratory replicates are created to support data quality assessments, all of the valid results that are produced are informative, and will be used to produce the most accurate possible estimate of the true concentration in a sample. When there are replicate results for a sample, the data will be averaged in a stepwise, or hierarchical, fashion. Because each level of the hierarchy represents a different source of variation, all the results at a single level are averaged together before results are averaged across other levels. The different levels of replication, and the source of variation that each represents, are as follows:

- Laboratory replicates—Variability of laboratory measurement methods
- Laboratory reanalyses—Variability of overall laboratory procedures
- Sample splits—Variability of field sample handling or homogenization procedures
- Field replicates—Spatial variability and variability of sample collection procedures.

Data will be summarized by successive averaging across these levels of replication, in the order given above. During the averaging process, data validation qualifiers and significant digits will be propagated. The rules for propagating the data validation qualifiers *U* (undetected), *J* (estimated), and *R* (rejected) are as follows:

- If both detected and undetected data are to be averaged, then if the detection limit of the undetected data is lower than the highest detected value, the value of the nondetect(s) will be taken at one-half the detection limit and averaged with the detected data, and the result will be identified as detected.
- If all data to be averaged are undetected, the result will be taken to be the lowest detection limit, and will be identified as undetected.
- If any of the data to be averaged are *J*-qualified data are averaged with non-*J*-qualified data, the final result will be *J*-qualified.
- If *R*-qualified data are averaged with non-*R*-qualified data, the result will be *R*-qualified.

10.3 DOCUMENTS

The project database will be used to store citations for all authoritative or finalized documents. The database will be used to record the name, authors, date, and other descriptive information for documents. Every project document will be assigned a unique identifier, and that identifier will be used as a key to the citation in the database. Electronic copies of project documents will be stored as PDF files and the document's file name will be included as part of the document description in the database. Paper copies of project documents will be filed by the document identifier.

10.3.1 Record Preservation

The storage of all source and reference documents within the project database allow for the preservation of all original values for each data set. This allows for both automated and interactive comparisons between original and summarized data.

11 ASSESSMENT/OVERSIGHT

This task will rely on the knowledge and expertise of the technical team, as described in Section 1. The field team and laboratories will stay in close verbal contact with the task manager and task laboratory coordinator during all phases of this task. This level of communication will serve to keep the management team informed about activities and events, and will allow for informal but continuous task oversight.

11.1 ASSESSMENTS AND RESPONSE ACTIONS

Assessment activities will include readiness reviews by the field coordinator prior to sampling, by the database administrator prior to release of the final data to the data users, and internal review while work is in progress. An informal technical systems audit may be conducted if problems are encountered during any phase of this project.

The first readiness review will be conducted by the field lead prior to field sampling to verify that all field equipment is ready for transfer to the Site. The field lead will also verify that the field team and any subcontractors have been scheduled and briefed and that the contracts for the subcontractors have been signed by both parties. Any deficiencies noted during this readiness review will be corrected prior to initiation of sampling activities.

The second readiness review will be completed by the database administrator before final data are released for use to verify that all results have been received from each laboratory, data validation has been completed for all of the data, and data qualifiers have been entered into the database and verified. Any deficiencies noted during this review will be corrected by the database administrator, or their designee. Data will not be released for final use until all data have been verified and validated. No report will be prepared in conjunction with the readiness reviews. However, the technical team coordinator and data users will be notified when the data are ready for use.

Technical review of intermediate and final work products generated for this task will be completed throughout the course of all sampling, laboratory, data validation, data management, and data interpretation activities to ensure that every phase of work is accurate and complete and follows the QA procedures outlined in this document. Any problems that are encountered will be resolved between the reviewer and the person completing the work. Any problems that cannot be easily resolved or that affect the final quality of the work product will be brought to the attention of the technical team coordinator and project coordinator.

Each laboratory will be required to have implemented a review system that serves as a formal surveillance mechanism for all laboratory activities. Details are provided in the laboratory QA plans (Attachments A-1 and A-2).

Technical system audits may be conducted if serious problems are encountered during sampling or analysis operations. If completed, these audits will be conducted by the task laboratory coordinator or designee, or by the laboratory, as appropriate. These audits may consist of onsite reviews of any phase of field or laboratory activities or data management. Results of any audits will be provided along with the associated laboratory data.

Any task team member who discovers or suspects a nonconformance is responsible for reporting the nonconformance to the project manager, the laboratory coordinator, or the laboratory project or QA manager, as applicable. The laboratory coordinator will ensure that no additional work dependent on the nonconforming activity is performed until a confirmed nonconformance is corrected. Any confirmed nonconformance issues will be relayed to the technical team coordinator.

11.2 REPORTS TO MANAGEMENT

Each laboratory has implemented routine systems of reporting nonconformance issues and their resolution. These procedures are described in the laboratory QA manuals (Attachments A-1 and A-2). The laboratories will keep the laboratory coordinator informed of any laboratory QC data outside of control limits and any corrective actions implemented and will provide descriptions and justification for any significant changes in methodology or QA/QC procedures. Laboratory nonconformance issues will also be described in the data report if they affect the quality of the data.

Data packages and EDDs will be prepared by each laboratory upon completion of analyses for each sample delivery group. The case narrative will include a description of any problems encountered, control limit exceedances (if applicable), and a description and rationale for any deviations from protocol. Copies of corrective action reports generated at the laboratory will also be included with the data package.

Data validation reports will be prepared following receipt of the complete laboratory data packages for each sample delivery group. These reports will be provided to the laboratory coordinator when validation is completed for each parameter. A summary of any significant data quality issues will be provided with the data report.

12 DATA REVIEW, VERIFICATION, AND VALIDATION

Data generated in the field and at the laboratory will be reviewed, verified, and validated according to the criteria and procedures described in this section.

12.1 DATA REVIEW AND VERIFICATION

Data review and verification will be performed by Integral in accordance with *Guidance on Environmental Data Verification and Validation* (USEPA 2002), which defines verification as "…the process of evaluating the completeness, correctness, and conformance/compliance of a specific data set against the method, procedural, or contractual requirements."

Field data will be verified during preparation of samples and COC forms. Field data and COC forms will be reviewed daily by the field supervisor. After field data are entered into the project database, 100 percent verification of the entries will be completed to ensure the accuracy and completeness of the database. All entries into the database will be verified. All errors found during the verification of field data, laboratory data, and the database will be corrected prior to release of the final data.

12.1.1 Laboratory Analyses and Tests

Review and verification of laboratory analyses and tests will be performed in conjunction with the data validation procedures described in Section 12.2.

12.1.2 Overall Sampling Results

Verification of the overall sampling results will be performed once all field sampling events are completed and all analytical data has been received. The FSP will be reviewed to verify that all planned samples were collected, including field QC samples. The project database will be reviewed to verify that all planned analytical data has been received and validated in accordance with the procedures described in Section 12.2.

12.2 DATA VALIDATION

All laboratory data will be validated by LDC of Carlsbad, California. The first data package generated for each matrix (MUA well water, groundwater, surface water, and sediment) will be fully validated, equivalent to a Stage 4 validation described in *Guidance for Labeling Externally Validated Laboratory Analytical Data for Superfund Use* (USEPA 2009). If no major problems are encountered during validation of these packages, subsequent data packages will be selected so that 10 percent of the packages are fully validated. Validation for the remaining data will be

based on review of the summary forms for sample and QC data, equivalent to a Stage 2B validation. If problems or questions are encountered during validation, the laboratory will be contacted for resolution. Additional full validation will be completed if required to fully assess the quality of the data or to verify that laboratory errors have been addressed. The accuracy and completeness of each data set will be verified at the laboratory when the EDDs are prepared and again as part of data validation. Ten percent of entries to the database from the laboratory EDDs will be checked against the hard-copy data packages.

No guidelines are available for validation of data for PFCs; the data will be validated using procedures described in the following U.S. Environmental Protection Agency (EPA) guidance documents for data validation:

- *Guidance on Environmental Data Verification and Validation* (USEPA 2002)
- *USEPA Contract Laboratory Program National Functional Guidelines for Superfund Organic Data Review* (USEPA 2008).

Data will be qualified as estimated as necessary if results for surrogates, laboratory control samples, matrix spike samples, matrix spike duplicates, or internal standards do not meet laboratory control limits. Results for other QC procedures will be qualified if they do not meet control limits outlined in the laboratory's SOP. Data will be qualified as undetected based on concentrations of target analytes detected in laboratory or field blanks, according to EPA's functional guidelines and SOPs for data validation.

Results for field split samples will be evaluated using control limits of 35 percent for aqueous samples and 50 percent for nonaqueous samples. Data will not be qualified as estimated if the measurement quality objectives are exceeded, but RPD results will be tabulated and any exceedances will be discussed in the data validation report. Equipment rinse blanks will be evaluated and data qualifiers will be applied in the same manner as method blanks, as described in the functional guidelines for data review (USEPA 2008).

Data will be rejected if control limits for acceptance of data are not met, as described in USEPA (2008).

13 RECONCILIATION WITH DATA QUALITY OBJECTIVES

The goal of data validation is to determine the quality of each data point and to identify data points that do not meet the project criteria. Nonconforming data may be qualified as estimated or rejected as unusable during data validation if criteria for data quality are not met. Data qualified as estimated will be used for all intended purposes and will be appropriately qualified in the final project database. These data may be less precise or less accurate than unqualified data. Rejected data will not be used for any purpose. An explanation of any rejected data will be included in the data validation report.

14 DATA USABILITY AND REPORTING

Following the completion of all sample analyses and data validation the data will be reported in a tabular format to the respective MUAs or NJDEP. A transmittal letter detailing any data limitations and their effect on data interpretation activities will accompany the data as will the data validation reports.

15 REFERENCES

NJDEP. 2005. Field sampling procedures manual. New Jersey Department of Environmental Protection, Trenton, NJ.

USEPA. 2002. Guidance on environmental data verification and validation. EPA QA/G-8. U.S. Environmental Protection Agency, Office of Environmental Information, Washington, DC.

USEPA. 2008. USEPA contract laboratory program functional guidelines for superfund organic methods data review. U.S. Environmental Protection Agency, Office of Emergency and Remedial Response, Washington, DC.

USEPA. 2009. Guidance for labeling externally validated laboratory analytical data for superfund use. U.S. Environmental Protection Agency, Office of Emergency and Remedial Response, Washington, DC.

FIGURES

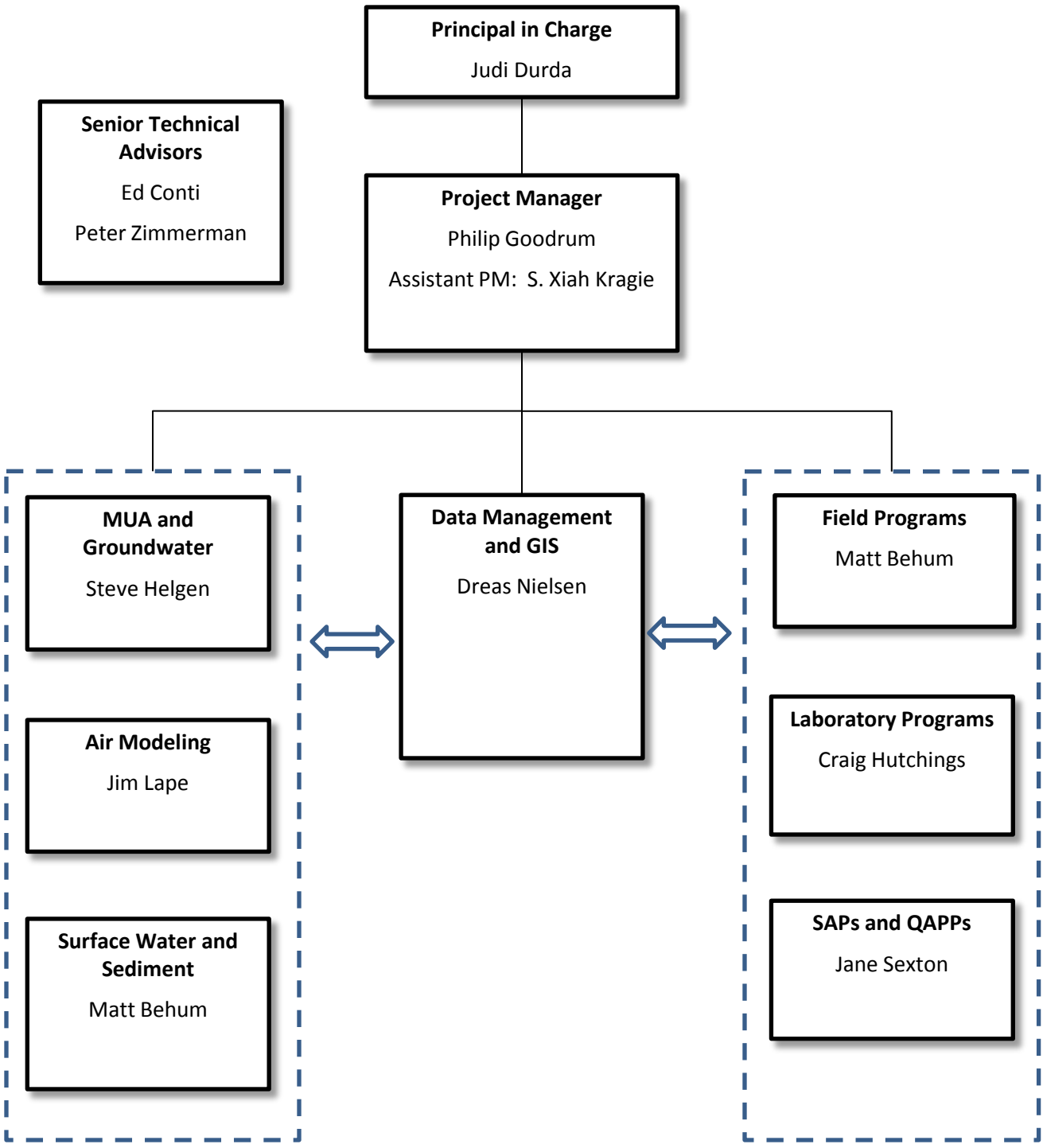
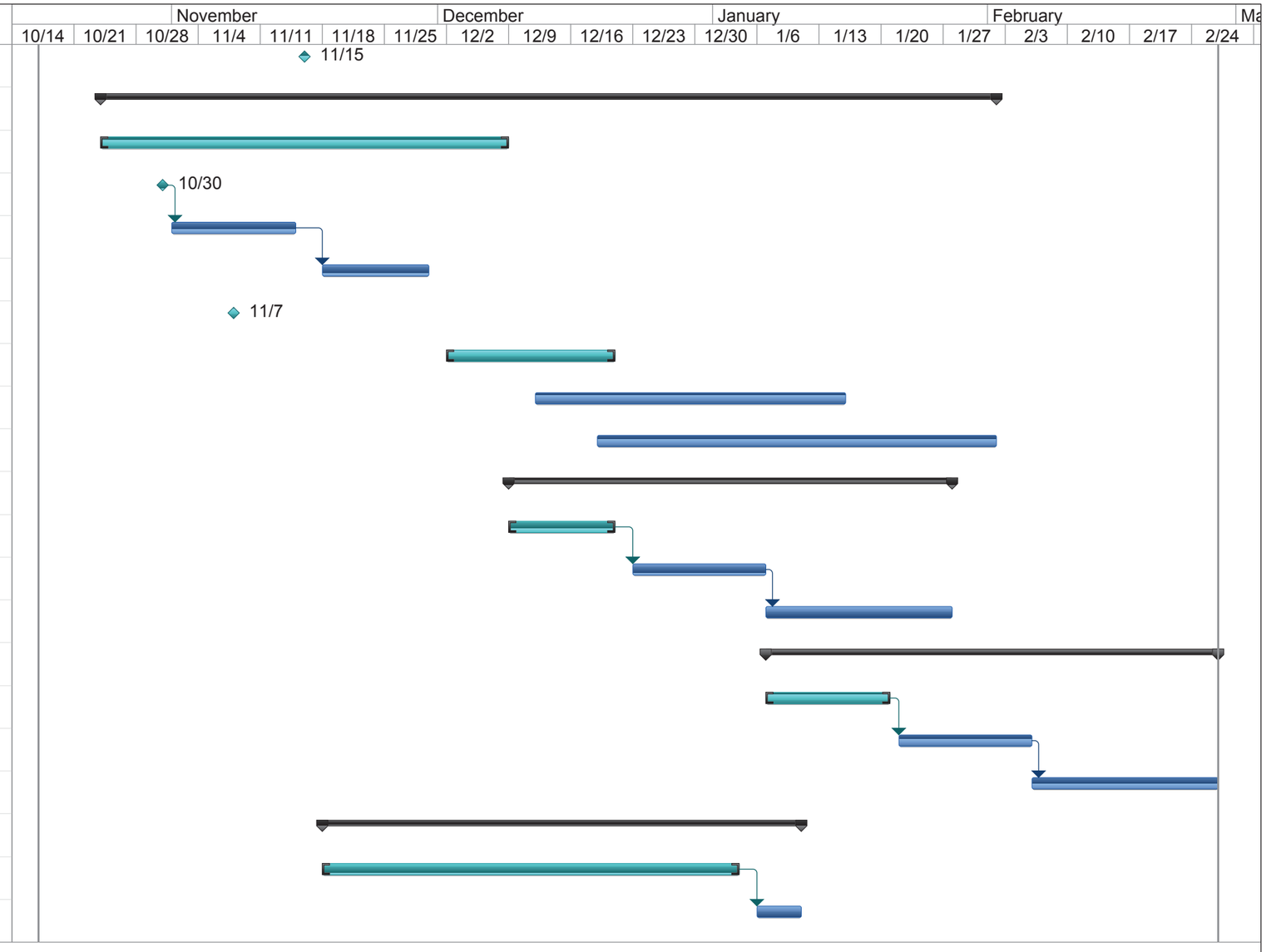


Figure A-1.
Integral Project Organizational Chart

ID	Task Name	Start	Finish
1	Work Plan to NJDEP	Fri 11/15/13	Fri 11/15/13
2	**Field MUA**	**Thu 10/24/13**	**Sat 2/1/14**
3	Municipal Outreach and Scheduling	Thu 10/24/13	Mon 12/9/13
4	W. Deptford - Sampling	Wed 10/30/13	Wed 10/30/13
5	W. Deptford - Lab Analysis and Reporting	Fri 11/1/13	Thu 11/14/13
6	W. Deptford - External Data Validation	Mon 11/18/13	Fri 11/29/13
7	NJDEP Letter to MUAs	Thu 11/7/13	Thu 11/7/13
8	Six MUAs - Sampling	Mon 12/2/13	Fri 12/20/13
9	Six MUAs - Lab Analysis and Reporting	Thu 12/12/13	Wed 1/15/14
10	Six MUAs - External Data Validation	Thu 12/19/13	Sat 2/1/14
11	**Field Groundwater Onsite Wells**	**Mon 12/9/13**	**Mon 1/27/14**
12	GW - Sampling	Mon 12/9/13	Fri 12/20/13
13	GW - Lab Analysis and Reporting	Mon 12/23/13	Mon 1/6/14
14	GW - External Data Validation	Tue 1/7/14	Mon 1/27/14
15	**Field Surface Water and Sediment**	**Tue 1/7/14**	**Wed 2/26/14**
16	SW & Sediment - Sampling	Tue 1/7/14	Mon 1/20/14
17	SW & Sediment - Lab Analysis and Reporting	Wed 1/22/14	Wed 2/5/14
18	SW & Sediment - External Data Validation	Thu 2/6/14	Wed 2/26/14
19	**Air Modeling**	**Mon 11/18/13**	**Fri 1/10/14**
20	Run Modeling Scenarios	Mon 11/18/13	Fri 1/3/14
21	Reporting	Mon 1/6/14	Fri 1/10/14



MUA - Municipal Utility Authority
GW - Groundwater
SW - Surface Water

integral consulting inc.

Figure A-2.
Gantt Chart of Proposed Project Schedule for Sampling and Lab Validation

TABLES

Table A-1. Chemicals of Interest

Analyte	CAS Number	Matrix			
		MUA Samples	Groundwater	Surface Water	Sediment
PFCs					
Perfluorooctanoic acid (PFOA;C8)	335-67-1	X	X	X	X
Perfluorononanoic acid (PFNA;C9)	375-95-1	X	X	X	X
Perfluorodecanoic acid (PFDA; C10)	335-76-2	X	X	X	X
Perfluoroundecanoic acid (PFUnDA; C11)	2058-94-8	X	X	X	X
Perfluorododecanoic acid (PFDoDA; C12)	307-55-1	X	X	X	X
Perfluorotridecanoic acid (PFTrDA; C13)	72629-94-8	X	X	X	X
Perfluorooctanesulfonic acid (PFOS; C8)	1763-23-1	X	X	X	X
Conventional Parameters					
Total organic carbon	7440-44-0			X	X
Total suspended solids	--			X	
pH	12408-02-5			X	
Hardness, Total	--			X	
Alkalinity	--			X	
Grainsize	--				X

Notes:

CAS = Chemical Abstracts Service registry number
MUA = Municipal Utility Authority
PFC = perfluorinated compound
X = samples will be collected and analyzed for these parameters
-- = not available

Table A-2. Sample Containers, Preservation, and Holding Time Requirements

Analyses	Container [a] Type	Container [a] Size	Preservation	Holding Time	Minimum Sample Size
Water Samples (MUA, Groundwater, Surface Water)					
PFCs (Eurofins)	PP	2 x 275 mL	≤10°C, greater than 0°C (not frozen); preserved with Trizma®	14 days to extraction 28 days from extraction to analysis	250 mL
PFCs (TestAmerica)	HDPE	2 x 250 mL	4 ± 2°C	7 days to extraction 40 days from extraction to analysis	250 mL
Total organic carbon	AG	250 mL	4 ± 2°C, H_2SO_4 to pH<2	28 days	50 mL
Total suspended solids	HDPE	1,000 mL	4 ± 2°C	7 days	100 mL
pH	HDPE	250 mL	4 ± 2°C	Analyze immediately	100 mL
Hardness, total	HDPE	500 mL	4 ± 2°C, HNO_3 to pH<2	6 months	50 mL
Alkalinity	HDPE	500 mL	4 ± 2°C	14 days	50 mL
Sediments					
PFCs (TestAmerica)	Wide Mouth HDPE	250 mL	4 ± 2°C	14 days to extraction 40 days from extraction to analysis	10 g
Grainsize	WMG	16 oz.	4 ± 2°C	6 months	500 g
Total organic carbon	WMG	4 oz.	4 ± 2°C	14 days	3 g

Notes:
AG = amber glass
HDPE = high density polyethylene
HNO_3 = nitric acid
H_2SO_4 = sulfuric acid
MUA = Municipal Utility Authority
PFC = perfluorinated compound
PP = polypropylene
WMG = wide mouth glass

[a] The size and number of containers may be modified by the analytical laboratory.

Table A-3. Laboratory Methods for MUA, Groundwater, Surface Water, and Sediment Samples

		Sample Preparation		Quantitative Analysis	
Analyses	Laboratory	Protocol	Procedure	Protocol	Procedure
Water Samples (MUA, Groundwater, Surface Water)					
PFCs	Eurofins	EPA 537	Solid phase extraction	EPA 537	LC-MS/MS
	TestAmerica Denver	Laboratory SOP: DV-LC-0012	Solid phase extraction	Laboratory SOP: DV-LC-0012	LC-MS/MS
Total organic carbon	TestAmerica Edison	SM 5310B	Heated-persulfate oxidation	SM 5310B	Infrared detector
Total suspended solids		SM 2540D	Filtration and drying	SM 2540D	Gravimetric
pH		--	--	SM 4500H^+ B	Electrometric
Hardness, Total		--	--	SM 2340 C	Titrimetric
Alkalinity		--	--	SM 2320 B	Titrimetric
Sediments					
PFCs	TestAmerica Denver	Laboratory SOP: DV-LC-0012	Sonication	Laboratory SOP: DV-LC-0012	LC-MS/MS
Grain size	TestAmerica Burlington	--	--	ASTM D422	Sieve and hydrometer
Total organic carbon	TestAmerica Edison	Lloyd Kahn	Acid pretreatment	Lloyd Kahn	TCD

Notes:
ASTM = American Society for Testing and Materials
EPA = U.S. Environmental Protection Agency
LC-MS/MS = liquid chromatography- tandem mass spectrometry
MUA = Municipal Utility Authority
PFC = perfluorinated compound
SM = Standard Methods for the Examination of Water and Wastewater
SOP = standard operating procedure
TCD = thermal conductivity detector

Table A-4. Analytes, Method Reporting Limits, and Method Detection Limits

Analyte	Units	MRL	MDL
MUA, Groundwater, and Surface Water Samples			
PFCs - Eurofins			
Perfluorooctanoic acid (PFOA)	µg/L	0.0025	0.0025
Perfluorononanoic acid (PFNA)	µg/L	0.0025	0.0006
Perfluorodecanoic acid (PFDA)	µg/L	0.0025	0.0003
Perfluoroundecanoic acid (PFUnDA)	µg/L	0.0025	0.0005
Perfluorododecanoic acid (PFDoDA)	µg/L	0.0025	0.0007
Perfluorotridecanoic acid (PFTrDA)	µg/L	0.0025	0.0010
Perfluorooctanesulfonic acid (PFOS)	µg/L	0.0025	0.0003
PFCs - TestAmerica Denver			
Perfluorooctanoic acid (PFOA)	µg/L	0.02	0.0098
Perfluorononanoic acid (PFNA)	µg/L	0.04	0.0174
Perfluorodecanoic acid (PFDA)	µg/L	0.02	0.0078
Perfluoroundecanoic acid (PFUnDA)	µg/L	0.02	0.0069
Perfluorododecanoic acid (PFDoDA)	µg/L	0.03	0.0149
Perfluorotridecanoic acid (PFTrDA)	µg/L	0.04	0.0177
Perfluorooctanesulfonic acid (PFOS)	µg/L	0.03	0.0133
Conventional Parameters			
Total organic carbon	mg/L	1.00	0.296
Total suspended solids	mg/L	10.0	--
pH	pH units	NA	NA
Hardness, Total	mg/L	5.00	1.62
Alkalinity	mg/L	5.00	--
Sediment Samples			
PFCs - TestAmerica Denver			
Perfluorooctanoic acid (PFOA)	µg/kg	0.80	0.23
Perfluorononanoic acid (PFNA)	µg/kg	0.80	0.22
Perfluorodecanoic acid (PFDA)	µg/kg	0.80	0.27
Perfluoroundecanoic acid (PFUnDA)	µg/kg	0.80	0.32
Perfluorododecanoic acid (PFDoDA)	µg/kg	2.00	0.57
Perfluorotridecanoic acid (PFTrDA)	µg/kg	0.80	0.32
Perfluorooctanesulfonic acid (PFOS)	µg/kg	0.80	0.14
Conventional Parameters			
Grainsize	percent	NA	NA
Total organic carbon	mg/kg	1,000	NA

Notes:

MDL = method detection limit

MRL = method reporting limit

MUA = Municipal Utility Authority

NA = not applicable

PFC = perfluorinated compound

-- = not available

Table A-5. Laboratory Control Limits for Matrix Spike and Laboratory Control Samples [a]

Analytes	MS/LFSM Recovery (%)	LCS/LFB Recovery (%)	Type of Duplicate	Control Limit RPD
MUA, Groundwater, and Surface Water Samples				
PFCs - Eurofins				
Perfluorooctanoic acid (PFOA)	70–130 [b]	70–130 [b]	LFSMD	30 [c]
Perfluorononanoic acid (PFNA)	70–130 [b]	70–130 [b]	LFSMD	30 [c]
Perfluorodecanoic acid (PFDA)	70–130 [b]	70–130 [b]	LFSMD	30 [c]
Perfluoroundecanoic acid (PFUnDA)	70–130 [b]	70–130 [b]	LFSMD	30 [c]
Perfluorododecanoic acid (PFDoDA)	70–130 [b]	70–130 [b]	LFSMD	30 [c]
Perfluorotridecanoic acid (PFTrDA)	70–130 [b]	70–130 [b]	LFSMD	30 [c]
Perfluorooctanesulfonic acid (PFOS)	70–130 [b]	70–130 [b]	LFSMD	30 [c]
PFCs - TestAmerica Denver				
Perfluorooctanoic acid (PFOA)	62–132	62–132	MSD	20
Perfluorononanoic acid (PFNA)	70–138	70–138	MSD	30
Perfluorodecanoic acid (PFDA)	64–146	64–146	MSD	30
Perfluoroundecanoic acid (PFUnDA)	70–138	70-138	MSD	30
Perfluorododecanoic acid (PFDoDA)	60–154	60–154	MSD	30
Perfluorotridecanoic acid (PFTrDA)	44–164	44–164	MSD	30
Perfluorooctanesulfonic acid (PFOS)	60–130	60-130	MSD	20
Conventional Parameters				
Total organic carbon	80-120	Vendor specified limits	MSD	10
Total suspended solids	NA	85–115	LD	5
pH	NA	Vendor specified limits	LD	10
Hardness, Total	NA	85–115	LD	10
Alkalinity	NA	Vendor specified limits	LD	27
Sediments				
PFCs - TestAmerica Denver				
Perfluorooctanoic acid (PFOA)	57–153	57–153	MSD	30
Perfluorononanoic acid (PFNA)	50–170	50–170	MSD	30
Perfluorodecanoic acid (PFDA)	51–169	51–169	MSD	30
Perfluoroundecanoic acid (PFUnDA)	67–152	67–152	MSD	30
Perfluorododecanoic acid (PFDoDA)	47–168	47–168	MSD	30
Perfluorotridecanoic acid (PFTrDA)	34–152	34–152	MSD	30
Perfluorooctanesulfonic acid (PFOS)	70–130	70–130	MSD	30
Conventional Parameters				
Grainsize	NA	NA	NA	NA
Total organic carbon	NA	Vendor specified limits	QD	20 [d]

Notes:

LCS = laboratory control sample
LD = lab duplicate
LFB = laboratory fortified blank
LFSM = laboratory fortified sample matrix
LFSMD = laboratory fortified sample matrix duplicate
MS = matrix spike
MSD = matrix spike duplicate
MUA = Municipal Utility Authority
NA = not applicable
PFC = perfluorinated compound
QD = laboratory quadruplicate
RPD = relative percent difference

[a] Control limits are updated periodically by the laboratories. Control limits that are in effect at the laboratory at the time of analysis will be used for sample analysis and data validation. These may differ slightly from the control limits shown in this table.

[b] EPA Method 537 requires the lab to rotate between low, medium, and high spike concentrations from batch to batch. Control limits of 70–130 percent apply to medium and high spike concentrations. For low spike concentrations control limits of 50–150 percent apply.

[c] EPA Method 537 requires the lab to rotate between low, medium, and high spike concentrations from batch to batch. A control limit of 30 percent applies to medium and high spike concentrations. For low spike concentrations a control limit of 50 percent applies.

[d] The laboratory quadruplicate will be evaluated based on the percent relative stanard deviation.

Table A-6. Laboratory Control Limits for Surrogates

Analytes	Surrogate Recovery (%)
MUA Samples - Eurofins	
Perfluor-n-[1,2-$^{13}C_2$]decanoic acid (^{13}C-PFDA)	70–130
Perfluor-n-[1,2-$^{13}C_2$]hexanoic acid (^{13}C-PFHxA)	70–130
MUA, Groundwater, and Surface Water Samples - TestAmerica Denver	
$^{13}C_8$- Perfluorooctanoic acid ($^{13}C_8$-PFOA)	60–155
$^{13}C_8$-Perfluorooctanesulfonate ($^{13}C_8$-PFOS)	45–130
Sediments - TestAmerica Denver	
$^{13}C_8$- Perfluorooctanoic acid ($^{13}C_8$-PFOA)	60–155
$^{13}C_8$-Perfluorooctanesulfonate ($^{13}C_8$-PFOS)	45–130

Notes:
MUA = Municipal Utility Authority

ATTACHMENT A1

EUROFINS QUALITY ASSURANCE MANUAL AND SOPS

TABLE 9.6 UCMR 3 QA/QC REQUIREMENTS FOR METHOD 537

Sample Description/Frequency	Acceptance Criteria	Corrective Action
Initial Calibration (As needed)	Peak Assymetry Factor is calculated on mid-Cal std. Peak asymmetry factor of 0.8 – 1.5. Back calculation is required. Recoveries should be within 70%-130% for all points except those at or below the MRL where the range is 50% to 150%. <20% RPD among IS responses in the lowest and highest calibration standards.	Recalibrate before sample analysis.
Quality Control Sample (QCS) (At least quarterly)	Analyze a standard from a second source at mid-calibration range. Results must be within 70-130% of true value.	Correct the problem source then repeat initial calibration.
Continuing Calibration Checks (CCC) (Low-level CCC analyzed at the beginning of each analysis batch, subsequent CCC's analyzed following every 10 field samples and at the end of batch)	The lowest CCC at or below MRL must be within ±50% of the true value. All other points must be within ±30%. High biased CCC acceptable if ND sample (only for random high biased failure, no pattern).	Instrument maintenance to recover sensitivity, followed by recalibration and verification of sensitivity by a low level CCC
Internal Standards (IS) (Added to all extracts, field samples, standards, and QC samples)	70-140% of the most recent previous CCC and ±50% of the average area in the initial calibration.	Check and optimize the instrument. Re-analyze out of control samples.
Surrogate Recovery (Added to all extracts, field samples, standards, and QC samples)	Acceptance criteria ±30%	Check and optimize the instrument. Re-analyze out of control samples.
Lab Reagent Blank (LRB) (Analyzed with each extraction/analysis batch)	All target analytes ≤1/3 MRL concentration.	Identify and correct source of problem.
Lab Fortified Blank (LFB) (LFB at ≤MRL level per extraction batch and each analysis batch)	LFB ≤ MRL: ±50% of the true value for each target analyte. All other concentrations must be within ±30% of the true value for all analytes.	Identify and correct source of problem.
Field Reagent Blank (FRB) (Analyzed only when there are sample hits)	All target analytes ≤1/3 MRL.	If FRB shows unacceptable contamination, then field samples collected with that FRB are invalid and new samples must be collected.

TABLE 9.6 UCMR 3 QA/QC REQUIREMENTS FOR METHOD 537 (CONT'D)

Sample Description/Frequency	Acceptance Criteria	Corrective Action
LFSM of a Field Sample (One per analysis batch)	At low-level fortification concentrations, 50-150% recovery. At mid-level fortification concentrations, 70-130% recovery. Low-level spike concentration is ±50% of the MRL level. Mid-level spike concentration is ±20% of the mid-level cal standard.	If % recovery is out of the designated range, but laboratory performance for all other QC is acceptable, the recovery problem is judged to be matrix related, but is still reportable. UCMR 3 does not have target analyte recovery acceptance criteria specified for LFSM results, thus not rejected. If IS or Surrogate fails, but all other pass, then LFSM and LFSMD cannot be reported. If IS or surrogate fails in LFSM, the LFSMD data should be substituted for the LFSM so accuracy can be assessed. If IS or surrogate for both LFSM and LFSMD fail, then no matrix spike data are reported. IS or surrogate recovery failure of the LFSM/LFSMD do not invalidate the associated sample results.
LFSMD of Field Sample (One per analysis batch)	RPD between LFSM and LFSMD ≤50% for low-level fortification concentrations and ≤30% for mid-level fortification concentrations.	If % recovery or RPD is out of the designated range, but laboratory performance for all other QC is acceptable, the recovery problem is judged to be matrix related, but is still reportable. UCMR 3 does not have target analyte recovery and RPD acceptance criteria specified for LFSM/ LFSMD results, thus not rejected. If IS or Surrogate fails, but all other pass, then LFSM and LFSMD cannot be reported. If IS or surrogate fails in LFSM, the LFSMD data should be substituted for the LFSM so accuracy can be assessed. If IS or surrogate for both LFSM and LFSMD fail, then no matrix spike data are reported. IS or surrogate recovery failure of the LFSM/LFSMD do not invalidate the associated sample results.

QC Type	Nonconformance****	Invalidated Data	UCMR 3 Lab Approval Requirements and Information Document 01/2012, v 2.0 / EEA UCMR3 QAPP
2. Continuing Calibration Checks (CCC)*** (cont'd)	Failure to meet the acceptance criteria for verifying calibration using CCC standards. (Table 4 – CCC acceptance criteria for all methods)	Reject any samples or extracts analyzed between the failing CCC standard and the last acceptable CCC standard.	p.30 / Attachment B p.2
	High biased CCC for ND samples.	Reject all ND data analyzed between the failing CCC standard and the last acceptable CCC standard. Only exception as defined by method to report ND data/high biased CCCs (exceptions used at random, <5% of analytical batches): →Methods 522 (allowed only for ending CCCs with high recoveries for target analytes and/or high surrogate recoveries) and 537 (allowed for CCC results and high recoveries exclusive to target analytes).	p.30
	Pattern of high biased allowance for ND results for methods 522 and 537 (>5% of analytical batches).	A formal corrective action is needed to investigate cause of high biased CCCs.	p.30

QC Type		Nonconformance****	Invalidated Data	UCMR 3 Lab Approval Requirements and Information Document 01/2012, v 2.0 / EEA UCMR3 QAPP
2.	Continuing Calibration Checks (CCC)*** (cont'd)	Failure to meet CCC frequency of every 10 field samples or less and at the end of an analytical batch.	Reject all data that are not bracketed by acceptable CCCs with the correct frequency.	p.30
3.	Surrogate standard	Failure to meet the acceptance criteria for surrogate recovery in an ending CCC. (Table 4)	Reject all data from the last acceptable CCC for the failing CCC.	p.30 / Attachment B p.2
		Failure to meet the acceptance criteria for surrogate recovery in a specific sample. (Table 5)	If the surrogate in a specific sample does not meet the acceptance criteria, then process a new aliquot of the sample. If this is not possible, then the data for that sample are considered suspect for the analysis in question, and is to be flagged as not meeting QC criteria. Data for samples with failed surrogate recovery must not be reported.	p.32 / Attachment B p.3
		Failure to meet the acceptance criteria for surrogate recovery in an LRB or LFB. (Table 5)	Reject all data in the batch.	p.33 / Attachment B p.3
		Failure to meet the acceptance criteria for surrogate recovery in an LFSM or LFSMD, but all other batch QCs pass.	Only the fortified matrix sample (LFSM or LFSMD) with the failed surrogate is impacted and cannot be reported.	p.33
		Failure to meet acceptance criteria for surrogate recovery in an LFSM (LFSMD surrogate is acceptable).	The LFSMD data should be substituted for the LFSM such that accuracy can be assessed.	p.33

QC Type	Nonconformance****	Invalidated Data	UCMR 3 Lab Approval Requirements and Information Document 01/2012, v 2.0 / EEA UCMR3 QAPP
3. Surrogate standard (cont'd)	Failure to meet acceptance criteria for surrogate recovery in both LFSM and LFSMD.	Both LFSM and LFSMD cannot be reported, but surrogate recovery failures of the LFSM and LFSMD do not invalidate the associated sample results if the associated sample meets the IS and surrogate QC criteria.	p.33
4. Internal Standard (IS)	Failure to meet the acceptance criteria for IS recovery in a specific sample. (Table 6)	Reject data for that one sample (the lab can reprocess another aliquot of sample to confirm result – failed IS, data is rejected).	p.34 / Attachment B p.4
	Failure to meet the acceptance criteria for IS recovery in an LRB or LFB. (Table 6)	Reject all data in the batch.	p.34 / Attachment B p.4
	Failure to meet the acceptance criteria for IS recovery in an LFSM or LFSMD, but all other batch QC pass.	Only the fortified matrix sample with the failed internal standard is impacted and cannot be reported.	p.34
	Failure to meet the acceptance criteria for IS recovery in an LFSM (LFSMD IS is acceptable).	The LFSMD data should be substituted for the LFSM such that accuracy can be assessed.	p.34
	Failure to meet the acceptance criteria for IS recovery in both LFSM and LFSMD.	Both LFSM and LFSMD cannot be reported, but IS recovery failures of the LFSM and LFSMD do not invalidate the associated sample results if the associated sample meets the IS and surrogate QC criteria.	p.34

QC Type		Nonconformance****	Invalidated Data	UCMR 3 Lab Approval Requirements and Information Document 01/2012, v 2.0 / EEA UCMR3 QAPP
5.	Laboratory Reagent Blank (LRB)	Failure to meet LRB acceptance criteria (No analyte >1/3 the MRL or >1/2 the MRL for 524.3). (Table 7)	Reject all data for samples processed with an LRB that fails the criteria.	p.35 / Attachment B p.5
6.	Laboratory Fortified Blank (LFB)***	Failure to meet acceptance criteria in the low-level LFB (≤MRL) for any analyte. (Table 8)	Immediate reanalysis of the LFB is allowed, but if the re-run fails again, the analyst must correct the problem before analyzing field samples. Reject all data for samples processed with an LFB that fails the criteria.	p.37 / Attachment B p.6
7.	Quality Control Sample (QCS)	Failure to analyze a QCS at least quarterly, a source external to the laboratory, and different from the source of calibration standards (or different lot from calibration standard used if no second source is available). (Table 9)	Samples are not rejected but viewed as a finding/deficiency during a lab audit.	p. 38 / Attachment B p.7
8.	Laboratory Fortified Sample Matrix (LFSM/ LFSMD)	Failure to meet the acceptance criteria for surrogate and/or IS recovery in an LFSM or LFSMD, but all other batch QC pass.	Only the fortified matrix sample (LFSM/LFSMD) with the failed surrogate and/or IS is impacted and cannot be reported.	p.33, 34
		Failure to meet acceptance criteria for surrogate and/or IS recovery in an LFSM (LFSMD meets acceptance criteria for surrogate/IS recovery).	The LFSMD data should be substituted for the LFSM such that accuracy can be assessed.	p.33, 34

QC Type		Nonconformance****	Invalidated Data	UCMR 3 Lab Approval Requirements and Information Document 01/2012, v 2.0 / EEA UCMR3 QAPP
8.	Laboratory Fortified Sample Matrix (LFSM/ LFSMD) (cont'd)	Failure to meet acceptance criteria for surrogate and/or IS recovery in both LFSM and LFSMD.	Both LFSM and LFSMD cannot be reported, but surrogate and IS recovery failures of the LFSM and LFMSD do not invalidate the associated sample results if meeting the surrogate and IS QC criteria.	p.33, 34
		Failure to meet recovery of fortified target analytes or the precision (%RPD) between the LFSM and LFSMD.	Data should not be rejected (no target analyte recovery or % RPD acceptance criteria specified) if IS and surrogate standard recoveries are acceptable and all other batch QC are valid.	p.38
9.	Field Blanks (FBs)	Failure to analyze FB as required in Table 10 where there is a hit in the associated sample. (Table 10)	Reject all data for samples with hits for methods that require FB.	p.41 / Attachment B p.7
		Failure to meet FB acceptance criteria (no analyte >1/3 the MRL).	Reject all data for samples with hits collected with the failing field blank.	p.41
10.	Sample Shipment	Failure to chill samples during shipment at ≤ 10°C, not frozen, within the first 48 hours.	If samples are received >10˚C, samples are rejected and should not be analyzed.	p.42
		Failure to ship samples packed on ice or with frozen gel packs if samples arrive at the lab on the same day of collection (not stabilized at ≤10°C when arrived at the lab).	Reject samples.	p.42
		Failure to receive samples at ≤6°C, not frozen, after 48 hours of sample collection.	Reject samples.	p.43

QC Type	Nonconformance****	Invalidated Data	UCMR 3 Lab Approval Requirements and Information Document 01/2012, v 2.0 / EEA UCMR3 QAPP
11. Improper Sampling Container	Failure to collect sample with the proper container.	Reject samples.	p.45

*EEA guideline is to verify preservation within 2 days of sample receipt. Method 524.3 is an exception since immediately checking for preservation upon receipt would compromise the sample, so for this VOC method the checks must be performed after analysis to avoid loss of analytes. The pH for 522 samples must be checked by the lab within 48 hours of sample collection if pH needs to be adjusted in the lab.

**524.3 EEA internal pH acceptance criteria is pH < 2.5; pH acceptance criteria for 200.8 is pH <2; pH acceptance criteria for 218.7 is pH >8 (>7.8 for small system samples); pH acceptance criteria for 522 is pH <4; pH acceptance criteria for 537 is pH 6.5-7.5.

***There is no difference between the LFB and the CCC for EPA 218.7 and EPA 524.3 methods since these 2 methods utilize procedural calibration standards.

****See Attachment B for the tables from the UCMR 3 Lab Approval Requirements and Information Document v 2.0 that are mentioned in the Nonconformance column.

QUALITY MANUAL

Version 35

For
Eurofins Eaton Analytical, Inc.
750 Royal Oaks Drive
Suite 100
Monrovia, CA 91016
(626) 386-1100

QUALITY MANUAL

Prepared by and for
Eurofins Eaton Analytical, Inc.
750 Royal Oaks Drive
Suite 100
Monrovia, CA 91016
(626) 386-1100

Prepared by: Nilda B. Cox **Date** 7/11/2013
Nilda Cox
Quality Manager

Approved by: for CJ Belt **Date** 7/11/13
Carol J. Belt
Asbestos Technical Manager

Approved by: **Date** 7/11/13
Dr. Andrew Eaton
Technical Director

Approved by: **Date** 7/11/2013
Ed Wilson
Laboratory Director

Issue Date: 7/11/13

Effective Date: 7/25/13

NELAP ACCREDITED TNI LABORATORY
01114CA

EEA: Controlled Copy
15 of 15 Copies
Initials aie Date 10/25/13

Revised July 2013

TABLE OF CONTENTS

2.1 LIST OF FIGURES

2.2 LIST OF TABLES

2.3 LIST OF APPENDICES

3.0 STATEMENT OF POLICY

3.1. INTRODUCTION

Eurofins Eaton Analytical, Inc. (EEA) is a premier TNI-NELAC Approved Lab, full-service drinking water and wastewater laboratory that serves a national and international clientele. EEA is located at 750 Royal Oaks Drive, Suite 100, Monrovia, CA 91016 and is an entity that can be held legally responsible. EEA provides organic, inorganic, microbial, and radiochemical analyses in support of the Safe Drinking Water Act (SDWA), Clean Water Act (CWA), National Pollutant Discharge Elimination EEA Systems (NPDES), Resource Conservation and Recovery Act (RCRA), Food and Drug Administration (FDA), and the World Health Organization (WHO) as well as the EPA Unregulated Contaminant Monitoring Regulation (UCMR) Programs. The Quality Assurance Project Plan (QAPP) for the UCMR program is discussed in a separate document as an addendum to the laboratory's comprehensive Quality Manual (QM). The essential elements of the Quality Systems Program of EEA and the quality control procedures utilized by the laboratory to ensure compliance to the UCMR program requirements are discussed in the UCMR QAPP. UCMR QAPPs are developed for specific UCMR programs.

EEA takes an active role in supporting the promulgation of improved methodologies and the practice of differentiating laboratories based on quality of data. EEA participates in the methods development and validation of Standard Methods.

3.2. QUALITY POLICY

Management's commitment to quality and to the management system is stated in the Quality Policy below, which is upheld through the application of related policies and procedures described in EEA's *Quality Manual*, SOPs and policies.

The foundation of the quality policy lies in the involvement and continuous improvement activities of all aspects at EEA. A system of monitoring, auditing, and reviewing processes is used to bring to light the opportunities for improvement.

The quality policy is signed and dated, and is issued under the authority of the highest level of laboratory management, which demonstrates management's commitment to integrity, ethics, the quality system and associated standards.

Quality Policy Statement

The objective of the quality system and the commitment of management is to consistently provide our customers with data of known and documented quality that meets their requirements. EEA is committed to the production of quality analytical data. The methods by which this is ensured are: 1) meeting or exceeding method performance criteria, 2) providing deliverables to our clients in a timely manner and 3) fostering a spirit of continuous improvement in all areas of management and operations. Our policy is to use good professional practices, to maintain quality, to uphold the highest quality of service, and to comply with the TNI Standard and ISO 17025. The laboratory ensures the personnel are free from any commercial, financial, and other undue pressures, which might adversely affect the quality of work. This policy is implemented and enforced through unequivocal commitment of management, at all levels, to the Quality Assurance (QA) principles and practices outlined in this manual. However, the primary responsibility for quality rests with each individual within the laboratory organization. Every laboratory employee must ensure that the generation and reporting of quality analytical data is a fundamental priority. Every laboratory employee is required to familiarize themselves with the quality documentation and to implement the policies and procedures in their work. All employees are trained annually on ethical principles and procedures surrounding the data that is generated. The laboratory maintains a strict policy of client confidentiality.

Ed Wilson
Laboratory Director

This Quality Manual defines the performance criteria and support procedures by which quality analytical data are generated. Standard Operating Procedures (SOPs) for individual analytical methodologies supplement this Quality Manual. Together they provide the documentation framework for ensuring the generation of uniform, comparable and quality data over time.

The foundation of the quality policy is in the involvement and continuous improvement activities of all personnel at EEA. Opportunities for improvement are showcased with a system of monitoring, auditing, and reviewing processes. The spirit of innovation is encouraged and viewed as paramount to the continued success of the laboratory in serving its clients.

3.3. MISSION STATEMENT

EEA will contribute to global health and safety by providing customers with high quality laboratory and advisory services whilst creating opportunities for EEA employees and generating sustainable shareholder value. EEA will provide outstanding client service and data of known and documented quality to all clients at all times.

3.4. CODE OF ETHICS AND POLICY/DATA INTEGRITY PROCEDURES

EEA was a founding member (1989) of actLABS, the California Association of Testing Laboratories and drafted one of the first lab ethics policies for actLABS. actLABS subsequently became part of ACIL (American Council of Independent Labs). Beginning in 1997 our increased geographic client base required us to give up our actLABS membership.

As a former actLABS member and a current TNI (the NELAC Institute) accredited laboratory, EEA is committed to ensuring the integrity of generated data, meeting the quality needs of clients, and setting high quality and ethical standards in the environmental industry. EEA is committed to managing its businesses by agreeing to:

- Produce results that are accurate and include QA/QC information which meets client predefined Data Quality Objectives.
- 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.
- Operate the laboratory to ensure its personnel are free from any commercial, financial and other undue pressure that might adversely affect the quality of the work.
- 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.

In addition any employee of EEA identified as not conforming to the code of ethics of the laboratory, committing fraud or improper data manipulation, falsifying data, or deviating from the contractual requirements will be subject to disciplinary procedures, including suspension and up to termination of employment. Any supervisor or employee applying undue pressure to another coworker that might adversely affect the quality of the work (TNI-EL-V1M2- 2009-4.1.5.b) (ISO/IEC 17025:2005(E)-4.1.5.b) will be subject to the same disciplinary procedures outlined above.

In order to meet the requirements of the TNI data integrity program (TNI-EL-V1M2-2009-5.2.7), the laboratory implements a proactive program for the prevention and detection of improper, unethical or illegal action. This program includes training courses on Laboratory Ethics and Data Integrity Procedures, and educating all personnel on questionable practices. Details of the Laboratory Ethics and Data Integrity Procedures are found in the laboratory SOP. The laboratory SOP includes the implementation of Data Integrity Procedures including:

- Management Responsibilities on Data Integrity Procedures/Signed Contract/Ethics Agreement for all laboratory personnel [TNI-EL-V1M2-2009-4.2.8.1, 4.2.8.1a, 4.2.8.1b].
- Control and documentation – Internal Audit/Periodic Monitoring of Data Integrity/Evidence of Vulnerabilities [TNI-EL-V1M2-2009-4.14.2, 4.14.3][ISO/IEC 17025:2005(E)- 4.14.2, 4.14.3].
- Data Integrity Training and documentation of Examples of Improper Practices in the Laboratory Ethics SOP [TNI-EL-V1M2-2009-5.2.7].

3.5. SERVICE TO THE CLIENT

The laboratory collaborates with clients and/or their representatives in clarifying their request and in monitoring of the laboratory performance related to their work. Each request is reviewed to determine the nature of the request and the laboratory's ability to comply with the request within the confines of prevailing statues and/or regulations without risk to the confidentiality of other clients.

3.5.1. Client Confidentiality

EEA recognizes its clients to be its contractors, the regulatory community, and the general public. The day to day operations are defined with considerations to the needs, goals and health of all clients. Protection of clients' confidential information and proprietary rights are considered. Where data are provided for external audits or for other similar reasons, the client's name and identity are concealed as necessary to protect client-confidential information.

In the event that the laboratory transfers ownership or goes out of business, the laboratory will notify all clients to ensure that records are maintained or transferred according to the client's instructions [TNI-EL-V1M2-2009-4.13.3.h].

3.6. REVIEW OF REQUESTS AND CONTRACTS/CONTRACT AMENDMENTS

EEA agrees to assert competency only for work for which adequate preparation has been made. Before commencing new work, the laboratory reviews all new work to ensure that it has the appropriate capability, facilities, resources, and the test method is applicable to the customer's needs. This process assures that all work will be given adequate attention without shortcuts that may compromise data quality.

A contract may be any written or oral agreement to provide a client with environmental testing. The laboratory reviews contracts and informs clients if there are any potential conflicts, deficiencies, lack of accreditations or inability to complete client work. The review also covers any work that will be subcontracted by the laboratory.

3.6.1. Procedure for the Review of Work Requests

3.6.1.1. Requests, tenders and contracts received by the laboratory are reviewed to ensure that the laboratory has the necessary personnel, information resources, facilities, equipment, PT, MDLs, QC and current applicable accreditation status [TNI-EL-V1M2-2009-4.4.1][ISO/IEC 17025:2005(E)-4.4.1].

3.6.1.2. For new clients and comprehensive testing, contracts are generated and appropriate lab personnel, such as the Lab Director or Technical Director, review the Contracts to assure that the lab is capable of providing testing prior to the start of work [TNI-EL-V1M2-2009-4.4.1][ISO/IEC 17025:2005(E)-4.4.1].

3.6.1.3. For repetitive, routine tasks the review needs to be made only at the initial inquiry stage or on granting of the contract for ongoing routine work performed under a general agreement with the client, provided that the client's requirements remain unchanged.

3.6.1.4. For any contract amendment for TNI compliance, the laboratory repeats the review process. The client is informed of any deviation from the contract including the test method or sample handling processes. If a contract needs to be amended after work has commenced, the same contract review process is reviewed and amendments are communicated to all affected personnel. If the laboratory's accreditation is suspended, revoked, or voluntarily withdrawn, the laboratory reports to clients any applicable changes of its accreditation status.

3.6.1.5. The designated Project Manager (PM) reviews client samples received by the laboratory and logged in the LIMS. Review of logged tests and methods are documented in the Sample Acknowledgement Report by affixing the PM's signature and/or initials and date of review. A Sample Acknowledgement Report is sent to the client to document approval of LOGGED samples and methods of analysis.

3.6.1.6. Refer to the Nonmethod 26 SOP for detailed Contract Review procedures.

3.6.2. **Documentation of Review**

3.6.2.1. Records of reviews, including any significant changes, shall be maintained. Records shall also be 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.

3.7. EEA STANDARD POLICY ON RESOLUTION OF COMPLAINTS

3.7.1. EEA reviews all complaints and determines appropriate action.

3.7.2. EEA will, if it is feasible and within holding times, arrange for repeat of all analyses that do not meet regulatory requirements. We hold ourselves responsible for reporting or re-reporting all results in a format that complies with regulatory requirements, and will

make every attempt to correct and when feasible will repeat work at no additional charge for all analyses compromised due to laboratory error in shipping, sample preparation, or analysis. In the event of a sample loss within the required sample collection window, we will discuss with clients the merits of available options for flagging data versus re-sampling for either the individual parameter or the entire suite of samples. In all circumstances, EEA will keep clients completely informed and aware of potential or actual problems as they arise, using e-mail or telephone.

3.7.3. Where a complaint or any other circumstance raises doubt concerning compliance with the laboratory's policies, with the requirement of the TNI and ISO 17025 Standards or otherwise concerning the quality of the laboratory's data, the EEA Quality Assurance Department will conduct an audit of the affected areas of activity.

3.7.4. Documentation of the complaints or initiating event, internal audit findings and resulting corrective action will be maintained by the EEA Quality Assurance Department (TNI-EL-V1M2-2009-4.11.3) (ISO/IEC 17025:2005(E)-4.11.3) and as appropriate be conveyed to the client.

3.8. CAPABILITIES

EEA has the capability to analyze drinking water and wastewater for clients in the private and public sector where work is dictated by the regulatory requirements for the Safe Drinking Water Act (SDWA), Resource Conservation and Recovery Act (RCRA), National Pollutant Discharge Elimination Systems (NPDES), Clean Water Act (CWA), Food and Drug Administration (FDA), World Health Organization (WHO) and the Superfund Amendments and Reauthorization Act (SARA) and the EPA Unregulated Contaminants Monitoring Regulations (UCMR) Program. Our specialized laboratory services include;

- Analysis and identification of inorganic & organic disinfection by-products, taste and odor compounds in drinking water
- Identification and quantitation of coliphage in drinking water and wastewater
- Comparability of alternate test procedures for drinking water and wastewater analysis.
- Analysis of emerging contaminants such as Pharmaceuticals and Personal Care Products (PPCPs), Endocrine Disrupter Compounds (EDCs), and perfluoro octanesulfonate (PFOS).
- Analysis of bottled water and beverage matrices for FDA and WHO regulated analyses.

3.9. CERTIFICATIONS

EEA is currently certified in 45 states and 2 territories to perform various analyses for regulated parameters. EEA is also NELAP accredited in 13 TNI states out of 14 TNI

States' accrediting bodies. EEA holds primary accreditation under California NELAP (01114 CA) and ELAP Program (Certificate No. 1422). Please refer to Table 3-1 for the list of the states, laboratory identification number, and the certification type. An updated list is available in the QA office.

A copy of EEA's NELAP Accreditation plus NELAP fields of accreditation (Fig. 4-3, Fig. 4-4) and a copy of the CA ELAP plus Fields of Testing are attached (Figures 4-5 and 14-6). The most recent certification is available in the QA office.

Arizona Dept of Health Services requires that a copy of EEA's AZ certification and License (AZ0778) be attached in the Lab QM. See the AZ License and list of license parameters in Appendix I. The most recent certification is available in the QA office.

Table 3-1 State Certifications

State Certification List

Item #	State	Lab ID	Drinking Water	Wastewater	Hazardous Waste
1.	Alabama	41060	X		
2.	Alaska	CA0006	X		
3.	Arizona	AZ0778	X	X	X
4	Arkansas		X		
5.	California – NELAP	01114CA	X	X	
	California – ELAP (Monrovia)	1422	X	X	X
	California – ELAP (Colton)	2641	X	X	
	California – ELAP (Folsom)	2820	X		
6.	Colorado		X		
7.	Commonwealth of Northern Mariana Islands	MP0004	X		
8.	Connecticut	PH-0107	X		
9.	Delaware	CA 006	X		
10.	Florida – NELAP	E871024	X	X	
11.	Georgia	947	X		
12.	Guam	12-006r	X		
13.	Hawaii		X		
14.	Idaho		X		
15.	Illinois – NELAP	200033	X		
16.	Indiana	C-CA-01	X		
	Iowa – Not Certified	Lab has not applied	NO		
17.	Kansas – NELAP	E-10268	X		
18.	Kentucky	90107	X		
19.	Louisiana – NELAP	LA130008	X		
20.	Maine	CA0006	X		
21	Maryland	224	X		
22.	Massachusetts	M-CA006	X		
23.	Michigan	9906	X		
	Minnesota – Not Certified - NELAP	Lab has not applied	NO		
24.	Mississippi		X		
	Missouri – Not Certified	Lab has not applied	NO		
25.	Montana (Chemistry)	Cert. 0035	X		
26.	Nebraska		X		
27.	Nevada	CA-06-2012	X	X	X
28.	New Mexico		X		
29.	New Hampshire – NELAP	2959	X		
30.	New Jersey – NELAP	CA 008	X	X	
31.	New York – NELAP	11320	X	X	
32.	North Carolina	06701	X		

Item #	State	Lab ID	Drinking Water	Wastewater	Hazardous Waste
33.	North Dakota	R-009	X		
	Ohio – Not Certified	Certifies only in-state labs (No certification program for out-of-state labs)	NO		
	Oklahoma – Not Certified	Lab has not applied	NO		
34.	Oregon – NELAP	CA 200003	X		
35.	Pennsylvania – NELAP	68-565	X		
36.	Rhode Island	LAO00326	X		
37.	South Carolina	87016001	X		
38.	South Dakota		X		
39.	Tennessee	TN02839	X		
40.	Texas – NELAP	TX104704230	X		
41.	Utah - NELAP	MONT-1	X		
42.	Vermont	VT0114	X		
43.	Virginia - NELAP	00210	X		
44.	West Virginia	9943C	X		
45.	Washington	C 838	X		
46.	Wisconsin	998316660	X		
47.	Wyoming	8TMS-Q	X		

EEA may accept, analyze, and report results for samples from states in which it is not certified if the results are intended for non-regulatory monitoring.

When there is a change in lab location or ownership, the laboratory will report in writing to the accrediting authorities within 30 calendar days of the change.

3.10. SUBCONTRACTED LABORATORY WORK

3.10.1. On occasion laboratory work may be subcontracted to certified laboratories approved by EEA. The subcontractor laboratory will be approved only if the laboratory meets all the necessary certification requirements required by the state where the samples are collected. For example, samples collected from Alaskan Public Water supplies for compliance monitoring must be analyzed by a laboratory certified by the State of Alaska or the USEPA (18 AAC 80.255). For any part of testing covered under NELAP, the laboratory sends the work to a subcontractor accredited under NELAP or to a laboratory that meets applicable satisfactory and regulatory requirements for performing the test and submitting the results of the tests performed [TNI-EL-V1M2-2009-4.5.1][ISO/IEC 17025:2005(E)-4.5.1]. For ISO 17025 subcontracted work, EEA subcontracts work to an ISO 17025 subcontractor or qualified non ISO 17025 accredited subcontractor. Refer

to the Nonmethod 31 SOP for the requirements for Non ISO 17025 accredited subcontractors.

3.10.2. Under no circumstances will work be subcontracted without client approval. The laboratory advises the client in writing of its intention to sub-contract any portion of the testing to another laboratory during the project bid proposal or purchase order procurement [TNI-EL-V1M2-2009-4.5.2][ISO/IEC 17025:2005(E)-4.5.2]. Test results provided by the subcontractor are identified by the subcontractor name or applicable accreditation number. The subcontractor shall report the results in writing or electronically [TNI-EL-V1M2-2009-5.10.6] [ISO/IEC 17025:2005(E)-5.10.6]. The laboratory shall make a copy of the subcontractor's report available to the client when requested by the client or when required by regulations.

3.10.3. Subcontracted work is documented in the chain of custody (COC). The COC and other appropriate records are included with the final data package as part of the final deliverables. To comply with California ELAP regulations (Title 22, Division 4, Chapter 19, Article 10, Section 64819), EEA's reports must include the original copies of reports prepared by the subcontracted laboratories. See section 14.4 for all the information required in the final test report.

3.10.4. To help ensure all subcontractors meet EEA's Data Quality Objectives and consistently produce documented data of known quality, EEA requires that the following documentation should be submitted by the vendor for review:

(1) NELAP laboratory accreditation, or state certifications that meet the applicable statutory and regulatory requirements
(2) Laboratory Quality Manual(QM) or at the minimum the signed cover page and table of contents of the lab QM (non-NELAP accredited facilities only)
(3) Notify EEA of 2 failed Proficiency Testing (PT) results or any changes of certification status either suspension or revocation for any relevant tests, if applicable.
(4) Recent state onsite audit findings for the relevant methods and corrective action report, if applicable
(5) For Non-NELAP lab, a copy of Data Integrity/Ethics Policy, if available

3.10.5. At a minimum, the lab's NELAP accreditation or states certification status and the signed cover page and table of contents of the lab QM is verified.

3.10.6. Data deliverables must meet EEA's project needs and requirements. EEA assumes responsibility to the client for the subcontractor's work, except in the case where a client or a regulating authority specified which subcontractor is to be used [TNI-EL-V1M2-2009-4.5.3][ISO/IEC 17025:2005(E)-4.5.3]. At a minimum, laboratory deliverables submitted to EEA must include final report, QC results and acceptance limits. Level 4 data deliverables may be requested by EEA for review as needed. Onsite audit of subcontract laboratory may also be conducted by EEA as needed.

3.10.7. Project managers and the designated subcontracting administrator must ensure all applicable quality documents specified in section 3.10.4 to evaluate subcontractor's qualifications are submitted to EEA for review by the subcontracting administrator. The subcontracting administrator will ensure that all approved subcontract labs and EEA's representatives have signed the required Subcontract Vendor Agreement/checklist. Before subcontracting samples, the designated subcontracting administrator shall review certifications to ensure that the laboratory's subcontractor's certification/ accreditation is current. If certification is not current, the subcontracting administrator shall contact the vendor for a current copy of the vendor's certification before shipping samples.

3.10.8. A register of all subcontractors and a record of evidence (such as NELAP accreditation or appropriate compliance to applicable regulatory requirements) are kept by the designated subcontracting administrator [TNI-EL-V1M2-2009-4.5.4][(ISO/IEC 17025:2005(E)-4.5.4]. A list of subcontracted laboratories approved by EEA is available in the server.

3.10.9. For samples originating in Massachusetts and subcontracted to another lab, EEA must identify, in writing, those samples needing special reports (e.g. MCL exceedance). The subcontract laboratory is responsible for notifying EEA and Massachusetts DEP of any MCL exceedances within 24 hours of obtaining valid data.

3.10.10. Refer to the Nonmethod 31 SOP for detailed Subcontracting procedures.

3.11. FACILITIES

3.11.1. ACCOMODATIONS

EEA's main laboratory is located at 750 Royal Oaks Drive, Suite 100 in Monrovia, California. It has more than 20,000 square feet of analytical laboratory workspace plus almost 15,000 square feet of support space with a staff of 109 in 2011. Figure 3-1 and Figure 3-2 contain the Floor Plans for the first and second floors, respectively of the Monrovia facility.

The Monrovia facility is controlled by access control locks which provide entry through plastic keycards stored with digital signatures of each employee.

Departments of the Main Laboratory include:

Asbestos
GC extractables/volatiles
GC/MS extractables/volatiles
Ion Chromatography
LC/MS/MS Extractables
Metals/Metals Prep

Microbiology
Organic extractions
Radiochemistry
Sample Disposal
Sample Receipt
Shipping – sample bottle preparation
Wet Chemistry (including General Physical)

In addition to the Monrovia facility, there are three service centers that are a part of the laboratory.

- The Inland Empire/Microbiology Lab located at 1012 E. Cooley Dr., Ste P, Colton, California, 92324;
- The Southwest Center located at 15953 N. Greenway Hayden Loop, Ste. C, Scottsdale, Arizona 85260;
- The Northern California Center is located at 180 Blue Ravine Road, Suite A & B, Folsom, CA 95630.

The management systems that are compliant with TNI Standards and ISO 17025 that are documented in the laboratory Quality Manual covers work carried out in the Monrovia facility and in-house sampling procedures associated with field activities.

3.11.2. ENVIRONMENTAL CONDITIONS

3.11.2.1. The laboratory ensures that the laboratory environment conditions do not invalidate the results or adversely affect the required quality of any measurement.

3.11.2.2. The laboratory monitors, controls and records environmental conditions as required by the relevant specifications, methods and procedures, or where they influence the quality of the results.

3.11.2.3. Biological sterility and dust are monitored in microbiology to ensure that environmental conditions do not jeopardize the results of the environmental tests and/or calibrations. The laboratory micro walls, floors, work surfaces are non-absorbent and easy to clean and disinfect.

3.11.2.4. Incompatible areas such as Volatiles, Sample Extraction, Microbiology, culture handling or incubation, Radiochemistry preparation areas are separated to prevent cross-contamination.

3.11.2.5. The laboratory work spaces are adequate, and appropriately clean to support environmental testing and ensure an unencumbered work area.

Figure 3-1 Floor Plan First Floor

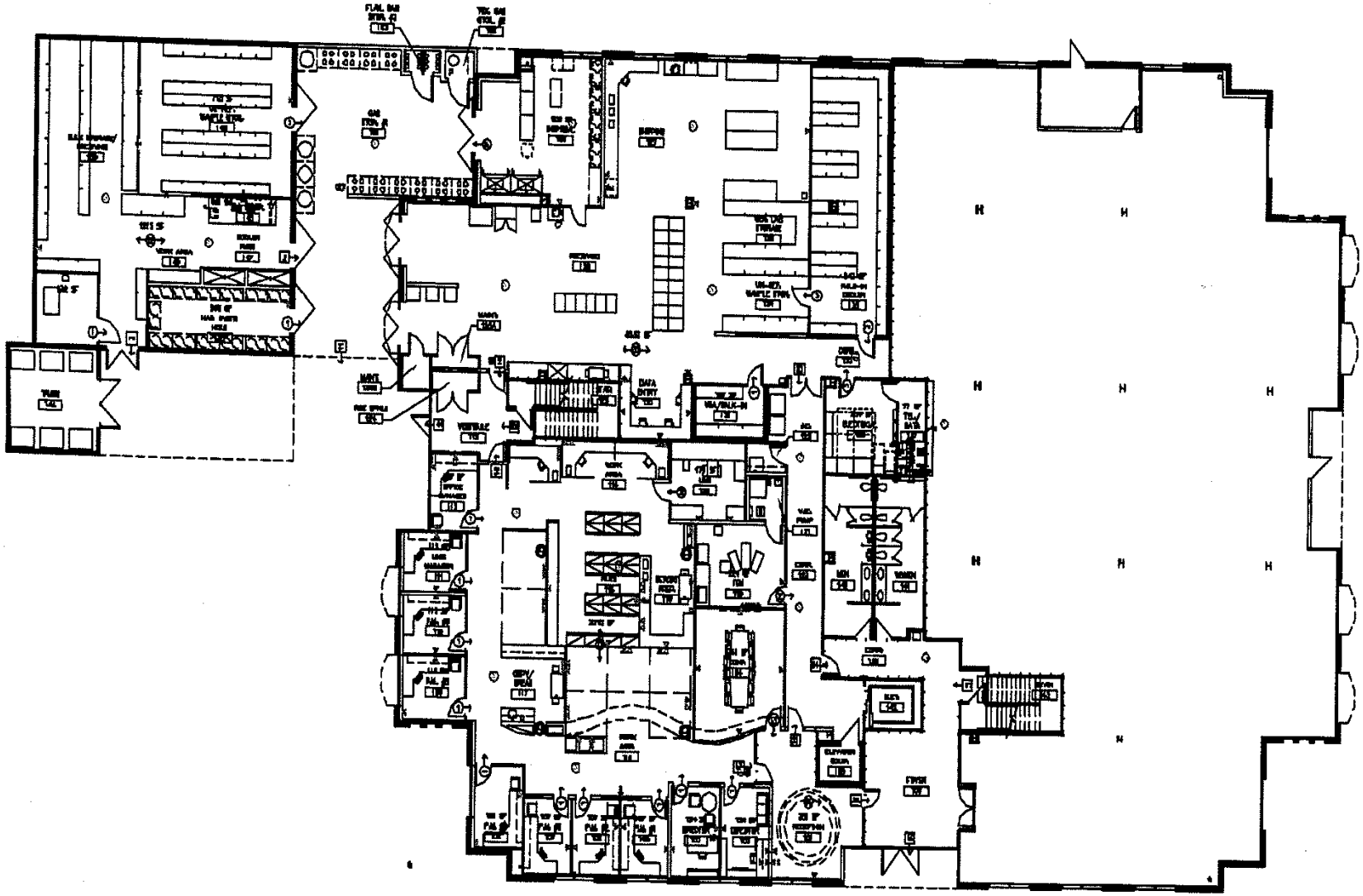
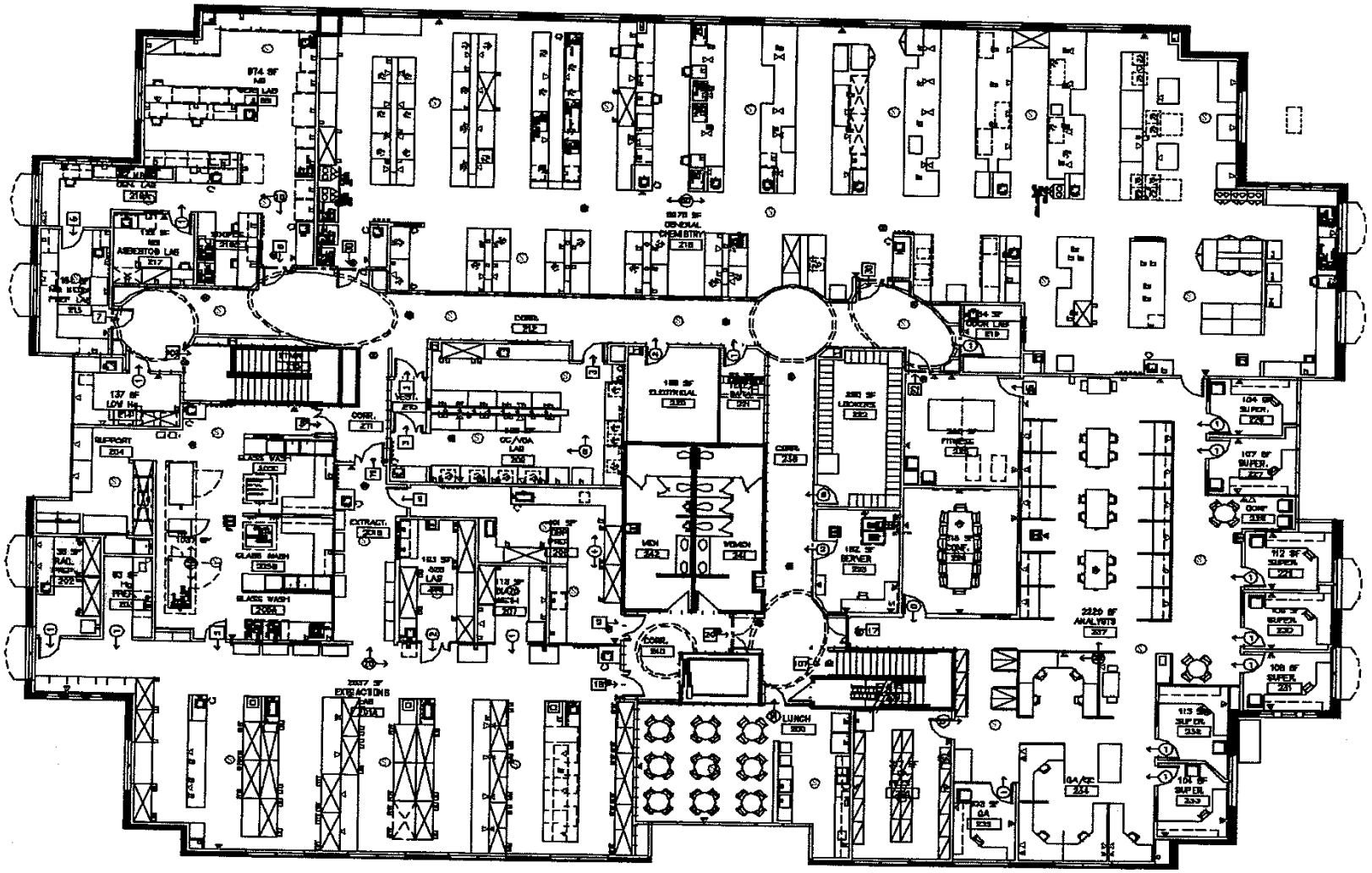


Figure 3-2 Floor Plan 2nd Floor



4.0 PROGRAM ORGANIZATION AND MANAGEMENT

All EEA's analysts and technicians analyzing drinking water samples meet the minimum qualifications specified in the Manual for the Certification of Laboratories Analyzing Drinking Water, Criteria and Procedures, Quality Assurance, 5th Edition. The organization and chain of command for the laboratory is shown in Figure 4-9. Details of assigned positions, responsibilities and qualifications for senior management personnel are summarized below. The laboratory is organized in such a way that managerial staff has the authority and resources needed to discharge their duties. Top Management is the Laboratory Director who makes decisions on policy, endorses the quality policy statement, and allocates resources to implement and maintain the quality system. The Technical Director, Quality Manager, Technical Managers, and deputies are part of the management staff. The Quality Manager reports directly to the EEA Laboratory Director and has the authority to make independent technical judgment not influenced by production, marketing and financing issues. Qualified supervisors are certified as to their educational and technical background and experience, to ensure that supervision is provided by persons familiar with the calibration or test methods and procedures, the objective of the calibration or test and the assessment of the results.

4.1. EEA'S LABORATORIES PERSONNEL

4.1.1. Laboratory Director: Mr. Ed Wilson

Mr. Wilson has over 35 years of environmental chemistry and laboratory management experience to the laboratory. He sets laboratory policy and is responsible for overall laboratory performance and direction. In his role as Lab Director, he has ultimate responsibility for ensuring the operational efficiency and accuracy of laboratory procedures, cost analysis, overhead control, marketing, and project management. His guided management principles are based on achieving outstanding Customer Service and Technical Excellence. Under his direction and leadership, EEA would have systems built on the most sophisticated information technology platform and would be proud to have the best technical staff in the industry.

4.1.2. Technical Director: Dr. Andrew Eaton

Dr. Andrew Eaton has over 30 years of analytical experience including over 20 years of managerial experience. In his capacity as Technical Director, Dr. Eaton certifies that personnel with appropriate educational and/or technical background perform all tests for which the laboratory is accredited. Such certification for each personnel is documented in the analyst's initial demonstration of capability (DOC) certification. The initial DOC certification statement was modified to include the certification for the analyst for having the appropriate educational and/or technical background. A copy of the certification statement is retained in the training files of each affected employee. Dr. Eaton is responsible for Project Management on large projects with significant technical issues, serves as a technical advisor to the laboratory staff and clients, works on special assignments such as productivity assessments and financial analyses, as well as marketing activities with clients whose projects are highly technical in nature. Dr. Eaton also serves as a member of the Joint Editorial Board for Standard Methods for the

Examination of Water and Wastewater (SM). In this capacity, he is responsible for recommending new methods for inclusion in SM and ensuring that all proposed methods include appropriate levels of QC and validation. He is a former member of the TNI Advocacy Committee. Formerly on the Board of actLABS, Dr. Eaton also served as a member of The Methods and Data Comparability Board, which reports to the National Water Quality Monitoring Council.

4.1.3. **Asbestos Technical Manager: Carol J. Belt**

Ms. Belt has over 30 years of environmental laboratory experience in EEA conducting microbiology and asbestos analyses. Her expertise includes analysis of drinking water and wastewater samples for microbiological testing and asbestos analysis. She is responsible for training analysts in various microbiological procedures and in the analytical method for the determination of asbestos fibers in water. As the Technical Manager for Asbestos analysis, Ms. Belt has the overall responsibility for the technical operation of the asbestos testing in the laboratory and currently oversees all aspects of the asbestos testing She is responsible for monitoring the performance of the entire procedure and accurate reporting of all samples received for asbestos analysis. She is also responsible to train other technicians on this methodology and to certify trained analysts as to their educational and technical background and demonstration of capability.

4.1.4. **Client Services Manager: Frederick Haley**

Mr. Haley has over 27 years of environmental laboratory experience. His experience has encompassed analytical method development for soils and water as well as development of mobile laboratory services. Mr. Haley has managed lab operations for both small and large lab settings. He oversaw the daily operations of a small satellite laboratory of 10 staff performing basic analytical methods and has also managed a large laboratory of over 150 personnel conducting wastewater, soil, hazardous waste and drinking water analysis. In addition Mr. Haley has managed projects requiring coordination of schedule, personnel, budget and compliance to technical specifications for local, state and federal agencies as well as private sector companies. Mr. Haley is responsible for the daily supervision of 7 project managers.

4.1.5. **Quality Manager/Regulatory Consulting Manager: Ms. Nilda B. Cox**

Ms. Cox has over 20 years of environmental experience in Quality Assurance/Quality Control including hazardous waste management and safety compliance in the laboratory. Her experience also includes eight years as senior chemist and supervisor of chemistry QA/QC Methods Development Group, and in-charge of the Industrial Hygiene Monitoring Program for a medical device company. Additional experience includes six years in Research and Development in the field of agriculture. Ms. Cox is responsible for providing QA solutions to our clients.

In addition to supporting internal QA/QC, Ms. Cox serves as a resource for numerous outside entities, providing consulting services in the area of QA/QC to assist them in the development of their own in-house QA Programs.

In the absence of the Quality Manager, Robert Dean and Yoon Cha are authorized as the Deputy Quality Managers.

4.1.6. **Senior Project Manager/Deputy Technical Director: Linda Geddes**

Ms. Geddes has over 30 years experience in the field of analytical chemistry related to environmental issues, including three years as the Quality Assurance Manager at another laboratory, over five years of experience in pharmaceutical chemistry, and 2 years as QA/QC Officer for EEA. Her experience has encompassed analytical methods development and validation for soils, sediments and water, maintaining a quality assurance program and managing Department of Defense site assessment projects. These projects have required coordination of schedule, personnel, budget, and compliance to technical specifications for local, state, and federal agencies, as well as private sector companies. These included compliance monitoring under the Coliform Rule, the Lead and Copper Rule, Phase II and V, the Information Collection Rule (ICR), and the Unregulated Contaminant Monitoring Rule (UCMR). Prior to becoming the Quality Assurance Officer, Ms. Geddes was a Project Manager at EEA for eight years.

In the absence of the Technical Director, Ms. Geddes is designated as the Deputy Technical Director.

4.1.7. **Technical Manager/LCMS Supervisor: Mr. Ali Haghani**

As EEA's Technical Manager/LCS Supervisor, Mr. Haghani is responsible for method development of new methods and for asset management and currently supervising 4 analysts. Mr. Haghani was previously responsible for overseeing six supervisors and a staff of over 50 analysts performing sample preparation and analysis of environmental samples for organics and a wide range of inorganic parameters. He was also responsible for the day-to-day scheduling of analysts workloads, providing guidance and technical expertise to the analyst, and checking the validity of their work. Mr. Haghani has over 17 years of experience in the environmental monitoring business and has technical expertise in inorganic and organic analytical chemistry.

4.1.8. **Technical Manager/Extraction and GC/MS Supervisor: Mr. Charles Grady**

Mr. Grady has over 20 years experience in environmental extraction, environmental wet chemistry, environmental GC and environmental GC/MS. He also has experience in hazardous waste, drinking water and waste water testing. Mr. Grady also has two years of experience as an instrument repair service technician.

As Technical Manager/Extraction and GC/MS Supervisor for EEA, Mr. Grady is responsible for supervising 18 analysts, meeting quality control and method requirements, scheduling work,

recruiting and training staff, and managing the group budget. He works closely with Client Services, the Lab Directors and department managers to schedule incoming work and to meet QC requirements and specific client needs.

4.1.9. **Technical Manager/GC/HPLC Supervisor: Ms. Sophia Liang**

As EEA's Technical Manager or GC/HPLC supervisor, Ms. Liang is responsible for day to day supervision of a staff of 8 analysts performing organic analysis by GC and HPLC (High Performance Liquid Chromatography). Ms. Liang schedules analysts' workloads to ensure that holding times are not exceeded, approves final data, and insures that all QA guidelines are met. Ms. Liang has over 8 years of experience performing organic analyses.

4.1.10. **Technical Manager/Inorganic Supervisor: Mr. Walter Hsieh**

As EEA's Metals/Radiochemistry/Wet Chemistry supervisor, Mr. Hsieh is responsible for day to day supervision of a staff of 24 analysts performing inorganic analyses such as metals, radiochemistry and wet chemistry. Mr. Hsieh schedules analysts' workloads to ensure that holding times are not exceeded, approves final data, and insures that all QA guidelines are met. Mr. Hsieh has over 20 years experience performing metal and organic analyses in environmental laboratories.

4.1.11. **Technical Manager/Microbiology Supervisor: Ms. Polly Barrowman**

Ms. Barrowman has over 5 years of microbiology and biology experience. She obtained her BS in Biology and Chemistry at Western Michigan University in 2003 and her MS in Environmental Biology at University of Aberdeen, Scotland in 2005. She has been a Microbiologist at EEA's since June 2009, with experience performing water suitability, inhibitory residues, standard plate counts, and coliform analyses. Ms. Barrowman ensures that all holding times are not exceeded and that all QA guidelines are met. Ms. Barrowman is responsible for the daily supervision of a staff of 9 laboratory personnel.

4.1.12. **LIMS Manager: Mr. Ryan Chang**

Mr. Chang has over 15 years of IT experience including technical support for several nationally syndicated radio shows, website implementation for several fortune 1000 companies, intra and internet e-commerce development and was a webmaster for UCLA Business Law Courses. He is skilled in multiple server-side, client-side, database, web server, app-server and e-commerce languages. He holds a Bachelor's of Science in Computer Science and Engineering and a Bachelor of Arts in Economics both, from UCLA.

4.1.13. **Deputy Lab Director: Mr. James Hein**

Mr. Hein has over 30 years of environmental laboratory experience. His experience has encompassed analytical methods development for soils, sediments and water, the development of data assessment procedures for validation of analytical data, and the implementation of numerous bench scale treatment studies for the removal of various environmental pollutants.

He has managed projects requiring coordination of schedule, personnel, budget and compliance to technical specifications for local, state and federal agencies as well as private sector companies. In the absence of the Lab Director, Mr. Hein is designated as the Deputy Lab Director.

4.2. QUALITY SYSTEMS PROGRAM AND ITS MANAGEMENT

The Quality Systems Program is dynamic and is updated frequently when changes to policy and procedures are necessary. The Quality Manager has direct access to the highest level of management, which is the Laboratory Director, where decisions are made on laboratory policy or resources [TNI-EL-V1M2- 2009-4.1.7.1]. It is the responsibility of the Quality Manager to oversee all aspects of this program and document the participation of all staff members. In order to administer and manage this program, the Quality Manager must be knowledgeable in the TNI Quality Systems and ISO 17025 Current Standards and their implementation [TNI-EL-V1M2- 2009-4.1.7.1, 4.2.8.2]. Attendance at the TNI Interim and annual Conferences should be documented in the training files of the Quality Manager.

Vital areas of the Quality Systems Program include:

4.2.1. Preparing annual reports to management on QA related activities in the laboratory. Through the annual report, the Quality Manager notifies the laboratory management of deficiencies in the Quality Systems and monitors corrective actions. (Section 16.4) This includes a periodic QA report, reports on internal and external PT samples, and verbal transmittal of QA information to the Laboratory Director and group supervisors during a weekly staff meeting.

4.2.2. Coordinating analyses of Proficiency Testing (PT) (i.e. water supply study-WS, water pollution study-WP) or blind performance samples; investigating any problems associated with the results; reviewing results, problems and corrective actions with the analytical and supervisory staff; providing timely response to certification authorities with respect to any identified problem areas. (Section 16.3)

4.2.3. Implementing procedures that allow for adequate documentation and control of specific documents. These procedures use a unique identification system that allows for tracking and traceability of official copies and the time period the procedure or document was in force. To ensure that the Quality Manual and SOPs remain controlled documents, the master SOPs and Quality Manual (original official version of the SOP and Quality Manual) and copies of the SOP and Quality Manual will be identified. The cover page of each copy will contain a unique identification indicating that the document is controlled copy ___ of ____ copies, initialed and dated by the Quality Manager (or designee) in red ink. This ensures that the analyst is using the current version. Refer to the Nonmethod 25 SOP for detailed Document Control procedures.

4.2.3.1. The Quality Manual and Standard Operating Procedures (SOP) of EEA are reviewed and updated if needed at least once a year to ensure continuing suitability and compliance with applicable requirements. The laboratory's document control system allows for the

amendment of documents by hand, pending the reissue of the documents. The changes are clearly marked, initialed and dated by the personnel that performed the original review. The revised document formerly reissued as soon as practicable [TNI-EL-V1M2- 2009-4.3.3.3, 4.3.3.1][ISO/IEC 17025:2005(E)-4.3.3.3, 4.3.3.1]. All appropriate laboratory personnel signs the Quality Manual Signature Page / SOP Training Documentation Form after the annual review of the Quality Manual / SOPs.

4.2.3.2. See Figure 4-1 QM Signature Page for a copy of the QM Signature Page. See Figure 4-2 SOP/Method Training Documentation for a copy of the SOP Training Documentation Form. See Table 4-1 for a list of SOPs.

4.2.3.3. A SOP/ QM Distribution Form is prepared for each SOP/ QM that includes the SOP/QM ID, control number, individual receiving the SOP/QM, date of issue and the date of completion of the analyst's SOP/QM training documentation.

4.2.4. Documenting participation and performance of the laboratory staff in initial and continuing training courses.

4.2.5. Overseeing and maintaining the training program files for each analyst at EEA.

4.2.6. Providing guidelines for the QS orientation program to newly hired personnel and ensuring that they are familiar with the quality systems program operating within the laboratory.

4.2.7. Interacting with auditors and certifying authorities for in-state programs, out-of-state programs, and internally to the laboratory. (16.2)

4.2.8. Serving as focal point for initiation, implementation, review and dissemination of QA/QC Guidelines to ensure that data quality meets the objectives of certifying authorities and maintaining documentation of those guidelines.

4.2.9. Maintaining copies of procedural write-ups and QA documentation files, and ensuring that all personnel working in the laboratory follow established standard operating procedures that do not compromise the quality of data submitted to clients or violate rules and guidelines from certifying agencies.

4.2.10. Ensuring that analysts are monitoring long-term quality control trends with quality control charts and insuring that corrective action is initiated whenever an out of control event occurs.

4.2.11. Ensuring that sample log-in and traceability are done correctly and that the chain of custody forms and other relevant documentation are properly maintained by periodic spot checks of the records.

4.2.12. Implementing a record management/archival system for control of laboratory notebooks; instrument logbooks; standard logbooks; records for data reduction, validation, storage, and reporting; training records for personnel no longer with the laboratory; outdated manuals and

SOPs; and the eventual removal of outdated documentation. Archived information is stored physically or electronically in-house for 3 months and then physical files are transferred off-site, for storage for 2 years for Arizona or 3 years for Wisconsin. Electronically scanned files are stored for 5 years as per NELAP, and additional 5 years as per Massachusetts, Hawaii and New York. All hard copies and electronic files for Asbestos test method are stored for 30 years.

4.2.13. Maintain a log of names, initials and signatures for all individuals responsible for signing or initialing any laboratory records is maintained by the QA group.

4.2.14. Writing or reviewing QA project specific plans.

4.2.15. Providing the staff with quality assurance information and updates.

4.2.16. Ensuring that all laboratory procedures currently in use are acceptable and will not compromise quality.

4.2.17. Where QA oversight is needed, the Quality Manager (or designee) functions independently from the laboratory operations. The Quality Manager evaluates data objectively and performs assessments without managerial influence. The Quality Manager may enlist the aid of various supervisors of the analytical groups in order to achieve these objectives. The Quality Manager and/or a designee should perform periodic audits of laboratory data or procedures to ensure that QA objectives are being met. The Quality Manager or designee must have a general knowledge of the analytical test methods for which the data review is performed and will arrange for or conduct annual internal audits per TNI-EL-V1M2-2009-4.1.7.1.e and 4.1.7.1.f.

4.2.17.1. Maintaining current certifications, licenses and accreditation materials. See section 3.9 for more information about certification.

4.3. STAFF RESPONSIBILITY

A comprehensive Quality Systems Program requires the involvement of all laboratory personnel. The level of involvement for each staff member is dependent upon his or her assignment within the laboratory. Laboratory analysts are responsible for quality control parameters that are done at the time of analysis. Laboratory management is responsible for monitoring and evaluating the results of the quality control procedures performed by the analysts.

The minimum level for qualifications, experience, and skills necessary for each position varies by job position. A list for each position is available in QA for review. The laboratory follows minimum requirements as per the EPA Drinking Water Manual and TNI Standards.

4.3.1. Initial Training

4.3.1.1. The objective for data generated by EEA is that the quality and consistency of the data produced be independent of the analyst performing the analysis. This can only occur when

all analyses are performed using SOPs, and the analyst performing the procedure has been properly trained and has demonstrated proficiency with the analysis. This is accomplished at EEA by having a training checklist for each group or set of analyses within a group.

4.3.1.2. This checklist is followed for each trainee analyst by the group supervisor with the help of an assigned analyst mentor. The trainee is issued a set of training materials (i.e. safety information, SOP, Ethics SOP, method reference etc.) and is given hands-on training under the direct supervision of the mentor analyst or supervisor. Progress is monitored closely for the first three to six months by using frequent performance reviews, quality control check samples, performance audits and bench sheet reviews.

4.3.1.3. IDC Certification serves as a record of Authorization and Competence [TNI-EL-V1M2-2009-5.2.5][ISO/IEC 17025:2005(E)-5.2.5]. All Analysts, including contracted personnel when hired, are required to undergo the same training (IDC, MDL Studies, ability to achieve a low background, the precision and accuracy required by the method and satisfactory performance on a PT sample), and IDC Certificate of Competence [TNI-EL-V1M2-2009-5.2.1][ISO/IEC 17025:2005(E)-5.2.1]. A copy is filed in the analyst training record. Demonstration of Capability will also be done for analysts working as a unit. Examples are extraction analysts preparing the IDC and MDL samples and the prepared sample analyzed by the appropriate GC, GCMS, or HPLC analysts. IDC certification is completed for the group of analysts.

4.3.1.4. Initial training for a field sampling personnel is done through overall sampling procedure technique review and through duplicate samples for each new method and/or matrix that each of the field sampling personnel first performed.

For field sampling testing, DOC and MDL studies are performed initially and repeated at the frequency that the specific method requires.

4.3.2. **On-going Training/Annual Competency Check**

The laboratory performs an annual competency check for each analyst to ensure that each technical employee demonstrates an initial and ongoing proficiency for the tests performed by the technical employee.

On-going proficiency checks are conducted to ensure that the training of personnel is kept up-to-date by the following:

4.3.2.1. A certification that the technical personnel have read, understood and agreed to perform the most recent version of the test method (the approved method or standard operating procedure) and documentation of continued proficiency by at least one of the following once per year:

4.3.2.1.1. Acceptable performance of a blind sample (single blind to the analyst).

4.3.2.1.2. Another initial demonstration of method performance

4.3.2.1.3. Successful analysis of a blind performance sample on a similar test method using the same technology (e.g., GC/MS Volatiles by purge and trap for 524.2, 624 or 5030B/8260) would only require documentation for one of the test methods [TNI-EL-V1M3 to V1M4-ISO-2009]. The laboratory must determine the acceptable limits of the blind performance sample prior to analysis. The laboratory uses the Provider acceptable TNI limits of any blind PT sample that is used to document the annual proficiency documentation for each analyst.

4.3.2.1.4. At least four consecutive laboratory control samples with acceptable levels of precision and accuracy as per method specified precision and accuracy limits.

4.3.2.1.5. If the previous item cannot be performed, because spiking is not an option or QC samples not available, analysis of authentic samples that have been analyzed by another trained analyst with statistically identical results or analysis of Proficiency Test samples obtained from NIST approved providers can be done.

4.3.2.1.6. For specialized situations where extraction analysts have to do the sample preparation for LCS and MDL samples and the analyses of the prepared samples are done by the analysts belonging to another group, such as GC or GCMS areas, the group as a unit completes a Demonstration of Capability.

4.3.2.2. Evidence on file that demonstrates that each employee has read, understood, and is using the latest version of the laboratory's in-house SOP documentation and all other documentation, which relates to his/her job responsibilities.

4.3.2.3. Training courses or workshops on specific equipment, analytical techniques or laboratory procedures shall all be documented.

4.3.3. **Training Records**

A training file for each analyst and method is kept in the QA department along with a training history form completed at the inception of the present training program or at the time of employment. Each analyst's training file includes; a resume indicating the analyst's qualifications, experience, transcript of records, job description, and an initial demonstration of capability (IDC) and continuing demonstration of proficiency for each analyst. Up-to-date training records of courses in ethical and legal responsibilities, including potential punishments and penalties for violations, are kept in the QA department.

Figure 4-1 QM Signature Page

Quality Manual Signature Page

This is to certify that I have read and understood Eurofins Eaton Analytical's Quality Manual.

I further certify that I will comply with the laboratory procedures and practices described in the manual for the generation of high quality data.

If you know any deviations in the laboratory practices, please notify your supervisor or Quality Manager to evaluate if the said deviation adheres to good laboratory practices and affects data quality.

If you find errors in any section applicable to you, please notify your supervisor or Quality Manager to correct them appropriately. The Quality Manual will be revised annually to reflect current laboratory practices.

Signature: _____ Date: _____

Name (print): _____

QM – Rev. #: 35 Effective Date: _____

QA FO 0020 (06/11/13)

Figure 4-2 SOP/Method Training Documentation Form

eurofins | Eaton Analytical

Laboratory SOP and Method Reference Training Documentation

I certify that I have read, understood and agreed to perform the techniques and procedures, including instrument calibration procedures if applicable, stated in the most recent version of the approved test method and the laboratory standard operating procedure.

SOP Title: _____

SOP ID: _____

SOP Revision No.: _____

Issue Date: _____

Effective Date: _____

EPA/SM Method Reference: _____

Method Revision No: _____

Method Date Revised: _____

Analyst Name (Print) _____

Analyst Signature _____

SOP Training Start Date _____

SOP Training Completion Date _____

Training Duration _____

Trainer Name(s): _____

Print:	Signature:	Date:
_____	_____	_____
Supervisor		

QA FO 0013 (06/06/13)

Table 4-1 List of SOPs

SOP No.	Analytes	Method	Issue Date	Revision No.
Wet Chem 01	Cyanide Analysis by Ion Selective Electrode (ISE)	EPA 9012B/ SM 4500-CN E,G	4/13/2012	13.0
Wet Chem 02	Fluoride by Ion Selective Electrode	EPA 9214/ SM 4500-F B,C	11/21/2012	13.0
Wet Chem 03	Alkalinity	SM 2320B	6/7/2013	12.0
Wet Chem 04	Total Dissolved Solids (TDS) in water	SM 2540C	11/20/2012	17.0
Wet Chem 05	Total Suspended Solids (TSS) in water	SM 2540D	6/12/2013	8.0
Wet Chem 06	Turbidity - Nephelometric	EPA 180.1/ SM 2130B	6/10/2013	10.0
Wet Chem 07	Total Solids (TS) in Aqueous Sample	SM 2540B	6/12/2013	7.0
Wet Chem 09	Settleable Solids	SM 2540F	11/11/2011	6.0
Wet Chem 11	Color	SM 2120 B	11/11/2011	9.0
Wet Chem 12	Conductivity (EC)	EPA 120.1, 9050A/ SM 2510B	11/21/2012	11.0
Wet Chem 13	Cyanide (Reflux-Distillation) Midi Distillation	EPA 335.4	11/20/2012	11.0
Wet Chem 14	Orthophosphate, Total, Suspended and Dissolved	EPA 365.1/ SM 4500-P F	11/21/2012	8.0
Wet Chem 15	Odor	SM 2150	1/10/2012	9.0
Wet Chem 16	Determination of Perchlorate in Drinking Water using Ion Chromatography	EPA 314.0/ CADHS 300.0 Modified	11/21/2012	10.0
Wet Chem 17	Biochemical Oxygen Demand	SM 5210B	6/12/2013	15.0
Wet Chem 19	Phenolics and Phenolics-Low	EPA 420.1 / 420.4	9/3/2010	13.0
Wet Chem 21	Determination of Nitrate / Nitrite by Flow Injection Analysis	EPA 353.2	11/21/2012	12.0
Wet Chem 22	Total Kjedahl Nitrogen by Colorimetric Analysis follwing Semi-Automated Digestion	EPA 351.2	11/21/2012	12.0
Wet Chem 25	Determination of Anions and Inorganic Disinfectant By-Products by Ion Chromatography	EPA 300.0, 9056	11/21/2012	20.0
Wet Chem 26	Total Volatile Solids/Volatile Suspended Solids in Liquid	EPA 160.4	3/26/2012	8.0
Wet Chem 27	Ammonia as Nitrogen by Rapid Flow Analyzer (RFA)	EPA 350.1/ SM 4500-NH3 D,H	11/28/2012	12.0
Wet Chem 28	pH Value	EPA 9040B, 150.1/ SM 4500-H+B	11/21/2012	7.0
Wet Chem 31	Surfactants, Anionic (MBAS)	SM 5540 C	6/12/2013	8.0
Wet Chem 32	Total Organic Carbon and Dissolved Organic Carbon by UV/ Persulfate Oxidation	EPA 9060A/ SM 5310C	11/21/2012	13.0
Wet Chem 34	Analytical method for Ultraviolet Absorption of Organic constituents at 254 nm	SM 5910B	1/10/2012	6.0
Wet Chem 35	Sulfide Determination (Methylene Blue)	EPA 9030B, 9034/ SM 4500-S2-D	8/20/2010	7.0
Wet Chem 36	Chemical Oxygen Demand (COD)	EPA 410.4/ SM 5220D	11/21/2012	11.0
Wet Chem 37	Determination of Total Cyanide by Semi-Automated Colorimetry	EPA 335.4	4/9/2012	12.0
Wet Chem 38	Determination of Total Phosphate by Flow Injection Analysis Colorimetry	EPA 365.1/ SM 4500-PF	1/18/2012	7.0
Wet Chem 39	Langelier Index by Calculation	SM 2330B	11/21/2012	4.0
Wet Chem 40	Determination of Inorganic Anions and trace Bromate in Drinking Water using Ion Chromatography by the addition of a Post-column reagent and Absorbance Detector in Series with an Electrochemical Detector	EPA 300.1/EPA 317.0	2/8/2013	22.0
Wet Chem 42	Dissolved Organic Halogen: Adsorption-Pyrolysis-Titrimetric Method	SM 5320B	8/20/2010	13.0

SOP No.	Analytes	Method	Issue Date	Revision No.
Wet Chem 43	Dissolved Oxygen, Membrane Electrode	SM 4500 OG	11/11/2011	4.0
Wet Chem 48	Determination of Low Level Perchlorate in Drinking Water using Ion Chromatography	EPA 314.0	11/28/2012	5.0
Wet Chem 49	Determination of Dissolved Hexavalent Chromium in Drinking Water, Groundwater, and Industrial Wastewater Effluents by IC	EPA 218.6	11/28/2012	5.0
Wet Chem 53	A Simplified and Rapid Method for Biodegradable Dissolved Organic Carbon Measurement (BDOC)	N/A	11/11/2011	3.0
Wet Chem 54	Determination of EDTA & NTA in Water Using Ion Chromatography by the Addition of a Post-column Reagent and Absorbance Detector	Metrohm Method	7/7/2010	0.0
Wet Chem 55	Chlorate by 300.1	EPA 300.1	6/18/2013	4.0
Wet Chem 56	UCMR3 – Determination of Hexavalent Chromium in Drinking Water by Ion Chromatography with Post Column Derivatization and UV/VIS Detection	EPA 218.7	6/18/2013	4.0
Extract 03	Liquid - Solid Extraction	EPA 525.2	2/28/2013	16.0
Extract 04	Determination of Endothall in Drinking Water by Solid Phase Extraction	EPA 548.1	11/26/2012	12.0
Extract 05	Liquid-Solid Extraction of Diquat and Paraquat	EPA 549.2	11/26/2012	11.0
Extract 11	Extraction BNA Continuous Liquid-Liquid Extraction	EPA 8270 C	11/19/2012	13.0
Extract 16	Solid Phase Extraction of Phenols in Drinking Water	EPA 528	4/13/2012	3.0
Extract 17	Solid Phase Extraction of Explosives in Drinking Water	EPA 529	1/25/2012	2.0
Extract 18	Solid Phase Extraction of Selected Pesticides and Flame Retardants in Drinking Water	EPA 527	1/19/2012	4.0
Extract 19	Determination of Nitrosamines in Drinking Water by Solid Phase Extraction (SPE)	EPA 521	12/31/2012	3.0
Extract 20	Measurement of Chloroacetanilide and Other Acetamide Herbicide Degradates in Drinking Water by Solid Phase Extraction	EPA 535	12/31/2012	4.0
Extract 21	Determination of MCPA, MCPB and MCPP in Drinking Water by Solid Phase Extraction (SPE) Modified SM 555	EPA 555	4/13/2012	2.0
Extract 22	Liquid-Solid Extraction	EPA 526	7/31/2012	2.0
Extract 23	Determination of Phenylurea Compounds in Drinking Water by SPE	EPA 532	4/13/2012	2.0
Extract 24	EDC4 by Continuous Liquid-Liquid Extraction	EDC4	11/19/2012	3.0
Extract 25	Determination of Nitrosamines in Drinking Water by Liquid-Liquid Phase Extraction	EPA 521	11/19/2012	2.0
Extract 26	Extraction for Determination of 2,3,7,8-TCDD by Isotope Dilution in Drinking Water by Capillary Column Gas Chromatography with Large Volume Injection and Electron Ionization Tandem Mass Spectrometry (EI/MS/MS) Modified EPA 1613B	EPA 1613B	11/19/2012	2.0
Extract 27	Determination of 1,4-Dioxane in Drinking Water by Solid Phase Extraction	EPA 522	6/18/2013	4.0
GC 03	EDB, DBCP and 1,2,3-TCP in water by Microextraction and Gas Chromatography	EPA 504.1	11/26/2012	14.0
GC 08	Chlorination Disinfection Byproducts and Chlorinated Organic Solvents by Liquid-Lquid Extraction and Gas Chromatography with Electron-Capture Detection	EPA 551.1	11/21/2012	20.0
GC 09	Haloacetic Acids	SM 6251 B	11/26/2012	17.0
GC 16	1,2,-Dibromoethane & 1,2-Dibromo-3-Chloropropane by Microextraction & Gas Chromatography	EPA 8011	11/26/2012	7.0
GC 27	Free and Total Chlorine Analysis and Chloramine Calculation	SM 4500-Cl-G	2/2/2012	8.0
GC 29	Formation of Trihalomethanes and other disinfection by-products. Modified Standard Method 5710 B and 5710D	SM 5710 B and SM 5710 D	8/20/2010	3.0
GC 33	Chlorine Dioxide Analysis	SM 4500-CLO2-D	2/2/2012	3.0
GC 34	Chlorinated Pesticides and PCBs	EPA 505	5/28/2013	13.0
GC 35	Chlorinated Acids in Drinking Water by Liquid-Liquid Extraction, Derivatization and Gas Chromatograph with Electron Capture Detection	EPA 515.4	11/26/2012	10.0

SOP No.	Analytes	Method	Issue Date	Revision No.
GC 36	Chlorine Demand, Modified Standard Method 2350B	SM 2350B	2/2/2012	2.0
GC 37	Aldehydes	EPA 556	2/11/2010	0.0
GC 38	Haloacetic Acids	EPA 552.3 Rev. 1.0	5/3/2010	0.0
GCMS 01	Volatile Organic Compounds in Drinking Water by GC/MS	EPA 524.2 (Modified)	6/7/2013	24.0
GCMS 01a	Determination of 1,2,3 Trichloropropane (TCP) in Drinking Water by Purge and Trap GC/MS in SIM Mode	EPA 524.2 (Modified)	11/21/2012	5.0
GCMS 01b	Determination of tert-Butanol, Epichlorohydrin, 1,2-Dichloropropane, 1,2,3-Trichloropropane and Cyanogen Chloride in Drinking Water by purge and trap GC/MS in SIM mode	EPA 524.2 (Modified)	11/21/2012	3.0
GCMS 02	Determination of Semivolatile Organic Compounds in Drinking Water by Gas Chromatography/Mass Spectrometry	EPA 525.2	6/7/2013	27.0
GCMS 03	Endothall Analysis by GCMS	EPA 548.1	6/7/2013	15.0
GCMS 04	Volatile Organic Compounds in Aqueous Matrix by GC/MS	EPA 624 (Modified)	10/24/2012	11.0
GCMS 05	Analysis of Semivolatile Organic Compounds by GCMS	EPA 625	9/16/2010	10.0
GCMS 07	Volatile Organic Compounds in Water by GC/MS	EPA 8260 B	11/21/2012	11.0
GCMS 08	Analysis of Semivolatile Organic Compounds by GCMS	EPA 8270 C	4/10/2012	9.0
GCMS 15	Determination of Selected Semivolatile Organic Compounds in Drinking Water by Solid Phase Extractions and Capillary Column GCMS	EPA 526	9/16/2010	5.0
GCMS 16	Determination of Phenols in Drinking Water by Capillary Column GCMS	EPA 528 Soda	8/20/2010	4.0
GCMS 17	Taste and Odor Analytes by Solid Phase Micro-Extraction and GCMS	SM 6040D	2/1/2013	5.0
GCMS 20	Determination of Nitrosamines in Drinking Water by Capillary Column GC with Large Volume Injection and Chemical Ionization Trap Mass Spectrometry	EPA 521	3/21/2013	4.0
GCMS 21	Determination of Explosives and Related Compounds in Drinking Water by Solid Phase Extractions and Capillary Column GCMS	EPA 529	9/3/2010	3.0
GCMS 22	Determination of Endocrine Disruptor Chemicals in Wastewater by GCMS Method 4	EDC 4SCR	9/16/2010	3.0
GCMS 23	Determination of Selected Pesticides and Flame Retardants in Drinking Water by Solid Phase Extraction and Capillary Column Gas Chromatography/Mass Spectrometry (GC/MS)	EPA 527	9/3/2010	3.0
GCMS 24	Determination of 2,3,7,8-TCDD in Drinking Water by Capillary Column Gas Chromatography with Large Volume Injection and Electron Ionization Tandem Mass Spectrometry (EI/MS/MS)	EPA 1613B (Modified)	6/7/2013	2.0
GCMS 25	Determination of Galaxolide in Wastewater Using USGS Endocrine Disrupter Chemicals Method 4 by Gas Chromatograph/Tandem Mass Spectrometry	Galaxolide	4/23/2010	0.0
GCMS 26	Determination of 1,4-Dioxane in Drinking Water by Solid Phase Extraction with GCMS with Selected Ion Monitoring (SIM)	EPA 522	6/18/2013	4.0
GCMS 27	Volatile Organic Compounds in Drinking Water by GC/MS	EPA 524.3	6/18/2013	4.0
HPLC 02	Glyphosate Analysis in Drinking Water by High Performance Liquid Chromatography (HPLC)	EPA 547	11/21/2012	15.0
HPLC 03	Diquat and Paraquat Analysis in Drinking Water by HPLC	EPA549.2	6/7/2013	16.0
HPLC 05	Carbamates Analysis in Drinking Water by HPLC with post column derivatization	EPA 531.2	11/26/2012	7.0
HPLC 06	Determination of Phenylurea Compounds in Drinking water by Solid Phase Extraction and HPLC with UV Detection	EPA 532	11/21/2012	4.0
HPLC 07/ LCMS 01	Determination of Perchlorate in Drinking Water by Liquid Chromatography/Electrospray Ionization Tandem Mass Spectrometry	EPA 331	2/8/2013	4.0
HPLC 08	Analysis of MCPA, MCPB and MCPP in Drinking Water by HPLC	EPA 555	11/15/2012	4.0
HPLC 09	Measurement of Chloroacetanilide and other Acetamide Herbicide Degradates in Drinking Water by Solid Phase Extraction (SPE) and Liquid Chromatography/Tandem Mass Spectrometry (LC/MS/MS)	EPA 535	8/19/2010	4.0
HPLC 10/ LCMS 02	Determination of Acrylamide in Drinking Water by Liquid Chromatography Electrospray Ionization/Mass Spectrometry	Acrylamide	8/27/2010	1.0
HPLC 11/ LCMS 03	Determination of Emerging Organic Pollutants in Environmental Matrices by Liquid Chromatography Mass Spectrometry in Tandem Analysis (LC/MS/MS)	EDC 2	8/27/2010	1.0

SOP No.	Analytes	Method	Issue Date	Revision No.
HPLC 12/ LCMS 04	Determination of Perfluorinated Pollutants in Environmental Matrices by Online Solid-Phase Extraction (SPE) coupled with HPLC/MS in Tandem	PFC	4/2/2010	4.0
HPLC 13/ LCMS 05	Determination of Selected Perfluorinated Alkyl Acids in Drinking Water by Solid Phase Extraction and Liquid Chromatography/Tandem Mass Spectrometry (LC/MS/MS)	EPA 537	11/15/2012	5.0
HPLC 14/ LCMS 06	The Determination of Personal Care Products, Pharmaceutical (PPCP) and Endocrine Disrupters Compounds (EDC), Herbicide and Degradates in Environmental Matrices by Online Solid-Phase Extraction Coupled with High-Performance Liquid Chromatography/Mass Spectrometry in Tandem	PPCP	4/23/2010	0.0
HPLC 16/LCMS 07	Determination of Hormones in Drinking Water by Solid Phase Extraction (SPE) and Liquid Chromatography Electrospray Ionization Tandem Mass Spectrometry (LC-ESI-MS/MS)	EPA 539	6/18/2013	4.0
LCMS 08	Determination of Iodide and Iodate in Drinking Water by Liquid Chromatography/Electrospray Ionization Tandem Mass Spectrometry			
LCMS 11	Determination of Selected Perfluorinated Alkyl Acids in Drinking Water by Solid Phase Extraction and Liquid Chromatography/Tandem Mass Spectrometry (LC/MS/MS)	UCMR3 537	6/18/2013	0.0
Met 01	Analysis of Trace Elements by ICP Emission Spectroscopy	ICP, EPA 200.7, 6010B, 6020A	11/19/2012	20.0
Met 02	Trace Metals by ICP/MS	ICP/MS, EPA 200.8, 6020A	6/7/2013	30.0
Met 04a	Mercury by Cold Vapor Atomic Absorption	SW846 Method 7470, EPA 245.1, 7470A	11/19/2012	19.0
Met 19	Hexavalent Chromium, Colorimetric Method	EPA 7196 A / SM 3500 CR-B /SM3500 CR - D	11/19/2012	11.0
Met 26	Silica by the Molybdosilicate Method	SM 4500-SiO2C	8/19/2010	5.0
Met 27	Hardness by Calculation	EPA 200.7/ SM 2340B	11/21/2012	7.0
Met 28	pH / Turbidity Check for Metals	pH paper/180.1	11/21/2012	4.0
Met 30	Heated Block Metals Digestion	EPA 200.7, 200.8, 6020A	11/15/12	11.0
Met 31	UCMR3 TRACE METALS ANALYSIS BY ICP/MS	EA 200.8	6/18/2013	2.0
Micro 01	Determination of Asbestos Fibers in Water	EPA 100.2	11/26/2012	19.0
Micro 02	Assimilable Organic Carbon Biossay	SM 9217 B	12/20/2011	8.0
Micro 05	Determination of Coliform in drinking water by the ONPG-MUG Method (Colilert)	SM 9223 B	1/10/2012	23.0
Micro 06	Determination of Total and Fecal Coliforms in water, wastewater and soil by Multiple Tube Fermentation Technique	SM 9221	1/10/2012	19.0
Micro 09	Determination of Fecal Streptococci and enterococci in water, wastewater and soil	SM 9230B	12/20/2011	12.0
Micro 11	Heterotrophic Plate Count	SM 9215 A, B	4/16/2012	12.0
Micro 13	Microscopic Particulate Analysis	EPA 910/9-92-029	7/1/2013	11.0
Micro 16	Determination of Coliforms in Water by the CPRG-MUG Method / Colisure	SM 9223	12/20/2011	10.0
Micro 17	Determination of Escherichia Coli in water and wastewater by Multiple Tube Fermentation Technique	SM 9221 F	12/20/2011	10.0
Micro 19	Water Suitability Test	SM 9020B	1/10/2012	7.0
Micro 20	Inhibitory Residues	SM 9020B	1/10/2012	8.0
Micro 21	Microbiology Demonstration of Capability	N/A	1/26/2012	4.0
Micro 23	Male-specific (F+) and Somatic Coliphage in water by Single Agar Layer (SAL) Procedure	EPA 1602 April 2001	1/26/2012	3.0
Micro 24	pH Check of Clean Glassware Using Bromthymol Blue	SM 9020B	1/26/2012	4.0
Micro 26	Determination of Coliforms in Drinking Water by the 18 Hour ONPG-MUG Method	SM 9223B	3/14/2012	5.0
Micro 27	Microcystin by ELISA analysis	USEPA Region 9 SOP	4/16/2012	1.0
Micro 28	Determination of Yeast and Mold in Water	Standard Methods, 21st Ed. (2005) Section 9610 D	4/16/2012	0.0

SOP No.	Analytes	Method	Issue Date	Revision No.
Micro 29	Algae Enumeration and Identification	SM 10200 F, SM 10900 C	8/23/2012	1.0
Micro 30	Cylindrospermopsin		11/1/2012	0.0
Micro 31	Saxitoxin		8/23/2012	0.0
Micro 32	Heterotrophic Plate Count - MF	Coca Cola, SM 9215A,D	6/26/2013	0.0
Micro 33	DETERMINATION OF PSEUDOMONAS AERUGINOSA IN DRINKING WATER BY THE PSEUDALERT METHOD	IDEXX Pseudalert/ IDEXX Quanti-Tray/2000		0.0
Micro Colton 1	Determination of Coliform in drinking water by the ONPG-MUG Method	SM 9223 B	12/22/2011	3.0
Micro Colton 2	Determination of Coliforms in Drinking Water by the 18-hr ONPG-MUG Method	SM 9223B	12/22/2011	3.0
Micro Colton 3	Determination of Coliforms in Water by the CPRG-MUG Method / Colisure	SM 9223	12/22/2011	3.0
Micro Colton 4	Determination of Coliform in water by Multiple Tube Fermentation Technique	SM 9221	12/22/2011	3.0
Micro Colton 5	Heterotrophic plate count	SM 9215 A, B	12/20/2011	3.0
Non Method 01	Sample Receiving and Log In	N/A	2/11/2013	22.0
Non Method 02	Chain of Custody	N/A	2/11/2013	11.0
Non Method 03	Preparation and Shipment of Sample Kits	N/A	2/11/2013	5.0
Non Method 04	Hazardous Waste Management and Sample Disposal Procedures	N/A	6/18/2013	9.0
Non Method 05	Laboratory Ethics/Data Integrity Plan	N/A	8/28/2012	9.0
Non Method 06	Environmental Monitoring for Microbiological Contaminants	N/A	2/11/2013	8.0
Non Method 07	Standards and Reagent Preparation, Documentation, and Labeling	N/A	2/11/2013	10.0
Non Method 08	Compositing and Subsampling in the Laboratory	N/A	2/2/2012	6.0
Non Method 11	Balance Maintenance	N/A	2/11/2013	8.0
Non Method 12	Manual Integration	N/A	2/11/2013	9.0
Non Method 13	Retention of Significant figures	N/A	2/11/2013	7.0
Non Method 14	Instrument Maintenance	N/A	2/11/2013	8.0
Non Method 15	Use of Class A glassware	N/A	2/11/2013	7.0
Non Method 16	Glassware Cleaning	N/A	1/25/2012	5.0
Non Method 19	Temperature Monitoring and Thermometer Calibration	N/A	2/1/2012	4.0
Non Method 20	Handling and Disposal of Foreign Soil Samples	N/A	2/9/2010	4.0
Non Method 22	States Certification & Performance Tests Requirements	N/A	4/23/2012	4.0
Non Method 23	Calibration	N/A	11/26/2012	0.0
Non Method 24	Handling of Controlled Substances	N/A	3/8/2013	2.0
Rad 02	Radon by Liquid Scintillation Counter	SM 7500-Rn	6/7/2013	10.0
Rad 06	Gross alpha and beta Radioactivity	EPA 900.0	6/7/2013	18.0
Rad 09	Ra-226 and Ra-228 by Gamma-Ray Spectrometry Using HPGE Detector	GA Tech	6/7/2013	4.0
UCMR3 PresCheck1	Standardized Procedure for pH, Free Chlorine, and Total Chlorine Checks for UCMR3 Samples	N/A	2/15/2013	1.0

Note: The most current SOP list is available in the QA Department for review.

Table 4-2 Other Certifications

#	AGENCY	LAB ID	EXPIRATION DATE
1	LACSD	10249	-------
2	Radioactive Material License	3069-19	March 15, 2020
3	Soil Permit	S-65114	February 26, 2013
4	CUPA Consolidate Permit/License to Operate	AR0036980	June 30, 2013
5	Drug Enforcement Administration (DEA)	RE0438158	August 31, 2013

The most current licenses are available in the QA Department for review.

Figure 4-3 State of California Accreditation

NELAP - RECOGNIZED

CALIFORNIA STATE

ENVIRONMENTAL LABORATORY ACCREDITATION PROGRAM BRANCH

CERTIFICATE OF NELAP ACCREDITATION

Is hereby granted to

Eurofins Eaton Analytical, Inc. (former M.W.H.)

750 Royal Oaks Drive, Suite 100

Monrovia, CA 91016

Scope of the Certificate is limited to the
"NELAP Fields of Accreditation"
which accompany this Certificate.

Continued accredited status depends on successful
ongoing participation in the program.

This Certificate is granted in accordance with provisions of
Section 100825, et seq. of the Health and Safety Code.

Certificate No.: **01114CA**

Expiration Date: **1/31/2014**

Effective Date: **2/1/2013**

Richmond, California
subject to forfeiture or revocation

David Mazzera, Ph.D., Assistant Division Chief
Division of Drinking Water and Environmental Management

Figure 4-4 List of California Accredited Analytes

State of California—Health and Human Services Agency
California Department of Public Health

RON CHAPMAN, MD, MPH
Director & State Health Officer

EDMUND G. BROWN JR.
Governor

January 9, 2013

Ed Wilson
Eurofins Eaton Analytical, Inc. (former M.W.H.)
750 Royal Oaks Drive, Suite 100
Monrovia, CA 91016

Dear Ed Wilson: Certificate No. 01114CA

This is to advise you that the laboratory named above has been accredited under National Environmental Laboratory Accreditation Program (NELAP) as an environmental testing laboratory pursuant to the provisions of the Health and Safety Code (HSC), Division 101, Part 1, Chapter 4, Section 100825, *et seq.*

The Fields of Accreditation for which this laboratory has been accredited are enclosed. The certificate shall remain in effect until **January 31, 2014** unless revoked by California Environmental Laboratory Accreditation Program Branch (ELAPB) or withdrawn at your written request. To maintain accreditation, the laboratory shall comply with the National Environmental Laboratory Accreditation Conference (NELAC) Standards and all associated California ELAPB regulations and statutes.

The application for renewal of this certificate must be received before the expiration date of this certificate to remain in force according to the HSC 100847(a).

Please note that your laboratory is required to notify California ELAPB of any major changes in key accreditation criteria within 30 calendar days of the change. This written notification includes, but is not limited to, changes in ownership, location, key personnel, and major instrumentation (HSC 100847(b), (c), (d), and NELAC Standard Section 4.3.2). The certificate must be returned to California ELAPB upon loss of accredited status.

Your continued cooperation with the above requirements is essential for maintaining the high quality of the data produced by environmental laboratories accredited by the State of California.

If you have any questions, please contact Bill Walker at (818) 551-2012.

Sincerely,

David Mazzera, Ph.D., for Assistant Division Chief
Division of Drinking Water and Environmental Management

Enclosure

Environmental Laboratory Accreditation Program Branch
MS 0511, Building P, 1st Floor, 850 Marina Bay Parkway, Richmond, CA 94804
(510) 620-3155, (510) 620-3165 Fax
www.cdph.ca.gov/certlic/labs/Pages/ELAP.aspx

NELAP RECOGNIZED

CALIFORNIA DEPARTMENT OF PUBLIC HEALTH

ENVIRONMENTAL LABORATORY ACCREDITATION PROGRAM BRANCH

NELAP Fields of Accreditation

Eurofins Eaton Analytical, Inc. (former M.W.H.)
750 Royal Oaks Drive, Suite 100
Monrovia, CA 91016
Phone: (626) 386-1100

Certificate No.: 01114CA
Renew Date: 1/31/2014

101 - Microbiology of Drinking Water			
101.010	001	SM9215B	Heterotrophic Bacteria
101.020	001	SM9221A,B	Total Coliform
101.021	001	SM9221E (MTF/EC)	Fecal Coliform
101.022	001	CFR 141.21(f)(6)(i) (MTF/EC+MUG)	E. coli
101.060	002	SM9223	Total Coliform
101.060	003	SM9223	E. coli
101.070	002	Colisure	Total Coliform
101.070	003	Colisure	E. coli
101.120	001	SM9221A,B,C	Total Coliform (Enumeration)
101.130	001	SM9221E (MTF/EC)	Fecal Coliform (Enumeration)
101.160	001	SM9223	Total Coliform (Enumeration)
101.200	001	SM9223B	E. coli (Enumeration)
101.210	001	SM9221B.1/SM9221F	E. coli (Enumeration)
102 - Inorganic Chemistry of Drinking Water			
102.020	001	EPA 180.1	Turbidity
102.022	001	SM2130B	Turbidity
102.030	001	EPA 300.0	Bromide
102.030	002	EPA 300.0	Chlorate
102.030	003	EPA 300.0	Chloride
102.030	004	EPA 300.0	Chlorite
102.030	005	EPA 300.0	Fluoride
102.030	006	EPA 300.0	Nitrate
102.030	007	EPA 300.0	Nitrite
102.030	010	EPA 300.0	Sulfate
102.040	001	EPA 300.1	Bromide
102.040	002	EPA 300.1	Chlorite
102.040	003	EPA 300.1	Chlorate
102.040	004	EPA 300.1	Bromate
102.045	001	EPA 314.0	Perchlorate
102.047	001	EPA 331.0	Perchlorate
102.050	001	EPA 335.4	Cyanide
102.060	001	EPA 353.2	Nitrate calc.

As of 1/9/2013 , this list supersedes all previous lists for this certificate number.
Customers: Please verify the current accreditation standing with the State.

Eurofins Eaton Analytical, Inc. (former M.W.H.)

Certificate No.: 01114CA
Renew Date: 1/31/2014

102.061	001	EPA 353.2	Nitrite
102.070	001	EPA 365.1	Phosphate, Ortho
102.100	001	SM2320B	Alkalinity
102.110	001	SM2330B	Corrosivity (Langlier Index)
102.120	001	SM2340B	Hardness
102.130	001	SM2510B	Conductivity
102.140	001	SM2540C	Total Dissolved Solids
102.163	001	SM4500-Cl G	Chlorine, Free and Total
102.180	001	SM4500-ClO2 D	Chlorine Dioxide
102.191	001	SM4500-CN F	Cyanide, Total
102.192	001	SM4500-CN G	Cyanide, amenable
102.200	001	SM4500-F C	Fluoride
102.210	001	SM4500-H+ B	pH
102.212	001	EPA 150.1	pH
102.240	001	SM4500-P E	Phosphate, Ortho
102.262	001	SM5310C	Total Organic Carbon
102.263	001	SM5310C	DOC
102.263	002	SM5310C	TOC/DOC
102.267	001	SM5310C-OO	TOC/DOC
102.270	001	SM5540C	Surfactants
102.280	001	SM5910B	UV254
102.520	001	EPA 200.7	Calcium
102.520	002	EPA 200.7	Magnesium
102.520	003	EPA 200.7	Potassium
102.520	004	EPA 200.7	Silica
102.520	005	EPA 200.7	Sodium
102.520	006	EPA 200.7	Hardness (calc.)
102.533	002	SM4500-Si D	Silica
102.542	002	SM4500-SiO2 C	Silica
102.545	001	EPA 317.0	Bromate
102.545	003	EPA 317.0	Chlorite
102.551	002	SM4500-Cl G	Chlorine, Free, Combined, Total
102.558	001	SM4500-Cl G-OO	Chlorine, Free, Combined, Total

103 - Toxic Chemical Elements of Drinking Water

103.130	001	EPA 200.7	Aluminum
103.130	003	EPA 200.7	Barium
103.130	004	EPA 200.7	Beryllium
103.130	005	EPA 200.7	Cadmium
103.130	007	EPA 200.7	Chromium
103.130	008	EPA 200.7	Copper

As of 1/9/2013 , this list supersedes all previous lists for this certificate number.
Customers: Please verify the current accreditation standing with the State.

Eurofins Eaton Analytical, Inc. (former M.W.H.)

Certificate No.: 01114CA
Renew Date: 1/31/2014

103.130	009	EPA 200.7	Iron
103.130	011	EPA 200.7	Manganese
103.130	012	EPA 200.7	Nickel
103.130	015	EPA 200.7	Silver
103.130	017	EPA 200.7	Zinc
103.140	001	EPA 200.8	Aluminum
103.140	002	EPA 200.8	Antimony
103.140	003	EPA 200.8	Arsenic
103.140	004	EPA 200.8	Barium
103.140	005	EPA 200.8	Beryllium
103.140	006	EPA 200.8	Cadmium
103.140	007	EPA 200.8	Chromium
103.140	008	EPA 200.8	Copper
103.140	009	EPA 200.8	Lead
103.140	010	EPA 200.8	Manganese
103.140	012	EPA 200.8	Nickel
103.140	013	EPA 200.8	Selenium
103.140	014	EPA 200.8	Silver
103.140	015	EPA 200.8	Thallium
103.140	016	EPA 200.8	Zinc
103.160	001	EPA 245.1	Mercury
103.301	001	EPA 100.2	Asbestos
103.310	001	EPA 218.6	Chromium (VI)

104 - Volatile Organic Chemistry of Drinking Water

104.030	001	EPA 504.1	1,2-Dibromoethane
104.030	002	EPA 504.1	1,2-Dibromo-3-chloropropane
104.030	003	EPA 504.1	1,2,3-Trichloropropane
104.040	000	EPA 524.2	Volatile Organic Compounds
104.040	001	EPA 524.2	Benzene
104.040	002	EPA 524.2	Bromobenzene
104.040	003	EPA 524.2	Bromochloromethane
104.040	006	EPA 524.2	Bromomethane
104.040	007	EPA 524.2	n-Butylbenzene
104.040	008	EPA 524.2	sec-Butylbenzene
104.040	009	EPA 524.2	tert-Butylbenzene
104.040	010	EPA 524.2	Carbon Tetrachloride
104.040	011	EPA 524.2	Chlorobenzene
104.040	012	EPA 524.2	Chloroethane
104.040	014	EPA 524.2	Chloromethane
104.040	015	EPA 524.2	2-Chlorotoluene

As of 1/9/2013 , this list supersedes all previous lists for this certificate number.
Customers: Please verify the current accreditation standing with the State.

Eurofins Eaton Analytical, Inc. (former M.W.H.)

Certificate No.: 01114CA
Renew Date: 1/31/2014

104.040	016	EPA 524.2	4-Chlorotoluene
104.040	018	EPA 524.2	Dibromomethane
104.040	019	EPA 524.2	1,3-Dichlorobenzene
104.040	020	EPA 524.2	1,2-Dichlorobenzene
104.040	021	EPA 524.2	1,4-Dichlorobenzene
104.040	022	EPA 524.2	Dichlorodifluoromethane
104.040	023	EPA 524.2	1,1-Dichloroethane
104.040	024	EPA 524.2	1,2-Dichloroethane
104.040	025	EPA 524.2	1,1-Dichloroethene
104.040	026	EPA 524.2	cis-1,2-Dichloroethene
104.040	027	EPA 524.2	trans-1,2-Dichloroethene
104.040	028	EPA 524.2	Dichloromethane
104.040	029	EPA 524.2	1,2-Dichloropropane
104.040	030	EPA 524.2	1,3-Dichloropropane
104.040	031	EPA 524.2	2,2-Dichloropropane
104.040	032	EPA 524.2	1,1-Dichloropropene
104.040	033	EPA 524.2	cis-1,3-Dichloropropene
104.040	034	EPA 524.2	trans-1,3-Dichloropropene
104.040	035	EPA 524.2	Ethylbenzene
104.040	036	EPA 524.2	Hexachlorobutadiene
104.040	037	EPA 524.2	Isopropylbenzene
104.040	038	EPA 524.2	4-Isopropyltoluene
104.040	039	EPA 524.2	Naphthalene
104.040	040	EPA 524.2	Nitrobenzene
104.040	041	EPA 524.2	N-propylbenzene
104.040	042	EPA 524.2	Styrene
104.040	043	EPA 524.2	1,1,1,2-Tetrachloroethane
104.040	044	EPA 524.2	1,1,2,2-Tetrachloroethane
104.040	045	EPA 524.2	Tetrachloroethene
104.040	046	EPA 524.2	Toluene
104.040	047	EPA 524.2	1,2,3-Trichlorobenzene
104.040	048	EPA 524.2	1,2,4-Trichlorobenzene
104.040	049	EPA 524.2	1,1,1-Trichloroethane
104.040	050	EPA 524.2	1,1,2-Trichloroethane
104.040	051	EPA 524.2	Trichloroethene
104.040	052	EPA 524.2	Trichlorofluoromethane
104.040	053	EPA 524.2	1,2,3-Trichloropropane
104.040	054	EPA 524.2	1,2,4-Trimethylbenzene
104.040	055	EPA 524.2	1,3,5-Trimethylbenzene
104.040	056	EPA 524.2	Vinyl Chloride

As of 1/9/2013 , this list supersedes all previous lists for this certificate number.
Customers: Please verify the current accreditation standing with the State.

Eurofins Eaton Analytical, Inc. (former M.W.H.)

Certificate No.: 01114CA
Renew Date: 1/31/2014

104.040	057	EPA 524.2	Xylenes, Total
104.040	058	EPA 524.2	Hexachloroethane
104.045	001	EPA 524.2	Bromodichloromethane
104.045	002	EPA 524.2	Bromoform
104.045	003	EPA 524.2	Chloroform
104.045	004	EPA 524.2	Dibromochloromethane
104.045	005	EPA 524.2	Trihalomethanes
104.050	002	EPA 524.2	Methyl tert-butyl Ether (MTBE)
104.050	004	EPA 524.2	tert-Amyl Methyl Ether (TAME)
104.050	005	EPA 524.2	Ethyl tert-butyl Ether (ETBE)
104.050	006	EPA 524.2	Trichlorotrifluoroethane
104.050	011	EPA 524.2	Oxygenates
104.055	001	EPA 524.3	Benzene
104.055	002	EPA 524.3	Carbon Tetrachloride
104.055	003	EPA 524.3	Chlorobenzene
104.055	004	EPA 524.3	1,2-Dichlorobenzene
104.055	005	EPA 524.3	1,4-Dichlorobenzene
104.055	006	EPA 524.3	1,2-Dichloroethane
104.055	007	EPA 524.3	cis-1,2-Dichloroethene
104.055	008	EPA 524.3	trans-1,2-Dichloroethene
104.055	009	EPA 524.3	Dichloromethane
104.055	010	EPA 524.3	1,2-Dichloropropane
104.055	011	EPA 524.3	Ethylbenzene
104.055	012	EPA 524.3	Styrene
104.055	013	EPA 524.3	Tetrachloroethene
104.055	014	EPA 524.3	1,1,1-Trichloroethane
104.055	015	EPA 524.3	Trichloroethene
104.055	016	EPA 524.3	Toluene
104.055	017	EPA 524.3	1,2,4-Trichlorobenzene
104.055	018	EPA 524.3	1,1-Dichloroethene
104.055	019	EPA 524.3	1,1,2-Trichloroethane
104.055	020	EPA 524.3	Vinyl Chloride
104.055	021	EPA 524.3	Xylenes, Total
104.055	022	EPA 524.3	1,2-Dibromo-3-chloropropane
104.055	023	EPA 524.3	1,2-Dibromoethane
104.055	024	EPA 524.3	Trihalomethanes, Total

105 - Semi-volatile Organic Chemistry of Drinking Water

105.010	001	EPA 505	Aldrin
105.010	002	EPA 505	Alachlor
105.010	004	EPA 505	Chlordane

As of 1/9/2013 , this list supersedes all previous lists for this certificate number.
Customers: Please verify the current accreditation standing with the State.

Eurofins Eaton Analytical, Inc. (former M.W.H.)

Certificate No.: 01114CA
Renew Date: 1/31/2014

105.010	005	EPA 505	Dieldrin
105.010	006	EPA 505	Endrin
105.010	007	EPA 505	Heptachlor
105.010	008	EPA 505	Heptachlor Epoxide
105.010	011	EPA 505	Lindane
105.010	012	EPA 505	Methoxychlor
105.010	014	EPA 505	Toxaphene
105.010	015	EPA 505	PCBs as Aroclors (screen)
105.010	016	EPA 505	PCB-1016
105.010	017	EPA 505	PCB-1221
105.010	018	EPA 505	PCB-1232
105.010	019	EPA 505	PCB-1242
105.010	020	EPA 505	PCB-1248
105.010	021	EPA 505	PCB-1254
105.010	022	EPA 505	PCB-1260
105.083	001	EPA 515.4	2,4-D
105.083	002	EPA 515.4	Dinoseb
105.083	003	EPA 515.4	Pentachlorophenol
105.083	004	EPA 515.4	Picloram
105.083	005	EPA 515.4	2,4,5-TP
105.083	006	EPA 515.4	Dalapon
105.083	007	EPA 515.4	Bentazon
105.083	008	EPA 515.4	Dicamba
105.083	009	EPA 515.4	Chlorinated Acids
105.090	001	EPA 525.2	Alachlor
105.090	002	EPA 525.2	Aldrin
105.090	003	EPA 525.2	Atrazine
105.090	004	EPA 525.2	Benzo(a)pyrene
105.090	005	EPA 525.2	Butachlor
105.090	006	EPA 525.2	Chlordane
105.090	007	EPA 525.2	Dieldrin
105.090	008	EPA 525.2	Di(2-ethylhexyl) Adipate
105.090	009	EPA 525.2	Di(2-ethylhexyl) Phthalate
105.090	010	EPA 525.2	4,4'-DDD
105.090	011	EPA 525.2	4,4'-DDE
105.090	012	EPA 525.2	4,4'-DDT
105.090	013	EPA 525.2	Endrin
105.090	014	EPA 525.2	Heptachlor
105.090	015	EPA 525.2	Heptachlor Epoxide
105.090	016	EPA 525.2	Hexachlorobenzene

Eurofins Eaton Analytical, Inc. (former M.W.H.)

Certificate No.: 01114CA
Renew Date: 1/31/2014

105.090	017	EPA 525.2	Hexachlorocyclopentadiene
105.090	018	EPA 525.2	Lindane
105.090	019	EPA 525.2	Methoxychlor
105.090	020	EPA 525.2	Metolachlor
105.090	021	EPA 525.2	Metribuzin
105.090	022	EPA 525.2	Molinate
105.090	023	EPA 525.2	Pentachlorophenol
105.090	024	EPA 525.2	Propachlor
105.090	025	EPA 525.2	Simazine
105.090	030	EPA 525.2	Adipates
105.090	031	EPA 525.2	Phthalates
105.090	034	EPA 525.2	Pesticides
105.101	001	EPA 531.2	Carbofuran
105.101	002	EPA 531.2	Oxamyl
105.101	003	EPA 531.2	Aldicarb
105.101	004	EPA 531.2	Aldicarb Sulfone
105.101	005	EPA 531.2	Aldicarb Sulfoxide
105.101	006	EPA 531.2	Carbaryl
105.101	007	EPA 531.2	3-Hydroxycarbofuran
105.101	008	EPA 531.2	Methomyl
105.120	001	EPA 547	Glyphosate
105.140	001	EPA 548.1	Endothall
105.150	001	EPA 549.2	Diquat
105.170	001	EPA 551.1	Bromochloroacetonitrile
105.170	005	EPA 551.1	Chloral Hydrate
105.170	007	EPA 551.1	Chloropicrin
105.170	008	EPA 551.1	Dibromoacetonitrile
105.170	010	EPA 551.1	1,2-Dibromo-3-chloropropane
105.170	011	EPA 551.1	1,2-Dibromoethane
105.170	012	EPA 551.1	Dichloroacetonitrile
105.170	013	EPA 551.1	1,1-Dichloro-2-propanone
105.170	015	EPA 551.1	Trichloroacetonitrile
105.170	018	EPA 551.1	1,1,1-Trichloro-2-propanone
105.175	001	EPA 551.1	Bromodichloromethane
105.175	002	EPA 551.1	Bromoform
105.175	003	EPA 551.1	Chloroform
105.175	004	EPA 551.1	Dibromochloromethane
105.175	005	EPA 551.1	Trihalomethanes
105.190	001	SM6251B	Bromoacetic Acid
105.190	002	SM6251B	Bromochloroacetic Acid

As of 1/9/2013 , this list supersedes all previous lists for this certificate number.
Customers: Please verify the current accreditation standing with the State.

Eurofins Eaton Analytical, Inc. (former M.W.H.)

Certificate No.: 01114CA
Renew Date: 1/31/2014

105.190	003	SM6251B	Chloroacetic Acid
105.190	005	SM6251B	Dibromoacetic Acid
105.190	006	SM6251B	Dichloroacetic Acid
105.190	007	SM6251B	Trichloroacetic Acid
105.190	008	SM6251B	Haloacetic Acids (HAA5)
105.191	001	SM6251B (20th)	Haloacetic Acids (HAA5)
105.201	001	EPA 552.3	Haloacetic Acids (HAA5)
105.201	002	EPA 552.3	Dalapon
105.230	001	EPA 1613	2,3,7,8-Tetrachlorodibenzo-p-dioxin (TCDD)
106 - Radiochemistry of Drinking Water			
106.010	001	EPA 900.0	Gross Alpha
106.010	002	EPA 900.0	Gross Beta
106.060	001	EPA 904.0	Radium-228
106.092	001	EPA 200.8	Uranium
106.270	001	SM7110C	Gross Alpha
106.610	001	SM7500-Rn	Radon-222
106.651	001	Georgia Inst. of Tech. rev 1.2	Radium-226
106.651	002	Georgia Inst. of Tech. rev 1.2	Radium-228
107 - Microbiology of Wastewater			
107.010	001	SM9215B	Heterotrophic Bacteria
107.020	001	SM9221B	Total Coliform
107.030	001	SM9221B	Total Coliform with Chlorine Present
107.040	001	SM9221C,E (MTF/EC)	Fecal Coliform
107.050	001	SM9221E	Fecal Coliform with Chlorine Present
107.100	001	SM9230B	Fecal Streptococci
107.100	002	SM9230B	Enterococci
107.245	001	SM9223	E. coli
108 - Inorganic Chemistry of Wastewater			
108.020	001	EPA 120.1	Conductivity
108.090	001	EPA 160.4	Residue, Volatile
108.110	001	EPA 180.1	Turbidity
108.112	001	EPA 200.7	Boron
108.112	002	EPA 200.7	Calcium
108.112	003	EPA 200.7	Hardness (calc.)
108.112	004	EPA 200.7	Magnesium
108.112	005	EPA 200.7	Potassium
108.112	006	EPA 200.7	Silica
108.112	007	EPA 200.7	Sodium
108.120	001	EPA 300.0	Bromide

As of 1/9/2013 , this list supersedes all previous lists for this certificate number.
Customers: Please verify the current accreditation standing with the State.

Eurofins Eaton Analytical, Inc. (former M.W.H.)

Certificate No.: 01114CA
Renew Date: 1/31/2014

108.120	002	EPA 300.0	Chloride
108.120	003	EPA 300.0	Fluoride
108.120	004	EPA 300.0	Nitrate
108.120	005	EPA 300.0	Nitrite
108.120	006	EPA 300.0	Nitrate-nitrite
108.120	008	EPA 300.0	Sulfate
108.183	001	EPA 335.4	Cyanide, Total
108.200	001	EPA 350.1	Ammonia
108.211	001	EPA 351.2	Kjeldahl Nitrogen
108.232	001	EPA 353.2	Nitrate-nitrite
108.232	002	EPA 353.2	Nitrite
108.260	001	EPA 365.1	Phosphate, Ortho
108.261	001	EPA 365.1	Phosphorus, Total
108.323	001	EPA 410.4	Chemical Oxygen Demand
108.360	001	EPA 420.1	Phenols, Total
108.362	001	EPA 420.4	Phenols, Total
108.385	001	SM2120B	Color
108.390	001	SM2130B	Turbidity
108.410	001	SM2320B	Alkalinity
108.420	001	SM2340B	Hardness (calc.)
108.430	001	SM2510B	Conductivity
108.440	001	SM2540B	Residue, Total
108.441	001	SM2540C	Residue, Filterable
108.442	001	SM2540D	Residue, Non-filterable
108.443	001	SM2540F	Residue, Settleable
108.465	001	SM4500-Cl G	Chlorine
108.473	001	SM4500-CN G	Cyanide, amenable
108.474	001	SM4500-CN F	Cyanide, Total
108.480	001	SM4500-F C	Fluoride
108.483	001	SM4500-F B	Fluoride
108.490	001	SM4500-H+ B	pH
108.493	001	SM4500-NH3 D or E (19th/20th)	Ammonia
108.498	001	SM4500-NH3 H (18th)	Ammonia
108.531	001	SM4500-O G	Dissolved Oxygen
108.540	001	SM4500-P E	Phosphate, Ortho
108.541	001	SM4500-P E	Phosphorus, Total
108.550	001	SM4500-Si D (18th/19th)	Dissolved Silica
108.551	001	SM4500-SiO2 C (20th)	Silica
108.580	001	SM4500-S= D	Sulfide
108.590	001	SM5210B	Biochemical Oxygen Demand

As of 1/9/2013 , this list supersedes all previous lists for this certificate number.
Customers: Please verify the current accreditation standing with the State.

Eurofins Eaton Analytical, Inc. (former M.W.H.)

Certificate No.: 01114CA
Renew Date: 1/31/2014

108.591	001	SM5210B	Carbonaceous BOD
108.602	001	SM5220D	Chemical Oxygen Demand
108.611	001	SM5310C	Total Organic Carbon
108.620	001	SM5320B	Total Organic Halides
108.640	001	SM5540C	Surfactants

109 - Toxic Chemical Elements of Wastewater

109.002	001	EPA 100.2	Asbestos
109.010	001	EPA 200.7	Aluminum
109.010	002	EPA 200.7	Antimony
109.010	004	EPA 200.7	Barium
109.010	005	EPA 200.7	Beryllium
109.010	007	EPA 200.7	Cadmium
109.010	009	EPA 200.7	Chromium
109.010	010	EPA 200.7	Cobalt
109.010	011	EPA 200.7	Copper
109.010	012	EPA 200.7	Iron
109.010	013	EPA 200.7	Lead
109.010	015	EPA 200.7	Manganese
109.010	016	EPA 200.7	Molybdenum
109.010	017	EPA 200.7	Nickel
109.010	021	EPA 200.7	Silver
109.010	024	EPA 200.7	Tin
109.010	026	EPA 200.7	Vanadium
109.010	027	EPA 200.7	Zinc
109.020	001	EPA 200.8	Aluminum
109.020	002	EPA 200.8	Antimony
109.020	003	EPA 200.8	Arsenic
109.020	004	EPA 200.8	Barium
109.020	005	EPA 200.8	Beryllium
109.020	006	EPA 200.8	Cadmium
109.020	007	EPA 200.8	Chromium
109.020	008	EPA 200.8	Cobalt
109.020	009	EPA 200.8	Copper
109.020	010	EPA 200.8	Lead
109.020	011	EPA 200.8	Manganese
109.020	012	EPA 200.8	Molybdenum
109.020	013	EPA 200.8	Nickel
109.020	014	EPA 200.8	Selenium
109.020	015	EPA 200.8	Silver
109.020	016	EPA 200.8	Thallium

As of 1/9/2013 , this list supersedes all previous lists for this certificate number.
Customers: Please verify the current accreditation standing with the State.

Eurofins Eaton Analytical, Inc. (former M.W.H.)

Certificate No.: 01114CA
Renew Date: 1/31/2014

109.020	017	EPA 200.8	Vanadium
109.020	018	EPA 200.8	Zinc
109.020	022	EPA 200.8	Tin
109.020	023	EPA 200.8	Titanium
109.104	001	EPA 218.6	Chromium (VI)
109.190	001	EPA 245.1	Mercury
109.809	002	SM3500-Cr B (20th)	Chromium (VI)
109.812	001	SM3500-Cr C (20th)	Chromium (VI)

110 - Volatile Organic Chemistry of Wastewater

110.040	001	EPA 624	Benzene
110.040	002	EPA 624	Bromodichloromethane
110.040	003	EPA 624	Bromoform
110.040	004	EPA 624	Bromomethane
110.040	005	EPA 624	Carbon Tetrachloride
110.040	006	EPA 624	Chlorobenzene
110.040	007	EPA 624	Chloroethane
110.040	008	EPA 624	2-Chloroethyl Vinyl Ether
110.040	009	EPA 624	Chloroform
110.040	010	EPA 624	Chloromethane
110.040	011	EPA 624	Dibromochloromethane
110.040	012	EPA 624	1,2-Dichlorobenzene
110.040	013	EPA 624	1,3-Dichlorobenzene
110.040	014	EPA 624	1,4-Dichlorobenzene
110.040	015	EPA 624	1,1-Dichloroethane
110.040	016	EPA 624	1,2-Dichloroethane
110.040	017	EPA 624	1,1-Dichloroethene
110.040	018	EPA 624	trans-1,2-Dichloroethene
110.040	019	EPA 624	1,2-Dichloropropane
110.040	020	EPA 624	cis-1,3-Dichloropropene
110.040	021	EPA 624	trans-1,3-Dichloropropene
110.040	022	EPA 624	Ethylbenzene
110.040	023	EPA 624	Methylene Chloride
110.040	024	EPA 624	1,1,2,2-Tetrachloroethane
110.040	025	EPA 624	Tetrachloroethene
110.040	026	EPA 624	Toluene
110.040	027	EPA 624	1,1,1-Trichloroethane
110.040	028	EPA 624	1,1,2-Trichloroethane
110.040	029	EPA 624	Trichloroethene
110.040	030	EPA 624	Trichlorofluoromethane
110.040	031	EPA 624	Vinyl Chloride

As of 1/9/2013 , this list supersedes all previous lists for this certificate number.
Customers: Please verify the current accreditation standing with the State.

Eurofins Eaton Analytical, Inc. (former M.W.H.)

Certificate No.: 01114CA
Renew Date: 1/31/2014

110.040	043	EPA 624	Other Volatile Organics
111 - Semi-volatile Organic Chemistry of Wastewater			
111.100	001	EPA 625	Acenaphthene
111.100	002	EPA 625	Acenaphthylene
111.100	003	EPA 625	Anthracene
111.100	004	EPA 625	Benzidine
111.100	005	EPA 625	Benz(a)anthracene
111.100	006	EPA 625	Benzo(b)fluoranthene
111.100	007	EPA 625	Benzo(k)fluoranthene
111.100	008	EPA 625	Benzo(g,h,i)perylene
111.100	009	EPA 625	Benzo(a)pyrene
111.100	010	EPA 625	Benzyl Butyl Phthalate
111.100	011	EPA 625	bis(2-chloroethoxy)methane
111.100	012	EPA 625	bis(2-chloroethyl) Ether
111.100	013	EPA 625	Bis(2-chloroisopropyl) Ether
111.100	014	EPA 625	Di(2-ethylhexyl) Phthalate
111.100	015	EPA 625	4-Bromophenyl Phenyl Ether
111.100	016	EPA 625	4-Chloro-3-methylphenol
111.100	017	EPA 625	2-Chloronaphthalene
111.100	018	EPA 625	2-Chlorophenol
111.100	019	EPA 625	4-Chlorophenyl Phenyl Ether
111.100	020	EPA 625	Chrysene
111.100	021	EPA 625	Dibenz(a,h)anthracene
111.100	025	EPA 625	3,3'-Dichlorobenzidine
111.100	026	EPA 625	2,4-Dichlorophenol
111.100	027	EPA 625	Diethyl Phthalate
111.100	028	EPA 625	2,4-Dimethylphenol
111.100	029	EPA 625	Dimethyl Phthalate
111.100	030	EPA 625	Di-n-butyl phthalate
111.100	031	EPA 625	Di-n-octyl phthalate
111.100	032	EPA 625	2,4-Dinitrophenol
111.100	033	EPA 625	2,4-Dinitrotoluene
111.100	034	EPA 625	2,6-Dinitrotoluene
111.100	035	EPA 625	Fluoranthene
111.100	036	EPA 625	Fluorene
111.100	037	EPA 625	Hexachlorobenzene
111.100	038	EPA 625	Hexachlorobutadiene
111.100	039	EPA 625	Hexachlorocyclopentadiene
111.100	040	EPA 625	Hexachloroethane
111.100	041	EPA 625	Indeno(1,2,3-c,d)pyrene

Eurofins Eaton Analytical, Inc. (former M.W.H.)

Certificate No.: 01114CA
Renew Date: 1/31/2014

111.100	042	EPA 625	Isophorone
111.100	043	EPA 625	2-Methyl-4,6-dinitrophenol
111.100	044	EPA 625	Naphthalene
111.100	045	EPA 625	Nitrobenzene
111.100	046	EPA 625	2-Nitrophenol
111.100	047	EPA 625	4-Nitrophenol
111.100	048	EPA 625	N-nitrosodimethylamine
111.100	049	EPA 625	N-nitroso-di-n-propylamine
111.100	050	EPA 625	N-nitrosodiphenylamine
111.100	051	EPA 625	Pentachlorophenol
111.100	052	EPA 625	Phenanthrene
111.100	053	EPA 625	Phenol
111.100	054	EPA 625	Pyrene
111.100	055	EPA 625	1,2,4-Trichlorobenzene
111.100	056	EPA 625	2,4,6-Trichlorophenol

112 - Radiochemistry of Wastewater

112.010	001	EPA 900.0	Gross Alpha
112.010	002	EPA 900.0	Gross Beta

Figure 4-5 Laboratory Certificate - State of California (ELAP)

CDPH
Public Health

CALIFORNIA STATE

ENVIRONMENTAL LABORATORY ACCREDITATION PROGRAM BRANCH

CERTIFICATE OF ENVIRONMENTAL ACCREDITATION

Is hereby granted to

MWH LABORATORIES, A DIVISION OF MWH AMERICAS, INC.

750 ROYAL OAKS DRIVE, SUITE 100

MONROVIA, CA 91016

Scope of the certificate is limited to the
"Fields of Testing"
which accompany this Certificate.

Continued accredited status depends on successful completion of on-site, proficiency testing studies, and payment of applicable fees.

This Certificate is granted in accordance with provisions of Section 100825, et seq. of the Health and Safety Code.

Certificate No.: **1422**

Expiration Date: **01/31/2013**

Effective Date: **02/01/2011**

Richmond, California
subject to forfeiture or revocation

George C. Kulasingam, Ph.D., Chief
Environmental Laboratory Accreditation Program Branch

Figure 4-6 California (ELAP) Field of Testing

State of California—Health and Human Services Agency
California Department of Public Health

RON CHAPMAN, MD, MPH
Director & State Health Officer

EDMUND G. BROWN JR.
Governor

January 5, 2012

Ed Wilson
MWH Laboratories, a Division of MWH Americas, Inc.
750 Royal Oaks Drive, Suite 100
Monrovia, CA 91016

Dear Ed Wilson: Certificate No. 1422

This is to advise you that the laboratory named above has been certified as an environmental testing laboratory pursuant to the provisions of the Health and Safety Code (HSC), Division 101, Part 1, Chapter 4, Section 100825, *et seq.*

The Fields of Testing for which this laboratory has been certified are indicated on the enclosed "Fields of Testing." The certificate shall remain in effect until **January 31, 2013** unless it is revoked. This certificate is subject to an annual fee as prescribed by HSC 100860.1(a).

The application for renewal of this certificate must be received before the expiration date of this certificate to remain in force according to the HSC 100845(a).

Any changes in laboratory location or structural alterations, which may affect adversely the quality of analysis in the Fields of Testing for which this laboratory has been granted a certificate, require prior notification. Notification is also required for changes in ownership or laboratory director within 30 days after the change (HSC, Section 100845(b) and (d)).

Your continued cooperation with the above requirements is essential for maintaining the high quality of the data produced by environmental laboratories certified by the State of California.

If you have any questions, please contact Rosalinda Lomboy at (818) 551-2014.

Sincerely,

Fred Choate for

George C. Kulasingam, Ph.D., Chief
Environmental Laboratory Accreditation Program Branch

Enclosure

CALIFORNIA DEPARTMENT OF PUBLIC HEALTH
ENVIRONMENTAL LABORATORY ACCREDITATION PROGRAM
Accredited Fields of Testing

MWH Laboratories, a Division of MWH Americas, Inc.
750 Royal Oaks Drive, Suite 100
Monrovia, CA 91016
Phone: (626) 386-1100

Certificate No.: 1422
Renew Date: 1/31/2013

Field of Testing: 101 - Microbiology of Drinking Water			
101.010	001	Heterotrophic Bacteria	SM9215B
101.020	001	Total Coliform	SM9221A,B
101.021	001	Fecal Coliform	SM9221E (MTF/EC)
101.022	001	E. coli	CFR 141.21(f)(6)(i) (MTF/EC+MUG)
101.060	002	Total Coliform	SM9223
101.060	003	E. coli	SM9223
101.070	002	Total Coliform	Colisure
101.070	003	E. coli	Colisure
101.120	001	Total Coliform (Enumeration)	SM9221A,B,C
101.130	001	Fecal Coliform (Enumeration)	SM9221E (MTF/EC)
101.160	001	Total Coliform (Enumeration)	SM9223
101.200	001	E. coli (Enumeration)	SM9223B
101.210	001	E. coli (Enumeration)	SM9221B.1/SM9221F
Field of Testing: 103 - Toxic Chemical Elements of Drinking Water			
103.130	018	Boron	EPA 200.7
103.140	018	Vanadium	EPA 200.8
103.310	001	Chromium (VI)	EPA 218.6
Field of Testing: 104 - Volatile Organic Chemistry of Drinking Water			
104.035	001	1,2,3-Trichloropropane	SRL 524M-TCP
104.050	007	tert-Butyl Alcohol (TBA)	EPA 524.2
104.050	008	Carbon Disulfide	EPA 524.2
104.050	009	Methyl Isobutyl Ketone	EPA 524.2
104.055	001	Benzene	EPA 524.3
104.055	002	Carbon Tetrachloride	EPA 524.3
104.055	003	Chlorobenzene	EPA 524.3
104.055	004	1,2-Dichlorobenzene	EPA 524.3
104.055	005	1,4-Dichlorobenzene	EPA 524.3
104.055	006	1,2-Dichloroethane	EPA 524.3
104.055	007	cis-1,2-Dichloroethene	EPA 524.3
104.055	008	trans-1,2-Dichloroethene	EPA 524.3
104.055	009	Dichloromethane	EPA 524.3
104.055	010	1,2-Dichloropropane	EPA 524.3
104.055	011	Ethylbenzene	EPA 524.3
104.055	012	Styrene	EPA 524.3
104.055	013	Tetrachloroethene	EPA 524.3

MWH Laboratories, a Division of MWH Americas, Inc.

Certificate No 1422
Renew Date: 1/31/2013

104.055	014	1,1,1-Trichloroethane	EPA 524.3	
104.055	015	Trichloroethene	EPA 524.3	
104.055	016	Toluene	EPA 524.3	
104.055	017	1,2,4-Trichlorobenzene	EPA 524.3	
104.055	018	1,1-Dichloroethene	EPA 524.3	
104.055	019	1,1,2-Trichloroethane	EPA 524.3	
104.055	020	Vinyl Chloride	EPA 524.3	
104.055	021	Xylenes, Total	EPA 524.3	
104.055	022	1,2-Dibromo-3-chloropropane	EPA 524.3	
104.055	023	1,2-Dibromoethane	EPA 524.3	
104.055	024	Trihalomethanes, Total	EPA 524.3	
104.055	025	Methyl Isobutyl Ketone	EPA 524.3	
104.055	026	Trichlorotrifluoroethane	EPA 524.3	
104.055	027	Nitrobenzene	EPA 524.3	
Field of Testing: 105 - Semi-volatile Organic Chemistry of Drinking Water				
105.090	028	Thiobencarb	EPA 525.2	
105.201	001	Haloacetic Acids (HAA5)	EPA 552.3	
105.201	002	Dalapon	EPA 552.3	
105.230	001	2,3,7,8-Tetrachlorodibenzo-p-dioxin (TCDD)	EPA 1613	
Field of Testing: 109 - Toxic Chemical Elements of Wastewater				
109.020	022	Tin	EPA 200.8	
Field of Testing: 114 - Inorganic Chemistry of Hazardous Waste				
114.010	001	Antimony	EPA 6010B	Aqueous Only
114.010	003	Barium	EPA 6010B	Aqueous Only
114.010	004	Beryllium	EPA 6010B	Aqueous Only
114.010	005	Cadmium	EPA 6010B	Aqueous Only
114.010	006	Chromium	EPA 6010B	Aqueous Only
114.010	007	Cobalt	EPA 6010B	Aqueous Only
114.010	008	Copper	EPA 6010B	Aqueous Only
114.010	009	Lead	EPA 6010B	Aqueous Only
114.010	010	Molybdenum	EPA 6010B	Aqueous Only
114.010	011	Nickel	EPA 6010B	Aqueous Only
114.010	013	Silver	EPA 6010B	Aqueous Only
114.010	015	Vanadium	EPA 6010B	Aqueous Only
114.010	016	Zinc	EPA 6010B	Aqueous Only
114.020	001	Antimony	EPA 6020	Aqueous Only
114.020	002	Arsenic	EPA 6020	Aqueous Only
114.020	003	Barium	EPA 6020	Aqueous Only
114.020	004	Beryllium	EPA 6020	Aqueous Only
114.020	005	Cadmium	EPA 6020	Aqueous Only
114.020	006	Chromium	EPA 6020	Aqueous Only
114.020	007	Cobalt	EPA 6020	Aqueous Only
114.020	008	Copper	EPA 6020	Aqueous Only

As of 1/10/2012 , this list supersedes all previous lists for this certificate number.
Customers: Please verify the current accreditation standing with the State.

MWH Laboratories, a Division of MWH Americas, Inc.

Certificate No 1422
Renew Date: 1/31/2013

114.020	009	Lead	EPA 6020	Aqueous Only
114.020	010	Molybdenum	EPA 6020	Aqueous Only
114.020	011	Nickel	EPA 6020	Aqueous Only
114.020	012	Selenium	EPA 6020	Aqueous Only
114.020	013	Silver	EPA 6020	Aqueous Only
114.020	014	Thallium	EPA 6020	Aqueous Only
114.020	015	Vanadium	EPA 6020	Aqueous Only
114.020	016	Zinc	EPA 6020	Aqueous Only
114.103	001	Chromium (VI)	EPA 7196A	Aqueous Only
114.106	001	Chromium (VI)	EPA 7199	Aqueous Only
114.140	001	Mercury	EPA 7470A	Aqueous Only
114.240	001	Corrosivity - pH Determination	EPA 9040B	Aqueous Only
Field of Testing: 116 - Volatile Organic Chemistry of Hazardous Waste				
116.010	000	EDB and DBCP	EPA 8011	Aqueous Only
116.080	000	Volatile Organic Compounds	EPA 8260B	Aqueous Only
116.080	120	Oxygenates	EPA 8260B	Aqueous Only
Field of Testing: 117 - Semi-volatile Organic Chemistry of Hazardous Waste				
117.110	000	Extractable Organics	EPA 8270C	Aqueous Only

Figure 4-7 LA County Fire Department License to Operate

LOS ANGELES COUNTY CERTIFIED UNIFIED PROGRAM AGENCY
ADMINISTERED BY LOS ANGELES COUNTY FIRE DEPARTMENT

UNIFIED PROGRAM FACILITY PERMIT

FISCAL YEAR: July 1, 2012 - June 30, 2013

ISSUED TO: MWH LABORATORIES DIV OF AMERIC
750 ROYAL OAKS DR
MONROVIA, CA 91016

LA Co. CUPA NO. AR: AR0036980

FACILITY OWNER: MWH GLOBAL INC
FACILITY SITE ADDRESS: 750 ROYAL OAKS DR # 100, MONROVIA, CA 91016

THIS PERMIT IS ISSUED FOR THE FOLLOWING PROGRAMS:

Administering Agency:	Program Description:
CITY OF MONROVIA	HAZARDOUS MATERIALS DISCLOSURE PROGRAM
LA COUNTY FIRE DEPARTMENT	HAZARDOUS WASTE GENERATOR PROGRAM
LA COUNTY FIRE DEPARTMENT	TIERED PERMIT - CONDITIONALLY EXEMPT (CE)

THIS PERMIT MUST BE CONSPICUOUSLY DISPLAYED AT THE FACILITY AT ALL TIMES.

ISSUED BY: Daryl L. Osby
County of Los Angeles Fire Chief

ISSUED ON: Nov 16, 2012

This permit is valid only for the above location and is subject to ALL REQUIRMENTS of State and Local Laws. The permit is non-transferrable and is void upon change in ownership or location.

Figure 4-8 Drug Enforcement Administration Certificate

EUROFINS EATON ANALYTICAL, INC
750 ROYAL OAKS DRIVE
SUITE 100
MONROVIA, CA 91016-0000-000

DEA REGISTRATION NUMBER	THIS REGISTRATION EXPIRES	FEE PAID
RE0438158	08-31-2013	$244

SCHEDULES	BUSINESS ACTIVITY	ISSUE DATE
3,3N,4,	ANALYTICAL LAB	12-03-2012

EUROFINS EATON ANALYTICAL, INC
750 ROYAL OAKS DRIVE
SUITE 100
MONROVIA, CA 91016-0000

CONTROLLED SUBSTANCE REGISTRATION CERTIFICATE
UNITED STATES DEPARTMENT OF JUSTICE
DRUG ENFORCEMENT ADMINISTRATION
WASHINGTON D.C. 20537

Sections 304 and 1008 (21 USC 824 and 958) of the Controlled Substances Act of 1970, as amended, provide that the Attorney General may revoke or suspend a registration to manufacture, distribute, dispense, import or export a controlled substance.

THIS CERTIFICATE IS NOT TRANSFERABLE ON CHANGE OF OWNERSHIP, CONTROL, LOCATION, OR BUSINESS ACTIVITY, AND IT IS NOT VALID AFTER THE EXPIRATION DATE.

CONTROLLED SUBSTANCE REGISTRATION CERTIFICATE
UNITED STATES DEPARTMENT OF JUSTICE
DRUG ENFORCEMENT ADMINISTRATION
WASHINGTON D.C. 20537

DEA REGISTRATION NUMBER	THIS REGISTRATION EXPIRES	FEE PAID
RE0438158	08-31-2013	$244

SCHEDULES	BUSINESS ACTIVITY	ISSUE DATE
3,3N,4,	ANALYTICAL LAB	12-03-2012

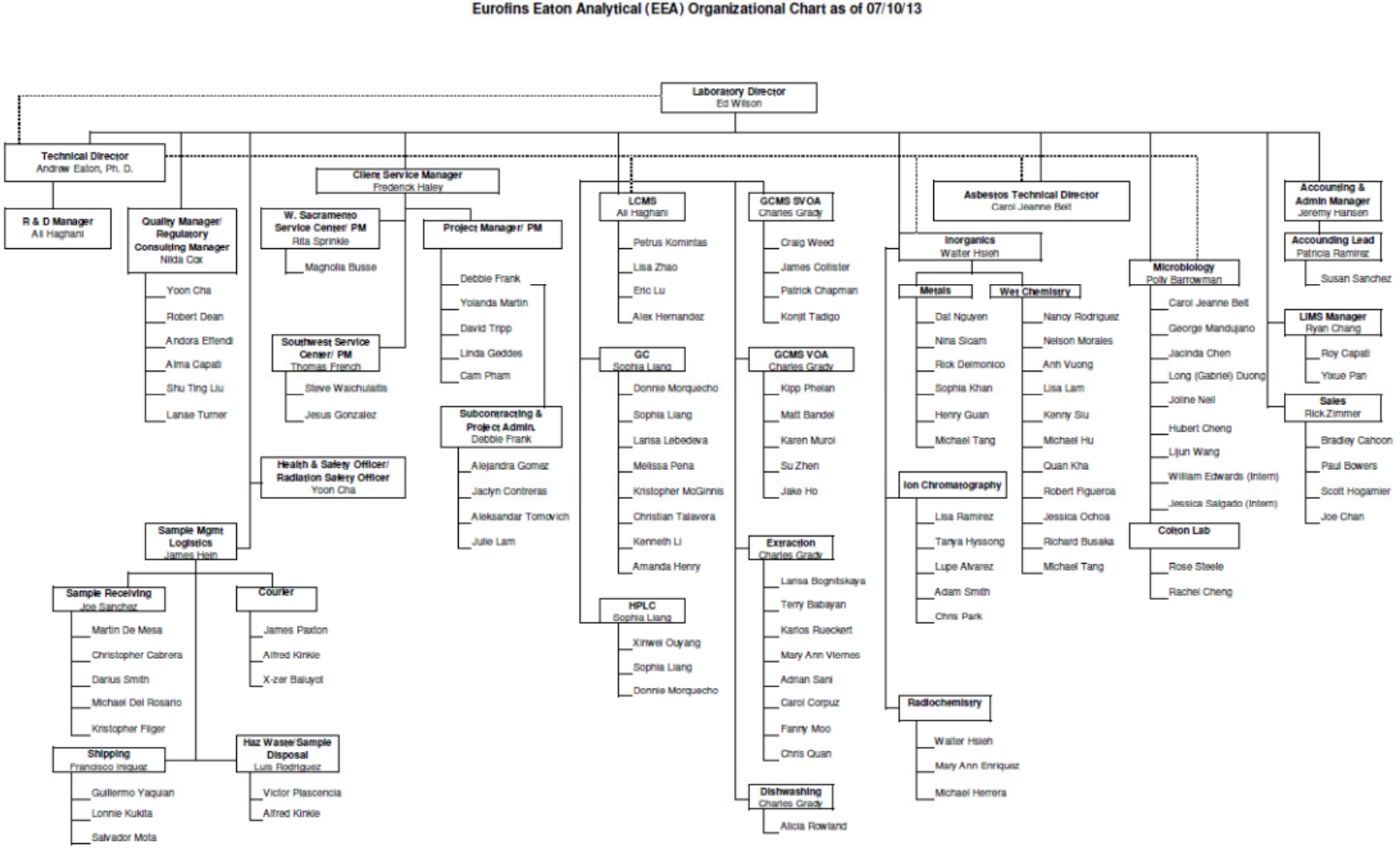
EUROFINS EATON ANALYTICAL, INC
750 ROYAL OAKS DRIVE
SUITE 100
MONROVIA, CA 91016-0000

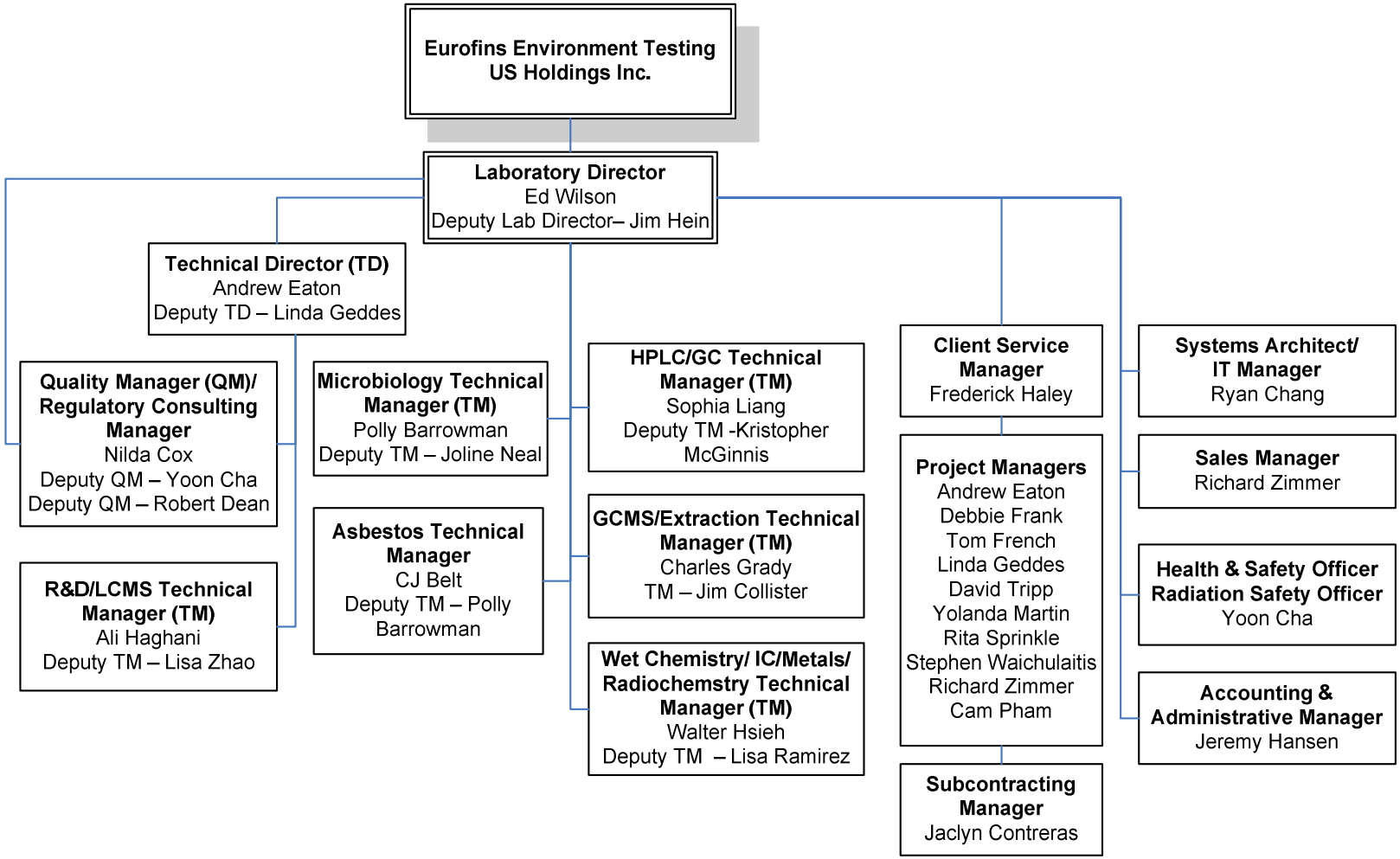
Sections 304 and 1008 (21 USC 824 and 958) of the Controlled Substances Act of 1970, as amended, provide that the Attorney General may revoke or suspend a registration to manufacture, distribute, dispense, import or export a controlled substance.

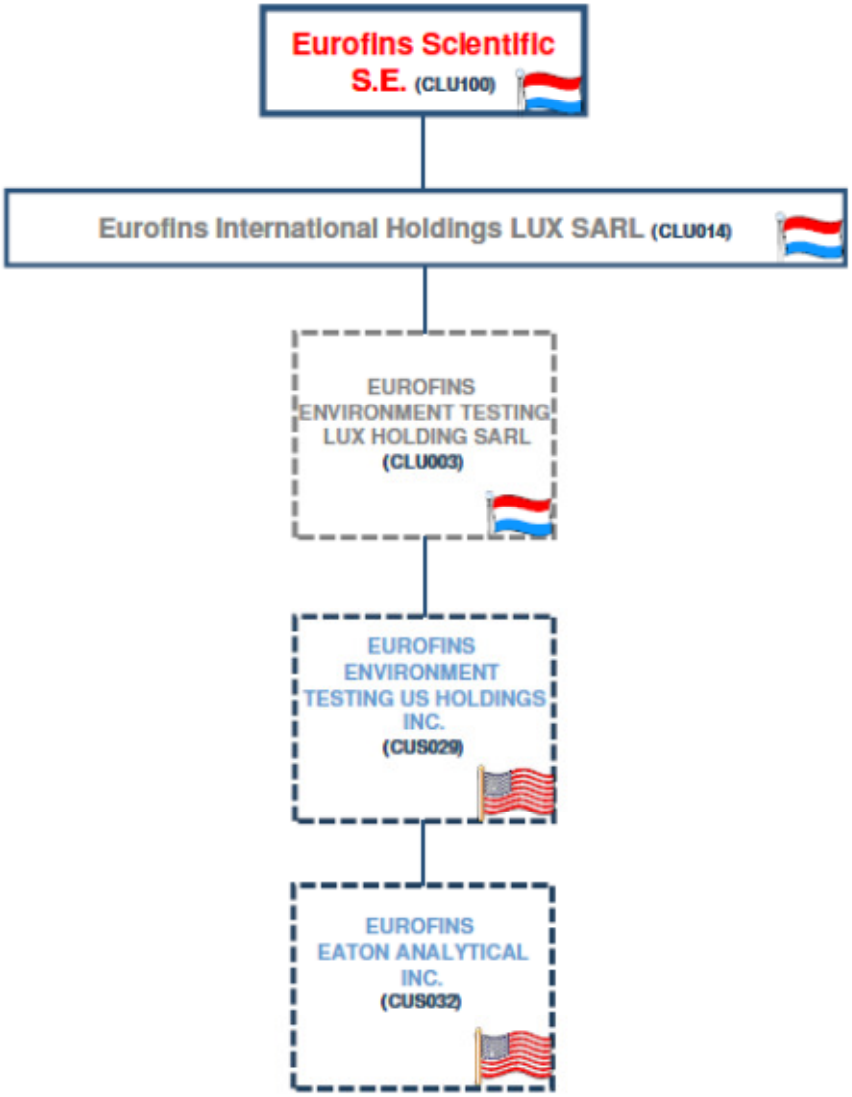
THIS CERTIFICATE IS NOT TRANSFERABLE ON CHANGE OF OWNERSHIP, CONTROL, LOCATION, OR BUSINESS ACTIVITY, AND IT IS NOT VALID AFTER THE EXPIRATION DATE.

Form DEA-223 (4/07)

Figure 4-9 EEA Organizational Chart




Eurofins Environment Testing US Holdings Inc.
Laboratory Director
Ed Wilson
Deputy Lab Director– Jim Hein
Technical Director (TD)
Andrew Eaton
Deputy TD – Linda Geddes
Quality Manager (QM)/ Regulatory Consulting Manager
Nilda Cox
Deputy QM – Yoon Cha
Deputy QM – Robert Dean
R&D/LCMS Technical Manager (TM)
Ali Haghani
Deputy TM – Lisa Zhao
Microbiology Technical Manager (TM)
Polly Barrowman
Deputy TM – Joline Neal
Asbestos Technical Manager
CJ Belt
Deputy TM – Polly Barrowman
HPLC/GC Technical Manager (TM)
Sophia Liang
Deputy TM -Kristopher McGinnis
GCMS/Extraction Technical Manager (TM)
Charles Grady
TM – Jim Collister
Wet Chemistry/ IC/Metals/ Radiochemstry Technical Manager (TM)
Walter Hsieh
Deputy TM – Lisa Ramirez
Client Service Manager
Frederick Haley
Project Managers
Andrew Eaton
Debbie Frank
Tom French
Linda Geddes
David Tripp
Yolanda Martin
Rita Sprinkle
Stephen Waichulaitis
Richard Zimmer
Cam Pham
Subcontracting Manager
Jaclyn Contreras
Systems Architect/ IT Manager
Ryan Chang
Sales Manager
Richard Zimmer
Health & Safety Officer Radiation Safety Officer
Yoon Cha
Accounting & Administrative Manager
Jeremy Hansen


Eurofins Scientific S.E. (CLU100)
Eurofins International Holdings LUX SARL (CLU014)
EUROFINS ENVIRONMENT TESTING LUX HOLDING SARL (CLU003)
EUROFINS ENVIRONMENT TESTING US HOLDINGS INC. (CUS029)
EUROFINS EATON ANALYTICAL INC. (CUS032)

5.0 QUALITY ASSURANCE OBJECTIVES

Before analytical data can be used, the quality of data produced by EEA is measured by the following characteristics: precision, accuracy, completeness, representativeness, comparability, timeliness, and documentation, used in the determination of the suitability of the data for a given purpose. EEA has set specific objectives for each of these characteristics as a means of meeting the data quality objectives of the client. A definition of each of the characteristics follows, along with the specific objectives for each of the characteristics.

Table 5-1 lists specific limit objectives for precision and accuracy for drinking water analyses.

Table 5-2 lists specific limit objectives for precision and accuracy for wastewater analyses.

Table 5-3 lists specific limit objectives for precision and accuracy for hazardous waste analyses.

Criteria for precision and accuracy included are only for representative reference methods. Criteria for the other methods and specific analytes can be found in relevant SOPs.

5.1. PRECISION

Analytical precision is an important component of overall data quality since it is a measure of how far an individual determination may be from the mean of replicate measurements (how well replicate analyses agree). If the precision of an analysis is poor, there is a good probability that the reported result will differ substantially from the true value even if there are no systematic errors leading to bias in the result. Precision is often directly related to concentration.

5.1.1. EEA uses Relative Percent Difference (RPD) to measure agreement between duplicate analyses. RPD is calculated as follows:

$$\textbf{RPD} = \frac{(S-D)}{(S+D)/2} \times 100$$

where;

RPD	=	Relative Percent Difference
S	=	First Sample Value (original)
D	=	Second Sample Value (duplicate)

5.1.2. The precision of a method is expressed as the Relative Standard Deviation (RSD) of the percent recoveries. Percent RSD (%RSD) is calculated as follows:

$$\%\mathbf{RSD} = \frac{S}{X_{avg}} \text{ X } 100$$

where:

X_{avg} = the arithmetic mean of the recovery values, and

$$S = \sqrt{\sum \frac{(X_i - X)^2}{n-1}}$$

where:

S	=	Standard Deviation
X_i	=	the individual recovery values
X	=	the arithmetic mean of the recovery values
n	=	the number of determinations

5.1.3. To assess precision in the laboratory, EEA uses the following:

- Duplicate Samples
- Duplicate Matrix Spikes
- Duplicate Laboratory Control Samples
- Control Charts

5.2. ACCURACY

Accuracy is the agreement between an experimentally determined value and the accepted reference value (deviation of the analytical value from the "true or known value). Analytical accuracy is a measure of analytical bias due to systematic errors. A measure of this bias along with a measure of the precision will provide the overall accuracy of the results.

The true value for field samples are never known, so accuracy measurements are made on the analysis of QC samples analyzed with field samples. The primary QC tools for assessing accuracy are control standards (LCSs), matrix spikes and spike duplicates (MS/MSD), and surrogate spikes.

5.2.1. Spike recoveries are calculated as follows:

$$\%R = \frac{SSR - SR}{SA} \times 100$$

Where;

%R	=	percent spike recovery
SSR	=	spiked sample result
SR	=	sample result
SA	=	spike amount added

5.2.2. For Laboratory Control Samples, percent recovery (%R) is calculated as follows:

$$\%R = \frac{\text{found concentration}}{\text{true concentration}} \times 100$$

5.2.3. Accuracy is monitored for nearly all methods by percent recoveries of the LCSs and plotted on control charts. The mean recovery ± 2 standard deviations are the warning limits, and the mean recovery ± 3 standard deviations are the control limits. In the event that the method has no acceptance criteria, control charts are reviewed and evaluated to establish internal limits or guidelines [TNI-EL-V1M4-2009-1.7.4.2.a].

To assess accuracy, EEA uses the following:

- MRL Checks
- Laboratory Control Samples
- Matrix Spikes
- Certified Reference Materials
- Blind QC Samples
- Control Charts

5.3. REPRESENTATIVENESS/SAMPLING OF SUB-ALIQUOT

All sample aliquots, which are analyzed, must be representative of the bulk sample from which they are taken [TNI-EL-V1M2-2009-5.7.1][ISO/IEC 17025:2005(E)-5.7.1]. Representativeness is easily achieved for aqueous samples free of suspended material. Obtaining a representative sample is a more difficult task for soils and sludge.

Unless a sample is known to be non-randomly heterogeneous in its composition, the most appropriate manner of obtaining a representative aliquot for analysis is by simple random sampling after the material has been mixed as thoroughly as possible. Thorough mixing is acceptable for inorganic analyses, but any samples requiring volatile or semi-volatile organic analyses must be handled in a manner which minimizes loss of these volatile compounds from the sample.

Representativeness is also impacted by conditions of sample receipt. EEA documents all samples that do not meet acceptance criteria (TNI-EL-V1M2-2009-5.8.3) (ISO/IEC 17025:2005(E)-5.8.3).

The laboratory documents the sampling techniques of aliquots from a submitted sample in the method SOPs to ensure that representativeness of samples are obtained. (TNI-EL-V1M2-2009-5.7.1)(ISO/IEC 17025:2005(E)-5.7.1).

5.4. COMPARABILITY

The characteristic of comparability determines whether analytical conditions are uniform for each analytical run to insure that all of the reported data will be consistent. This requires temporal stability of analytical conditions within the laboratory.

To insure temporal stability, uniform analytical and quality control protocols will be closely adhered to for each analytical run. In addition, traceable standards are used as part of every analytical run. Every analyst is required to demonstrate his precision and accuracy for a particular analysis by analyzing four replicate matrix spiked samples. All newly trained or backup analysts must demonstrate comparable precision and accuracy.

5.5. COMPLETENESS

The characteristic of completeness is a measure of the percentage of specified data which are valid. Valid data are obtained when samples are analyzed in accordance with the quality control procedures outlined in this manual and none of the quality control criteria is exceeded.

Sample data which does not meet the specified quality control criteria will automatically be reanalyzed if sufficient quantity of sample is available and analytical holding times have not been exceeded. The laboratory strives for a completeness percentage of 100%.

5.6. TIMELINESS

EPA guidelines require that samples be analyzed for constituents within specified holding times. These holding times represent a compromise between allowance of a realistic time to perform the analysis and minimization of elapsed time to insure sample integrity.

EEA has adopted a computerized sample tracking system and supervisory review process to insure that samples are scheduled for extraction and analysis within the EPA holding times. In the unforeseen circumstance of instrument performance problems, EEA will do everything possible to meet EPA holding times without compromising the quality of the reported data. The client is notified if a holding time is exceeded.

5.7. DOCUMENTATION

Proper documentation is a vital component in supporting the integrity of analytical results. All of the proceeding quality control components will not support reported data unless they have been fully documented for subsequent review. EEA maintains documentation of sample handling, chain of custody (if applicable), analytical procedures, raw and calculated data, supporting chromatograms, quality control data, and final reports. Please see section 14 for data reduction, validation, and reporting procedures.

NOTE: When the method does not specify the Acceptance Limit, recovery limits are based on control charts.

Table 5-1 Precision and Accuracy for Drinking Water for Mid or High Level Spikes

(A) Inorganics - Wet Chemistry

Parameter Method Name	Method Number	Parameters/ Analytes	Accuracy		Precision RPD Maximum
			LCS/LFB	MS/LFM	
			% Rec.	% Rec.	
Alkalinity	SM 2320B	Bicarbonate	90 - 110	80 - 120	10
		Carbonate	90 - 110	80 - 120	10
		Hydroxide	90 - 110	80 - 120	10
Bromate, BrO3	EPA 317	Bromate	90 - 110	75 - 125	20
Bromate, BrO3	EPA 300.1	Bromate	90 - 110	75 - 125	20
Bromide, Br	EPA 300.0	Bromide	90 - 110	80 - 120	20
Bromide, Br	EPA 300.1	Bromide	90 - 110	85 - 115	20
Chloride, Cl	EPA 300.0	Chloride	90 - 110	80 - 120	20
Chlorine Dioxide	SM 4500-ClO2 D	Chlorine Dioxide	85 - 115	85 - 115	15
Chlorite, ClO2	EPA 300.0	Chlorite	90 - 110	80 - 120	20
Chlorite, ClO2	EPA 300.1	Chlorite	90 - 110	85 - 115	20
Chlorite, ClO2	EPA 317.0	Chlorite	90 - 110	85 - 115	20
Chlorate, ClO3	EPA 300.0	Chlorate	90 - 110	80 - 120	20
Chlorate, ClO3	EPA 300.1	Chlorate	90 - 110	85 - 115	20
Color	SM 2120B	Color	-	-	+1 unit (0-10)
					+5 units (10-110)
					+10 units (>110)
Conductivity	SM2510B	Conductivity	95 - 105	-	20
Corrosivity (Langlier Index)	SM 2330B	Corrosivity	85 - 115	85 - 115	15
Cyanide	SM4500CN-F, G	Cyanide	80 - 120	80 - 120	20
	EPA335.4	Cyanide	90 - 110	90 - 110	20
Fluoride	SM 4500 F-C	Fluoride	81 - 116	73 - 124	20
Free & Total Chlorine	SM 4500 Cl G	Free & Total Chlorine	85 - 115	85 - 115	15
Hardness	EPA 200.7/SM 2340B	Calcium Hardness	-	-	-
Nitrate	EPA300.0/353.2	Nitrate	90 - 110	80 - 120	20
Nitrate & Nitrite	EPA 353.2	Nitrate & Nitrite	90 - 110	90 - 110	20
Nitrite	EPA300.0	Nitrite	90 - 110	80 - 120	20
	EPA353.2	Nitrite	90 - 110	90 - 110	20
Odor	SM 2150B	Odor	-	-	20
o-Phosphate	365.1	o-Phosphate	90 - 110	90 - 110	20
	SM4500 P-E, PF	o-Phosphate	90 - 110	90 - 110	20

Parameter Method Name	Method Number	Parameters/ Analytes	Accuracy		Precision RPD Maximum
			LCS/LFB	MS/LFM	
			% Rec.	% Rec.	
Perchlorate	EPA 314.0	Perchlorate	85 - 115	80 - 120	15
pH	SM4500-HB	pH	98 - 102	-	+ 0.1 pH unit
Phenols	EPA 420.1/420.4	Phenols	70 - 130	70 - 130	10
Residual Disinfectant (Total/ Free Residual Chlorine)	SM4500 Cl-G	Residual Disinfectant	85 - 115	-	20
Silica	EPA200.7	Silica	85 - 115	70 - 130	-
	SM 4500 SiO2C	Dissolved /Reactive Silica	85 - 115	70 - 130	20
Total Dissolved Solids (TDS)	SM 2540C	Total Dissolved Solids (TDS)	80 - 114	-	20
Total Suspended Solids (TSS)	SM 2540D	Total Suspended Solids (TSS)	80 - 120	-	10
Sulfate	EPA 300.0	Sulfate	90 - 110	80 - 120	20
Total Organic Carbon	SM 5310C/EPA 415.3	TOC	80 - 120	80 - 120	20
Dissolved Organic Carbon	SM 5310C/EPA 415.3	DOC	90 - 110	80 - 120	20
Turbidity	EPA180.1	Turbidity	90 - 110	N/A	20
UV 254	SM 5910 B/EPA 415.3	UV/SUVA	82 - 134	N/A	15 (6.0 mg/L/DOC)

(B) Inorganics – Metals

Parameter Method Name	EPA Method Number	Parameters/ Analytes	Accuracy		Precision RPD Maximum
			LCS/LFB	MS/LFM	
			% Rec	% Rec	
Chromium VI	EPA 218.6	Chromium VI (Dissolved)	90 - 110	90 - 110	20
Mercury	EPA 245.1	Mercury, Hg	85 - 115	70 - 130	20
Metals	EPA200.7	Aluminum, Al	85 - 115	70 - 130	20
		Barium, Ba	85 - 115	70 - 130	20
		Beryllium, Be	85 - 115	70 - 130	20
		Boron, B	85 - 115	70 - 130	20
		Calcium, Ca	85 - 115	70 - 130	20
		Cadmium, Cd	85 - 115	70 - 130	20
		Chromium, Cr	85 - 115	70 - 130	20
		Copper, Cu	85 - 115	70 - 130	20
		Iron, Fe	85 - 115	70 - 130	20
		Magnesium, Mg	85 - 115	70 - 130	20

Parameter Method Name	EPA Method Number	Parameters/ Analytes	Accuracy		Precision RPD Maximum
			LCS/LFB	MS/LFM	
			% Rec	% Rec	
Metals (con't.)	EPA200.7	Manganese, Mn	85 - 115	70 - 130	20
		Nickel, Ni	85 - 115	70 - 130	20
		Potassium, K	85 - 115	70 - 130	20
		Silica, SiO2	85 - 115	70 - 130	20
		Silver, Ag	85 - 115	70 - 130	20
		Sodium, Na	85 - 115	70 - 130	20
		Thallium, Ti	85 - 115	70 - 130	20
		Tin, Sn	85 - 115	70 - 130	20
		Zinc, Zn	85 - 115	70 - 130	20
Metals	EPA200.8	Aluminum, Al	85 - 115	70 - 130	20
		Antimony, Sb	85 - 115	70 - 130	20
		Arsenic, As	85 - 115	70 - 130	20
		Barium, Ba	85 - 115	70 - 130	20
		Beryllium, Be	85 - 115	70 - 130	20
		Cadmium, Cd	85 - 115	70 - 130	20
		Chromium, Cr	85 - 115	70 - 130	20
		Copper, Cu	85 - 115	70 - 130	20
		Lead, Pb	85 - 115	70 - 130	20
		Manganese, Mn	85 - 115	70 - 130	20

(C) Microbiology/Microscopy Tests

Parameter Method Name	Method Number	Analyte Parameter	Accuracy		Precision RPD Maximum
			LCS/LFB	MS/LFM	
			% Rec.	% Rec.	
Asbestos	EPA 100.2	Asbestos	-	-	-
Fecal Coliforms--EC Medium, MTF	SM9221E	Fecal Coliforms EC Medium (Enumeration)	-	-	-
Heterotrophic Plate Count (Standard Plate Count)	SM9215B	Heterotrophic Plate Count	-	-	10
Total Coliform by Multiple Tube Fermentation (MF)	SM9221AB	Total Coliform/Enumeration	-	-	-
Total Coliform/ E-Coli (Colilert)	SM 9223B	Total Coliforms (Present or Absent)	-	-	-
Total Coliform/Colilert (Enumeration)	SM 9223B	Total Coliform (Enumeration)	-	-	-
Total Coliforms (MTF) Enumeration	SM9221A, B	Total Coliforms	-	-	-

Parameter Method Name	Method Number	Analyte Parameter	Accuracy		Precision RPD
Total Coliform and E-Coli (Colisure)	SM 9223B	Total Coliform and E-Coli	-	-	-
Coliphage	1602	Coliphage	-	-	-

(D) Organics

Parameter Method Name	EPA Method Number	Parameters/ Analytes	Accuracy		Precision RPD Maximum
			LCS/LFB	MS/LFM	
			% Rec.	% Rec.	
DBCP/EDB	EPA504.1	1,2-Dibromo-3-chloropropane	70 - 130	60 - 140	20
		1,2-Dibromoethane (EDB)	70 - 130	60 - 140	20
		1,2,3-Trichloropropane (1,2,3-TCP)	70 - 130	60 - 140	20
Organohalide Pesticides and Commercial Polychlorinated Biphenyl (PCB)	EPA 505	Alachlor	70 - 130	65 - 135	20
		Aldrin	70 - 130	65 - 135	20
		Chlordane	70 - 130	65 - 135	20
		Dieldrin	70 - 130	65 - 135	20
		Endrin	70 - 130	65 - 135	20
		Heptachlor	70 - 130	65 - 135	20
		Heptachlor Epoxide	70 - 130	65 - 135	20
		Lindane	70 - 130	65 - 135	20
		Methoxychlor	70 - 130	65 - 135	20
		Cis-Nonachlor	70 - 130	65 - 135	20
		Trans-Nonachlor	70 - 130	65 - 135	20
		Toxaphene	70 - 130	65 - 135	20
		Aroclor 1016	58 - 145	65 - 135	20
		Aroclor 1221	65 - 132	65 - 135	20
		Aroclor 1232	56 - 152	65 - 135	20
		Aroclor 1242	70 - 130	65 - 135	20
		Aroclor 1248	63 - 130	65 - 135	20
		Aroclor 1254	78 - 136	65 - 135	20
		Aroclor 1260	70 - 130	65 - 135	20
Chlorinated Acids	EPA 515.4	2,4,5-TP (Silvex)	70 - 130	70 - 130	30
		2,4,5-T	70 - 130	70 - 130	30
		2,4-D	70 - 130	70 - 130	30
		2,4-DB	70 - 130	70 - 130	30
		Acifluorfen	70 - 130	70 - 130	30
		DCPA	70 - 130	70 - 130	30
		Dichloroprop	70 - 130	70 - 130	30
		Dinoseb	70 - 130	70 - 130	30
		4-Nitrophenol	-	-	30
		Pentachlorophenol	70 - 130	70 - 130	30
		Picloram	70 - 130	70 - 130	30

Parameter Method Name	EPA Method Number	Parameters/ Analytes	Accuracy		Precision RPD Maximum
			LCS/LFB	MS/LFM	
			% Rec.	% Rec.	
Chlorinated Acids (con't.)	EPA 515.4	2,4-Dichlorophenylacetic Acid	70 - 130	70 - 130	30
		3,5-Dichlorobenzoic Acid	70 - 130	70 - 130	30
		Bentazon	70 - 130	70 - 130	30
		Dalapon	70 - 130	70 - 130	30
		Dicamba	70 - 130	70 - 130	30
Purgeable Organic Compounds/ Halogenated & Aromatic Volatiles/ Trihalomethanes, Di-Isopropyl Ether (DIPE), Tertiary Amyl methyl Ether (TAME) Tert-Butyl ethyl ether (ETBE)	EPA524.2	1,1,1-Trichloroethane	70 - 130	70 - 130	20
		1,1,2,2-Tetrachloroethane	70 - 130	70 - 130	20
		1,1,1,2-Tetrachloroethane	70 - 130	70 - 130	20
		1,1,2-Trichloroethane	70 - 130	70 - 130	20
		1,1-Dichloroethane	70 - 130	70 - 130	20
		1,1-Dichloroethylene	70 - 130	70 - 130	20
		1,2,3-Trichlorobenzene	70 - 130	70 - 130	20
		1,2,4 Trichlorobenzene	70 - 130	70 - 130	20
		1,2,3- Trichloropropane	70 - 130	70 - 130	20
		1,2,4- Trimethylbenzene	70 - 130	70 - 130	20
		1,3,5 Trimethyl benzene	70 - 130	70 - 130	20
		1,1-Dichloropropene	70 - 130	70 - 130	20
		1,2-Dichloropropane	70 - 130	70 - 130	20
		1,3-Dichloropropane	70 - 130	70 - 130	20
		2,2-Dichloropropane	70 - 130	70 - 130	20
		Benzene	70 - 130	70 - 130	20
		Bromobenzene	70 - 130	70 - 130	20
		Bromochloromethane	70 - 130	70 - 130	20
		Bromodichloromethane	70 - 130	70 - 130	20
		Bromoform	70 - 130	70 - 130	20
		Bromomethane	70 - 130	70 - 130	20
		Carbon Tetrachloride	70 - 130	70 - 130	20
		Chlorobenzene	70 - 130	70 - 130	20
		Chlorodibromomethane	70 - 130	70 - 130	20
		Chloroform (Trichloromethane)	70 - 130	70 - 130	20
		Chloroethane	70 - 130	70 - 130	20
		Chloromethane (Methyl Chloride)	70 - 130	70 - 130	20
		Dichloromethane	70 - 130	70 - 130	20
		Dibromomethane	70 - 130	70 - 130	20
		Dichlorodifluoromethane	70 - 130	70 - 130	20
		Ethylbenzene	70 - 130	70 - 130	20
		Fluorotrichloromethane (Freon	70 - 130	70 - 130	20
		Hexachlorobutadiene	70 - 130	70 - 130	20

Parameter Method Name	EPA Method Number	Parameters/ Analytes	Accuracy		Precision RPD Maximum
			LCS/LFB % Rec.	MS/LFM % Rec.	
Purgeable Organic Compounds/ Halogenated & Aromatic Volatiles/ Trihalomethanes, Di-Isopropyl Ether (DIPE), Tertiary Amyl methyl Ether (TAME) Tert-Butyl ethyl ether (ETBE) (con't.)	EPA524.2	Isopropylbenzene	70 - 130	70 - 130	20
		Methyl Tert-Butyl Ether (MTBE)	70 -130	70 - 130	20
		m-Dichlorobenzene (1,3-DCB)	70 -130	70 - 130	20
		Naphthalene	70 -130	70 - 130	20
		n-Butylbenzene	70 -130	70 - 130	20
		n-Propylbenzene	70 -130	70 - 130	20
		Styrene	70 -130	70 - 130	20
		Tetrachloroethylene (PCE)	70 -130	70 - 130	20
		Tert-Butyl Alcohol (TBA)	70 -130	70 - 130	20
		Carbon Disulfide	70 -130	70 - 130	20
		Methyl Isobutyl Ketone (MIBK)	70 -130	70 - 130	20
		Toluene	70 -130	70 - 130	20
		Trichloroethylene	70 -130	70 - 130	20
		1,1,2-Trichloro-1,2,2-trifluoroethane (Freon 113)	70 -130	70 - 130	20
		Vinyl Chloride	70 -130	70 - 130	20
		cis-1,2-Dichloroethylene	70 -130	70 - 130	20
		cis-1,3-Dichloropropene	70 -130	70 - 130	20
		sec-Butylbenzene	70 -130	70 - 130	20
		m,p-Xylenes	70 -130	70 - 130	20
		1,2-Dichlorobenzene	70 -130	70 - 130	20
		o-Chlorotoluene	70 -130	70 - 130	20
		o-Xylene	70 -130	70 - 130	20
		p-Chlorotoluene	70 -130	70 130	20
		p-Isopropyltoluene	70 -130	70 - 130	20
		1,4-Dichlorobenzene	70 -130	70 - 130	20
		2-Butanone (MEK)	70 -130	56 - 85	20
		4-Methyl-2-Pentanone	70 -130	70 - 130	20
		trans-1,2-Dichloroethylene	70 -130	85 - 129	20
		trans-1,3-Dichloropropene	70 -130	80 - 131	20
		tert-Butylbenzene	70 -130	70 - 130	20
		Di-Isopropyl Ether (DIPE)	70 -130	70 - 130	20
		Tertiary Amyl methyl ether (TAME)	70 -130	70 130	20
		Tertiarry Butyl ethyl Ether (ETBE)	70 -130	70 130	20
		Nitrobenzene	80 -120	70 - 130	20
		Hexachloroethane	80 -120	70 - 130	20
		1,2-Dichlorobenzene	80 -120	70 - 130	20

Parameter Method Name	EPA Method Number	Parameters/ Analytes	Accuracy		Precision RPD Maximum
			LCS/LFB % Rec.	MS/LFM % Rec.	
(con't.)	EPA 524.2	1,2-Dichloroethane	80 - 120	70 - 130	20
TCP-Low (5ppt)	CA DHS SRLPT/ GCMS	1,2,3-Trichloropropane	80 - 120	-	20
Semi-Volatile Organics Acid/Base Neutrals	EPA 525.2	Acenaphthylene	70 - 130	70 - 130	20*
		Alachlor	70 - 130	70 - 130	20*
		Aldrin	70 - 130	70 - 130	20*
		Anthracene	70 - 130	70 130	20*
		Atrazine	70 - 130	70 130	20*
		Benzo(a)anthracene	70 - 130	70 - 130	20*
		Benzo(a)pyrene	70 - 130	70 - 130	20*
		Benzo(b)fluoranthene	70 - 130	70 - 130	20*
		Benzo(g,h,i)perylene	70 - 130	70 130	20*
		Benzo(k)fluoranthene	70 - 130	70 130	20*
		Butylbenzylphthalate	70 - 130	80 - 131	20*
		Caffeine	70 - 130	70 - 130	-
		a-Chlordane	70 - 130	70 - 130	-
		g-Chlordane	70 - 130	70 130	20*
		Chrysene	70 - 130	70 130	20*
		Di-(2-Ethylhexyl)phthalate	70 - 130	70 - 130	20*
		Di-(2-Ethylhexyl)adipate	70 - 130	70 - 130	20*
		Di-n-Butylphthalate	70 - 130	70 - 130	20*
		Dibenzo(a,h)anthracene	70 - 130	70 - 130	20*
		Diethylphthalate	70 - 130	70 - 130	20*
		Dimethylphthalate	70 - 130	70 - 130	20*
		Endrin	70 - 130	70 - 130	20*
		Fluorene	70 - 130	70 - 130	20*
		Butachlor	70 - 130	70 - 130	20*
		4,4-DDD	70 - 130	70 - 130	20*
		4,4-DDE	70 - 130	70 - 130	20*
		4,4-DDT	70 - 130	70 - 130	20*
		Metolachlor	70 - 130	70 - 130	20*
		Metribuzin	70 - 130	70 - 130	20*
		Propachlor	70 - 130	70 - 130	20*
		Heptachlor	70 - 130	70 - 130	20*
		Heptachlor Expoxide	70 - 130	70 - 130	20*
		Hexachlorobenzene	70 - 130	70 - 130	20*
		Hexachlorocyclopentadiene	70 - 130	70 - 130	20*
		Indeno(1,2,3,c,d)pyrene	70 - 130	70 - 130	20*

Parameter Method Name	EPA Method Number	Parameters/ Analytes	Accuracy		Precision RPD Maximum
			LCS/LFB % Rec.	MS/LFM % Rec.	
Semi-Volatile Organics Acid/Base Neutrals (con't.)	EPA 525.2	Lindane	70 - 130	70 - 130	20*
		Methoxychlor	70 - 130	70 - 130	20*
		Molinate	70 - 130	70 - 130	20*
		Pentachlorophenol	70 - 130	70 - 130	20*
		Phenanthrene	70 - 130	70 - 130	20*
		Pyrene	70 - 130	70 - 130	20*
		Simazine	70 - 130	70 - 130	20*
		Thiobencarb	70 - 130	70 - 130	20*
		trans-Nonachlor	70 - 130	70 - 130	20*
		Perylene-d12 (surr)	70 - 130	70 - 130	-
N-methylcarbamoyloximes and N-Methylcarbamates	EPA531.2	3-Hydroxycarbofuran	70 - 130	70 - 130	20
		Aldicarb (Temik)	70 - 130	70 - 130	20
		Aldicarb Sulfone	70 - 130	70 - 130	20
		Aldicarb Sulfoxide	70 - 130	70 - 130	20
		Baygon	70 - 130	70 - 130	20
		Carbaryl	70 - 130	70 - 130	20
		Carbofuran (Furadan)	70 - 130	70 - 130	20
		Methiocarb	70 - 130	70 - 130	20
		Methomyl	70 - 130	70 - 130	20
		Oxamyl (Vydate)	70 - 130	70 - 130	20
		4-Bromo-3,5-Dimethylphenyl-N-Methylcarbamate (BDMC)	70 - 130	70 - 130	20
Glyphosate	EPA547	Glyphosate	70 - 130	70 - 130	-
Endothall	EPA548.1	Endothall	63 - 144	38 - 157	-
Diquat & Paraquat	EPA549.2	Diquat	80 - 120	70 - 130	20
		Paraquat	80 - 120	70 - 130	20
Trihalomethanes, Chloral Hydrate, Haloacetonitrile, EDB (1-2,dibromoethane) DBCP (1,2-dibromo-3-chloropropane	551.1	Bromodichloromethane	80 - 120	80 - 120	20
		Bromoform	80 - 120	80 - 120	20
		Chloral Hydrate	80 - 120	80 - 120	20
		Chloroform	80 - 120	80 - 120	20
		Dibromochloromethane	80 - 120	80 - 120	20
		Dibromoacetonitrile	80 - 120	80 - 120	20
		Dichloroacetonitrile	80 - 120	80 - 120	20
		1,1-Dichloro-2-propanone	80 - 120	80 - 120	20
		Trichloroacetonitrile	80 - 120	80 - 120	20
		1,1-Trichloro-2-propanone	80 - 120	80 - 120	20
		EDB (1-2,dibromoethane)	80 - 120	80 – 120	20
		DBCP (1,2-dibromo-3-chloropropane	80 – 120	80 - 120	20

Parameter Method Name	EPA Method Number	Parameters/ Analytes	Accuracy		Precision RPD Maximum
			LCS/LFB % Rec.	MS/LFM % Rec.	
Haloacetic Acids **	SM6251B	Bromochloroacetic Acid	85 -115	84 - 123	20
		Chlorodibromoacetic Acid	85 -115	70 - 130	-
		Dibromoacetic Acid	85 -115	84 - 122	20
		Dichloroacetic Acid	85 -115	79 - 123	20
		Monobromoacetic Acid	85 -115	81 - 122	20
		Monochloroacetic Acid	85 -115	75 - 126	20
		Tribromoacetic Acid	85 -115	70 - 130	
		Trichloroacetic Acid	85 -115	82 - 124	20

* RPD-LCS
** Low Level LFB/LCS 50-150 % Recovery

(E) Radiochemistry

Parameter Method Name	EPA Method Name	Parameters/ Analytes	Accuracy		Precision RPD Maximum
			LCS/LFB % Rec.	MS/LFM % Rec.	
Uranium	EPA 200.8 (Screen)	Uranium	85 - 115	80 - 120	20
Gross Alpha	EPA 900.0	Gross Alpha	80 - 120	70 - 130	20
Gross Beta	EPA 900.0	Gross Beta	80 - 120	70 - 130	20
Radium 228	EPA 904	Radium 228	80 - 120	70 - 130	20
Radon 222, Liquid Scintillation	SM7500-Rn	Radon 222	80 - 120	-	20

Note: Refer to individual SOPs for precision and accuracy details for all methods

Table 5-2 Precision and Accuracy for Wastewater for Mid or High Level Spikes

(A) Inorganics – Wet Chemistry

Parameter Method Name	Method Name	Parameters/ Analytes	Accuracy		Precision RPD Maximum
			LCS/LFB % Rec.	MS/LFM % Rec.	
Alkalinity	SM2320B	Bicarbonate	90 - 110	80 - 120	20
		Carbonate	90 - 110	80 - 120	20
		Hydroxide	90 - 110	80 - 120	20
Ammonia	EPA350.1 / SM4500NH3H/G	Ammonia	90 - 110	90 - 110	20
Biochemical Oxygen Demand (BOD)	EPA 405.1 / SM5210B	Biochemical Oxygen Demand	85 - 115	-	-
Carbon Biochemical Oxygen Demand (CBOD)	SM5210B	Carbon Biochemical Oxygen Demand	85 - 115	-	-
Chemical Oxygen Demand (COD)	EPA410.4 / 5220 D	Chemical Oxygen Demand (COD)	90 - 110	90 - 110	20
Chloride	EPA300.0	Chloride	90 - 110	80 - 120	20
Chlorine, Total Residual	SM4500 Cl G	Chlorine, Total Residual	85 - 115	-	-

Parameter Method Name	Method Name	Parameters/ Analytes	Accuracy		Precision RPD Maximum
			LCS/LFB % Rec.	MS/LFM % Rec.	
Chromium VI	EPA 218.6/ SM3500 Cr-B, Colorimetric	Chromium VI	90 - 110	90 - 110	20
Specific Conductance	SM2510B / EPA 120.1	Specific Conductance	90 - 110	-	20
Cyanide, Total	EPA 335.4	Cyanide, Total	90 - 110	90 - 110	20
Cyanide, Amenable to Chlorination	SM 4500CN-G	Cyanide, Amenable to Chlorination	80 - 120	80 - 120	20
Fluoride	SM4500 F-C	Fluoride	81 - 116	73 - 124	20
Hardness	SM2340B/EPA 200.7	Hardness	90 - 110	80 - 120	20
Total Kjeldahl Nitrogen	EPA351.2	Kjeldahl Nitrogen	90 - 110	90 - 110	20
Nitrate	EPA353.2	Nitrate + Nitrite	90 - 110	90 - 110	20
	EPA300.0	Nitrate	90 - 110	80 - 120	20
Nitrite	EPA300.0	Nitrite	90 - 110	80 - 120	20
	EPA 353.2	Nitrite	90 - 110	90 - 110	20
Orthophosphate	EPA365.1/ SM4500 P-E/PF	Orthophosphate	90 - 110	90 - 110	20
Perchlorate	EPA 314	Perchlorate	85 - 115	80 - 120	20
pH	SM4500-HB	pH	98 - 102	-	+ 0.1 pH unit
Phenols	EPA 420.1 / 420.4	Phenols	90 - 110	90 - 110	20
Phosphorus, Total	EPA365.1/ SM4500 P-F	Phosphorus, Total	90 - 110	90 - 110	20
Dissolved Silica	SM 4500 SiO2C	Dissolved Silica	85 - 115	70 - 130	-
Residue, Filterable (Total Dissolved Solids--TDS)	SM2540C	TDS	80 - 114	-	20
Residue, Non-filterable (Total Suspended Solids--TSS)	SM2540D	TSS	80 - 120	-	10
Residue, Settleable (Settleable Solids)	SM2540F	Residue, Settleable (Settleable Solids)	-	-	-
Sulfate	EPA300.0	Sulfate	90 - 110	80 - 120	20
Sulfide (Total & Soluble)	SM 4500S-2D/ EPA 376.2	Sulfide	90 - 110	80 - 120	20
Total Residue	SM 2540 B	Total Solids	85 - 115	-	10
Total Organic Carbon (TOC)	SM5310C	Total Organic Carbon (TOC)	90 - 110	80 - 120	20
Total Organic Halide (TOX)	SM 5320B	Total Organic Halide (TOX)	85 - 115	90 - 110	-
Dissolved Oxygen	SM 4500-O G	Dissolved Oxygen	85 - 115	70 - 130	-
Color	SM 2120B	Color	-	-	-
Surfactants	SM 5540C	Surfactants	90 - 110	80 - 120	20
Turbidity	SM 2130B/ EPA 180.1	Turbidity	90 - 110	-	-

(B) Inorganics – Metals

Parameter Method Name	Method Name	Parameters/ Analytes	Accuracy		Precision RPD Maximum
			LCS/LFB	MS/LFM	
			% Rec.	% Rec.	
Asbestos	EPA 100.2	Asbestos	-	-	2.0X Poisson standard deviation
Metals	EPA200.7	Aluminum, Al	85 - 115	70 - 130	20
		Antimony, Sb	85 115	70 130	20
		Barium, Ba	85 - 115	70 - 130	20
		Beryllium, Be	85 - 115	70 - 130	20
		Boron, B	85 - 115	70 - 130	20
		Cadmium, Cd	85 - 115	70 - 130	20
		Calcium, Ca	85 - 115	70 - 130	20
		Chromium, Cr	85 - 115	70 - 130	20
		Cobalt, Co	85 - 115	70 - 130	20
		Copper, Cu	85 - 115	70 - 130	20
		Iron, Fe	85 - 115	70 - 130	20
		Magnesium, Mg	85 - 115	70 - 130	20
		Manganese, Mn	85 - 115	70 - 130	20
		Molybdenum, Mo	85 115	70 130	20
		Nickel, Ni	85 - 115	70 - 130	20
		Potassium, K	85 - 115	70 - 130	20
		Silica, SiO2	85 - 115	70 - 130	20
		Silver, Ag	85 - 115	70 - 130	20
		Sodium, Na	85 - 115	70 - 130	20
		Tin, Sn	85 - 115	70 - 130	20
		Vanadium, V	85 - 115	70 - 130	20
		Zinc, Zn	85 - 115	70 - 130	20
Metals	EPA 200.8	Aluminum, Al	85 - 115	70 - 130	20
		Antimony, Sb	85 - 115	70 - 130	20
		Arsenic, As	85 - 115	70 - 130	20
		Barium, Ba	85 - 115	70 - 130	20
		Beryllium, Be	85 - 115	70 - 130	20
		Cadmium, Cd	85 - 115	70 - 130	20
		Chromium, Cr	85 - 115	70 - 130	20
		Cobalt, Co	85 - 115	70 - 130	20
		Copper, Cu	85 - 115	70 - 130	20
		Lead, Pb	85 - 115	70 - 130	20
		Manganese, Mn	85 - 115	70 - 130	20
		Molybdenum, Mo	85 - 115	70 - 130	20

Parameter Method Name	Method Name	Parameters/ Analytes	Accuracy		Precision RPD Maximum
			LCS/LFB	MS/LFM	
			% Rec.	% Rec.	
Metals (con't.)	EPA 200.8	Nickel, Ni	85 - 115	70 - 130	20
		Selenium, Se	85 - 115	70 - 130	20
		Silver, Ag	85 - 115	70 - 130	20
		Thallium, Tl	85 - 115	70 - 130	20
		Vanadium, V	85 - 115	70 - 130	20
		Zinc, Zn	85 - 115	70 - 130	20
Mercury	EPA 245.1/7470A	Mercury,Hg	85 - 115	70 - 130	20
Chromium VI	SM 3500Cr B (20th)	Chromium VI	85 - 115	70 - 130	20
Silica, Dissolved	SM 4500SiO2C	Silica, Dissolved	85 - 115	70 - 130	20

(C) Microbiology/Microbiology Tests

Parameter Method Name	Method Name	Parameters/ Analytes	Accuracy		Precision RPD Maximum
			LCS/LFB	MS/LFM	
			% Rec.	% Rec.	
Fecal Coliforms By Multiple Tube Fermentation /EC Medium	SM9221C, E (MTF/EC)	Fecal Coliforms	-	-	-
Fecal Streptococci/ Enterococci by MTF	SM9230B	Fecal Streptococci/ E-Coli by MTF	-	-	-
Heterotrophic Plate Count	SM9215B	Heterotrophic Plate Count	-	-	5
Total Coliforms Multiple Tube Fermentation (MTF)	SM9221B	Total Coliforms	-	-	-

(D) Radiochemistry

Parameter Method Name	EPA Method Name	Parameters/ Analytes	Accuracy		Precision RPD Maximum
			LCS/LFB	MS/LFM	
			% Rec.	% Rec.	
Gross Alpha/Proportional Counting	EPA900.0	Gross Alpha	80 - 120	80 - 120	20
Gross Beta	EPA900.0	Gross Beta	80 - 120	80 - 120	20

Note: Refer to individual SOPs for precision and accuracy details for all methods

Table 5-3 Precision and Accuracy for Hazardous Waste for Mid or High Level Spikes

(A) Inorganics – Wet Chemistry

Parameter Method Name	EPA Method Name	Parameters/ Analytes	Accuracy		Precision RPD Maximum
			LCS/LFB	MS/LFM	
			% Rec.	% Rec.	

Total Organic Halogen	EPA 9020B	Total Organic Halogen	85 - 115	70 - 130	20

(B) Inorganics – Metals

Parameter Method Name	EPA Method Name	Parameters/ Analytes	Accuracy		Precision RPD Maximum
			LCS/LFB	MS/LFM	
			% Rec.	% Rec.	
Metals, Total	EPA6010B	Aluminum, Al	85 - 115	70 - 130	20
		Antimony, Sb	85 - 115	70 - 130	20
		Barium, Ba	85 - 115	70 - 130	20
		Beryllium, Be	85 - 115	70 - 130	20
		Cadmium, Cd	85 - 115	70 - 130	20
		Chromium, Cr	85 - 115	70 - 130	20
		Cobalt, Co	85 - 115	70 - 130	20
		Copper, Cu	85 - 115	70 - 130	20
		Manganese, Mn	85 - 115	70 - 130	20
		Molybdenum, Mo	85 - 115	70 - 130	20
		Nickel, Ni	85 - 115	70 - 130	20
		Silver, Ag	85 - 115	70 - 130	20
		Strontium, Sr	70 - 130	70 - 130	20
		Tin, Sn	85 - 115	70 - 130	20
		Titanium, Ti	70 - 130	70 - 130	20
		Vanadium, V	85 - 115	70 - 130	20
		Zinc, Zn	85 - 115	70 - 130	20
Metals , Total	EPA6020	Antimony, Sb	85 - 115	70 - 130	20
		Arsenic, As	85 - 115	70 - 130	20
		Barium, Ba	85 - 115	70 - 130	20
		Beryllium, Be	85 - 115	70 - 130	20
		Cadmium, Cd	85 - 115	70 - 130	20
		Chromium, Cr	85 - 115	70 - 130	20
		Cobalt, Co	85 - 115	70 - 130	20
		Copper, Cu	85 - 115	70 - 130	20
		Lead, Pb	85 - 115	70 - 130	20
		Molybdenum, Mo	85 - 115	70 - 130	20
		Nickel, Ni	85 - 115	70 - 130	20

Parameter Method Name	EPA Method Name	Parameters/ Analytes	Accuracy		Precision RPD Maximum
			LCS/LFB	MS/LFM	
			% Rec.	% Rec.	
Metals , Total (con't.)	EPA6020	Selenium, Se	85 - 115	70 - 130	20
		Silver, Ag	85 - 115	70 - 130	20
		Thallium, Tl	85 - 115	70 - 130	20
		Vanadium, V	85 - 115	70 - 130	20
		Zinc, Zn	85 - 115	70 - 130	20
Chromium VI	EPA 7196A EPA 7199	Hexavalent Chromium	85 - 115	70 - 130	20
Mercury	EPA7470A	Mercury, Hg	85 - 115	70 - 130	20

(C) Organics

Parameter Method Name	EPA Method Name	Parameters/ Analytes	Accuracy		Precision RPD Maximum
			LCS/LFB	MS/LFM	
			% Rec.	% Rec.	
Halogenated/ Aromatic Volatiles	EPA8260B EPA 624	Acetone	70 - 130	70 - 130	30
		Acrolein (Propenal)	70 - 130	70 - 130	30
		Acrylonitrile (screen)	-	-	30
		Benzene	70 - 130	70 - 130	30
		Bromodichloromethane	70 - 130	70 - 130	30
		Bromoform	70 - 130	70 - 130	30
		Bromomethane	70 - 130	70 - 130	30
		2-Butanone (MEK)	70 - 130	70 - 130	30
		Carbon disulfide	70 - 130	70 - 130	30
		Carbon tetrachloride	70 - 130	70 - 130	30
		Chlorobenzene	70 - 130	70 - 130	30
		Chlorodibromomethane	70 - 130	70 - 130	30
		Chloroethane	70 - 130	70 - 130	30
		2-Chloroethyl vinyl ether	70 - 130	70 - 130	30
		Chloroform	70 - 130	70 - 130	30
		Chloromethane	70 - 130	70 - 130	30
		Acetone	70 - 130	70 - 130	30
		Dibromomethane	70 - 130	70 - 130	30
		1,2-Dichlorobenzene	70 - 130	70 - 130	30
		1,3-Dichlorobenzene	70 - 130	70 - 130	30

Parameter Method Name	EPA Method Name	Parameters/ Analytes	Accuracy		Precision RPD Maximum
			LCS/LFB % Rec.	MS/LFM % Rec.	
Halogenated/ Aromatic Volatiles (con't.)	EPA8260B EPA 624	1,4-Dichlorobenzene	70 - 130	70 - 130	30
		Dichlorodifluoromethane	70 - 130	70 - 130	30
		1,1-Dichloroethane	70 - 130	70 - 130	30
		1,2-Dichloroethane	70 - 130	70 - 130	30
		1,1-Dichloroethylene	70 - 130	70 - 130	30
		cis-1,2-Dichloroethene	70 - 130	70 - 130	30
		trans-1,2-Dichloroethene	70 - 130	70 - 130	30
		1,2-Dichloropropane	70 - 130	70 - 130	30
		cis-1,3-Dichloropropene	70 - 130	70 - 130	30
		trans-1,3-Dichloropropene	70 - 130	70 - 130	30
		Ethylbenzene	70 - 130	70 - 130	30
		2-Hexanone	70 - 130	70 - 130	30
		Methylene chloride	70 - 130	70 - 130	30
		4-Methyl-2-pentanone (MIBK)	70 - 130	70 - 130	30
		Naphthalene	70 - 130	70 - 130	30
		2-Pentanone	70 - 130	70 - 130	30
		Styrene	70 - 130	70 - 130	30
		1,1,2,2-Tetrachloroethane	70 - 130	70 - 130	30
		Tetrachloroethene	70 - 130	70 - 130	30
		Toluene	70 - 130	70 - 130	30
		1,2,4-Trichlorobenzene	70 - 130	70 - 130	30
		1,1,1-Trichloroethane	70 - 130	70 - 130	30
		1,1,2-Trichloroethane	70 - 130	70 - 130	30
		Trichloroethene	70 - 130	70 - 130	30
		Trichlorofluoromethane	70 - 130	70 - 130	30
		Vinyl acetate	70 - 130	70 - 130	30
		Vinyl chloride	70 - 130	70 - 130	30
		o-Xylene	70 - 130	70 - 130	30
		m-Xylene	70 - 130	70 - 130	30
		p-Xylene	70 - 130	70 - 130	30
		1,2-Dichloroethane-d4 (surr)	80 - 120	80 - 120	-
		Toluene-d8 (surr)	88 - 110	88 - 110	-
		4-Bromofluorobenzene (surr)	86 - 115	86 - 115	-

6.0 QUALITY OF TEST RESULTS

6.1. ESSENTIAL QUALITY CONTROL PROCEDURES

The laboratory has established a quality control program that is designed to provide two different types of information about a particular analysis. The ability to confidently evaluate laboratory performance in terms of analytical bias and precision is accomplished through the use of both laboratory control samples (LCS), in the absence of sample matrix effects, and the traditional approach of using matrix spikes and duplicate (MS/MSD) analyses.

The quality control program implemented at EEA recognizes the problems associated with the use of matrix spikes and duplicates, and thus decisions regarding method data quality, when matrix effects are present, are made using data obtained from all control samples. The types and frequencies of control samples used at EEA are summarized below. Method Acceptance Limits are used to validate analytical results for each test. When method reference does not specify acceptance limits for the QC type, control limits are calculated and used as acceptance limits. The Control limits are recalculated at least annually for drinking water and waste water matrices and every 6 months for Hazardous Waste matrix. (See relevant SOP for the current control limits).

6.1.1. NEGATIVE CONTROL

6.1.1.1. Method Blanks

A method blank consists of laboratory pure water containing all of the reagents utilized in the analytical procedure. The method blank is prepared in the same manner as a sample and is processed through all of the analytical steps. All reagents are dated upon receipt in the laboratory and each new lot of reagents is checked by performance of method blanks.

Method blanks are processed along with the associated samples (minimum one MB per prep batch or analytical batch of ≤ 20 samples) performed to determine whether there is reagent contamination or instrument contamination due to sample carryover. The method blanks must remain below the MRL for each analyte of interest. Some analyses (see specific SOPs) have a more stringent requirement (e.g. < ½ or < 1/3 MRL). If samples require a preparatory procedure such as a digestion or extraction prior to analysis, a method blank must be carried through the entire process and analyzed in addition to the instrumental calibration blanks.

When a blank is determined to be contaminated, the cause must be investigated and measures taken to minimize or eliminate the problem. Samples associated with a contaminated blank shall be evaluated as to the best corrective action for the samples (e.g. reprocessing or data qualifying codes). In all cases the corrective action must be documented [TNI-EL-V1M4-2009-1.7.4.1.c].

Method blanks are analyzed as part of the initial or daily calibration process (calibration blanks) and after every 20 samples for each matrix type to monitor the overall procedural blank as well as the purity of the reagents. If analyte in method blanks is >MRL and is >1/10 of amount measured in sample and if blank contamination affects samples or individual data, quality, objectives, the problem is eliminated and reprocessed or affected samples appropriately qualified.

6.1.1.2. **Travel Blanks**

The trip blank is required to be analyzed in the event of any detects in the associated field samples. For example, both methods 504.1 and 524.2 for volatiles determination require a trip blank with each set of samples.

When running method 525.2 for phthalates determination for compliance monitoring purposes, the laboratory runs a trip blank if any of the samples are found positive for phthalates. This is necessary to show that samples were not contaminated from bottle caps, the HCl used for preservation, or the latex gloves worn during sampling. If the samples show the presence of phthalates and there was no trip blank with the set of samples then subsequent resamples from the site must be accompanied by a trip blank. If the samples are not to be analyzed for phthalates, the laboratory does not need to run a trip blank.

If a client has submitted a trip blank and wishes it to be analyzed automatically, the sample is logged in with the appropriate tests and with the log-in ID "Trip Blank" so that analysts will know to analyze and report them.

If a trip blank is submitted and is only to be analyzed in the event of hits, the sample is logged in with an ID of "Trip Blank-Hold."

For the analysis of ethylene dibromide and dibromochloropropane by Method 504.1 and phthalates by method 525.2, the analyst and supervisor ensure that if hits are detected in the associated samples, the trip blank is analyzed and reported within holding times.

Because of the relatively short holding times for VOAs by Method 524.2 and 504.1, the trip blanks are usually analyzed (unless specified by client) whether or not there are hits in the associated sample. In this way, Trip Blanks are always analyzed within holding times.

If there is adequate holding time remaining the analyst may elect to not analyze the trip blank. However in this case, the data should be reduced immediately and if there are hits, the sample should be analyzed on the next run, still within holding time.

In the event that no hits are present in the associated client samples the analyst and supervisor enter NA for the trip blank and preferably place a comment on the sample “not analyzed, no hits in field samples".

In the event that an analyte is detected in the trip blank, the analyst gets the associated stationary blank from shipping, if available, and runs that immediately to confirm that the hits are not due to lab contamination when the blank was prepared. The information to associate the proper trip blank to the sample(s) is be found on the sample bottle label, through the LIMS numbering system, and/or on the COC.

6.1.1.3. Field Blanks

Field blanks are used to identify contamination that may have occurred during the sample collection process. Empty containers with the applicable preservatives are sent to the field and filled at the sampling location at the time of sampling from a bottle with analyte-free water that was prepared at the laboratory as per client's request. The empty sample bottle and field blank sample bottle will be provided.

6.1.1.4. Sample Blanks

Sample blanks are used with spectrophotometric methods where sample characteristics such as color may give erroneous results. The absorbance of a sample is measured before and after the color development process. The absorbance before is subtracted from the absorbance after to give the true absorbance. Sample blanks are analyzed on an as-needed basis.

6.1.1.5. Calibration Blanks (CB)

For non-chromatographic analysis, calibration blanks are prepared along with the calibration standards and differ from the standards only in that the calibration blank does not contain any of the analyte(s) of interest. The calibration blank, by definition, provides the "zero point" in the calibration curve.

6.1.2. Positive Control

6.1.2.1. Laboratory Control Sample (LCS)/Laboratory Fortified Blank (LFB)

The LCS is used to evaluate the performance of the total analytical system, including all preparation and analysis [TNI-EL-V1M4-2009-1.7.3.2.1].

Laboratory control samples (LCSs) are defined as an interference free matrix spiked with a particular set of method-specific target compounds at a level 5-10 times above the minimum reporting limit. The matrix used to prepare aqueous LCS samples is laboratory reagent water (deionized water - carbon-filtered for organic analyses) and all method preservatives. In some cases LCS must be from a second or independent source, but other methods allow for the use of same sources. LCS is run at a frequency of one LCS per prep batch or analytical batch of ≤ 20 samples.

The purpose of the LCS matrix is not to duplicate the sample matrix, but more importantly to provide a consistent matrix with which baseline performance data for an analysis can be generated. This feature of the LCS provides one of the most significant advantages over the use of matrix spikes and spike duplicates. The variable matrix interferences inherent to matrix spikes and spike duplicates are manifested in the extremely wide control limits presented in the methods. This variability results in a large relative standard deviation in the data used to calculate the control limits which forces the control limits to become wider. The control of this variability significantly reduces the relative standard deviation of the data and results in control limits that are representative of laboratory precision alone.

6.1.2.2. **Matrix Spike and Matrix Spike Duplicate Samples (MS/MSD)**

MS/MSD samples are defined as a sample matrix spiked with a particular set of method-specific target compounds at a level 5-10 times above the minimum reporting limit. Samples are generally divided into two types of matrices, aqueous and non-aqueous.

Matrix spikes and spike duplicates are prepared using a sample matrix that is representative of the sample type being analyzed for a particular method. Frequency of the analysis of matrix spikes is as per method specifications or as per contract review.

6.1.2.3. **LCS and MS/MSD Concentration Levels**

When the method reference does not specify the LCS and MS/MSD concentration, the following criteria (in order of descending preference) are to be applied when determining the appropriate concentration of any particular analyte in the designated control sample:

6.1.2.3.1. If no MCL exists, or the MCL represents an impractical level relative to MDL or calibration range, the selected level should be set at the corresponding level used in the EPA's reference methods.

6.1.2.3.2. The level selected should be equal to any existing federal maximum contaminant level (MCL). This may not always be practical (as in the case of thallium [Tl]) when the MCL is too close to our actual MRL to yield consistent accuracy and precision.

6.1.2.3.3. If there is no EPA protocol for a particular method, or this level is inappropriate for the method, then the selected level should be near the midpoint of the calibration range. Optimally, this would be equivalent to the MCL, unless the calibration range spans more than 2 orders of magnitude.

6.1.2.3.4. If the calibration range spans 2 or more orders of magnitude, the selected level should be set at approximately 10 times the MRL for each analyte.

In some cases multiple levels (MRL, midpoint, high) are used to monitor control throughout the calibration range.

6.1.2.4. **Selection of Spike Analytes**

Any analyte reported must be included in the LCS and MS spiked sample for drinking water matrix samples. The selection of specific analytes to be spiked should be based on the following scheme:

6.1.2.4.1. If there are regulatory or method specific monitoring requirements for any of the target compounds, these compounds should be included.

6.1.2.4.2. If there are no regulatory or method specific monitoring requirements or additional analytes required to meet the absolute number to be included in the subset, follow TNI-EL-V1M4-2009-1.7.3.2.3 requirements for LCS spiking composition and TNI-EL-V1M4- 2009-1.7.3.3.1 for MS spiking composition.

6.1.2.4.3. As per the TNI Standard, for those test methods that have extremely long lists of analytes, a representative number may be chosen. The analytes selected should be representative of all analytes reported. The following criteria shall be used for determining the minimum number of analytes to be spiked for LCS and MS. However, the laboratory shall ensure that all targeted components are included in the spike mixture over a 2-year period. This can be applied for non-compliance samples, or other non-drinking water matrix samples.

- For methods that include 1-10 targets, spike all components;
- For methods that include 11-20 targets, spike at least 10 or 80%, whichever is greater;
- For methods with more than 20 targets, spike at least 16 components.

6.1.2.4.4. If neither of the above criteria apply, then the analytes should be selected for the subset so that all the different classes of compounds in the list of target compounds for the method are represented.

6.1.2.4.5. Any unique, method-specific problem analyte or element (such as potential loss of a particular analyte during extraction, digestion, or cleanup step or an element subject to severe inter-element interference on the ICP) should be represented in the subset.

6.1.2.4.6. In the absence of specified spiking components, for those components that interfere with an accurate assessment such as spiking simultaneously with technical chlordane, toxaphene and PCBs, the spike chosen represents the chemistries and elution patterns of the components to be reported. (TNI-EL-V1M4- 2009-1.7.3)

6.1.2.5. **Sample Preparation of LCS/LFB and MS/MSD**

The intent of this program is to set our control sample analytes and concentration levels such that a single concentrated stock mix is (1) independently prepared (preferably from different neat materials) from calibration stock solutions, and (2) can be used to prepare LCS samples as well as MS/MSD samples for both aqueous and non-aqueous environmental samples.

The ratio of spiked concentrate to sample aliquot used to prepare MS/MSD samples must be 1 to 10%, depending on the method specifications. In the case of matrix spikes, this practice ensures that we are not diluting the environmental sample to such an extent that we are diluting out any matrix interferences. The purpose of the matrix spike is to provide information regarding the ability to recover an analyte from a particular matrix.

6.1.2.5.1. **Stock Source of LCS/LFB and MS/MSD**

In order to serve its purpose as an external verification (reference) of the calibration, it is essential that the stock solutions used to prepare LCS and matrix spike samples be prepared independently of calibration stocks unless a method specifies a contrary approach. In the organics area, there is a lack of independent sources from which reference materials are obtained but the stock solutions should be prepared independently although they may share a common source.

The source of control sample reference materials should be selected in the following order of preference:

6.1.2.5.1.1. The neat compound must be prepared from either a completely independent sources. For example, a 1000-mg/L stock As solution obtained from Fisher is used to prepare As calibration standards, while a 1000 mg/L stock As solution obtained from Spex is used to prepare the control sample concentrate.

6.1.2.5.1.2. If a completely independent source cannot be obtained, the same vendor may be used, but the solution shall be from a completely different lot (second lot).

6.1.2.5.1.3. If it is impossible to obtain the reference material from two independent sources, or from two different lots, then the material from a single source can be used provided that a different analyst than the one who prepared the calibration stock is responsible for preparing the control sample solution.

6.1.2.6. **Frequency of MS/MSD**

MS/MSD samples are run at a frequency of one pair for every sample batch of 20 or less of a similar matrix. In cases where there is insufficient sample to run a MS/MSD as well as the original, a pair of LCS samples may be substituted to fulfill this requirement. There is often insufficient sample for aqueous samples to have a MS/MSD set up due to the large volumes of sample required for analysis. EEA encourages clients who require

precision and accuracy information based on a particular matrix to make arrangements to submit adequate sample volumes for this purpose. By supplying these samples, the client is able to obtain not only specific information regarding laboratory performance (from LCS sample data), but also a measure of the applicability of the sample matrix to the analytical method used (from the matrix spike and duplicate data). If the matrix spike is used in place of the LCS, the acceptance criteria must be as stringent as the LCS [TNI-EL-V1M4-2009-1.7.3.2.3].

6.1.2.7. **Frequency of LCS/LFB**

Laboratory control samples are analyzed throughout a run at a frequency of 5%-10% for environmental samples of a similar matrix. Bias information is provided based on recovery data for the LCS and precision information is available by comparing LCS sample results using a RPD calculation. The frequencies are consistent with the requirements of most methods referenced in Standard Methods, EPA Manual for Chemical Analysis of Water and Waste, 40 CFR 136 for the wastewater methods, and EPA Manual for the Certification of Laboratories Analyzing Drinking Water, 5th Edition. Additional measures of precision and bias are obtained from other control samples, as specified in the SOP's.

In order to ensure that some measure of analytical control is provided with each batch of samples going through a pre-analysis preparation step, an LCS is prepared with each set of 20 samples extracted or digested for these analyses. In each case, an LCS will be associated with each set of samples prepared, to allow documentation of control of the analytical procedures. Some methods require varying concentrations of LCS throughout a run.

6.1.2.7.1. Analyses with a preliminary treatment step (i.e. extraction or digestion):

6.1.2.7.1.1. LCS frequency is one for every 10 to 20 samples (see individual method SOPs) or at least one for every preparation batch of ≤ 20 samples.

6.1.2.7.1.2. MS/MSD or LCS pair (in cases where there is insufficient sample volume for a MS/MSD) is prepared for every sample batch of 20 samples or as per method specifications.

6.1.2.7.2. Analyses not requiring pretreatment:

6.1.2.7.2.1. A LCS must be run with each analytical run at a frequency of no less than one for every 10 or 20 samples (see individual method SOPs).

6.1.2.7.2.2. A MS/MSD or LCS pair must be run for every batch of 20 samples as defined in method specifications or TNI standards.

6.1.2.7.2.3. Any exceptions to this frequency on a given run must be documented on a corrective action form.

6.1.2.8. **Evaluation Criteria of MS/MSD**

MS acceptance criteria are compared to the acceptance criteria as published in the mandated test method if not specified in the method. Advisory limits for each method are established initially based on method validation data. Initial control limits are defined as the mean recovery (accuracy) ± 3 times the standard deviation obtained from the analysis of 4 (or more) replicates spiked at approximately 10x MRL during the method validation process. Warning limits are set as the mean recovery (accuracy) ±2 times the standard deviation.

Firm acceptance criteria, based upon actual laboratory data, are established once a minimum of 20 data points has been generated. These historical control limits are compared to any method specified or recommended limits to assess their feasibility. Control limits are re-calculated at least yearly to verify that there has been no significant change in performance.

Precision is determined as the relative percent difference (RPD) between LCS pairs or MS/MSD samples. By linking a LCS or MS/MSD pair to each batch of 20 environmental samples, it is possible to link a measure of analytical precision (and two measures of analytical accuracy) to each environmental sample analyzed.

Precision control limits for some analytes have been adopted from the EPA CLP program where they exist, otherwise, control limits are set after the analysis of 20 MS/MSD or LCS pairs of samples (40 control samples). Control limits are set as the mean ± 3 standard deviations of the RPD from the 20-30 "pairs", with warning limits set at the mean ± 2 standard deviations. Until such time as 20-30 data points have been accumulated, interim acceptance criteria should be set as 3 times the standard deviations of the RPD obtained during the method validation process.

Whenever MS/MSD or LCS pairs do not meet these limits, an analysis may have a potential problem. Samples with failing LCS shall be reprocessed and reanalyzed or data reported with data qualifying codes. The source of any problems must be investigated and documented by preparing a corrective action or procedural variance report.

For drinking water method, when there is no method specification, the spike level should not be less than the concentration of the sample selected for fortification unless specified by the method. If the sample concentration is unknown or less than detectable, the analyst should choose an appropriate concentration. If the spike level is less than the concentration of the sample selected, the spike recovery value is unusable since the analyte concentration in the sample is disproportionate to the spike level.

6.1.2.9. Evaluation Criteria of LCS/LFB – Marginal Exceedances

If a large number of analytes are in the LCS, it becomes statistically likely that a few will be outside control limits. This may not indicate that the system is out of control, therefore corrective action may not be necessary. Upper and lower marginal exceedance (ME) limits can be established to determine when corrective action is necessary. A ME is defined as being beyond the LCS control limit (3 standard deviations), but within the ME limits. ME limits are between 3 and 4 standard deviations around the mean.

The number of allowable marginal exceedances is based on the number of analytes in the LCS. If more analytes exceed the LCS control limits than is allowed, or if any one analyte exceeds the ME limits, the LCS fails and corrective action is necessary. This marginal exceedance approach is relevant for methods with long lists of analytes. It will not apply to target analyte lists with fewer than 11 analytes.

The number of allowable marginal exceedances is as follows [TNI-EL-V1M4-2009-1.7.4.2]:

- >90 analytes in LCS, 5 analytes allowed in ME of the LCS control limit;
- 71 – 90 analytes in LCS, 4 analytes allowed in ME of the LCS control limit;
- 51 – 70 analytes in LCS, 3 analytes allowed in ME of the LCS control limit;
- 31 – 50 analytes in LCS, 2 analytes allowed in ME of the LCS control limit;
- 11 – 30 analytes in LCS, 1 analytes allowed in ME of the LCS control limit;
- < 11 analytes in LCS, no analytes allowed in ME of the LCS control limit.

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.

6.2. Sample Specific Controls

6.2.1. Internal and Surrogate Standards

Internal standards are run with GC/MS, GC, and HPLC analyses to monitor the efficiency of the analytical procedure for each sample matrix encountered and to monitor retention time shifts and the efficiency of the auto-sampler injection. Surrogate standards are run with GC/MS, GC, and HPLC analyses to monitor the efficiency of the extraction for each sample matrix encountered. When there are no established criteria for surrogates from the method, the lab determines internal limits through control charts.

Control limits are re-established annually for surrogates based on historical laboratory data from environmental sample matrices. Internal and surrogate standards are added to each sample analyzed by EPA Methods as recommended and run in accordance with the method procedures. For references to specific compounds used for internal and surrogate standards please reference the SOP.

Current surrogate acceptance limits may be found in Table 6-1.

6.2.2. **Spikes – Recoveries, RPDs**

Spiked sample analyses (MS/MSD) are performed to evaluate the effect of the sample matrix on the analytical methodology. A known amount of the analyte(s) of interest is added to an aliquot of sample, which is then analyzed along with the unspiked sample. Spiked samples are prepared and subjected to the same process as the original sample. Spike recoveries are calculated, and used to determine whether the sample matrix interferes with the method.

Spike recoveries are calculated as follows:

$$\mathbf{\%R} = \frac{SSR - SR}{SA} \times 100$$

Where;

%R	=	percent spike recovery
SSR	=	spiked sample result (corrected for any dilution in spiking)
SR	=	sample result
SA	=	spike amount added

The Laboratory documents the percent (%) recoveries and %RPD for MS/MSD samples [TNI-EL-V1M4-2009-1.7.4.3.a].

6.2.3. **Duplicates, Duplicate Spikes**

Duplicate analysis of a sample has traditionally been used to obtain a measure of analytical precision in the form of a relative percent difference (RPD) calculation between the two values. EEA routinely will analyze duplicate spiked control samples, MS/MSD to meet specific client's QC requirements such as Arizona.

Since no precision information is obtained when either or both of the duplicates have analyte concentrations below the MRL, duplicate analysis of the spiked samples makes the most sense. While still subject to interference problems the advantage of duplicate matrix spikes is clearly the ability to obtain calculated RPD values specific for a particular sample matrix. Clients are encouraged to submit sufficient sample for the analysis of MS/MSD samples by specific request when a RPD value for their particular matrix is desirable.

Ongoing analytical precision is evaluated by tracking the difference between the MS/MSD (or LCS pairs) analyzed with each batch of 20 samples. These differences are compared to control limits established for each analysis from historical monitoring. In the

event that the method does not specify the criteria, control charts are reviewed to set laboratory internal/default QC criteria [TNI-EL-V1M4-2009-1.7.4.3.a].

For those analyses for which MS/MSD or LCS samples are not prepared, sample duplicates are analyzed to monitor performance.

The relative percent difference between duplicates or duplicate spikes is calculated as follows:

$$\textbf{RPD} = \frac{|S-D|}{(S+D)/2} \times 100$$

where;

RPD	=	Relative Percent Difference
S	=	First Sample Value (original)
D	=	Second Sample Value (duplicate)

6.2.4. External Reference Samples/Quality Control Sample (QCS)

Reference samples such as those available from NIST and EPA or other EEA approved vendors are analyzed to verify the accuracy of calibration standards. Reference standards with matrices comparable to the samples being analyzed are also included in the run whenever available.

External reference samples are analyzed immediately following the calibration standards for all inorganic and organic analyses. Appropriate reference samples for organics analyses by GC and GC/MS are less readily available and are only run when a new stock standard is prepared to verify its accuracy

6.2.5. Confirmation

Confirmation is performed to verify the compound identification when positive results are detected on a sample from a location that has not been previously tested by the laboratory. Confirmations are performed on GC organic tests such as pesticides or herbicides. GC confirmation is done following method requirements or recommendations. See method SOPs for detailed discussion of the confirmation methods. Confirmation is not required when a sample is analyzed by mass spectrometer methods. All confirmation is documented in appropriate log books/work books.

6.2.6. Retention Time Windows

Absolute retention time and relative retention time aid in the identification of components in chromatographic analyses and to evaluate the effectiveness of a column to separate constituents. The laboratory ensures that it meets the method acceptance

criteria for retention time windows. If the method does not specify acceptance criteria for retention time windows, the laboratory gathers a minimum of 30 data points and calculates the acceptance criteria range using 3 times the standard deviation of the average ($\bar{x} \pm 3sd$).

6.3. DEMONSTRATION OF CAPABILITY (DOC)

6.3.1. Method Detection Limits (MDL) / Limit of Detection (LOD)

6.3.1.1. The laboratory shall utilize MDL determination by 40 CFR Part 136 as one option to provide an LOD for each analyte that is appropriate and relevant for the intended use of the data. An LOD is not required for a test method when test results are not reported outside the calibration range. LOD shall be determined by the protocol in the mandated test method or applicable regulation. If the protocol for determining LOD is not specified, the selection of the procedure must reflect instrument limitations and the intended application of the test method [TNI-EL-V1M4-2009-1.5.2.1].

6.3.1.2. The MDL shall be initially determined for the compounds of interest in each test method in a quality system matrix in which there are not target analytes nor interferences at a concentration that would impact the results of the MDL must be determined in the quality system matrix of interest [TNI-EL-V1M4-2009-1.5.2.1.d].

6.3.1.3. Method Detection Limits (MDLs) will be determined as per 40CFR, part 136, Appendix B. Essentially, this requires that an estimate of the detection limit be determined for each target analyte based on analytical experience or published references. Seven replicates of DI water must then be spiked at this estimated MDL for each method analyte carried through the entire procedure over a minimum of 3 separate analysis/extraction days. The MDL is then calculated as the standard deviation of the 7 replicates multiplied by the statistical "t-value" associated with the actual number of replicates analyzed assuming N-1 degrees of freedom (for exactly 7 replicates, the t-value is 3.143; 40 CFR, Part 136).

6.3.1.4. MDL study must be verified annually as per the EPA Manual at a minimum (or more frequently if stated in the Method such as EPA 300.0 and 353.2 where the MDL study has to be repeated every 6 months). A copy of all associated data must be submitted to the QA group for filing.

6.3.1.5. An MDL study must be repeated for each new analyst trained in a particular method, or if there is a change in the instrumentation or the test method that is used for the analysis in question. This is a necessary requirement to ensure that each new analyst has received sufficient training such that the data generated will be comparable to that of former analysts. It is necessary to repeat the MDL process with a change in instrumentation to ensure that the new instrumentation is capable of achieving equivalent sensitivity. An MDL study must also be repeated when there is any significant change in background or instrument response.

6.3.1.6. A minimum of a three-point calibration will be performed prior to the MDL study. One of the points must be at the MDL spike level. The calibration must meet all criteria outlined in the Calibration Policy.

6.3.1.7. The spiked level must be within 10 times the calculated MDL or the process must be repeated at a lower spike concentration. The spike level should be greater than the calculated level.

6.3.1.8. If there is a significant blank level, the spike level for the MDL determination must be at least three times greater than the blank concentration.

6.3.2. **Minimum Reporting Limits (MRL) / Limits of Quantification (LOQ)**

6.3.2.1. The Minimum Reporting Limit (MRL) is the lowest concentration normally reported to the client. It represents the reporting value linked to a specific analyte for aqueous matrix in the LIMS system. The MRL represents a conservative, nominal reporting limit designed to be representative of the minimum quantifiable concentration level for a particular analyte in a real environmental matrix as opposed to the statistically derived MDL calculation.

6.3.2.2. The MRL will generally be established by multiplying the statistically derived MDL by a factor of 2 or 3. The rationale for this approach is that the resultant value becomes approximately 10 times the standard deviation obtained during the MDL study; the EPA frequently refers to this concentration as the "Limit of Quantification (LOQ)", and defines it as the level above which accurate quantitation can be achieved. This level is also more similar to the SW-846 and SDWA concept of "Practical Quantitation Limits" (PQL). At a minimum, the MRL needs to be greater than or equal to the MDL.

6.3.2.3. Perform an MRL check and calculate the acceptance criteria for recovery of spiked analyte at MRL is 50-150 % or ± 3 standard deviations, whichever is greater if the method does not specify acceptance limits. MRL Check is run daily as per EPA Manual for the Certification of Laboratories Analyzing Drinking Water, 5th Edition.

6.3.2.4. Final MRLs should only be established after receiving input from the Group Technical Manager, Client Services Manager, Lab Director and Quality Manager. This ensures that all relevant issues regarding the selection of MRLs have been considered. These issues include specific minimum reporting limits required by a particular state or regulatory body, contractually required reporting limits for a specific client, the need to provide consistent reporting limits for our clients that have historically submitted samples associated with long-term monitoring efforts, as well as to remain competitive in the market. Thus a specific client may require that we use an MDL on our reports rather than an MRL. This deviation must be documented on

client reports. A "J" flag is used to qualify results greater than MDL, but less than MRL (>MDL, <MRL). A "U" flag is used to qualify results not detected at the MDL.

6.3.3. Initial Demonstration of Capability(IDC)

6.3.3.1. An IDC is performed for each analyst and instrument. The IDC for each analyst includes a demonstration of the ability to achieve a low background, the precision and accuracy required by the method, the method detection limit (MDL) in accordance with procedure in 40 CFR 136, Appendix B and satisfactory performance on an unknown sample as on-going proficiency test result are also filed.

6.3.3.2. The IDC is repeated when there is a change in analyst, test method or instrument.

6.3.3.3. All initial demonstrations of capability and method certification shall be documented [TNI-EL-V1M3 to V1M7-2009-1.6]. A copy of the certification should be retained in the personnel records of each affected employee [TNI-EL-V1M3 to V1M7-2009-1.6.1].

6.3.3.4. Initial demonstration of method performance is completed each time there is a significant change in instrument type, personnel, or test method.

6.3.3.5. Continuing demonstration of method performance (such as laboratory control and matrix spike samples) is monitored by use of control charts.

6.3.3.6. The QC sample used for the IDC analysis is obtained from an outside source. If an external vendor is not available, the laboratory prepares the QC sample independent of the instrument calibration standard.

6.3.3.7. The QC sample concentration prepared for the IDC is approximately 1-4 times the MRL for spike concentration if not specified by the method or regulations. Four aliquots of the sample are analyzed concurrently (same day) or over a period of days. Average recovery and standard deviation for each parameter of interest are calculated in the units used for reporting to clients. The resulting average recovery and standard deviation must meet the acceptance criteria for the method.

6.3.3.8. When it is not possible to determine mean and standard deviations, such as for presence/absence and logarithmic values, the laboratory assesses performance against established and documented criteria. If there is no mandatory criteria in the method, either reference or laboratory generated limits are used.

6.3.3.9. If standards cannot be prepared, as for Microbiology, QC samples or PE samples obtained from NIST or other approved PT providers are used for the IDC. The laboratory retains all associated supporting data necessary to reproduce analytical results summarized in the IDC certification statement. The Microbiology DOC SOP provides the details of the DOC procedure.

6.3.3.10. Analysis of actual samples is not done until all parameters of interest for the IDC meet acceptance criteria. If one or more of the test parameters do not meet the acceptance criteria, the problem is corrected, followed by repeated analysis of the four aliquots for those that failed to meet criteria. If the repeat analyses fail acceptance criteria the laboratory investigates, corrects the problem and repeats the test for all parameters.

6.4. METHOD SPECIFIC QUALITY CONTROL

6.4.1. Gravimetric

6.4.1.1. All laboratory analytical balances and thermometers of ovens are calibrated annually with Class S weights and a certified thermometer. Records of this balance calibration are maintained by the balances and periodically turned in to the QA Officer for filing as records are completed. Balances are verified on each day of use.

6.4.1.2. A sufficient number of dessicators are maintained to insure that samples are not crowded to the point where they cannot cool to room temperature at the end of the specified drying period. Desiccant replacement is based on color changes.

6.4.1.3. LCS samples are analyzed at a frequency of 5 or 10% and are specified in each method SOP. At least one LCS is analyzed for each analytical run.

6.4.1.4. MS/MSD samples (or LCS pairs) are analyzed at the rate of once every batch of 20 samples of a similar matrix, or at other frequency, depending on the method requirements.

6.4.2. Titration

6.4.2.1. Use of an automated titrator set to proper delivery speed insures that every sample is titrated to the same endpoint. For manual titration, selection of the proper endpoint is achieved by comparing the color of the sample currently being titrated with the color of the previously titrated sample. The analyst must be particularly careful when performing a titration with a fading endpoint. In such instances, it is important to complete the titration as rapidly as possible.

6.4.2.2. An external reference sample is analyzed with each new set of standards or titrant to verify the accuracy of the titrant standardization and the endpoint determination. In addition, the endpoint pH is checked for each sample.

6.4.2.3. LCS samples are analyzed at a frequency of 5 or 10% and are specified in each method SOP. At least one LCS is analyzed for each analytical run.

6.4.2.4. MS/MSD samples (or LCS pairs) are analyzed at the rate of once every batch of 20 samples of a similar matrix, or at other frequency, depending on the method requirements.

6.4.3. Colorimetric Spectrophotometry

6.4.3.1. The alignment of the cell holder and light source is checked when absorbancy indicates a problem.

6.4.3.2. A minimum of three standards plus a blank, equally spaced over the concentration range, are used to calibrate the spectrophotometer in the absorbance mode, except where methods specify the use of one standard only.

6.4.3.3. The analyst records the absorbance reading for the top standard and notes on the form if a gradual increase or decrease in the absorbance of this standard is occurring. A gradual decrease in absorbance values from week to week is usually indicative of a deteriorating standard or the initial stage of lamp failure.

6.4.3.4. The rate of color development and color stability of spectrophotometric procedures varies considerably. The allowable time interval for reading the absorbance of the sample is specified in the method and must be rigidly adhered to in order to obtain accurate results.

6.4.3.5. Measuring a blank and a calibration standard after every twenty samples checks the stability of the spectrophotometer. If the baseline absorbance or the standard absorbance value has changed by more than 0.005 absorbance units or 10% from the initial calibration standard, whichever is greater, the instrument must be recalibrated and all samples analyzed since the last acceptable calibration check must be reanalyzed.

6.4.3.6. Some water samples have a natural color or turbidity which absorbs appreciably at the wavelength used in the analysis. If the sensitivity of a procedure is sufficiently high, it is usually possible to minimize this interference by diluting the sample. If the sensitivity is not adequate to permit sample dilution, the turbidity or color interference is corrected for, by reading the absorbance of the sample carried through the procedure without addition of the indicator reagent when instrumentation permits it. This absorbance reading is then subtracted as a blank from the absorbance reading of the sample.

6.4.3.7. LCS samples are analyzed at a frequency of 5 or 10% and are specified in each method SOP. At least one LCS is analyzed for each analytical run.

6.4.3.8. MS/MSD samples (or LCS pairs) are analyzed at the rate of once every batch of 20 samples of a similar matrix, or at other frequency, depending on the method requirements.

6.4.4. ICP Emission Spectroscopy & ICPMS

6.4.4.1. The sensitivity of each element is recorded in order to detect deficiencies in the instrument or operating conditions.

6.4.4.2. Reagent blanks followed by a calibration check standard are run for each metal determined with a frequency of 10%. If there is a difference of >10% from the initial standard reading, the instrument must be recalibrated and all samples that were analyzed after the last acceptable calibration check must be reanalyzed.

6.4.4.3. For ICP analysis using the simultaneous system, inter-element correction factors must be available for each wavelength used. Background correction must be used for each element.

6.4.4.4. LCS samples are analyzed at a frequency of 5 or 10% as specified in each method SOP. At least one LCS is analyzed for each analytical run.

6.4.4.5. MS/MSD samples (or LCS pairs) are analyzed at the rate of once every batch of 20 samples of a similar matrix, or at other frequency, depending on the method requirements.

6.4.5. Radiochemistry

6.4.5.1. The laboratory participates in performance studies for gross alpha and beta, Uranium and radium. Results must be within the control limits established by the vendor for each analysis.

6.4.5.2. The laboratory monitors monthly radiation measurement of laboratory instrumentation for radioactive contamination [TNI-EL-V1M6-2009-1.7.1.d]. The procedure is discussed in the CHP Manual including criteria and corrective action procedure.

6.4.5.3. Efficiency curves are run at least annually and the data recorded in the radiation notebook.

6.4.5.4. A background is run monthly for gas proportional counter, and each day of use for scintillation counter) and a known reference sample is run with each batch of radiation samples analyzed. Background check measurements shall be performed at least weekly for gas proportional counter [TNI-EL-V1M6-2009-1.7.1.c.iii]. EEA performs background check measurements each day of use for gas proportional counter. Method blank shall be performed at a frequency of at least one per preparation batch. If the acceptance criteria specified in the SOP are not met, the specified corrective action and contingencies shall be followed and the result reported with appropriate data qualifying codes [TNI-EL-V1M6- 2009-1.7.3.1.c].

6.4.5.5. LCS samples are analyzed at a frequency of 5 or 10% and are specified in each method SOP. At least one LCS is analyzed for each analytical run. The activity of LCS shall be at least10 times the Minimum Detectable Activity (MDA) or at a level comparable to that of the routine samples if the sample activities are expected to exceed 10 times the detection limit [TNI-EL-V1M6-2009-1.7.2.2.e].

6.4.5.6. Gross alpha and gross beta require MS for aqueous samples. When there is not sufficient sample aliquot size to perform a matrix spike, it shall be noted on the lab report [TNI-EL-V1M6-2009-1.7.2.3.a.iv]. The activity of the matrix spike analytes shall be greater than five times the MDA [TNI-EL-V1M6-2009-1.7.2.3.a.v].

6.4.5.7. The laboratory standards used to prepare LCS and MS shall be from a source independent of the laboratory standards used for instrument calibration [TNI-EL-V1M6-2009-1.7.2.2.f]. The MS shall be prepared by adding a known activity of target analyte.

6.4.5.8. Replicate shall be performed at a frequency of one per preparation batch where there is sufficient sample to do so. The replicate result shall be assessed against the specific acceptance criteria specified in the laboratory SOP. For low level samples (less than approximately three times the MDA) the laboratory may analyze duplicate laboratory control samples or a replicate matrix spike (matrix spike and a matrix spike duplicate) to determine reproducibility within a preparation batch [TNI-V1M6-2009-1.7.2.3.b.iv].

6.4.5.9. Consistent test conditions for RAD testing are maintained through a radiological control program that addresses analytical radiological control (See EEA's Radiation Safety Program Manual). The program shall address the procedures for segregating samples with potentially widely varying levels of radioactivity. The radiological control program shall explicitly define how low level and high level samples will be identified, segregated and processed in order to prevent sample cross-contamination. The radiological control program shall include the measures taken to monitor and evaluate background activity or contamination on an ongoing basis [TNI-EL-V1M6-2009-1.7.2.7.c].

6.4.6. Gas Chromatography

6.4.6.1. A laboratory water blank is analyzed for all analyses to check for artifacts from the GC system and for the presence of impurities in the water blank making it unsuitable for LCS preparation.

6.4.6.2. A field or travel blank should be analyzed for each set of field samples taken. With each set of travel blanks sent out, a stationary travel blank is kept in the laboratory for analysis to demonstrate that the water sent out was free of contamination.

6.4.6.3. A series of continuing calibration standards are run with the analysis each day for all GC analyses.

6.4.6.4. LCS and/or MS/MSD samples for assessing precision and accuracy are determined by carrying the control samples or spike and spike duplicates through the extraction procedure as well as the instrumental analysis.

6.4.6.5. LCS samples are analyzed at a frequency of 5 or 10% and are specified in each method SOP. At least one LCS is analyzed for each analytical run.

6.4.6.6. MS/MSD samples (or LCS pairs) are analyzed at the rate of once every batch of 20 samples of a similar matrix.

6.4.7. Gas Chromatography/Mass Spectrometry

6.4.7.1. GC/MS Tuning Specifications

The mass spectrometer must be shown to be properly tuned during each daily 12 hour shift. This insures that the masses and abundance's, which the data system determines, are accurate. The EPA has suggested criteria for tuning the GC/MS with two standard compounds, decafluorotriphenylphosphine (DFTPP) and 1-bromo-4-fluorobenzene (BFB). Tuning criteria are shown in Table 11-3.

The following settings are maintained:

- Emission Current: 0.5 ma
- Electron Energy: 70 ev
- Electron Multiplier: 1000-2000 volts as required for sensitivity
- Dynodes: 3000 V

For HPLC and LCMS tuning, please consult the appropriate Standard Operating Procedure for the method in question

6.4.7.2. Quantitation of Identified Compounds/Quantitation from Initial Instrument Calibration

The calibration procedure for GCMS is based on the EPA Methods Reference, for example 524.2, 525.2, 624, and 625. A minimum five point standard curve is run for all analytes. For each calibration compound a response factor (Rf) and the %RSD are calculated.

The procedure to be employed for evaluation of the acceptability of the initial calibration curve based on the EPA Methods Reference, see individual SOPs for specific examples.

All quantitation are done from initial instrument calibration and not from continuing calibration unless required by the method, regulation or program [TNI-EL-V1M4-2009-1.7.1.1.c].

6.4.7.3. Internal and Surrogate Standards (IS and SS)

The internal standard area counts are recorded for all volatile and semi-volatile samples.

If any sample is found to have an IS beyond ± 50% based on ICAL (± 30% for CCV) of the IS counts for the daily continuing calibration standard, the sample is re-analyzed unless an obvious matrix problem can be documented.

Surrogate standards are utilized in both the volatile and semi-volatile analysis.

Any volatile sample surrogate recovery that falls outside of the lab limits is immediately re-analyzed. If surrogate recoveries are still outside of the limits, a QIR is written and the report is annotated. If the second result is within the control limits, this result is reported.

For semi-volatile samples with unacceptable surrogate recoveries, the extraction run logs are examined for matrix related or other documented problems. In addition, the LCS recoveries are reviewed for the sample extraction set. If none of these indicate a matrix problem, the sample is re-extracted if still within holding times. If the analysis of the re-extract shows unacceptable surrogate recoveries, a QIR form is generated, then the sample report is annotated and the data reported.

6.4.7.4. Criteria for Tentatively Identified Compounds (TIC's)

A primary advantage of GC/MS is the ability to identify compounds for which the retention time and mass spectra are not well known to the operator. This is accomplished by performing a library search using the EPA/NIST library of mass spectra and comparing unknown to these spectra. The library search program gives five or ten of the "best fits". The best fits are determined by comparing the top eight mass fragments in the unknown to the spectra in the library. The program matches the mass numbers and the abundances at each mass number to those in the library. The program lists the possible identifications along with the numbers, which can be used by the MS operator to determine the quality of the identification. The fit is the degree to which the peaks and intensities in the unknown match those of a particular compound in the library. A perfect match would be 1000 or 1.000, depending on the software. EEA utilizes CLP criteria and method specifications for determining identification of unknowns. This includes the presence of all major ions greater than 10% relative intensity, agreement of ± 20% for major ions in the sample and reference spectra, and the review of all ions present in the sample spectrum for possible background contamination or interference.

In general a computer fit of 850 or 0.850 should be the minimum used for identification. It should be noted that even with computer library searches, there is no substitute for the judgment of a trained analyst.

6.4.7.5. Control Samples

LCS samples are analyzed at a frequency of 5%. At least one LCS is analyzed for each analytical run.

MS/MSD samples (or Duplicate) are analyzed at the rate of once every batch of 20 samples of a similar matrix, as required by TNI. Duplicates are usable only when target analytes are positives.

6.4.7.6. Blanks

Laboratory reagent water blank is normally the first sample analyzed at the beginning of each working day to demonstrate that the system is free from contamination. If the blank result indicates contamination, the system is cleaned by running additional water blanks or if necessary, finding an alternate source of contaminant free water.

6.4.8. Total Organic Carbon (TOC)

6.4.8.1. Samples are diluted to fall within the linear range of the standards.

6.4.8.2. Every tenth sample is an LCS and %recoveries must fall within acceptable control limits. MS/MSD samples (or LCS pairs) are analyzed at the rate of once every batch of 20 samples of a similar matrix as per method requirements.

6.4.9. Total Organic Halogen (TOX)

6.4.9.1. Three carbon blanks (carbon packed adsorption columns washed with nitrate-wash solution only) are analyzed at the beginning of each workday. All values must be within 20% of the average blank value obtained before standards can be run.

6.4.9.2. Each day, a set of three calibration standards is analyzed prior to analysis of samples. Calculated values for the standards must fall within 5% of the nominal value except for the 1.0 standard, which is allowed a 10% range.

6.4.9.3. Every eighth sample is, alternately, a continuing calibration standard or a carbon blank.

6.4.9.4. All samples are analyzed in duplicate. If the net values of the duplicates are not within acceptance criteria of 20%, a third and possibly a fourth replicate is analyzed.

Results are compared to the first and second replicate and the average of the two closest samples is reported.

6.4.9.5. The titration cell is revitalized by rinsing with fresh cell solution after every twenty analyses or sooner if necessary.

6.4.9.6. Samples are diluted to fall within the linear range of the standards.

6.4.9.7. Two or three serial adsorption columns from each sample adsorption are analyzed separately to determine if any organic halogen breakthrough is occurring. In the event of breakthrough, an additional diluted sample is analyzed. Every tenth sample is an LCS and %recoveries must fall within acceptable control limits.

6.4.9.8. MS/MSD samples (or LCS pairs) are analyzed at the rate of once every batch of 20 samples of a similar matrix.

6.4.9.9. The purity and adsorption capacity of each new batch of carbon purchased is assessed by duplicate analysis of an adsorption efficiency standard. This adsorption efficiency standard (standards injected into reagent water then filtered) must be within 5% of the standard value. In addition, duplicate carbon blank results must be less than 1 µg Cl-.

6.4.10. General Microbiology - Use of Commercial Dehydrated Powder for Coliform Testing

6.4.10.1. The individual collecting samples should be aware of the sampling precautions outlined in Standard Methods.

6.4.10.2. Specific sampling instructions are available from the EEA Microbiology Department. They list required precautions to follow to maintain the integrity of the samples and prevent contamination.

6.4.10.3. The maximum holding time for microbiological samples is 30 hours for drinking water and 6 hours for water/wastewater, and 8 hours for source water.

6.4.10.4. The bottles should be shipped sealed in strong plastic zip lock or bubble bags. This keeps the melting ice from contaminating the samples. Ice cubes or their equivalent must be placed around the samples but care must be taken that the samples do not freeze.

6.4.10.5. Sterility check on sample containers shall be performed on at least one container for each lot of purchased pre-sterilized sample containers. For containers prepared and sterilized in the lab, a sterility check shall be performed on one container per sterilized batch with non-selective growth media [TNI-EL-V1M5-2009-1.7.3.1.b.iii]. Microbiology sample containers are disposable high clarity polystyrene vessels with sodium thiosulfate sufficient to neutralize at minimum 5 mg/L of chlorine (IDEXX Cat No. WS216PS) for drinking water and 15 mg/L of chlorine for wastewater

samples. Containers from each lot of "ready to use" are tested to ensure efficacy of Na2S2O3 to 5 mg/L Cl2 for drinking water and 15 mg/L Cl2 for wastewater. Thus, samples received in the lab are not tested for additional residual Cl2 testing [TNI-EL-V1M5-2009-1.7.5.b]. When the residual chlorine concentration is checked in the field, the result is documented in the COC.

6.4.10.6. A sterilization indicator is used during each autoclave cycle. If problems exist as indicated by a failure of the sterilization indicator, none of the items from that autoclave load is used and the group leader is notified. Demonstration of effective sterilization is provided by the use of biological indicators at least once per month of use [TNI-EL-V1M5-2009-1.7.3.7.b.ii].

6.4.10.7. Culture media are prepared from commercial dehydrated powders or ready to use media such as colilert medium. The laboratory does not prepare media or its culture media from basic ingredients. [TNI-EL-V1M5-2009-1.7.3.5.a]

6.4.10.8. Only nanopure water is used for the preparation of media. Once opened, the powdered media is tightly recapped to prevent hydration.

6.4.10.9. Prepared liquid medium is stored in the dark at refrigeration of 4°C and used within 3 months. The media is labeled with the type of medium, date prepared and the initials of the analyst who weighed out the dehydrated powder.

6.4.10.10. Prepared agar plates are stored in plastic bags, agar up, in the refrigerator. The bag is labeled to identify the type of medium, date prepared and the initials of the analyst who prepared it.

6.4.10.11. When bacteriological samples are incubated in a water bath or incubator, the temperature is recorded each morning and afternoon on the appropriate temperature sheet.

6.4.10.12. A thermometer calibrated at 44.5˚C is used for the water bath when fecal coliforms are incubated.

6.4.10.13. A positive control culture obtained from the American Type Culture Collection is inoculated for each batch of media including chromofluorogenic medium, incubated and read to indicate the acceptability of a media to a particular bacteria type. A negative control consisting of an inoculation of sterile phosphate buffer or an un-inoculated portion of media is also incubated to demonstrate the absence of contamination prior to first use of the medium.

6.4.10.14. When membrane filtration methods are used to analyze samples, a control blank of sterile dilution water is analyzed at the beginning of each set of samples. For membrane filter or plate media, duplicate counts shall be performed monthly on one positive sample for each month that the test is performed. If more than one analyst,

each analyst shall count typical colonies on the same plate and count must be within 10%. If only one analyst, sample plate shall be counted twice by the analyst, with <5% difference between counts.

6.4.10.15. The laboratory analyzes a bacteriological proficiency test sample from ERA semi-annually for NELAP accreditation. The coliform test, standard plate count, is conducted on this reference sample.

6.4.10.16. A completed test is conducted on 10% of all positive coliform samples for wastewater matrix. If no positives are found, at least one positive source water or control sample is completed quarterly.

6.4.10.17. Environmental monitoring is conducted weekly using PCA plates to measure background contamination occurring from bacteria, yeast and mold carried in the air. The number of colonies on the air density plate should not exceed 15 colonies/plate/15 minutes of exposure.

6.4.11. Asbestos

6.4.11.1. The sampling technique follows the methods outlined by EPA in Method 100.2-Analytical Method for Determining Asbestos Fibers in Water EPA-600/R-94/134, June 1994. All samples are to be stored at 4°C until filtration and completion of analysis.

6.4.11.2. Specific sampling instructions are available from the Microbiology Department. They list precautions to follow in order to maintain the integrity of the samples and prevent contamination.

6.4.11.3. The procedure is outlined in the Method 100.2. All modifications of procedures including reasons for modifications are recorded in the SOP.

6.4.11.4. All counts for calculations and report generation are entered into LIMS to eliminate inconsistency in the final report.

6.4.11.5. The manufacturers' manuals for proper operation of all equipment used in asbestos analyses are properly filed and accessible. Records of periodic inspection, calibration and service of equipment are maintained in appropriate logbooks. Phone numbers for instrument service are posted by each instrument.

6.4.11.6. Blank using fiber-free water is processed each day that samples are filtered as stated in Method 100.2. The criterion for acceptability of bottle and process blanks is < 0.01 MFL > 10 microns in length. If this limit is exceeded, the samples filtered on the same day as the blank must be re-filtered.

6.4.11.7. All samples are filtered within 48 hours of sample collection. Samples received past 48 hours of collection are treated with O3 –UV.

6.4.11.8. The absolute (HEPA) filtration system is monitored daily and filters are changed when needed.

6.4.11.9. Asbestos glassware is prepared using sonication as stated in the method.

Table 6-1 Example of Surrogate Acceptance Limits

Method	Compound	Acceptance Limits, %
504.1/8011	1,2-Dibromopropane	60-140
524.2	4-Bromofluorobenzene	70-130
	1,2-Dichloroethane-d4	70-130
	Toluene-d8	70-130
525.2	perylene-d12	70-130
	1,3-dimethyl-2-nitrobenzene	70-130
	triphenylphosphate	70-130
531.2	BDMC	70-130
551.1	1,2-Dibromopropane	80-120
624	4-Bromofluorobenzene	70-130
	1,2-Dichloroethane-d4	70-130
	Toluene-d8	70-130
625/8270	Nitrobenzene-d5	24-118
	2-Fluorobiphenyl	24-117
	Terphenyl-d14	27-149
	2-Fluorophenol	11-126
	Phenol-d5	20-118
	2,4,6-Tribromophenol	24-141
6251 B	3,5-Dichlorobenzoic Acid	70-130
8260B	4-Bromofluorobenzene	70-130
	1,2-Dichloroethane-d4	70-130
	Toluene-d8	70-130

Note: Refer to individual SOPs for detailed Surrogate Acceptance Limits.

7.0 SAMPLE COLLECTION, PRESERVATION, IDENTIFICATION, HANDLING, AND STORAGE

Sample collection and sample handling techniques are important aspects of the overall sample analysis process and have a major impact on the validity of the results. Specific containers and preservatives are used to ensure that the analytes originally present in the sample are not lost through degradation or do not become more concentrated. In addition, contaminants that would interfere with the analysis or give erroneously high results must be mitigated.

The laboratory provides sampling instructions to all clients to guide clients on the appropriate sample collection procedures. If a client chooses to collect their own samples, experienced lab staff can brief clients by telephone or in writing through EEA's sampling collection instruction on the proper methods of sample collection. If a client chooses to hire the laboratory to do the sampling, the sampling will be done by trained sampling personnel.

7.1. SAMPLE COLLECTION AND BOTTLE PREPARATION

Production of quality analytical data requires that the collected sample is representative of the sampled area. Sampling procedures should adhere to the guidelines established by EPA and other regulatory agencies and be appropriate for the sample matrix and types of analytical parameters to be determined.

The laboratory provides sampling instructions to all clients to guide clients on the appropriate sample collection procedures. If a client chooses to collect their own samples, experienced lab staff can brief clients by telephone or in writing through our sampling collection instruction on the proper methods of sample collection. If a client chooses to hire the laboratory to do the sampling, the sampling will be done by a trained sampling personnel.

Sample bottles for all analyses except bacteriological are purchased pre-cleaned according to EPA Protocol specifications from various vendors. Certification statements for each lot of bottles are kept on file in the shipping department and each bottle is marked with its lot number. Each new lot of bottles used for volatiles analyses are checked for volatiles and trace metals contamination. All files regarding Bottle Testing are kept in the QA Files. Glass bottles are wrapped in bubble bags to prevent breakage and normally shipped to the sampling site in coolers with gel packs for chilling samples. A copy of the original kit order is included with each shipment and should be returned with properly cooled samples to the laboratory along with a properly completed chain of custody form (COC). The kit order specifies the numbers of bottles sent for each analysis and is used during the log in procedure in the laboratory.

7.2. CONTAINERS, PRESERVATIVES, HOLDING TIMES AND SAMPLE KITS

EEA supplies the appropriate sample containers, preservatives, chain-of-custody forms, coolers, and packing materials to a client upon request. The container types, bottle sizes, preservatives, container closures, and recommended holding times are shown in Table 6-1 for Drinking Water, Table 6-2 for Wastewater, and Table 6-3 for Hazardous Waste. These specifications follow CFR 136-149, Required Containers, Preservation Technique and Holding times July 1, 2011 edition and updates. Also followed is the Manual for the Certification of Laboratories Analyzing Drinking Water, Fifth Edition. Arrangements for sample kits may be made through the Client Services department. Preservatives are shipped to clients only in the specified container; bulk preservatives are not normally shipped. Only reagent grade (or better) preservatives are used. The chemicals used as preservatives are as follows:

Ascorbic Acid	Nitric acid	Sodium sulfite
Ammonium chloride	Potassium Citrate	Trizma Buffers
Copper Sulfate	Sodium hydroxide	Zinc acetate
Ethylenediamine	Sodium thiosulfate	Sodium azide
Hydrochloric acid	Sulfuric acid	Maleic Acid

Containers are delivered to the client by the following methods:

(1) Client comes to laboratory to take sample kit,
(2) Containers are sent to client by courier,
(3) Containers are shipped (via UPS/FedEx/DHL) in coolers meeting all DOT regulations.

To ensure that samples meet the temperature requirements, the laboratory checks and records the sample temperature upon receipt on the COC. The temperature check documents that the samples are kept cold (>1°C, ≤6°C), not frozen, during transport. Samples that arrive on the same day of collection and have not reached <6°C must have evidence of cooling to be acceptable.

7.3. SAMPLE STORAGE

7.3.1. Under normal circumstances storage is maintained in a refrigerator kept at 4 ± 2°C for one month from receipt [TNI-EL-V1M2-2009-5.8.9.a.i]. All samples are normally retained for at least 2 months after sample receipt or until holding times have expired, whichever is shorter. A different storage period can be arranged at the request of the client. All samples are kept in the proper storage environment for one month from receipt and then stored in the waste storage area until disposal.

7.3.2. Samples are kept in refrigerators or if storage at ambient temperature is permitted, on shelving in the designated area. Samples in the designated areas are available for the analyst to take as necessary. Documentation that these samples have been taken is available in the run log. The analyst uses their run log (Figure 8-7) as a means of tracking their samples.

7.3.3. Samples designated for volatile analysis are not kept in the same refrigerators as samples designated for non-volatile analysis.

7.3.4. Temperature in the cold storage areas is monitored twice a day at least 4 hours apart to ensure all samples meet storage temperature requirements. Storage temperatures are recorded in appropriate logbooks [TNI-EL-V1M2-2009-5.9.3.a.viii].

7.4. SAMPLE DISPOSAL

7.4.1. All laboratory wastes including excess samples, excess calibration standards, any excess test items, digestates, leachates, extracts or other sample preparation products are identified by their composition. Six waste streams are identified in the laboratory; extraction solvent, Methylene chloride wastewater, chloroform, Freon, rapid flow analyzer, corrosive acids and bases, HPLC, and flammable. Each type of waste is placed into a separate, clearly identified steel drum located in a secure area outside the laboratory. Each drum also has a characterization sheet (manifest) attached. This sheet is completed every time a waste is introduced into the drum. Drums are taken for disposal/recycling once the drum is 75 % full or every 90 days from the start date of accumulation.

7.4.2. A large majority of samples received by EEA are raw or potable waters. Residual samples, if not extracted, are disposed of by neutralizing with sodium hydroxide (NaOH) or nitric acid (HNO_3). The type and amount of waste is recorded in a logbook.

7.4.3. A continuous pH meter is attached to the effluent outfall into the city sewer to record pH of all outgoing fluids from the laboratory.

7.4.4. Hazardous waste is disposed of in 55 gallon drums. Characterization sheet is available for each type of waste or waste profile.

7.4.5. Sample disposal procedures details are available in the disposal area and available through our SOP titled, “Hazardous Waste Management and Sample Disposal Procedures”. The SOP describes the requirements for the safe and effective disposal of all sample, extract and digestate waste contained in the laboratory. Means of disposal include dispensing into manifested 55 gallon drums.

7.4.6. All samples that are considered to be potentially hazardous based upon analytical results or matrix will be disposed of through a hazardous waste disposal company or a client may request that the samples be returned to them for disposal. All disposal arrangements should be made with a project manager. All samples are disposed of in accordance to RCRA and county regulations.

Table 7-1 Preservation and Holding Times for Drinking Water

(A) Inorganics – Wet Chemistry – Drinking Water

No Extract Holding Time

Parameter/ Method Name	EPA/SM Method Number	Preservative	Sample Holding Time	Recommended Minimum Sample Size	Type of Container
Alkalinity	SM2320B	Cool, 4 ± 2°C	14 days	125 mL	Plastic
Bromate	EPA 300.1/ EPA 317.0	5mg Ethylene Diamine (EDA)/ 125 mL	28 days	125 mL	Plastic
Bromide	EPA 300.0/ EPA 300.1	None or EDA	28 days	125 mL	Plastic
Chloride	EPA300.0	None	28 days	125 mL	Plastic
Chlorate	EPA 300.0/ EPA 300.1	5 mg Ethylene Diamine/125 mL	28 days	125 mL	Plastic
Chlorite	EPA 300.0/ EPA 300.1/ EPA 317.0	5 mg Ethylene Diamine/ 125 mL Cool, 4 ± 2°C	14 days	125mL	Plastic
Color	SM2120B	Cool, 4 ± 2°C	48 hours	500 mL	Glass
Conductivity	SM2510B	Cool, 4 ± 2°C	28 days	125 mL	Plastic
Cyanide	SM4500CN-F/ EPA335.4	Cool, 4 ± 2°C, 1 mL Ascorbic acid. (if chlorinated), 1 mL NaOH, pH>12	14 days	1 L	Plastic
Fluoride	SM4500 F-C	None	28 days	125 mL	Plastic
Foaming Agents Surfactant (MBAS)	SM5540C	Cool, 4 ± 2°C	48 hours	500 mL	Plastic
Nitrate (chlorinated)	EPA300.0/ EPA 353.2	Cool, 4 ± 2°C	14 days	125 mL	Plastic
Nitrate (non-chlorinated)	EPA300.0/ EPA 353.2	Cool, 4 ± 2°C	48 hours	125 mL	Plastic
Nitrate + Nitrite	EPA 353.2 EPA 300.0	Cool, 4 ± 2°C, 0.5 mL H_2SO_4, pH<2	28 days	125 mL	Plastic
Nitrite	EPA300.0 EPA 353.2	Cool, 4 ± 2°C	48 hours	125 mL	Plastic
Odor	SM2150B	Cool, 4 ± 2°C	24 hours	500 mL	Glass
Perchlorate	EPA 314	None	28 days	125 mL	Plastic
Perchlorate	EPA 331	Sterile, Cool, 4 ± 2°C	28 days	125 mL	Plastic
pH	SM4500-HB	Cool, 4 ± 2°C	15 minutes*	125 mL	Plastic

Parameter/ Method Name	EPA/SM Method Number	Preservative	Sample Holding Time	Recommended Minimum Sample Size	Type of Container
o-Phosphate	SM4500 P-E	Filter immediately, Cool, 4 ± 2°C	48 hours	125 mL	Polyglass Glass
Residual Disinfectant (Total/Free Residual Chlorine)	SM 4500 Cl-G	None (Analyzed on the day of collection)	15 minutes*	200 mL	Amber Glass Bottle
Silica Dissolved/ Reactive Silica	EPA 200.7 SM 4500Si-D	Cool, 4 ± 2°C	28 days	125 mL	Plastic
Solids (TDS)	SM 2540C	Cool, 4 ± 2°C	7 days	125 mL	Plastic
Sulfate	EPA 300.0	Cool, 4 ± 2°C	28 days	125 mL	Plastic
Turbidity	EPA 180.1	Cool, 4 ± 2°C	48 hours	125 mL	Plastic
Total Organic Carbon/ Dissolved Organic Carbon (DOC)	SM 5310 C/ EPA 415.3	0.5 ml H_2SO_4 to pH<2 Cool, 4 ± 2°C	28 days	125 mL	Amber Glass Bottle Teflon lined cap
UV 254/SUVA	SM 5910 B/ EPA 415.3	Cool, 4 ± 2°C	48 hours	125 mL	Amber Glass Bottle Teflon lined cap

* Must be analyzed immediately in the field for compliance.

(B) Inorganics – Metals – Drinking Water

No Extract Holding Time

Parameter/ Method Name	EPA/SM Method Number	Preservative	Sample Holding Time	Recommended Minimum Sample Size	Type of Container
Metals (except Hg)	EPA200.7/ EPA200.8	0.5 mL HNO_3, pH<2	6 months	1 L	Plastic
Metals (Ca, Mg, K, Na)	EPA200.7	0.5 mL HNO_3, pH<2	6 months	500 mL	Plastic
Mercury	EPA245.1	2 mL HNO_3, pH<2	28 days	500 mL	Plastic
Chromium VI (Dissolved)	EPA218.6	Ammonium Sulfate/Ammonium Hydroxide Buffer, NaOH, 4 ± 2°C, pH 9.2-9.7	28 days (24 hours for DW)	125 mL	Plastic
Hardness	EPA200.7/ SM 2340B	0.5 mL HNO_3, pH <2	28 days	500 mL	Plastic

(C) Microbiology/Microscopy Tests – Drinking Water

No Extract Holding Time

Parameter/ Method Name	EPA/SM Method Number	Preservative	Sample Holding Time	Recommended Minimum Sample Size	Type of Container
Asbestos	EPA 100.2	Cool, 4 ± 2°C	48 hours	1 L	1 L Plastic Bottle
Drinking Water Source Enumeration	SM9223 (Colilert) SM9221BE (MTF)	Cool, 4 ± 2°C, 0.2 mL of 3% $Na_2S_2O_3$	8 hours	100 mL	Sterile Plastic Bottle
Fecal Coliforms--EC Medium	SM9221E (MTF)	Cool, 4 ± 2°C, 0.2 mL 3% $Na_2S_2O_3$	30 hours	100 mL	Sterile Plastic Bottle
Heterotrophic Plate Count (Standard Plate Count)	SM9215B	Cool, 4 ± 2°C, 0.2 mL 3% $Na_2S_2O_3$	8 hours	100 mL	Sterile Plastic Bottle
Total Coliforms; By Multiple Tube Fermentation (MTF)	SM9221AB	Cool, 4 ± 2°C, 0.2 mL 3% $Na_2S_2O_3$	30 hours	100 mL	Sterile Plastic Bottle
Total Coliforms--E. Coli	SM9223	Cool, 4 ± 2°C	30 hours	100 mL	Sterile Plastic Bottle
Total Coliforms--E. Coli	SM 9223B - Colisure	Cool, 4 ± 2°C	30 hours	100 mL	Sterile Plastic Bottle
Coliphage	EPA 1602	Cool, 4 ± 2°C	48 hours	1000 mL	Sterile Plastic Bottle

(D) Organics – Drinking Water

Parameter/ Method Name	EPA/SM Method Number	Preservative	Sample Holding Time	Extract Holding Time	Recommended Minimum Sample Size	Type of Container
DBCP/EDB	EPA504.1	3 mg Sodium Thiosulfate Cool, 4 ± 2°C	14 days	4 °C, 24 hours	40 mL	Glass with Teflon Lined Septum

Parameter/ Method Name	EPA/SM Method Number	Preservative	Sample Holding Time	Extract Holding Time	Recommended Minimum Sample Size	Type of Container
Organohalide Pesticides and PCB	EPA 505	3 mg Sodium Thiosulfate Cool, 4 ± 2°C	14 days/ 7 days for heptachlor	4 °C, 24 hours	40 mL	Vial with PTFE-lined Screw caps
Chlorinated Herbicides (GC with Electron Capture	EPA515.4	Sodium Sulfite, dark, cool ≤ 10°C (first 48 hours, 4 ± 2°C after 48 hours)	14 days	≤0°C dark, 21 days	1 L	Amber Glass with Teflon lined Cap
Nitrosamines	EPA 521	80 – 100 mg sodium thiosulfate; Cool, 10°C (first 48 hours, 4 ± 2°C after 48 hours)	40 days	7 days	1 L	Amber glass with PTFE-lined Screw caps
Purgeable Organic Compounds/ Halogenated Aromatics, THMs, Di-Isopropyl Ether (DIPE), Tertiary Amyl methyl ether (TAME), Tert Butyl ethyl ether (ETBE) Low level TCP	EPA 524.2	25 mg Ascorbic Acid, then HCl pH < 2; Cool, 4 ± 2°C	14 days	NA	2x40 ml	Teflon Lined Septum
	EPA 524.3	25 mg Ascorbic Acid, Maleic Acid; Cool, 4 ± 2°C	14 days	NA	3x 40 mL	Teflon Lined Septum
Low Level TCP (GC/MS)	EPA 524.2/ CA DHS	Cool, 4 ± 2°C or thiosulfate, HCL pH<2	14 days	NA	2x40 ml	Teflon Lined Septum
Semi-Volatile Organics Acid/Base Neutrals, including thiobencarb (GC/MS)	EPA525.2	40-50 mg Sodium Sulfite, Dark, Cool, 4 ± 2°C, HCl, pH<2. HCL must be added after sample dechlorination	14 days	≤ 4˚C 30 days from collection	1 L	Amber Glass with teflon lined Cap

Parameter/ Method Name	EPA/SM Method Number	Preservative	Sample Holding Time	Extract Holding Time	Recommended Minimum Sample Size	Type of Container
Acetanilide Pesticide Parent Compounds	EPA 525.2	40-50 mg Sodium Sulfite, Dark, Cool, 4 ± 2°C, HCl, pH<2. HCL must be added after sample dechlorination	14 days	≤ 4˚C 14 days	1 L	Amber Glass with teflon lined Cap
Pesticides and Flame Retardants	EPA 527	0.10 g/L of L-Asorbic Acid, 0.35 g/L of Trisodium EDTA, and 9.4 g/L of Potassium dihydrogen citrate; Cool, 10°C (first 48 hours, 4 ± 2°C after 48 hours)	28 days	14 days	1 L	Amber glass with PTFE-lined Screw caps
Explosives	EPA 529	0.5 g/L of Copper Sulfate pentahydrate, 5.0 g of Trizma buffer; Cool, 10°C (first 48 hours, 4 ± 2°C after 48 hours)	30 days	14 days	1 L	Amber glass with PTFE-lined Screw caps
Carbamates	EPA 531.2	0.38 g/40-mL vial Potassium dihydrogen citrate If residual chlorine is present, 6-mg of sodium thiosulfate/40-mL vial	Cool, <10°C first 48 hrs; 4 ± 2°C thereafter; dark; 28-days; pH - 3.8	< 6°C; 28-days	40 mL	Vial with PTFE-lined Screw caps
Acetanilide Pesticide Degradation Products	EPA 535	25 – 30 mg ammonium chloride; Cool, 10°C (first 48 hours, 4 ± 2°C after 48 hours)	28 days	14 days	250 mL	Amber glass with PTFE-lined Screw caps

Parameter/ Method Name	EPA/SM Method Number	Preservative	Sample Holding Time	Extract Holding Time	Recommended Minimum Sample Size	Type of Container
Glyphosate (HPLC with Fluorescence Detector)	EPA547	6 mg Sodium Thiosulfate Cool, 4 ± 2°C	14 days (18 mo. If frozen)	NA	60 mL	Amber Glass with teflon lined septum
Endothall (GC/MS)	EPA548.1	Sodium Thiosulfate (HCl, pH 1.5-2 if high bio activity) Cool, 4 ± 2°C, Dark	7 days	14 days ≤4°C	250 mL	Amber Glass with teflon lined septum
Diquat & Paraquat (HPLC with Photoiode, Array Detector)	EPA549.2	100 mg Sodium Thiosulfate (H_2SO_4, pH<2 if bio active) Cool, 4 ± 2°C, Dark	7 days	21 days	1 L	Amber Plastic
THMs & EDB (1-2,dibromoethane) DBCP (1,2-dibromo-3-chloropropane	EPA 551.1	Sodium Sulfite, 10-50 mg NH4Cl/40 mL + 400-mg phosphate buffer/ 40 ml, Cool, 4 ± 2°C	14 days	14 days	3x60 ml	Amber Glass with Teflon lined cap
Haloacetic Acids	SM6251B	65 mg NH4Cl / 40 ml Cool, 4 ± 2°C	14 days	7 days	2 x 40 mL	Amber Glass with teflon lined cap
1,4-Dioxane	EPA 522	50 mg/L Sodium Sulfite ; 1 g/LSodium Bisulfate	14 days	30 days	2 x 250mL	Amber Glass with teflon lined cap
Dioxin	EPA 1613B	Sodium Thiosulfate Cool, 0-4°C	NA	40 days	1 L	Amber Glass with PTFE lined cap
Aldehyde	SM 6252	Cool, 4 ± 2°C	14 days	7 days	2 x 40 mL	Amber glass containers with

Parameter/ Method Name	EPA/SM Method Number	Preservative	Sample Holding Time	Extract Holding Time	Recommended Minimum Sample Size	Type of Container
		If residual chlorine is present, 10 – 50 mg of ammonium chloride/40-mL vial				teflon-faced septa and open top screw caps

(E) Radiochemistry – Drinking Water
No Extract Holding Time

Parameter/ Method Name	EPA/SM Method Number	Preservative	Sample Holding Time	Recommended Minimum Sample Size	Type of Container
Uranium	EPA 200.8	0.5 mL HNO_3 to pH<2	6 months	125 mL	Plastic
Gross Alpha	EPA 900.0	2.0 mL HNO_3 to pH<2	6 months	1 L	Plastic
Gross Beta	EPA 900.0	2.0 mL HNO_3 to pH<2	6 months	1 L	Plastic
Radium 228	EPA 904.0	2-mL HNO_3 per liter; pH <2	6-months, if unpreserved; after 5-days, preserve and hold in the original container for minimum of 16-hrs.before analysis	1 L	Plastic
Radon 222	SM 7500 Rn	None, no headspace	4 days	250 ml	Glass

Note: Refer to individual SOPs for preservation and holding times for all other methods not listed in Quality Manual.

Table 7-2 Preservation and Holding Times for Wastewater

(A) Inorganics – Wet Chemistry – Waste Water
No Extract Holding Time

Parameter/ Method Name	EPA/SM Method Number	Preservative	Sample Holding Time	Recommended Minimum Sample Size	Type of Container
Alkalinity, (Bicarbonate, Carbonate, & Total Hydroxide)	SM 2320B	Cool, > 0°C , < 6°C not frozen	14 days	125 mL	Plastic
Ammonia	EPA350.1 SM4500NH3-H	Cool, > 0°C , < 6°C not frozen, 0.5 mL of H_2SO_4 to pH < 2	28 days	125 mL	Plastic
Biochemical Oxygen Demand (BOD)	SM5210B	Cool, > 0°C , < 6°C not frozen	48 hours	500 mL	Plastic
Bromide	EPA300.0	None	28 days	125 mL	Plastic
Carbon Biochemical Oxygen Demand (CBOD)	SM5210B	Cool, > 0°C , < 6°C not frozen	48 hours	500 mL	Plastic
Chemical Oxygen Demand (COD)	EPA410.4/ SM 5220D	Cool, > 0°C , < 6°C not frozen, 0.5 mL of H_2SO_4 to pH < 2	28 days	125 mL	Plastic
Chloride	EPA300.0	None	28 days	125 mL	Plastic
Chlorine, Total Residual	SM4500 Cl G	Cool, > 0°C , < 6°C not frozen	15 minutes * (immediately)	250 mL	Amber Glass
Chromium VI	SM 3500Cr-D/ EPA 218.6	Cool, > 0°C , < 6°C not frozen, Ammonia Sulfate buffer, NaOH, pH 9.3-9.7,	28 days	125 mL	Plastic
Cyanide, Total	EPA 335.4	Cool, > 0°C , < 6°C not frozen, 4 mL NAOH to pH>12, 0.6 g Ascorbic Acid (if chlorinated)	14 days	1 L	Plastic
Cyanide, Amenable to Chlorination	EPA 335.1/ SM 4500 CN-G	Cool, > 0°C , < 6°C not frozen, 4 mL of NAOH to pH>12, 0.6 g Ascorbic Acid (if chlorinated)	14 days	1 L	Plastic
Fluoride	SM4500 F-C	None	28 days	125 mL	Plastic
Hardness	EPA 200.7/ SM 2340B	1.0 mL HNO_3 to pH< 2	6 months	250 mL	Plastic
Kjeldahl Nitrogen	EPA 351.2	Cool, > 0°C , < 6°C not frozen, 0.5 mL of $H_2SO_4$to pH < 2	28 days	125 mL	Plastic

Parameter/ Method Name	EPA/SM Method Number	Preservative	Sample Holding Time	Recommended Minimum Sample Size	Type of Container
Nitrate	EPA 353.2/ EPA 300.0	Cool, > 0°C , < 6°C not frozen	48 hours	125 mL	Plastic
Nitrite	EPA300.0/ EPA 354.1/ 353.2	Cool, > 0°C , < 6°C not frozen	48 hours	125 mL	Plastic
Orthophosphate	EPA 365.1/ SM4500 P-F	Filter Immediately, Cool, > 0°C , < 6°C not frozen	48 hours	125 mL	Plastic
Perchlorate	EPA 314.0	None	28 days	125 mL	Plastic
pH	SM4500-HB	None	15 minutes *	125 mL	Plastic
Phenols	EPA 420.4/ EPA 420.1	Cool, > 0°C , < 6°C not frozen, 2.0 mL H_2SO_4 to pH < 2	28 days	500 mL	Amber Glass
Phosphorus, Total	EPA 365.1/ SM4500 P-F	Cool, > 0°C , < 6°C not frozen,0.5 mL H_2SO_4 to pH < 2	28 days	125 mL	Plastic
Residue, Filterable (Total Dissolved Solids--TDS)	SM2540C	Cool, > 0°C , < 6°C not frozen	7 days	500 mL	Plastic
Residue, Non-filterable (Total Suspended Solids,TSS)	SM 2540D	Cool, > 0°C , < 6°C not frozen	7 days	500 mL	Plastic
Residue, Settleable (Settleable Solids)	EPA 160.5/ SM 2540F	Cool, > 0°C , < 6°C not frozen	48 hours	500 mL	Plastic
Specific Conductance	SM 2510B	Cool, > 0°C , < 6°C not frozen	28 days	125 mL	Plastic
Sulfate	EPA300.0	Cool, > 0°C , < 6°C not frozen	28 days	125 mL	Plastic
Sulfide (Total & Soluble)	SM 4500 S-2D	Cool, > 0°C , < 6°C not frozen, Zinc Acetate, plus NaOH to pH > 9	7 days	125 mL	Plastic
Total Organic Carbon (TOC)	SM 5310C	Cool, > 0°C , < 6°C not frozen,0.5 mL H_2SO_4 to pH < 2	28 days	125 mL	Amber Glass
Total Organic Halide (TOX)	SM 5320B	Sulfite & H_2SO_4	14 days	250 mL	Amber Glass
Turbidity	EPA180.1	Cool, > 0°C , < 6°C not frozen	48 hours	125 mL	Plastic

* Must be analyzed immediately in the field for compliance.

(B) Inorganics – Metals – Waste Water

No Extract Holding Time

Parameter/ Method Name	EPA/SM Method Number	Preservative	Sample Holding Time	Recommended Minimum Sample Size	Type of Container
Metals (except Hg)	EPA200.7 EPA200.8	0.5 mL HNO_3 to pH< 2	6 months	125 mL	Plastic
Metals (Ca, Mg, K, Na)	EPA200.7	0.5 mL HNO_3 to pH< 2	6 months	125 mL	Plastic
Mercury, Hg	EPA245.1	2.0 mL HNO_3 to pH< 2	28 days	500 mL	Plastic

(C) Microbiology/Microscopy Tests – Waste Water

No Extract Holding Time

Parameter/ Method Name	EPA/SM Method Number	Preservative	Sample Holding Time	Recommended Minimum Sample Size	Type of Container
Asbestos	EPA 100.2	Cool, > 0°C , < 6°C not frozen	48 hours	800 mL	Plastic (1 L)
Fecal Coliforms By Multiple Tube	SM9221E	Cool, > 0°C , < 6°C not frozen; 0.2 mL 3% $Na_2S_2O_3$ (if chlorinated)	6 hours	100 mL	Sterile Plastic
Fecal Streptococci/ Enterococci by MTF	SM9230B	Cool, > 0°C , < 6°C not frozen; 0.2 mL 3% $Na_2S_2O_3$ (if chlorinated)	6 hours	100 mL	Sterile Plastic
Heterotrophic Plate Count	SM9215B	Cool, > 0°C , < 6°C not frozen; 0.2 mL 3% $Na_2S_2O_3$ (if chlorinated)	6 hours	100 mL	Sterile Plastic
Total Coliforms By Multiple Tube Fermentation (MTF)	SM9221B	Cool, > 0°C , < 6°C not frozen; 0.2 mL 3% $Na_2S_2O_3$ (if chlorinated)	6 hours	100 mL	Sterile Plastic

(D) Organics – Waste Water

Parameter/ Method Name	EPA/SM Method Number	Preservative	Sample Holding Time	Extract Holding Time	Recommended Minimum Sample Size	Type of Container
Halogenated Volatiles/ Aromatic Volatiles	EPA 624	Cool, > 0°C , < 6°C not frozen, 10 mg $Na_2S_2O_3$ for residual Cl_2,HCl** pH < 4-5	14 days	NA	40 mL	Amber Glass/ Teflon lined Septum
Semi-Volatiles, Acid and Base/ Neutral Compounds	EPA 625	Cool, > 0°C , < 6°C not frozen, 80 mg $Na_2S_2O_3$ for residual Cl_2	7 days	40 days	1 L	Amber Glass/ Teflon lined Cap

**HCl must be added after sample dechlorination

(E) Radiochemistry – Waste Water

No Extract Holding Time

Parameter/ Method Name	EPA/SM Method Number	Preservative	Sample Holding Time	Recommended Minimum Sample Size	Type of Container
Uranium	EPA200.8	0.5 ml HNO_3 to pH <2	6 months	125 ml	Plastic
Gross Alpha	EPA900.0	4.0 mL HNO_3 (18%) to pH<2	6 months	1 L	Plastic
Gross Beta	EPA900.0	4.0 mL HNO_3 (18%) to pH<2	6 months	1 L	Plastic
Radon 222	SM 7500 Rn-B	None	4 days	250 ml	Glass

Note: Refer to individual SOPs for preservation and holding times for all other methods not listed in Quality Manual.

Table 7-3 Preservation and Holding Times for Hazardous Waste (Aqueous Matrix Only)

(A) Inorganics – Wet Chemistry – Hazardous Waste (Aqueous)

No Extract Holding Time

Parameter/ Method Name	Matrix	EPA/SM Method Number	Preservative	Sample Holding Time	Sample Size	Type of Container

Parameter/ Method Name	Matrix	EPA/SM Method Number	Preservative	Sample Holding Time	Sample Size	Type of Container
Chromium VI	Aqueous	EPA 7196A EPA 7199	Cool, > 0°C , < 6°C not frozen, add buffer or NAOH	24 hours	125 mL	Plastic
Conductivity	Aqueous	EPA 9050A	Cool, > 0°C , < 6°C not frozen	28 days	125 mL	Plastic
Cyanide, Total	Aqueous	EPA 9012A	4 mL NaOH to pH > 12, Cool, > 0°C , < 6°C not frozen	14 days	1 L	Plastic
Fluoride	Aqueous	EPA 9214	Cool, > 0°C , < 6°C not frozen	28 days	125 mL	Plastic
Nitrate as N	Aqueous	EPA 9056	Cool, > 0°C , < 6°C not frozen	48 hours	125 mL	Plastic
Perchlorate	Aqueous	EPA 314/ EPA 331	Sterile, > 0°C , < 6°C not frozen	28 days	125 mL	Plastic
pH	Aqueous	EPA 9040B	None	7 days	125 mL	Plastic
Phenol	Aqueous	EPA 9066	Cool, >0°C, < 6°C not frozen, 2.0 mL H_2SO_4 to pH < 2	28 days	500 mL	Amber Glass
Sulfide, Total	Aqueous	EPA 9030B	Zinc Acetate, NaOH pH > 9, Cool, > 0°C , < 6°C not frozen	7 days	125 mL	Plastic
Total Organic Halides (TOX)	Aqueous	EPA 9020B	Sulfite & H_2SO_4	14 days	250 mL	Amber Glass
Chloride, Chlorite, Sulfate, Nitrite	Aqueous	EPA 9056	Cool, > 0°C , < 6°C not frozen	48 hours	125 ml	Plastic

(B) Inorganics – Metals – Hazardous Waste (Aqueous)
No Extract Holding Time

Parameter/ Method Name	Matrix	EPA/SM Method Number	Preservative	Sample Holding Time	Sample Size	Type of Container
Arsenic, As, Dissolved	Aqueous	EPA 6020	0.5 mL HNO_3 to pH < 2	6 months	125 mL	Plastic
Arsenic, As, Total						
Mercury, Total	Aqueous	EPA 7470A	2.0 mL HNO_3 to pH < 2	28 days	500 mL	Plastic

Parameter/ Method Name	Matrix	EPA/SM Method Number	Preservative	Sample Holding Time	Sample Size	Type of Container
Mercury, Dissolved			Filtered on site, 2.0 mL HNO_3 to pH < 2			
Metals, Total *	Aqueous	EPA 6010B EPA 6020	0.5 mL HNO_3 to pH < 2	6 months	125 mL	Plastic
Metals, Dissolved *	Aqueous	EPA6010B EPA6020	Filtered on site, HNO_3 to pH < 2	6 months	125 mL	Plastic

* Aluminum, Antimony, arsenic, barium, beryllium, cadmium, chromium, cobalt, copper, lead, manganese, molybdenum, nickel, selenium, silver, strontium, thallium, tin, titanium, vanadium and zinc.

(C) Organics – Hazardous Waste (Aqueous)

Parameter/ Method Name	Matrix	EPA/SM Method Number	Preservative	Sample Holding Time	Extract Holding Time	Recommended Minimum Sample Size	Type of Container
EDB/DBCP	Aqueous	EPA 8011	3 mg sodium thiosulfate, Cool, > 0°C , < 6°C not frozen	14 days	< 6°C, 24 hours	40 mL	Glass/ Teflon lined septum
Halogenated Volatiles & Aromatic Volatiles	Aqueous	EPA8260B	10 mg $Na_2S_2O_3$ for residual chlorine, HCl, pH < 2 Cool, > 0°C , < 6°C not frozen	14 days	NA	40 mL	Amber Glass/ Teflon lined Septum
Semi-Volatile Organic Compounds	Aqueous	EPA8270C	80 mg $Na_2S_2O_3$, Cool, > 0°C , < 6°C not frozen	7 days	40 days	1 L	Amber Glass/ Teflon lined Cap
1,4-Dioxane	Aqueous	50 mg/L Sodium Sulfite ; 1 g/LSodium Bisulfate	EPA 522	50 mg/L Sodium Sulfite ; 1 g/LSodium Bisulfate	30 days	2 x 250mL	Amber Glass with teflon lined cap

Note: Refer to individual SOPs for preservation and holding times for all other methods not listed in Quality Manual.

8.0 SAMPLE MANAGEMENT

8.1. SAMPLE RECEIPT AND LOG-IN/SAMPLE RECEIPT PROTOCOL

EEA receives all samples through its sample control group. Upon receipt of samples, the sample control group inspects each sample for breakage or leakage, inverted septa, inappropriate caps or bottles, air bubbles in volatile organics samples, incomplete sample labels, incomplete paperwork, or discrepancies between the sample labels and the paperwork. The sample custodian checks the sample temperature to ensure that the required temperature is maintained during transport. EPA requires for most methods that a sample temperature of 4 ± 2°C (TNI > 0, < 6˚C) shall be maintained during transport. The sample custodian records the sample temperature on the Chain of Custody. If the reading is above 6°C, the Project Manager (PM) is notified who then notifies the client regarding his sample condition. For samples that arrive at the laboratory > 6°C, the client will be notified that the affected samples are unacceptable for regulatory compliance purposes, if not received on the same day of collection with evidence of cooling, and analysis is at the discretion of the client. (Acceptance criteria as per MUR, March 12, 2007 is ≤ 6°C).

The sample custodian also screens all hazardous waste and wastewater samples from a new client with the Geiger Counter meter for presence of radiation levels above background. For additional details refer to Sample Receiving and Log-In SOP. Any sample receipt problems are recorded either on the Chain of Custody (COC) Form (Figure 8-6) for Level I or on COC and Sample Cooler Receipt Form (Figure 8-1) for Level II samples. The Client Services Manager or designated Project Manager is notified about the problems. The client is informed of these problems, the appropriate course of action is determined and a decision is made immediately whether re-sampling is required.

Sample control employees are designated to receive all shipments and deliveries to the laboratory. The procedure for receiving samples is detailed in the Sample Receipt SOP kept on file in the log-in area and central QA files. A EEA Kit Order Form (KO) is filled out for each client's samples. An example of the KO is shown in Figure 8-2. A computer assigned laboratory number is placed on each sample bottle and the bottles are stored in refrigerators segregated by receipt date.

8.1.1. Sample Labeling System

Sample bottles must be clearly labeled so that the laboratory tracking system can function optimally. All sample bottles are shipped with labels containing the particular parameters to be tested from each bottle as well as any preservative information. The client must fill in the sampling date and sample site, and the client name/identification, on the label. The sample control group insures that all returned samples contain sample site identifications.

After log-in, the sample control group attaches a label with the laboratory sample tracking number to each sample bottle. All sample bottles collected for a particular

sample site normally receive the same base laboratory sample tracking number and a stamped label with this number is attached to each bottle. When analysts run a sample work schedule for their particular analysis, they receive a computer printout listing the laboratory sample numbers requiring that analysis. The analyst must then find the samples with these assigned numbers in their appropriate containers in refrigerated or non-refrigerated storage. The work schedule printout also gives the name of the client and sample ID that is compared with the information printed on the sample label to insure a proper identification.

The assigned laboratory numbers utilized for sample tracking are always a twelve-digit number. The first eight digits represent the year, month and day the sample was logged in. The remaining four digits are utilized to give each sample a unique identification number and these numbers are assigned consecutively from 1 to 9999 by the computer when the samples are logged in. These last four digits are reset back to one (1) at the beginning of each day. The laboratory also assigns a unique laboratory identification number to each sample and subsample container, and attaches a durable label to each sample container. The assignment of unique laboratory ID is done for each subsample except for samples that have short holding times. All laboratory ID codes assigned to each sample are documented in each appropriate logbooks/workbook for related laboratory activities such as sample preparation calibration and analysis.

8.1.2. Sample Receipt Acceptance Criteria:

8.1.2.1. The laboratory establishes and implements a sample acceptance/rejection policy per TNI-EL-V1M2-2009-5.8.6. The laboratory accepts a sample when the following criteria are met:

8.1.2.1.1. Proper, full, and complete documentation, which shall include sample identification, the location, date and time of collection, collector's name, preservation type, sample type and any special remarks concerning the sample;

8.1.2.1.2. Proper sample labeling to include unique identification and a labeling system for the samples with requirements concerning the durability of the labels (water resistant) and the use of indelible ink;

8.1.2.1.3. Use of appropriate sample containers;

8.1.2.1.4. Adherence to specified holding times;

8.1.2.1.5. Adequate sample volume. Sufficient sample volume must be available to perform the necessary tests.

8.1.2.1.6. Procedures to be used when sample shows signs of damage or contamination.

8.1.2.1.7. All samples, which require thermal preservation, shall be considered acceptable if the arrival temperature is ≤ 6°C, not frozen or the method specified range. For samples with a specified temperature of 4°C, samples with a temperature ranging from just above the freezing temperature of water to 6°C shall be acceptable. Samples that are hand delivered to the laboratory immediately after collection may not meet these criteria. In these cases, the samples shall be considered acceptable if there is evidence that the chilling process has begun such as arrival on ice.

8.1.2.1.8. The laboratory implements procedures for checking chemical preservation using readily available techniques, such as pH or free chlorine, prior to or during sample preparation or analysis [TNI-EL-V1M4-2009-1.7.5.b]. Residual Free Chlorine and pH testing are done for Volatile samples (524.2). Also samples for semivolatiles by 525.2 analysis and THMs by 551.1 are verified for proper preservation by checking the pH of the sample at the analyst's sample preparation area.

8.1.2.2. Results of all checks are recorded in the appropriate logbooks. If the sample does not meet the laboratory sample receipt acceptance criteria, the laboratory either:

8.1.2.2.1. Retains correspondence and/or records of conversations concerning the final disposition of rejected samples; or

8.1.2.2.2. Fully documents any decision to proceed with the analysis of samples not meeting acceptance criteria.

8.1.2.2.2.1. The condition of these samples shall, at a minimum, be noted on the chain of custody or transmittal form and laboratory receipt documents.

8.1.2.2.2.2. The analysis data shall be appropriately "qualified" on the final report.

8.1.2.3. After LIMS entries have been completed for a group, a sample acknowledgment is printed out (see figure 8-9). The original acknowledgment is emailed to the client immediately after log-in and reviewed by the client's project manager. The sample acknowledgment report allows the clients to confirm if methods and tests assigned to the samples are correct.

8.2. CHAIN OF CUSTODY

Chain of custody procedures provides legal evidence that tampering with a sample has not occurred. This is achieved by documenting an accurate written record tracing possession of the sample from collection through its final analysis and disposal. The EEA's chain of custody form provided with sample bottle shipments is presented in Figure 8-6. The laboratory maintains two levels of custody. As a standard protocol, the laboratory utilizes Level I chain of custody. Level II chain of custody is available upon request only and at an additional charge.

8.2.1. Level I

This process relies on the fact that the laboratory is a secure building. The laboratory either has custody of the sample, or not. Evidence of laboratory custody is shown through the receipt signatures on the chain of custody form. Documentation is available in the laboratory for the tracking and disposition of a sample, however this information is not intended to withstand rigorous legal scrutiny. Level I chain of custody is consistent with EPA's definition of custody. Documentation associated with this level of custody includes:

8.2.1.1. A copy of the Chain of Custody is kept in the project file.

8.2.1.2. Run logs indicating when samples were handled/analyzed.

8.2.2. Level II

Also known as Legal Chain of Custody, this process requires that the disposition of each sample be defined in terms of time and possession for the life span of the sample; from sample bottle preparation to the disposal or complete depletion of the sample during analysis. Documentation associated with this level of custody includes:

8.2.2.1. Requirements for Level I followed

8.2.2.2. Chain of custody signed by sample control personnel upon receipt of sample(s)

8.2.2.3. Airbills and/or courier receipts filed in the project file by sample control

8.2.2.4. Internal custody logbook and key to secure and separate storage refrigerators maintained by sample control personnel; all sample/extract/digestate transfers, including those to secured storage, recorded herein

8.2.2.4.1. This storage area is locked and entry is permitted only upon signing for the custody of the sample(s)/extract(s)/digestate(s).

8.2.2.5. Internal custody logbook entries include client, client sample ID, date sampled, analyses, laboratory ID, internal dates and times transferred, initials (all samples are returned at the end of each shift) see Figure 8-4.

8.2.2.6. Upon disposal the technician will complete the custody notebook (all client identifying label(s) on the container defaced or removed)

8.2.2.7. Errors deleted by drawing a single line through the item, dating and initialing and reasons clearly indicated.

8.2.2.8. Disposal of samples occur only with the concurrence of the affected legal authority, sample data user and/or submitter of the sample

8.2.2.9. Conditions of disposal and all correspondence between all parties concerning final disposition of the physical sample recorded and retained by the laboratory

8.2.2.10. Level II chain of custody sample disposal logbook (Figure 8-5) which indicates the date of disposal, nature of disposal (such as sample depleted, sample disposed in hazardous waste facility or sample returned to client, and the name of the individual who performed the task

8.2.3. Sub-contract Laboratories

When samples are sent to a sub-contract laboratory, a chain of custody is initiated by sample control or the subcontract administration group. The original chain of custody is filed in the project file with a reference to the second chain of custody. This sample is tracked internally and is identified as a subbed-out sample from an entry made into LIMs by sample control. All information from the original chain of custody is transferred to the second chain of custody in addition our internal Laboratory IDs are referenced. If samples were extracted at EEA and the extracts sent out, then the QC set for that extraction batch is sent out to the sub-contract laboratory also.

8.2.4. The Quality Manager or the Project Manager periodically inspects the chain of custody logbook to verify that analysts are signing samples back into custody the same day they are removed.

8.3. SAMPLE STORAGE AND DISPOSAL

Sample storage and disposal procedures are found in section 7.3 Sample Storage and 7.4 Sample Disposal.

8.4. SAMPLE TRACKING

When samples pass initial inspection, they are logged into the computer running the lab LIMS system. This system tracks samples from the time they arrive in the laboratory until final data are transmitted to the client. Multiple queries can be made of the database, and new routines can be written for retrieving certain information in a specified format. The following are example queries made from LIMS, printouts of these queries are available for personnel, on demand:

8.4.1. **Sample Disposition** - Shows which analyses have been performed on a given sample, which results have been validated by the manager/supervisor, and the results.

8.4.2. **Due Date/Hold time Date** - Allows analysts to schedule tests by accessing sample information according to priority date (hold time/turnaround time); query can be made per test, per group, per client, or per prompted date.

8.4.3. **QC Data** - Accessibility to QC information which can be tabulated and used to derive acceptability ranges, trend analyses, control charts etc.

8.4.4. **Formats** - Data is available for clients in various hard-copy layouts and/or electronic data format.

8.5. LABORATORY INFORMATION MANAGEMENT SYSTEM (LIMS)

8.5.1. The current Laboratory Information Management Software (LIMS) used by EEA is the STARLIMS software package developed by STARLIMS Corporation located in Hollywood, FL. The XML web based system is written in a proprietary scripted language and accesses a Microsoft SQL server.

8.5.2. The STARLIMS system provides functions to access client accounts, tests/analyses, sample tracking, test backlog generation, data entry/verification, data validation, client data in a variety of formats, monthly financial and statistical reports, and archival storage of data.

8.5.3. The security of the information contained in the LIMS is maintained through the restricted use of the database. All personnel have a unique access ID and password. The type of information entered or queried is dependent upon the level of access associated with the user.

8.5.4. LIMS has several defined user types. Access to key areas is restricted and based on the user type designated.

8.5.4.1. **Analyst/Reviewer** – Original data is entered by an analyst on an analytical batch basis (batch is no more than 20 samples). Once entered, the analyst cannot change the data nor can the analyst access the data at a higher level to review. Review must be conducted by a peer or supervisor.

8.5.4.2. **Manager/Validation** – After the secondary check, the group manager validates the data. Upon validation, the data is available to the client.

8.5.4.3. **User** – Personnel who only query the database, rather than enter data, are assigned this third level of access.

8.5.5. Final reports are uploaded to an external server (DMZ) for the client to access via a secure password and unique ID. Hardcopies are mailed upon request. Reports are electronically signed by the Project Manager before uploading to the DMZ. Invoices are mailed or electronically delivered based on client preference.

8.5.5.1. **Report Storage** – The LIMS generates an electronic file that is uploaded to an internal server. The files are stored by client/project and file type. Version numbers are noted on the file name extensions. Reports are kept electronically for 10 years. Hardcopies are no longer printed and scanned as the STARLIMS system can electronically merge all components of a final report.

8.5.5.2. **Electronic Data Deliverables (EDDs**) – Electronic data or magnetic medium data are delivered to the client using e-mail or by the client accessing the DMZ via a unique ID and password. The LIMS generates the final data report and merges this with the Chain of Custody, Acknowledgement sheet, subcontract report, case narrative, and any explanations regarding the visual sample condition into one file for the client to access.

8.5.5.3. **LIMS Maintenance** – The LIMS hardware is maintained by the IT Department of EEA and the Systems Architect/IT Manager on-site at the laboratory. The software is maintained by STARLIMS and upgrades are provided by the vendor for a yearly fee. Software validation is performed by the vendor prior to installation of the product.

A hardware/software maintenance logbook is kept with the manager of computer services. In addition to this record, all servicing performed by STARLIMS or outside vendors is documented by their staff and is available for our use.

8.5.6. Sample Status

Samples are logged into the system upon receipt in the laboratory. A laboratory number is assigned to each sample by the computer and the required tests are scheduled. Each sample then appears on the work schedule for the appropriate department. Turnaround time is automatically assigned to each sample test based on the sampling date and time and EPA holding times.

The work schedule is the primary means of checking the backlog for the analyst. The analyst can schedule the samples according to priority date, which is calculated according to the laboratory turnaround time and priority. An example of a computer generated work schedule is shown in Figure 8-8.

Operations meetings are held weekly to discuss the status of data. An Operations Report (Figure 8-10) is used by the supervisors and Project Managers during operations meetings. The Operations Report includes the group No., Client ID, Total number of Tests, Tests ready to be validated and, incomplete tests by department. The Operations Reports allow the supervisor and the project manager to monitor sample status. Also during the Operations meeting, Project managers are informed of any issues that may have arisen so that they can proactively contact the client. A list of samples with short turnaround time, 72 hours or less, is kept at sample control. Sample control contacts the analyst when short holding time samples arrive. Bottle orders are completed when clients

request containers and supplies. This allows sample control to monitor the amount of samples due to arrive in the near future.

8.5.7. Data Entry and Report Generation

Data entry is accomplished through a variety of interactive sub-systems. Some situations require the entry of raw data and the system performs calculations, and reports final results and detection limits. In other cases, final data is entered either manually or via instrument interfaces. When the final scheduled test result goes into the system, the Technical Manager/Group Supervisor passes on the reports to the validation section within the system for approval. In all cases, client reports are generated and printed automatically after the verification and approval by the supervisor of each analytical group.

Results are stored in LIMS in such a manner that immediate access is available to these reports. A list of all reports completed, indexed by client number, is maintained on the system. A few keystrokes can recall every report produced for a given client. Additionally, the system provides constant information on laboratory performance. This includes turnaround times reports for every analysis done by the laboratory, and productivity reports grouped into cost isolation accounts. A weekly laboratory Turnaround time report allows the tracking of turnaround time per department to ensure that the laboratory continuously improves its turnaround time and meets client needs. See example of weekly Lab Turnaround Time Report (Figure 8-11).

The system provides several levels of security. The first level is the entry of a password to initially log on to the computer, and then the person must be designated as a qualified user. Additionally, the department to which a person is assigned governs/accesses the various functions of the system. The system also provides for read-only access to results to further protect the data from unauthorized modification or deletion.

Figure 8-1 Cooler Receipt Form

EUROFINS EATON ANALYTICAL, INC. COOLER RECEIPT FORM

PROJECT:_____ DATE RECEIVED:_____

Use back of form to note check-in problems and describe action(s) regarding the resolution(s) of problems.

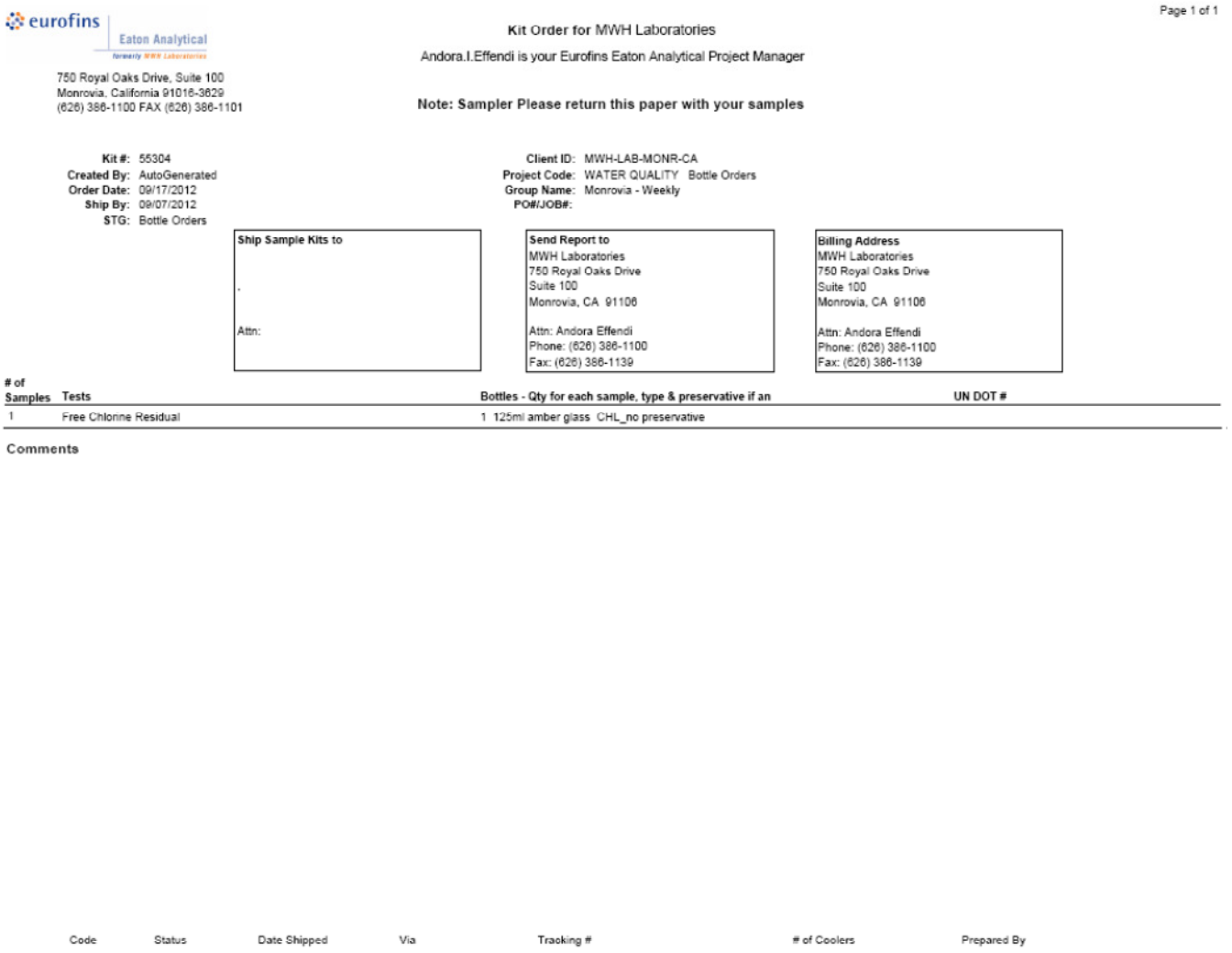
A. PRELIMANARY EXAMINATION
Date Cooler Opened: _____
By (print)_____ (sign)_____

1.	Did cooler come with shipping slip (air bill, etc.)? If yes, attach and enter carrier and air bill number here:_____	YES	NO
2.	Were custody seals on outside of cooler? If yes, how many and where:_____ If yes, enter the following: seal date:_____ seal name:_____	YES	NO
3.	Were custody seals unbroken and intact at delivery?	YES	NO
4.	Were custody papers sealed in bag and taped to lid?	YES	NO
5.	Were custody papers filled out properly (ink, etc.)?	YES	NO
6.	Did you sign custody papers in the appropriate place?	YES	NO
7.	Was project identifiable from custody papers?	YES	NO
8.	Have designated person(s) initial and acknowledge receipt: _____date:_____		

B. LOGIN PHASE
Date samples were logged in:_____
By (print)_____ (sign)_____

9.	Describe packing: _____		
10.	If required, was enough ice used?	YES	NO
11.	Were all bottles sealed in separate plastic bags?	YES	NO
12.	Did all bottles arrive unbroken and in good condition?	YES	NO
13.	Were all bottle labels complete (ID, date, sign, preservative)?	YES	NO
14.	Did all bottle labels agree with custody papers? If no, list on back.	YES	NO
15.	Were correct containers used for the analytes?	YES	NO
16.	Were correct preservatives used when required?	YES	NO
17.	Was sufficient amount of sample sent for tests?	YES	NO
18.	Bubbles absent in VOA vials? If no, list by sample ID on back.	YES	NO
19.	Was Client Services informed of problems?	YES	NO

Figure 8-2 Kit Order Form



Page 1 of 1

eurofins | Eaton Analytical
formerly MWH Laboratories

750 Royal Oaks Drive, Suite 100
Monrovia, California 91016-3629
(626) 386-1100 FAX (626) 386-1101

Kit Order for MWH Laboratories

Andora.I.Effendi is your Eurofins Eaton Analytical Project Manager

Note: Sampler Please return this paper with your samples

Kit #: 55304
Created By: AutoGenerated
Order Date: 09/17/2012
Ship By: 09/07/2012
STG: Bottle Orders

Client ID: MWH-LAB-MONR-CA
Project Code: WATER QUALITY Bottle Orders
Group Name: Monrovia - Weekly
PO#/JOB#:

Ship Sample Kits to

.

Attn:

Send Report to
MWH Laboratories
750 Royal Oaks Drive
Suite 100
Monrovia, CA 91106

Attn: Andora Effendi
Phone: (626) 386-1100
Fax: (626) 386-1139

Billing Address
MWH Laboratories
750 Royal Oaks Drive
Suite 100
Monrovia, CA 91106

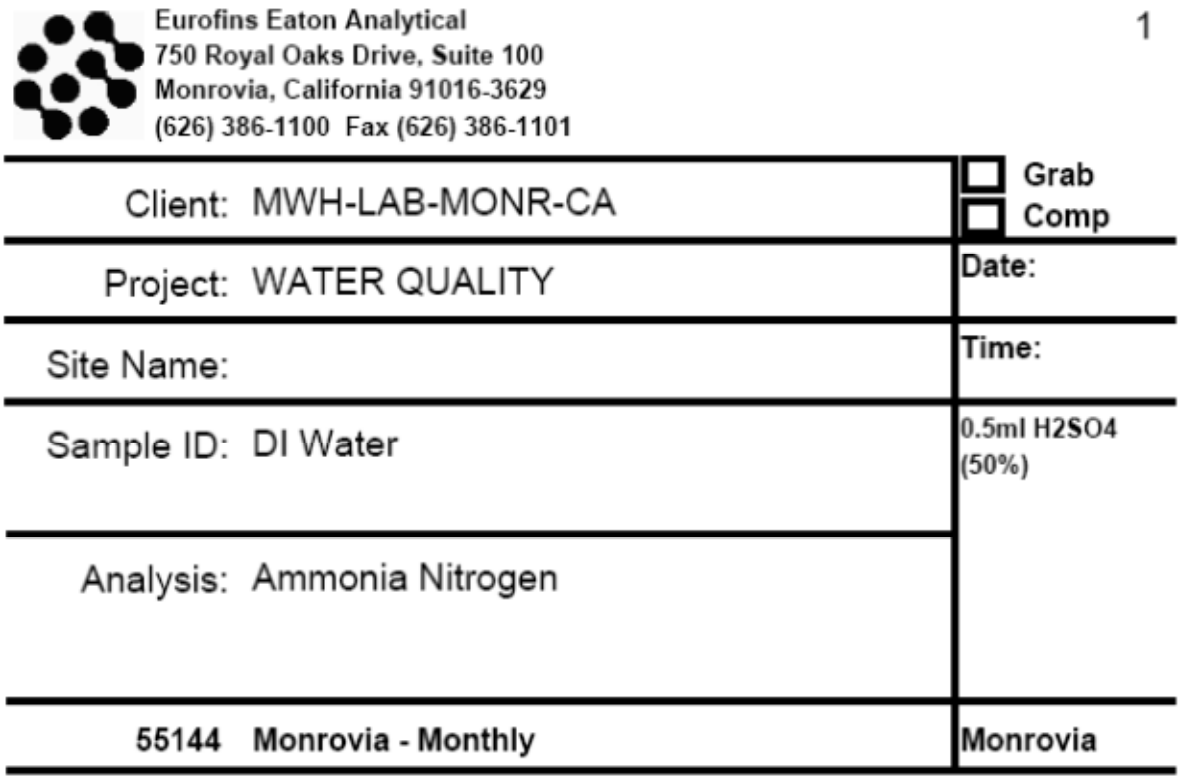
Attn: Andora Effendi
Phone: (626) 386-1100
Fax: (626) 386-1139

# of Samples	Tests	Bottles - Qty for each sample, type & preservative if an	UN DOT #
1	Free Chlorine Residual	1 125ml amber glass CHL_no preservative	

Comments

Code	Status	Date Shipped	Via	Tracking #	# of Coolers	Prepared By

Figure 8-3 Example Sample Labels



Eurofins Eaton Analytical
750 Royal Oaks Drive, Suite 100
Monrovia, California 91016-3629
(626) 386-1100 Fax (626) 386-1101

1

Client: MWH-LAB-MONR-CA	☐ Grab ☐ Comp
Project: WATER QUALITY	Date:
Site Name:	Time:
Sample ID: DI Water	0.5ml H_2SO_4 (50%)
Analysis: Ammonia Nitrogen	
55144 Monrovia - Monthly	Monrovia

Figure 8-4 Internal Custody Logbook

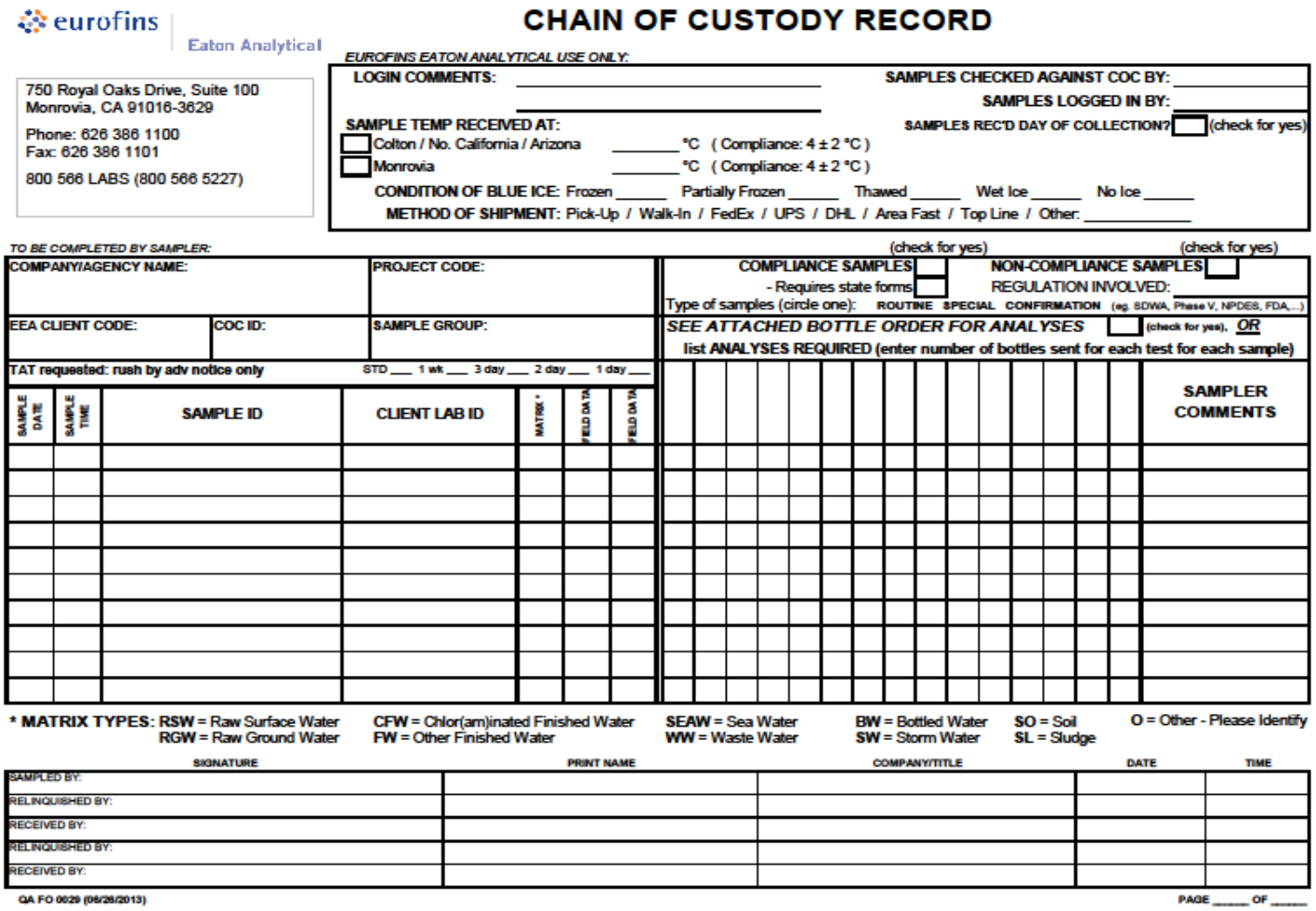
Internal COC														
Log In Received Date	Client	Client Sample ID	Sampling Date	Analysis	Laboratory Sample ID	Removal Code	Bottle #	Volume Taken	By (Staff Initial)	Date/Time Stored	Date/Time Removed	Date/Time Returned	Volume Returned	Peer Review Date/Time/Initial

Figure 8-5 Internal Sample Disposal (Level II)

Level II Chain of Custody Sample Disposal Logbook

Log in Rec'd Date	Client	Lab Sample ID	Authorized to Dispose By:	Tag Label		Date of Disposal	Nature of Disposal				Initials	Signature
				Yes	No		Sample Depleted	Hazwaste Drum	Sewer	Returned to Client		

Figure 8-6 Chain-of-Custody Form



eurofins | Eaton Analytical

CHAIN OF CUSTODY RECORD

750 Royal Oaks Drive, Suite 100
Monrovia, CA 91016-3629
Phone: 626 386 1100
Fax: 626 386 1101
800 566 LABS (800 566 5227)

EUROFINS EATON ANALYTICAL USE ONLY:

LOGIN COMMENTS: _____ SAMPLES CHECKED AGAINST COC BY: _____
SAMPLES LOGGED IN BY: _____
SAMPLES REC'D DAY OF COLLECTION? ☐ (check for yes)
SAMPLE TEMP RECEIVED AT:
☐ Colton / No. California / Arizona _____ °C (Compliance: 4 ± 2 °C)
☐ Monrovia _____ °C (Compliance: 4 ± 2 °C)
CONDITION OF BLUE ICE: Frozen _____ Partially Frozen _____ Thawed _____ Wet Ice _____ No Ice _____
METHOD OF SHIPMENT: Pick-Up / Walk-In / FedEx / UPS / DHL / Area Fast / Top Line / Other: _____

TO BE COMPLETED BY SAMPLER:

COMPANY/AGENCY NAME: | PROJECT CODE:
EEA CLIENT CODE: | COC ID: | SAMPLE GROUP:
TAT requested: rush by adv notice only STD ___ 1 wk ___ 3 day ___ 2 day ___ 1 day ___

(check for yes) (check for yes)
COMPLIANCE SAMPLES ☐ NON-COMPLIANCE SAMPLES ☐
- Requires state forms ☐ REGULATION INVOLVED: _____
Type of samples (circle one): ROUTINE SPECIAL CONFIRMATION (eg. SDWA, Phase V, NPDES, FDA,...)
SEE ATTACHED BOTTLE ORDER FOR ANALYSES ☐ (check for yes), *OR*
list ANALYSES REQUIRED (enter number of bottles sent for each test for each sample)

SAMPLE DATE	SAMPLE TIME	SAMPLE ID	CLIENT LAB ID	MATRIX *	FIELD DATA	FIELD DATA												SAMPLER COMMENTS

* **MATRIX TYPES:** **RSW** = Raw Surface Water; **RGW** = Raw Ground Water; **CFW** = Chlor(am)inated Finished Water; **FW** = Other Finished Water; **SEAW** = Sea Water; **WW** = Waste Water; **BW** = Bottled Water; **SW** = Storm Water; **SO** = Soil; **SL** = Sludge; **O** = Other - Please Identify

	SIGNATURE	PRINT NAME	COMPANY/TITLE	DATE	TIME
SAMPLED BY:					
RELINQUISHED BY:					
RECEIVED BY:					
RELINQUISHED BY:					
RECEIVED BY:					

QA FO 0029 (06/26/2013) PAGE _____ OF _____

Figure 8-7 Run Logbook

No.,	Sample Name,	Time,	Comment	Dil.Fac.,	Amount SO4, ECD_1			
1,	WASH	12/21/11 14:00,		1,	n.a.			
2,	autocal1	12/21/11 14:12,		1,	n.a.			
3,	autocal2	12/21/11 14:24,		1,	0.2429			
4,	autocal3	12/21/11 14:37,		1,	0.4945			
5,	autocal4	12/21/11 14:49,		1,	0.9313			
6,	autocal5	12/21/11 15:02,		1,	18.7837			
7,	autocal6	12/21/11 15:14,		1,	3.7761			
8,	autocal7	12/21/11 15:26,		1,	9.3196			
9,	autocal5	12/21/11 15:39,		1,	2.2017			
10,	autocal8	12/21/11 15:51,		1,	19.0900			
11,	autocal9	12/21/11 16:04,		1,	50.7623			
12,	autocal10	12/21/11 16:16,		1,	99.8682			
13,	autocal11	12/21/11 16:28,		1,	190.8361			
14,	20PPM/LDR	12/22/11 10:12,		1,	339.7175		DNR	
15,	HCV2	12/22/11 10:24,		1,	160.5565	100%	90%-110%	
16,	HCV1	12/22/11 10:36,		1,	99.5146	100%	90%-110%	
17,	MBLK	12/22/11 10:49,		1,	n.a.			
18,	MRLLW	12/22/11 11:01,		1,	0.2818	113%	50%-150%	
19,	MRL_CHK	12/22/11 11:14,	@ANION28	1,	0.9902	99%	50%-150%	
20,	LCS1	12/22/11 11:26,	632011	1,	51.1789	102%	90%-110%	
21,	LCS2	12/22/11 11:38,	@anion48	1,	51.3993	103%	90%-110%	
22,	MCV	12/22/11 11:51,	631998	1,	41.2474	103%	90%-110%	
23,	CCB	12/22/11 12:03,		1,	n.a.			
24,	MRLCHECK	12/22/11 12:38,		1,	0.9579	96%	50%-150%	
25,	201112210080	12/22/11 12:51,		1,	2.6984			
26,	201112210075	12/22/11 13:03,		1,	21.0028			
27,	201112210076	12/22/11 13:15,	H3	1,	8.5469			
28,	201112220169	12/22/11 13:28,		1,	48.0434			
29,	201112220153 12/21/11	12/22/11 13:40,		1,	34.1957			
30,	201112220157_1/2	12/22/11 13:53,		2,	258.0393	DNR		
31,	201112220164 SW	12/22/11 14:05,		1,	4.7805			
32,	201112220298_1/2	12/22/11 14:17,	HA	2,	135.9263			
33,	201112220132	12/22/11 14:30,		1,	10.5028			
34,	201112220140_1/5	12/22/11 14:42,	H3	5,	59.9622			
35,	201112220140MS	12/22/11 14:55,		5,	194.5431	26.9	108%	80%-120%
36,	201112220140MSD	12/22/11 15:07,		5,	194.8758	27.0	108%	80%-120%
37,	MCV	12/22/11 15:19,		1,	42.0281	105%	90%-110%	
38,	CCB	12/22/11 15:32,		1,	n.a.			
39,	201112220155	12/22/11 15:44,	ic under presure	1,	n.a.			
40,	MRLlow	12/22/11 16:05,		1,	0.2804	112%	50%-150%	
41,	201112220149	12/22/11 16:18,		1,	56.2977			
42,	201112220096_1/2	12/22/11 16:30,		2,	60.0654			
43,	201112220147_1/2	12/22/11 16:42,		2,	115.9345			
44,	201112220151_1/2	12/22/11 16:55,		2,	29.3873			
45,	201112220151MS	12/22/11 17:07,		2,	86.0782	28.3	113%	80%-120%
46,	201112220151MSD	12/22/11 17:20,		2,	84.4530	27.5	110%	80%-120%
47,	201112220162_1/2	12/22/11 17:32,		2,	121.0277			
48,	201112220160_1/2	12/22/11 17:44,		2,	93.2843			
49,	201112220171	12/22/11 17:57,		1,	12.7606			

Figure 8-8 Example Work Schedule Printout

Departmental Daily Report for: Chlorite by EPA 317

Purple/Italic = RUSH Order #
Red / Underline = Priority 1

Folder #	Sample #	Client (Project)	Sampled	Hold	Sample ID	Status	Analyze By	Profile	Method	Prepped	Run #
312174	200908140307	MIKE (CONTRACT FEES)	8/14	8/28	dumpty	Logged	08/28/2009	Chlorite by EPA 317	EPA 317		
312686	200908200575	LONGBEACHWD (DRINKING)	8/19	9/02	MF PERMEATE	Logged	08/28/2009	Chlorite by EPA 317	EPA 317		
312686	200908200576	LONGBEACHWD (DRINKING)	8/19	9/02	NF NORTH TRAIN 1ST PASS	Logged	08/28/2009	Chlorite by EPA 317	EPA 317		
312686	200908200577	LONGBEACHWD (DRINKING)	8/19	9/02	NF NORTH TRAIN 1ST	Logged	08/28/2009	Chlorite by EPA 317	EPA 317		
312686	200908200578	LONGBEACHWD (DRINKING)	8/19	9/02	NF NORTH TRAIN (PERMEATE)	Logged	08/28/2009	Chlorite by EPA 317	EPA 317		

Printed On: 8/26/2009

Page 1 of 1

Figure 8-9 Sample Acknowledgement

eurofins | Eaton Analytical
formerly MWH Laboratories

Acknowledgement of Samples Received

Addr: **MWH Laboratories**
750 Royal Oaks Drive
Suite 100
Monrovia, CA 91106
Attn: Andora Effendi
Phone: (626) 386-1100

Client ID: MWH-LAB-MONR-CA
Folder #: 405936
Project: WS (WS PT)
Sample Group: WS PT
Project Manager: Alma.B.Capati
Phone:
PO #: ERA WS192 PT - Inorganics

The following samples were received from you on **August 17, 2012**. They have been scheduled for the tests listed below each sample. If this information is incorrect, please contact your service representative. Thank you for using Eurofins Eaton Analytical.

Sample #	Sample ID	Sample Date
201208170474	WS Inorganics	08/17/2012 0000
	Sample Point ID: ERA WS 192 PT_Inorganics	
	Nitrate + Nitrite as N by RFA / Nitrate as N by RFA (calc)	

Test Description

Reported: 08/21/2012

Page 1 of 1

Figure 8-10 Operations Report

Due	PM	Client ID	Folder	Sampled	Order	Sample ID	Test	Run #	Department	Prep Date	Status
-16	AIE	MWH-LAB-MONR-CA	310880	7/26/2009	200907290297	Source Water E.Coli	E. Coli Bacteria	0	Micro/Asbestos		Logged
-41	AIE	MWH-LAB-MONR-CA	310553	7/10/2009	200907230384	Source Water E.Coli	MICR_COLI	0	Micro/Asbesto-P		Logged
-29	AIE	MWH-LAB-MONR-CA	309978	7/13/2009	200907160491	WP Simple Nutrients	Nitrate as N by RFA	517052	Metal/Wet		Logged
-17	AIE	MWH-LAB-MONR-CA	311411	8/4/2009	200908050946	PRD 1029	@ICPMS	521611	Metals		Logged
-16	AIE	MWH-LAB-MONR-CA	310879	7/26/2009	200907290296	Source Water E.Coli	E. Coli Bacteria	0	Micro/Asbestos		Logged
-35	AIE	MWH-LAB-MONR-CA	310552	7/10/2009	200907230383	Source Water E.Coli	@COLI10	0	Micro/Asbestos		Need Prep
-35	AIE	MWH-LAB-MONR-CA	310554	7/10/2009	200907230388	MicrobE D	@MPN10	0	Micro/Asbestos	8/17/2009	Logged
-41	AIE	MWH-LAB-MONR-CA	310552	7/10/2009	200907230383	Source Water E.Coli	MICR_COLI	0	Micro/Asbesto-P		Logged
-35	AIE	MWH-LAB-MONR-CA	310554	7/10/2009	200907230386	MicrobE B	@MPN10	0	Micro/Asbestos	8/17/2009	Logged
-35	AIE	MWH-LAB-MONR-CA	310554	7/10/2009	200907230389	MicrobE E	@MPN10	0	Micro/Asbestos	8/17/2009	Logged
-17	AIE	MWH-LAB-MONR-CA	311411	8/4/2009	200908050952	PRD 1082	@ICPMS	521611	Metals		Logged
-17	AIE	MWH-LAB-MONR-CA	311411	8/4/2009	200908050955	PRD 1111	@ICPMS	521611	Metals		Logged
-17	AIE	MWH-LAB-MONR-CA	310790	7/28/2009	200907280135	DI Water	PH (H3=past HT not compliant)	0	Wet Chemistry		Logged
-17	AIE	MWH-LAB-MONR-CA	310790	7/28/2009	200907280136	Nano Pure Water	PH (H3=past HT not compliant)	0	Wet Chemistry		Logged
-17	AIE	MWH-LAB-MONR-CA	311411	8/4/2009	200908050950	PRD 1069	@ICPMS	521611	Metals		Logged
-17	AIE	MWH-LAB-MONR-CA	311411	8/4/2009	200908050953	PRD 1102	@ICPMS	521611	Metals		Logged
-17	AIE	MWH-LAB-MONR-CA	311411	8/4/2009	200908050954	PRD 1109	@ICPMS	521611	Metals		Logged
-16	AIE	MWH-LAB-MONR-CA	310882	7/26/2009	200907290300	Source Water E.Coli	E. Coli Bacteria	0	Micro/Asbestos		Logged
-17	AIE	MWH-LAB-MONR-CA	311411	8/4/2009	200908050951	PRD 1074	@ICPMS	521611	Metals		Logged
-17	AIE	MWH-LAB-MONR-CA	311411	8/4/2009	200908050947	PRD 1031	@ICPMS	521611	Metals		Logged
-35	AIE	MWH-LAB-MONR-CA	310554	7/10/2009	200907230385	MicrobE A	@MPN10	0	Micro/Asbestos	8/17/2009	Logged
-29	AIE	MWH-LAB-MONR-CA	309978	7/13/2009	200907160513	WP Total Phenolics	Phenolic Compounds-low level	0	Metal/Wet	7/21/2009	Need Prep
-29	AIE	MWH-LAB-MONR-CA	309978	7/13/2009	200907160483	WP Hardness	@ICP	0	Metals		Done
-17	AIE	MWH-LAB-MONR-CA	311411	8/4/2009	200908050948	PRD 1049	@ICPMS	521611	Metals		Logged
-35	AIE	MWH-LAB-MONR-CA	310553	7/10/2009	200907230384	Source Water E.Coli	@COLI10	0	Micro/Asbestos		Need Prep
-17	AIE	MWH-LAB-MONR-CA	311411	8/4/2009	200908050945	PRD 1025	@ICPMS	521611	Metals		Logged
-17	AIE	MWH-LAB-MONR-CA	311411	8/4/2009	200908050949	PRD 1055	@ICPMS	521611	Metals		Logged
0	AIE	MWH-LAB-MONR-CA	312685	8/20/2009	200908200573	Lot# B-9-196-01VB	@TCP_PREP	0	Volatiles-P		Logged
2	AIE	MWH-LAB-MONR-CA	312401	8/18/2009	200908180396	Lot# 915507	GCVO_HAA	521751	GC Volatiles-P		Logged
-35	AIE	MWH-LAB-MONR-CA	310554	7/10/2009	200907230387	MicrobE C	@MPN10	0	Micro/Asbestos	8/17/2009	Logged
-17	AIE	MWH-LAB-MONR-CA	311411	8/4/2009	200908050956	PRD 1112	@ICPMS	521611	Metals		Logged
2	AIE	MWH-LAB-MONR-CA	312401	8/18/2009	200908180397	Lot# 915507	GCVO_HAA	521751	GC Volatiles-P		Logged
4	AIE	MWH-LAB-MONR-CA	312401	8/18/2009	200908180396	Lot# 915507	@HAA	0	GC Volatiles		Need Prep
6	AIE	MWH-LAB-MONR-CA	312685	8/20/2009	200908200573	Lot# B-9-196-01VB	@TCP	0	Volatiles		Need Prep
6	AIE	MWH-LAB-MONR-CA	312685	8/20/2009	200908200572	Lot# B-9-134-01VB - Acid Lot No. #24	@551	0	GC Volatiles		Need Prep
4	AIE	MWH-LAB-MONR-CA	312401	8/18/2009	200908180397	Lot# 915507	@HAA	0	GC Volatiles		Need Prep
4	AIE	MWH-LAB-MONR-CA	312685	8/20/2009	200908200572	Lot# B-9-134-01VB - Acid Lot No. #24	GCVO_551	0	GC Volatiles-P		Logged

Figure 8-11 Weekly Lab Turnaround Time

HPLC Department

Page 2 of 6

1/14/12 0:00 - 1/20/12 0:00

Total Test		OnTime (%)		Re-Test	
Analyzed	Approved	Analyzed	Approved		
22	40	100	95	0	@531
16	16	100	100	0	@531LOW
7	7	71	71	0	@532
0	40	0	88	0	@549
9	9	100	100	0	@ML555
30	40	100	100	0	Glyphosate

		On Time		Re-Test	
Analyzed	84	82	97.6%	0	
Approved	152	143	94.1%		

Average Days to Complete Peer Review: 3.0 +/- 1.5

Median: 4.00

9.0 ANALYTICAL PROCEDURES

9.1. SOURCES FOR METHODS

9.1.1. Standard Methods

9.1.1.1. The laboratory shall evaluate the Precision and Bias of a standard method for each analyte of concern for each quality system matrix according to the single-concentration four-replicate recovery study procedures in TNI-EL-V1M3 to V1M6-2009-1.6.2.2 (or alternate procedure documented in the quality manual when the analyte cannot be spiked into the sample matrix and QC samples are not commercially available) [TNI-EL-V1M4 and V1M6-2009-1.5.3.a].

9.1.1.2. The analytical methods performed by EEA are based primarily on methods specified by various federal, state, and local regulations. If more stringent standards or requirements are included in the mandated test method or by regulation than the TNI standard, the laboratory ensures that all SOPs meet such requirements. If it is unclear which requirements are more stringent, the laboratory follows the requirements from the method or regulation. All analysts must follow all the Quality Control protocols and all essential QC measures specified by the laboratory's SOPs. The majority of methods come from the U.S. Environmental Protection Agency. Other methods are from Standard Methods for the Examination of Water and Wastewater, 19th, 20th, and online Editions. Additional methods may be used when appropriate.

Methods from the EPA are listed in section 9.6, the references section.

9.1.2. Non Standard Methods

9.1.2.1. Methods not covered by standard methods are properly validated before use. Non-standard methods when used by the laboratory are subjected to agreement with the Client incorporating the Client's specification requirements, including the purpose of the environmental test. The method is validated appropriately before use [TNI-EL-V1M3 to V1M4-2009-1.4].

9.1.2.2. For laboratory-developed test methods or non-standard test methods as defined in TNI-EL-V1M2- 2009-5.4.3 and ISO/IEC 17025:2005(E)-5.4.3 and TNI-EL-V1M3 to V1M7-2009-1.4 that were not in use by the laboratory before July 2003, the laboratory must have a documented procedure to evaluate precision and bias. The laboratory must also compare results of the precision and bias measurements with criteria established by the client, by criteria given in the reference method or criteria established by the laboratory.

9.1.2.3. Laboratory developed methods may be used when the client does not specify the method to be used or where methods are employed that are not required by regulations, as in the Performance Based Measurement System Approach, the

methods shall be fully documented and validated (TNI-EL-V1M2-2009-5.4.2) (ISO/IEC 17025:2005(E)-5.4.2) and TNI-EL-V1M3 to V1M7-2009-1.4), and be available to the Client and other recipients of the relevant reports. The laboratory shall select appropriate methods that have been published either in international, regional or national standards, or by reputable technical organizations, or in relevant scientific texts or journals, or as specified by the manufacturer of the equipment. Laboratory-developed methods or methods adopted by the laboratory are used only if appropriate to the intended use and are validated. The laboratory informs the Client as to the method chosen. [TNI-EL-V1M2-2009-5.4.2][ISO/IEC 17025:2005(E)-5.4.2]

9.1.2.4. The laboratory informs the Client when the method proposed by the Client is considered to be inappropriate or out of date. [TNI-EL-V1M2-2009-5.4.2][ISO/IEC 17025:2005(E)-5.4.2]

9.1.3. The introduction of environmental test and calibration methods developed for the laboratory for its own use is a planned activity and is assigned to qualified personnel equipped with adequate resources.

9.2. INITIAL TEST METHOD EVALUATION PROCEDURES

For all test methods (other than microbiology or methods where LOD/LOQ determinations are not relevant) the following LOD and LOQ requirements apply.

9.2.1. Limit of Detection (LOD)

9.2.1.1. The laboratory shall determine the LOD by performing the MDL studies determination to conform to CFR136 for the method for each target analyte of concern in the quality system matrices. All sample-processing steps of the analytical method shall be included in the determination of the LOD.

9.2.1.2. The validity of the LOD shall be confirmed by quantitative identification of the analyte(s) in a QC sample in each quality system matrix containing the analyte at no more than 1-3X the LOD for single analyte tests and 1-4X the LOD for multiple analyte tests. This verification must be performed on every instrument that is to be used for analysis of sample and reporting data. LOD verification Acceptance Criteria is detection (>0).

9.2.1.3. An LOD study is not required for any component for which spiking solutions or quality control samples are not available such as temperature, or, when test results are not to be reported to the LOD (versus the method reporting limit or working range of instrument calibration). Where an LOD study is not performed, the laboratory may not report a value below the Limit of Quantitation. Since the EPA Manual for Drinking Water 5th Edition requires MDL studies, the laboratory conducts MDL determinations for all drinking water methods where applicable. EEA does not report down to the LOD but reports down to the LOQ.

9.2.2. Limit of Quantitation (LOQ)

9.2.2.1. The laboratory shall determine the LOQ for each analyte of concern according to a defined, documented procedure. LOQ/MRL is 2-3x LOD/MDL. At a minimum, LOQ/MRL > LOD.

9.2.2.2. The LOQ study is not required for any component or property for which spiking solutions of quality control samples are not commercially available or otherwise inappropriate (e.g., pH).

9.2.2.3. As per TNI Std, the validity of the LOQ shall be confirmed by successful analysis of a QC sample containing the analytes of concern in each quality system matrix 1-2 times the claimed LOQ. A successful analysis is one where the recovery of each analyte is within the established test method acceptance criteria or client data quality objectives for accuracy. This single analysis is not required if the bias and precision of the measurement system is evaluated at the LOQ. EEA verifies the validity of the MRL/LOQ daily at the MRL level as per EPA Manual for DW 5th edition.

9.2.2.4. Precision and Bias

Precision and bias measurements must evaluate the method across the analytical calibration range of the method. The laboratory must also evaluate precision and bias in the relevant quality system matrices and must process the samples through the entire measurement system for each analyte of interest [TNI-EL-V1M4 and V1M6-2009-1.5.3].

Examples of a systematic approach to evaluate precision and bias could be the following for non-standard methods:

9.2.2.4.1. Analyze QC samples in triplicate containing the analytes of concern at or near the limit of quantitation, at the upper-range of the calibration (upper 20%) and at a mid-range concentration. Process these samples on different days as three sets of samples through the entire measurement system for each analyte of interest. Each day one QC sample at each concentration is analyzed. A separate method blank shall be subjected to the analytical method along with the QC samples on each of the three days. (Note that the three samples of the MRL concentration can demonstrate sensitivity as well). For each analyte, calculate the mean recovery for each day, for each level over days, and for all nine samples. Calculate the relative standard deviation for each of the separate means obtained. Compare the standard deviations for the different days and the standard deviations for the different concentrations. If the different standard deviations are all statistically insignificant (e.g., F-test), then compare the overall mean and standard deviation with the established criteria from above.

9.2.2.4.2. A validation protocol such as the Tier I, Tier II, and Tier III requirements in US EPA Office of Water's Alternate Test Procedure (ATP) approval process.

9.2.2.5. Selectivity

The laboratory evaluates selectivity by following the checks established within the method, which may include mass spectral tuning, second column confirmation, ICP inter-element interference checks, chromatography retention time windows, sample blanks, spectrochemical absorption or fluorescence profiles, co-precipitation evaluations, and electrode response factors [TNI-EL-V1M4-2009-1.5.4].

9.2.3. Detection Limits

9.2.3.1. The method used in the quantitation of detection limits is as described in 40 CFR 136 Appendix B, which in summary is the analysis of at least seven replicates from which a statistically derived Method Detection Limit (MDL) is calculated. The replicates are determined over at least a 3 day period. This statistically derived limit is based on 3.143 times the standard deviation of 7 low concentration replicates (3-5 times the calculated detection limit). It is the laboratory's policy to be conservative when reporting a method detection limit on a non-detected sample.

9.2.3.2. Consequently, the laboratory has implemented the concept of minimum reporting levels (MRLs). The limit used on a laboratory report must be at or above the lowest standard associated with that analytical run. This ensures that all data reported as "detected" will have some degree of analytical precision associated with it. Data reported below these levels must be appropriately qualified. Copies of current MRLs for the laboratory are available upon request. An MRL can be no lower than the calculated MDL.

9.2.3.3. For newer methods, a Lowest Concentration Minimum Reporting Level (LCMRL) may be required before client samples may be run on the method, annually, and for any new analysts. The LCMRL is essentially a more strict verification of the MRL. See the appropriate method for the exact requirements of an LCMRL and its verification. Section 9.2.4 of EPA Method 524.3 details the requirements for the 524.3 LCMRL, for example.

9.3. ESTIMATION OF UNCERTAINTY

Estimation of uncertainty consists of the sum (combining the components) of the uncertainties of the numerous steps of the analytical process, including, but not limited to, sample plan variability, spatial and temporal sample variation, sample heterogeneity, calibration/calibration check variability, extraction variability, and weighing variability.

The laboratory estimates uncertainty using 2 times standard deviation calculated from 20 routine quality control samples with a guidance of <20% RSD. Refer to the Nonmethod 29 SOP for detailed Measurement Uncertainty procedures.

9.4. METHOD VALIDATIONS [TNI-EL-V1M3 to V1M7-2009-1.5]

9.4.1. The laboratory shall validate non-standard methods, laboratory-designed/developed methods, standard methods used outside their published scope, and amplifications and modifications of standard methods to confirm that the methods are fit for the intended use. The validation shall be as extensive as is necessary to meet the needs of the given application or field of application. The initial test method evaluation requirements given in V1M3 to V1M7 1.6 of TNI Standard 2009 and 2011 discussed in Section 4.4, MDL and IDC requirements for new analysts are done in validating new methods and non-standard methods [TNI-EL-V1M3 to V1M7-2009-1.5]. This is also applicable when an analyte not currently found on the laboratory's list of accredited analytes is added to an existing accredited test method. Initial evaluation must be performed for that analyte [TNI-EL-V1M4 and V1M6-2009-1.6.2.2.g]. The laboratory records the results obtained for the IDC, MDL, LOD and LOQ studies. The method is fit for the intended use when the results meet all the MDL and IDC criteria for the method.

9.4.2. The range and accuracy of the values obtainable from validated methods (e.g. the uncertainty of the results, detection limit, selectivity of the method, linearity, limit of repeatability and/or reproducibility, robustness against external influences and/or cross-sensitivity against interference from the matrix of the sample/test object), are assessed for the intended use, and relevant to the Client's needs [TNI-EL-V1M7-2009-1.4].

9.5. METHOD VALIDATION/MICROBIOLOGY

To demonstrate the suitability of a test method for its intended purpose, the laboratory meets the acceptance criteria by the EPA or State program requirements. Also, the laboratory must meet the following criteria as per TNI-EL-V1M5-2009-1.5.2 and 1.5.3:

9.5.1. Accepted (official) test methods or commercialized test kits for official methods from recognized national or international standards organizations do not require a specific validation. However to demonstrate proficiency with the test method prior to first use, the laboratory performs comparison to a method already approved for use in the laboratory, or by analyzing a minimum of ten spiked samples whose matrix is representative of those normally submitted to the laboratory, or by analyzing and passing one proficiency test series provided by an approved proficiency sample provider. The laboratory shall maintain this documentation as long as the method is in use and for at least 5 years past the date of last use [TNI-EL-V1M5-2009-1.5], or 10 years to meet Massachusetts, Hawaii, and New York requirements.

9.5.2. The laboratory participates in the proficiency test programs identified by NELAP [TNI-EL-V1M1-2009-4.1.1 and 4.2.1.a] or [TNI-EL-V1M2-2009-5.9.1.b][ISO/IEC

17025:2005(E)-5.9.1.b]. The results of these analyses are used to evaluate the ability of the laboratory to produce acceptable data.

9.6. METHODS USED/SCOPE OF TESTING

9.6.1. The analytical methods used by EEA can be grouped into three major categories: drinking water methods, wastewater methods, and methods for hazardous wastes and solid samples. The following tables provide method descriptions and method numbers for the methods used in these three major groups:

Table 9-1 Method Description for Drinking Water

Table 9-2 Method Description for Wastewater

Table 9-3 Method Description for Hazardous Waste

9.7. METHOD MODIFICATIONS

All method modifications are documented fully in individual SOPs. Methods are modified if and only if the original method goals for precision and accuracy have been met or better. Modifications are usually implemented due to available resources, or to expedite the process without sacrificing quality. Methods are validated prior to analyzing client samples. Validation is based on the method as described in the internal SOP. The validation includes an MDL study, an analyst precision and accuracy study, and subsequent review by the Technical Manager, Lab Director and Quality Manager.

9.8. REFERENCES

Ref	Method Description
1	These methods are available from USEPA, EMSL, Cincinnati, OH 45268. The identical methods were formerly in "Methods for Chemical Analysis of Water and Wastes," EPA-600/4-79-020, March 1983.
2	"Methods for the Determination of Metals in Environmental Samples - Supplement I," EPA-600/R-94-111, May 1994. Available at NTIS, PB 94-184942.
3a	USEPA "Methods for the Determination of Organic Compounds in Drinking Water - Supplement I". EPA-600/4-90-020, July 1990. (547, 551)
3b	USEPA "Methods for the Determination of Organic Compounds in Drinking Water - Supplement II." EPA-600/R-92-129, August 1992. (524.2, 548.1, 549.1)
3c	USEPA "Methods for the Determination of Organic Compounds in Drinking Water, Method 525.2, 504.1, and 508.1"
3d	USEPA “Methods for the Determination of Organic Compounds in Drinking Water, Supplement III (502.2, 504.1, 505, 507, 508, 524.2, 525.2, 531.1, 551.1), EPA/600/R-95/131, 08/95. For 1,2,3-TCP low level, CA DHS “Determination for 1,2,3-Trichloropropane in Drinking Water by Purge and Trap Gas Chromatography/ Mass Spectroscopy,” (524.2), 02/02.

4 Standard Methods for the Examination of Water and Wastewater, 20th Edition, 1998, American Public Health Association, 1015 Fifteenth Street NW, Washington, D.C. 20005.

4a Standard Methods for the Examination of Water and Wastewater, 18th Edition, 1992, American Public Health Association, 1015 Fifteenth Street NW, Washington, D.C. 20005.

4b Standard Methods for the Examination of Water and Wastewater, 19th Edition, 1995, American Public Health Association, 1015 Fifteenth Street NW, Washington, D.C. 20005.

4c Standard Methods for the Examination of Water and Wastewater, 21st Edition, 2005, American Public Health Association, 800 I Street, NW, Washington, D.C. 20001.

4d Standard Methods for the Examination of Water and Wastewater, Online Edition, 2005, American Public Health Association, 800 I Street, NW, Washington, D.C. 20001.

4e Standard Methods for the Examination of Water and Wastewater, 22nd Edition, 2012, American Public Health Association, 800 I Street, NW, Washington, D.C. 20001.

5 Available from Books and Open-File Reports Section, U.S. Geological Survey, Federal Center, Box 25425, Denver, CO 80225-0425.

6 "Methods for the Determination of Inorganic Substances in Environmental Samples," EPA-600/R-93-100, August 1993. Available at NTIS, PB94-121811.

7 USEPA “Methods for Chemical Analysis of Water and Wastewater,” EPA-600/4-79-020, 1983.

8 Method 100.2, "Determination of Asbestos Structure Over 10-mm In Length in Drinking Water," EPA-600/R-94-134, June 1994. Available at NTIS, PB 94-201902.

9 Industrial Method No. 129-71W, "Fluoride in Water and Wastewater," December 1972, and Method No. 380-75WE, "Fluoride in Water and Wastewater," February 1976, Technician Industrial Systems, Tarrytown, NY 10591.

10 40 CFR Parts 100, 136 to 141. July 1, 1995.

11 "Prescribed Procedures for Measurement of Radioactivity in Drinking Water", EPA-600/4-80-032 (1980), US EPA, August 1980.

12 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, EPA SW-846, 2nd edition, revised April 1985 and 3rd edition, September 1986.

13 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, EPA SW-846, Update III.

14 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, EPA SW-846, Update II

14a Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, EPA SW-846, Update I

14b Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, EPA SW-846, Update IV

14c Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, EPA SW-846, On-line

15 Methods for the Determination of Nonconventional Pesticides in Municipal and Industrial Wastewater – Volume 1 – EPA 821/R-93-010A. August 1993. Revision 1. Method 614. The Determination of Organophosphorus Pesticides in Municipal and Industrial Wastewater.

16 Method 300.1 Determination of Inorganic Anions in Drinking Water by Ion Chromatography, Revision 1.0 1997 (Stand Alone Method)

17 Federal Register, 12/1/99, USEPA 40 CFR Parts 141 & 143 National Primary & Secondary Drinking Water Regulations: Analytical Methods for Chemical & Microbiological Contaminants & Revisions to Laboratory Certification Requirements; Final Rule

17a Methods Update Rule, March 12, 2007, 40 CFR Parts 122, 136 and 141. Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act; National Primary Drinking Water Regulations; and National Secondary Drinking Water Regulations; Analysis and Sampling Procedures; Final Rule.

17b Methods Update Rule, March 18, 2012, 40 CFR Parts 136, 260, 423, 430, and 435. Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act; Analysis and Sampling Procedures; Final Rule.

18 Method 515.4 Determination of Chlorinated Acids in Drinking Water by Liquid-liquid Microextraction, Derivatization, And Fast Gas Chromatography with Electron Capture Detection, Revision 1.0, April, 2000, EPA 815-R-00-014

19 Method 531.2 Measurement of n-Methyl Carbamoyloximes and n-Methylcarbamates in Water by Direct Aqueous Injection – HPLC with Postcolumn Derivitization, Revision 1.0, September, 2001, EPA 815-B-01-002

20 USEPA "April 2000 draft – Method 1602." April, 2000.

21 EPA Method 522 Determination of 1,4-Dioxane in drinking water by solid phase extraction (SPE) and gas chromatography/mass spectrometry (GC/MS) with selected ion monitoring (SIM), Revision 1.0, September 2008, EPA/600/R-08/101

22 EPA Method 521 Determination of Nitrosamines in Drinking Water by Solid Phase Extraction and Capillary Column Gas Chromatography with Large Volume Injection and Chemical Ionization Tandem Mass Spectrometry (MS/MS) , Revision 1.0, September 2004, EPA/600/R-05/054

23 US ENVIRONMENTAL PROTECTION AGENCY. Jul 2001. Determination of Inorganic Oxyhalide Disinfection By-products in Drinking Water Using Ion Chromatography with the Addition of a Postcolumn Reagent for Trace Bromate Analysis, Draft Method 317.0, EPA/815/B-01/001. Technical Support Center Office of Ground Water and Drinking Water, Cincinnati, Ohio.

24 EPA Method 549.2 Determination of Diquat and Paraquat by High Performance Liquid Chromatography, Revision 1.0, June 1997

25 EPA Method 1613B Determination of 2,3,7,8-TCDD in Drinking Water by Capillary Column Gas Chromatography with Large Volume Injection and Electron Ionization Tandem Mass Spectrometry (EI/MS/MS), Version 1.0 Sept. 1994.

26 EPA Method 524.3, Measurement of Purgeable Organic Compounds in Water by Capillary Column Gas Chromatography/Mass Spectrometry. Version 1.0 June

2009

27 EPA Method 527 Determination of Selected Pesticides and Flame Retardants in Drinking Water by Solid Phase Extraction and Capillary Column Gas Chromatography/Mass Spectrometry (GC/MS), Revision 1.0, April 2005

28 EPA Method 552.3 Determination of Haloacetic Acids and Dalapon in Drinking Water by Liquid-Liquid Microextraction, Derivatization, and Gas Chromatography with Electron Capture Detection, Revision 1.0

29 EPA Method 535 Measurement of Chloroacetanilide and Other Acetamide Herbicide Degradates in Drinking Water by Solid Phase Extraction and Liquid Chromatography/Tandem Mass Spectrometry (LC/MS/MS), Version 1.1, April 2005

30 EPA Method 529 Determination of Explosives and Related Compounds in Drinking Water by Solid Phase Extractions (SPE) and Capillary Column Gas Chromatography/Mass Spectrometry (GC/MS), Revision 1.0, September, 2002

31 Method for the Determination of Radium-228 and Radium-226 in Drinking Water by Gamma-ray Spectrometry Using HPGE or Ge(Li) Detectors, December, 2004, Revision 1.2, Environmental Resource Center, Georgia Institute for Technology, Atlanta, GA.

Table 9-1 Method Description for Drinking Water

(A) Inorganics – Wet Chemistry – Drinking Water

Parameter/Method Name	Method Number	Method Description	Reference
Alkalinity	SM2320B	Titrimetric	4/4e
Ammonia	EPA350.1	Colorimetric	1
Bromate	EPA 300.0 / 300.1 / 317.0	Ion Chromatography	6/16/23
Bromide	EPA300.0/300.1	Ion Chromatography	6/16
Chloride	EPA300.0	Ion Chromatography	6
Chlorate	EPA 300.0/300.1	Ion Chromatography	6/16
Chlorite	EPA300.0/300.1 / 317.0	Ion Chromatography	6/16/23
Chromium VI (Dissolved)	EPA 218.6/ SM 3500 Cr-B	Ion Chromatography	2/4
Color	SM2120B	Visual	4
Conductivity	SM2510B	Wheatstone Bridge	4
Cyanide	SM4500CN-F,G	Selective Electrode Method	4
Cyanide	EPA335.4	Manual Distillation, Spectrophotometric	6
Fluoride	SM4500 F-C	Potentiometric - Ion Selection Electrode	4
Fluoride	EPA 300.0	Ion Chromatography	6
Foaming Agents/ Surfactant (MBAS)	SM5540C	Colorimetric	4
Nitrate/Nitrite	EPA300.0	Ion Chromatography	6
Nitrite/Nitrate + Nitrite	EPA 353.2	Automated Cadmium Reduction, RFA	1
Odor	SM2150B	Odor	4
Perchlorate	EPA 314.0 EPA 331	Ion Chromatography LCMS	6
pH	EPA 150.1/SM4500-HB	Electrometric	1/4
o-Phosphate	EPA 300.0	Ion Chromatography	6
o-Phosphate	SM4500 P-E EPA 365.1	Color, Ascorbic Acid	4/6
Residual Chlorine (Total/Free Chlorine)	SM4500 Cl-G	DPD Colorimetric/HaCH	4
Silica	EPA200.7	ICP	2
Dissolved Silica/Reactive Silica	SM 4500 SiO2C SM 4500 SiD	Molybdosilicate	4
Solids (TDS)	SM2540C	Gravimetric	4
Sulfate	EPA300.0	Ion Chromatography	6
Temperature	SM2550B	Thermometric	4
Total Organic Carbon(TOC)/ Dissolved Organic Carbon (DOC)	SM5310C	UV Persulfate	4

Parameter/Method Name	Method Number	Method Description	Reference
Turbidity	EPA180.1 SM 2130B	Nephelometric	6/4/4e
UV 254	SM5910B	Determination of UV absorbing organic constituents by UV absorption method at 254 nm	4
TOX (Total Organic Halogen) or Dissolved Organic Halogen (DOX)	SM 5320B	Adsorption-Pyrolysis-Titrimetric Method	4

(B) Inorganics – Metals – Drinking Water

Parameter/Method Name	Method Number	Method Description	Reference
Metals (except Hg)	EPA 200.7	ICP (Inductively Coupled Plasma)	2
Metals (except Hg)	EPA 200.8	ICPMS (Inductively Coupled Plasma Mass	2
Mercury	EPA 245.1	Manual Cold Vapor	2

(C) Microbiology/Microscopy Tests – Drinking Water

Parameter/Method Name	Method Number	Method Description	Reference
Drinking Water Source Enumeration (MTF)	SM9221B,C	Multiple Tube Fermentation (MTF)	4/4d
Drinking Water Source Enumeration/Colilert 24 hr & 8 hr	SM9223B	MMO-MUG Test/Colilert	4/4d
Fecal Coliforms/EC Medium	SM9221E	Multiple Tube fermentation (MTF) / EC Medium	4/4d
Heterotrophic Plate Count	SM9215B	Pour Plate Count	4/4d
Total Coliform & E. Coli	SM9223B	Colisure	4/4d
Total Coliforms	SM9221A, B	Multiple Tube Fermentation (MTF)	4/4d
Total Coliforms + --E. Coli / Present or Absent	SM9223B	MMO-MUG Test/Colilert	4/4d
Coliphage	EPA 1602	Coliphage	20
Asbestos	EPA 100.2	TEM (Transmission Electron Microscopy)	8

(D) Organics – Drinking Water

Parameter/Method Name	Method Number	Method Description	Reference
DBCP/EDB	EPA504.1	Microextraction, GC/ECD	3d
Organohalide Pesticides and Commercial Polychlorinated Biphenyl (PCB) Products in water by Microextraction and Gas Chromatography	EPA505	Microextraction, GC/ECD	3d

Parameter/Method Name	Method Number	Method Description	Reference
Chlorinated Herbicides	EPA515.4	GC, Electron Capture Detector (ECD)	18
Purgeable Organic Compounds/ Halogenated & Aromatic Volatiles/Trihalomethanes/Di-isopropyl Ether(DIPE),Tertiary Amyl Methyl Ether (TAME), Tert-Butyl ethyl ether (ETBE), TBA, CS2, MIBK 1,2,3-Trichloropropane (TCP)	EPA524.2 CA DHS 524.2-SIM	Purge and Trap capillary Column, GCMS	3d
Semi-Volatile Organics -- Acid/Base Neutrals including ThioBencarb	EPA525.2	Liquid Solid Extraction (LSE), capillary column, GCMS	3d
N-Methylcarbamoyloximes and N-Methylcarbamates	EPA531.2	HPLC with Fluorescence Detector	19
Glyphosate	EPA547	HPLC/Post Column Reactor - Fluorescence Detector	3a
Endothall	EPA548.1	GCMS, Liquid Solid Extraction (LSE)	3b
Diquat & Paraquat	EPA549.2	HPLC, Liquid Solid Extraction (LSE) UV Detector	24
Trihalomethanes & EDB/DBCP	EPA 551.1	GC, Electron Capture Detector (ECD), liquid liquid extraction	3d
Haloacetic Acids	SM6251B	GC, Electron Capture Detector (ECD)	4
1,4-Dioxane	EPA 522	GCMS, Solid Phase Extraction (SPE)	21
Nitrosamines	EPA 521	GCMS-MS, Solid Phase Extraction (SPE)	22
Dioxin	EPA 1613B	GC/EI/MS/MS, Solid Phase Extraction (SPE)	25
Volatile Organic Compounds	EPA 524.3	GC/MS	26
Selected Pesticides and Flame Retardants	EPA 527.0	GC/MS, Solid Phase Extraction (SPE)	27
Haloacetic Acids and Dalapon	EPA 552.3	GC, Electron Capture Detector	28
Chloroacetanilide and Other Acetamide Herbicide Degradates	EPA 535	LC/MS/MS, Solid Phase Extraction (SPE)	29
Explosives	EPA 529	GC/MS, Solid Phase Extraction (SPE)	30

(E) Radiochemistry – Drinking Water

Parameter/Method Name	Method Number	Method Description	Reference
Uranium	EPA 200.8	ICP MS	2

Parameter/Method Name	Method Number	Method Description	Reference
Gross Alpha	EPA900.0	Proportional Counting	11
Gross Beta	EPA900.0	Proportional Counting	11
Radium 226/228	Georgia Inst. Of Tech, rev 1.2	Gamma-ray Spectrometry Using HPGE Detector	31

Table 9-2 Method Description for Wastewater

(A) Inorganics – We Chemistry – Wastewater

Parameter/ Method Name	Method Number	Method Description	Reference
Alkalinity, Total (Bicarbonate, Carbonate, & Hydroxide)	SM2320B	Titrimetric, Potentiometric	4/4e
Ammonia	EPA350.1/SM 4500 NH3H and D (18th)	Colorimetric	1/4a
Biochemical Oxygen Demand (BOD)	SM5210B	BOD/Probe	4
Boron	EPA200.7	ICP	2
Bromide	EPA300.0	Ion Chromatography	6
Carbonaceous Biochemical Oxygen Demand (CBOD)	SM5210B	BOD/Probe with Nitrification Inhibitor	4
Chemical Oxygen Demand (COD)	EPA410.4, SM5220D	Colorimetric	1/4
Chloride	EPA300.0	Ion Chromatography	6
Chlorine, Total Residual	SM4500 Cl G	Spectrophotometric, DPD, HACH	4
Chromium VI	EPA 218.6/ SM3500D Cr-B	0.45 micron Filtration Followed by Colorimetric or Ion Chromatography	2/4
Color	SM2120B	Visual	4
Cyanide, Total	EPA 335.4	Manual Distillation followed by Auto Spectrophotometric	1
Cyanide	SM4500-CN F	Ion Selective Electrode	4
Cyanide, Amenable to Chlorination	SM 4500CN G	Automated Colorimetric after treatment	4
Dissolved Oxygen (DO)	SM 4500-OG	Membrane Electrode	4
Fluoride	SM4500 F-C	Ion Selective Electrode	4
Fluoride	EPA 300.0	Ion Chromatography	6
Foaming Agents/ Surfactant (MBAS)	SM5540C	Colorimetric	4

Parameter/ Method Name	Method Number	Method Description	Reference
Hardness	EPA 200.7/SM 2340B	Calculation Ca plus Mg as CO3-	2/4
Kjeldahl Nitrogen	EPA351.2	Colorimetric, Semi-auto block digester	1
Nitrate	EPA353.2 EPA300.0	Cadmium Reduction Ion Chromatography	1/6
Nitrite	EPA300.0 EPA 353.2	Ion Chromatography Cadmium Reduction	6/1
Total Residue	SM 2540B	Gravimetric	4
Orthophosphate	SM4500 P-E/PF, EPA 365.1, EPA300.0/HACH 8048	Manual Single Reagent Ion Chromatography	4/6
Perchlorate	EPA 300.0/314.0	Ion Chromatography	6
Phenols	EPA 420.1/420.4	Manual Distillation Followed by Colorimetric	1
pH	SM4500-HB	Electrometric	4
Phosphorus, Total	SM4500 PE EPA 365.1	Persulfate Digestion followed by Manual Colorimetric	4/6
Residue, Filterable (Total Dissolved Solids--TDS)	SM2540C	Gravimetric	4
Residue, Non-filterable (Total Suspended Solids--TSS)	SM2540D	Gravimetric	4
Residue, Settleable (Settleable Solids)	SM 2540F	ImHoff Cone	4
Residue, Volatile	EPA160.4	Gravimetric	1
Specific Conductance	EPA120.1/SM2510 B	Wheatstone Bridge	¼
Sulfate	EPA300.0	Ion Chromatography	6
Sulfide (Total & Soluble)	SM 4500S-2D	Colorimetric	4
Total Organic Carbon (TOC)	SM5310C	UV Persulfate	4
TOX (Total Organic Halogen) or Dissolved Organic Halogen (DOX)	SM5320B	Adsorption-Pyrolysis-Titrimetric Method	4
Turbidity	EPA 180.1 SM2130B	Nephelometric	6/4/4e

(B) Inorganic – Metals – Wastewater

Parameter/Method Name	Method Number	Method Description	Reference
Metals (except Hg)	EPA200.7 EPA200.8	Digestion, Inductively Coupled Plasma Emission Spectroscopy (ICP) Digestion, Inductively Coupled Plasma/Mass Spectrometry (ICPMS)	2
Mercury, Hg	EPA245.1	Digestion, Cold Vapor Manual	1
Silica Dissolved	SM4500SiO2C SM4500 SiD	Molybdosilicate	4/4b

(C) Microbiology/Microscopy Tests – Wastewater

Parameter/Method Name	Method Number	Method Description	Reference
Asbestos	EPA 100.2	Transmission Electron Microscopy	8
Total Coliforms By Multiple Tube Fermentation (MTF)	SM9221B	Multiple Tube Fermentation (MTF)	4/4d
Fecal Coliforms By Multiple Tube/EC	SM9221E	MTF (EC Medium)	4/4d
E. Coli	SM9223	Colisure	4/4d
Fecal Streptococci and Enterococci by MTF	SM9230B	Multiple Tube Fermentation (MTF)	4/4d
Heterotrophic Plate Count	SM9215B	Pour Plate Count	4/4d

(D) Organics – Wastewater

Parameter/Method Name	Method Number	Method Description	Reference
Halogenated/Aromatic Volatiles	EPA624	GC/MS	10
Semi-Volatiles Acid and Base/ Neutral Compounds	EPA625	GC/MS	10

(E) Radiochemistry – Wastewater

Parameter/Method Name	Method Number	Method Description	Reference
Gross Alpha	EPA900.0	Proportional Counting	11
Gross Beta	EPA900.0	Proportional Counting	11

Table 9-3 Method Description for Hazardous Waste (Aqueous)

(A) Inorganics – Wet Chemistry – Hazardous Waste (Aqueous)

Parameter/Method Name	Method Number	Method Description	Reference
Anions	EPA9056	Determination of Inorganic Anions by Ion Chromatography	14

Chromium VI	EPA7196A/7199	Colorimetric/Ion Chromatography	14a/13
Conductivity	EPA 9050A	Specific Conductance	13
Corrosivity	EPA 9040C	pH Electronic Measurement	13
Cyanide	EPA 9012B	Total and Amenable Cyanide (Automated Colorimetric, with Offline Distillation)	13
Fluoride	EPA 9214	Potentiometric Determination of Fluoride in Aqueous Samples with Ion-Selective Electrode	13
pH	EPA 9040B	pH Electronic Measurement	14
Solids (TDS)	SM 2540C	Gravimetric	4
Sulfide	EPA 9030B EPA 9034 SM 4500-S2-D	Acid-Soluble and Acid-Insoluble Sulfides: Distillation Titrimetric Procedure for Acid-Soluble and Acid-Insoluble Sulfides Colorimetric	13 13 4
Total Organic Carbon	EPA 9060A	Total Organic Carbon by Carbonaceous Analyzer	13
Total Organic Halides	EPA 9020 B	Absorption - Pyrolysis - Titrimetric Method	14
Total Phenolics	EPA 9066	Colorimetric, Automated 4-AAP with Distillation	14a

(B) Inorganics – Metals – Hazardous Waste (Aqueous)

Parameter/ Method Name	Method Number	Method Description	Reference
Aluminum, Al	EPA6010B EPA6020,A	EPA3005A/3010A Acid Digestion, Inductively Coupled Plasma Emission Spectroscopy (ICP) EPA3005A/3010A Acid Digestion, Inductively Coupled Plasma/Mass Spectrometry (ICPMS)	13 14/14b
Antimony, Sb	EPA6010B EPA6020,A	EPA3005A/3010A Acid Digestion, Inductively Coupled Plasma Emission Spectroscopy (ICP) EPA3005A/3010A Acid Digestion, Inductively Coupled Plasma/Mass Spectrometry (ICPMS)	13 14/14b
Arsenic, Ar	EPA6020,A	EPA3005A/3010A Acid Digestion, Inductively Coupled Plasma/Mass Spectrometry (ICPMS)	14/14b
Barium, Ba	EPA6010B EPA6020,A	EPA3005A/3010A Acid Digestion, Inductively Coupled Plasma Emission Spectroscopy (ICP) EPA3005A/3010A Acid Digestion, Inductively Coupled Plasma/Mass Spectrometry (ICPMS)	13 14/14b

Parameter/ Method Name	Method Number	Method Description	Reference
Beryllium, Be	EPA6010B EPA6020,A	EPA3005A/3010A Acid Digestion, Inductively Coupled Plasma Emission Spectroscopy (ICP) EPA3005A/3010A Acid Digestion, Inductively Coupled Plasma/Mass Spectrometry (ICPMS)	13 14/14b
Boron, Br	EPA6010B	EPA3005A/3010A Acid Digestion, Inductively Coupled Plasma Emission Spectroscopy (ICP)	13
Cadmium, Cd	EPA6010B EPA6020,A	EPA3005A/3010A Acid Digestion, Inductively Coupled Plasma Emission Spectroscopy (ICP) EPA3005A/3010A Acid Digestion, Inductively Coupled Plasma/Mass Spectrometry (ICPMS)	13 14/14b
Chromium, Cr	EPA6010B EPA6020,A	EPA3005A/3010A Acid Digestion, Inductively Coupled Plasma Emission Spectroscopy (ICP) EPA3005A/3010A Acid Digestion, Inductively Coupled Plasma/Mass Spectrometry (ICPMS)	13 14/14b
Cobalt, Co	EPA6010B EPA6020,A	EPA3005A/3010A Acid Digestion, Inductively Coupled Plasma Emission Spectroscopy (ICP) EPA3005A/3010A Acid Digestion, Inductively Coupled Plasma/Mass Spectrometry (ICPMS)	13 14/14b
Copper, Cu	EPA6010B EPA6020,A	EPA3005A/3010A Acid Digestion, Inductively Coupled Plasma Emission Spectroscopy (ICP) EPA3005A/3010A Acid Digestion, Inductively Coupled Plasma/Mass Spectrometry (ICPMS)	13 14/14b
Iron, Fe	EPA6010B	EPA3005A/3010A Acid Digestion, Inductively Coupled Plasma Emission Spectroscopy (ICP)	13
Lead, Pb	EPA6010B EPA6020,A	EPA3005A/3010A Acid Digestion, Inductively Coupled Plasma Emission Spectroscopy (ICP) EPA3005A/3010A Acid Digestion, Inductively Coupled Plasma/Mass Spectrometry (ICPMS)	13 14/14b
Magnesium, Mg	EPA6010B	EPA3005A/3010A Acid Digestion, Inductively Coupled Plasma Emission Spectroscopy (ICP)	13
Manganese, Mn	EPA6010B EPA6020,A	EPA3005A/3010A Acid Digestion, Inductively Coupled Plasma Emission Spectroscopy (ICP) EPA3005A/3010A Acid Digestion, Inductively Coupled Plasma/Mass Spectrometry (ICPMS)	13 14/14b
Mercury, Hg	EPA7470A	Manual Cold Vapor/Solid or Semi Solid (CV)	14

Parameter/ Method Name	Method Number	Method Description	Reference
Molybdenum, Mo	EPA6010B EPA6020,A	EPA3005A/3010A Acid Digestion, Inductively Coupled Plasma Emission Spectroscopy (ICP) EPA3005A/3010A Acid Digestion, Inductively Coupled Plasma/Mass Spectrometry (ICPMS)	13 14/14b
Nickel, Ni	EPA6010B EPA6020,A	EPA3005A/3010A Acid Digestion, Inductively Coupled Plasma Emission Spectroscopy (ICP) EPA3005A/3010A Acid Digestion, Inductively Coupled Plasma/Mass Spectrometry (ICPMS)	13 14/14b
Potassium , K	EPA6010B	EPA3005A/3010A Acid Digestion, Inductively Coupled Plasma Emission Spectroscopy (ICP)	13
Selenium, Se	EPA6020,A	EPA3005A/3010A Acid Digestion, Inductively Coupled Plasma/Mass Spectrometry (ICPMS)	14/14b
Silver, Ag	EPA6010B EPA6020,A	EPA3005A/3010A Acid Digestion, Inductively Coupled Plasma Emission Spectroscopy (ICP) EPA3005A/3010A Acid Digestion, Inductively Coupled Plasma/Mass Spectrometry (ICPMS)	13 14/14b
Sodium, Na	EPA6010B	EPA3005A/3010A Acid Digestion, Inductively Coupled Plasma Emission Spectroscopy (ICP)	13
Strontium, Sr	EPA6010B	EPA3005A/3010A Acid Digestion, Inductively Coupled Plasma Emission Spectroscopy (ICP)	13
Thallium, Tl	EPA6020,A	EPA3005A/3010A Acid Digestion, Inductively Coupled Plasma Emission Spectroscopy (ICP)	14/14b
Total Metals	EPA3010A	Acid Digestion of Aqueous Samples and Extracts for Total Metals for Analysis by FLAA or ICP Spectroscopy	14a
Tin, Sn	EPA6010B EPA6020,A	EPA3005A/3010A Acid Digestion, Inductively Coupled Plasma Emission Spectroscopy (ICP) EPA3005A/3010A Acid Digestion, Inductively Coupled Plasma/Mass Spectrometry (ICPMS)	13 14/14b

Parameter/ Method Name	Method Number	Method Description	Reference
Titanium, Ti	EPA6010B EPA6020,A	EPA3005A/3010A Acid Digestion, Inductively Coupled Plasma Emission Spectroscopy (ICP) EPA3005A/3010A Acid Digestion, Inductively Coupled Plasma/Mass Spectrometry (ICPMS)	13 14/14b
Vanadium, V	EPA6010B EPA6020,A	EPA3005A/3010A Acid Digestion, Inductively Coupled Plasma/Mass Spectrometry (ICPMS) EPA3005A/3010A Acid Digestion, Inductively Coupled Plasma Emission Spectroscopy (ICP)	13 14/14b
Zinc, Zn	EPA6010B EPA6020,A	EPA3005A/3010A Acid Digestion, Inductively Coupled Plasma/Mass Spectrometry (ICPMS) EPA3005A/3010A Acid Digestion, Inductively Coupled Plasma/Mass Spectrometry (ICPMS)	13 14/14b

(C) Organics – Hazardous Waste (Aqueous)

Parameter/Method Name	Method Number	Method Description	Reference
Halogenated Volatiles	EPA8260B	Purge & Trap, GC/MS	13
Aromatic Volatiles	EPA8260B	Purge & Trap, GC/MS	13
Semi-Volatile Organic Compounds (BNAs)	EPA8270C	EPA3550A Extraction, GC/MS	13
EDB/DBCP	EPA 8011	Microextraction, GC/ECD	14a
Purge and Trap for Aqueous Samples	EPA 5030C	Purge and Trap for Aqueous Samples	14c

10.0 PURCHASING SERVICES AND SUPPLIES/ MEASUREMENT TRACEABILITY

10.1. PURCHASING SERVICES AND SUPPLIES

10.1.1. Documented procedures for the purchase, receipt and storage of reagents and standards (consumable materials) used for the technical operations of the laboratory must be followed by all personnel as per TNI-EL-V1M2-2009-5.6.4.2. Refer to the Nonmethod 27 SOP for detailed procedures for Purchasing Services and Supplies.

10.1.2. Purchased supplies and services that affect the quality of environmental tests are of the required quality by using approved suppliers and products.

10.2. REAGENTS AND REFERENCE STANDARDS

10.2.1. All chemicals used by EEA are ACS Reagent Grade, or better. Wherever possible, standards are from sources that are traceable to the National Institute for Standards and Technology (NIST). The laboratory ensures the use of reagents of same or better purity than that specified in the method. Thus, the analyst checks the label of the container to verify that the purity of the reagents meets the requirements of the particular method. The purchased supplies and reagents that affect the quality of the tests are not used until they are inspected or otherwise verified as complying with requirements defined in the test method.

10.2.2. Procedures shall be in place to ensure prepared reagents meet the requirements of the test method [TNI-EL-V1M2-2009-5.6.4.2.e]. If the method does not specify reagent quality, at a minimum the laboratory uses analytical “Reagent Grade” or better quality for all reagents.

10.2.3. **Calibration Standards**

10.2.3.1. Stock standards are obtained from the EPA Repository, or suppliers traceable to NIST, for the organic compounds. The metal stock solutions are obtained from NIST traceable sources. Initial calibration verification standards are obtained from a second “Manufacturer or lot” if lot can be demonstrated from the manufacturer as prepared independently from other lots [TNI-EL-V1M4-2009-1.7.1.1.d]. Stock solutions for surrogate parameters and other inorganic compounds are made up by the analysts from the appropriate reagent grade chemical specified in the procedure.

10.2.3.2. Stock standards are utilized to make working standards of lower concentration, which are then used to make calibration standards for the analytical run. The holding periods of stock standards, working standards, and calibration standards for the different analyses are provided in Table 10-2.

10.2.3.3. Stock standards, working standards, and calibration standards are all prepared in accordance with the method procedure. A logbook is maintained for standards

preparation providing the initials of the analyst preparing the standard, the date of preparation, the concentration made up, and the lot numbers and suppliers. Since only one set of working standards is prepared at a time, the date of an analytical run can be keyed to the date of the working standards preparation to provide traceability to the particular lots of reagents from which the calibration standards were derived.

10.2.3.4. Calibration standards are run at the beginning of each day's analysis and a single standard is run every 10 samples throughout the analysis and at the end of the run to check for instrument drift. This "check" standard can also be used as an additional measure of analytical precision in addition to the LCS. Beginning and ending check standards must be at varying concentration within the established calibration range. If an internal standard is used, one CCV check must be analyzed per batch and an ending CCV may not need to be run unless required by method.

10.2.3.5. At the beginning of each day of analysis, all instruments must be calibrated. The calibration standards used must encompass a range of low, mid and high level concentrations to determine the calibration curve. The low level standard must be at or below the MRL value, the high level standard must be at the high end of the calibration range range and the mid level standard must be approximately midway between the low and high concentrations. Calibration procedures vary for the different instrumental methods and are summarized on Table 11-1. Section 11.1 for some representative methods.

10.2.4. **Policy on Verification of Standards**

All information relating to standards preparation and verification must be documented in the Standards Preparation notebook for that analysis. All documentation required must be examined by the analyst and signed off by the section supervisor. All documentation for each group must be stored in a central location (i.e. the standards preparation room). For microbiology, performance checks including the organisms used, their culture collection reference, date of issue of specification, or statements assuring that the relevant batch meets the product specifications is verified [TNI-EL-V1M5-2009-1.7.3.6].

10.2.4.1. **Mixtures**

New standard mix preparations must be compared to the previous mix. The concentrations calculated for the new standard should be within 10% of the "true" value (or as per the specific SOP). If the new standard does not agree within 10%, a third standard must be prepared by a different analyst and compared to the previous two. The third standard should agree with either the "old" standard or the "new" standard. If the third standard agrees with the "old" standard the third standard is used as the "new" standard. If the third standard agrees with the "new" standard the "old" standard is discarded and both the "new" and third standards can be used. In both cases the "new" standard must be verified by comparing to a "known" reference

standard before discarding the old standard. Note that for some methods it may not be possible for the new standard to agree within 10% (see the specific SOP).

A new calibration curve must be prepared analyzing both the new standard and a known reference sample. The calculated value must fall within the acceptance limits for the reference sample.

10.2.4.2. **Neat Compounds**

The identity and purity of any new bottle of neat material must be verified either by the method it will be used to monitor or, preferably, by a different method.

For Organics, a solution of the new neat material must be compared to the old standard as a check on identity and purity. Acceptance criteria are detailed in the previous Mixtures section. For inorganics the new stock standard must be compared to the old stock standard as a check on concentration.

10.3. DOCUMENTATION RECORDS OF REAGENTS AND STANDARDS

10.3.1. A logbook is maintained for all standards. Each log contains the date of fresh stock preparation, the manufacturer's lot number and supplier, the preparer's initials, the weight of material and the final volume used to prepare the stock.

10.3.2. The laboratory shall retain records for all standards, reagents, reference materials and media including the manufacturer/vendor, the manufacturer's Certificate of Analysis or purity (if supplied), the date of receipt, recommended storage conditions, and an expiration date after which the material shall not be used unless it is verified by the laboratory [TNI-EL-V1M2-2009-5.6.4.2].

10.3.3. Original containers (such as provided by the manufacturer or vendor) shall be verified and labeled with an expiration date [TNI-EL-V1M2-2009-5.6.4.2.b].

10.3.4. Records shall be maintained on standard and reference material preparation. These records shall indicate traceability to purchased stocks or neat compounds, reference to the method of preparation, date of preparation, expiration date and preparer's initials.

10.3.5. Where traceability to NIST is not applicable, the laboratory shall provide satisfactory evidence of correlation of results, example participation in proficiency testing or independent analysis of reference standards and reference materials [TNI-EL-V1M2-2009-5.6.4.1].

10.3.6. All containers of prepared standards and reference materials must bear a unique identifier and expiration date, and be linked to documentation requirements in 10.3 above [TNI-EL-V1M2-2009-5.6.4.2.d].

10.3.7. All containers of prepared reagents must bear a preparation date. An expiration date shall be defined on the container or documented elsewhere as indicated in the laboratory's quality manual or SOP [TNI-EL-V1M2-2009-5.6.4.2.d and 5.9.3.a.vi].

10.4. REAGENT STORAGE AND DISPOSAL

10.4.1. Standards are stored in designated refrigerators or freezers. Samples/extracts/digestates are not stored in these refrigerators due to the potential for cross-contamination.

10.4.2. All reagents, solvents and reactive chemicals are stored in their original containers in appropriate cabinets or storage closets specifically designed for this use. See Table 10-1, for storage instruction. Date received and date opened must be recorded on each reagent container.

Table 10-1 Reagent and Standard Storage

Chemical	Method of Storage
Nitric Acid	Stored in original containers in cabinet designed for acid storage.
Hydrochloric Acid	Stored in original containers in cabinet designed for acid storage.
Sulfuric Acid	Stored in original containers in cabinet designed for acid storage.
Flammable Solvents	Stored in original containers in flammable storage cabinets.
Oxidizers	Stored separately from flammable in cabinet designed for oxidizers.
Ethyl Ether	Stored in original containers in flammable storage cabinets. New lots are tested for peroxides. Each bottle is tested before and after peroxide removal with an activated alumna column
Stock Standard Solutions	Stored in freezer at 0°C in unbroken ampoules
Working Standard Solutions	Stored in refrigerator at 4°C labeled with prep information and expiration date.
Reagent Chemicals	Stored in cabinets in air conditioned laboratory areas
Hazardous Chemicals	Any chemical which is a health toxin and a known carcinogen, is stored in a secured area with restricted access

Table 10-2 Standard Storage and Holding Periods for Stock and Working Standard Solutions

Analyte	Stock Standard	Source Storage	Working Standard	Storage	Calibration Standard
ICP Metals	Expiration date	RT	6 months	RT	1-month
ICPMS Metals	Expiration date	RT	6 months	RT	1-month
Volatile 524.2	Expiration date	FZ	Monthly	FZ	Monthly
BNA Compounds	3 months if opened	FZ	Monthly if opened	FZ	3 months
	Expiration date If sealed	FZ	6 months If sealed	FZ	
Pesticides/PCBs/HAAs					
505	Expiration date	FZ	6 months	RF	6 months
525.2	Expiration date	FZ	6 months	RF	6 months
535	Expiration date	RF	6 months	RF	Daily
HAA's	2 months	FZ	2-Months	FZ	Daily
Inorganics					
300.0/300.1	6 Months	RF	Daily	RT	Daily
Nutrients	Semi-annually	RT	Monthly	RT	Daily
Phenol, Cyanide	Semi-annually	RT	Monthly	RT	Daily
TOX	Yearly	RT	Monthly	RT	Daily
TOC	Yearly	RF	6 Months	RF	Daily
NO2/Nitrate	1 Month	RF	Daily	RT	Daily
Chlorine	Yearly	RF	Daily	RT	Daily
UV 254	Yearly	RF	Monthly	RF	Daily
Microcystin	2 years	FZ	3 Months	RF	3 months
Cylindrospermopsin	1 year	RF	Daily	RF	Daily
Saxitoxin	10 months	RF	Daily	RF	Daily

* Bimonthly - every two months
* Biweekly - every two weeks

RT - Room Temperature
RF - Refrigerated at 4°C
FZ - Frozen at 0°C

Table 10-3 Sources of Standard Materials

Analysis	Vendor Source
ICAP/ICPMS Metals	JT-Baker
Volatile Gases	Ultra Scientific, EM Science Ampules
Volatiles	Ultra Scientific, EM Science Ampules
BNA Compounds	Ultra Scientific, Accu Standard, Absolute Standard
Pesticides/PCBs	Accu-Standards
Anions	EM Science/Baker, Fisher
Nutrients	EM Science/Baker
Phenol, Cyanide	EM Science/Baker
TOX,TOC	CPI
Radiochemistry	Eckert Ziegler/ Isotope Products

11.0 CALIBRATION PROCEDURES AND FREQUENCY

The production of analytical data of known, defensible and documented quality requires adherence to standardized procedures, which cover all aspects of laboratory operation. The following sections provide details of the standardized procedures relating to instrumentation calibration.

11.1. INITIAL INSTRUMENT CALIBRATION

Prior to use, every instrument must be calibrated according to a specified procedure found in the method-specific SOP. Table 12-1 lists all major laboratory equipment. Table 11-1 lists the minimum calibration frequency of use and the acceptance criteria for the various calibration techniques, on a method by method basis. Table 11-2 also summarizes the calibration methods and number of calibration standards that are used on an instrument basis. Table 11-3 lists the ion abundance tune criteria, which must be met during calibration, for mass spectroscopy methods such as 524.2 and 525.2. Calibration frequency and criteria included in the tables are only for representative test methods. Calibration procedures for other methods can be found in relevant SOPs.

Each instrument, and support equipment including reference standards of measurements such as Class S weights or equivalent weights, and traceable thermometers are marked and identified to indicate its calibration status such as "Calibration due date."

11.1.1. **Applicability**

11.1.1.1. The creation of this or any other policy is designed to be a guideline to ensure that all data are treated alike, and thus ensuring that data generated on any particular day of analysis are representative of the norm. The policies are not intended to be absolute criteria for the acceptance or rejection of any analytical data.

11.1.1.2. There is no substitute for the inherent familiarity that each analyst has with his or her specific analysis, and consequently their assessment of the data must be considered in cases where the acceptance criteria outlined in policy or SOPs cannot be achieved. Data generated in situations where one or more of the requirements outlined cannot be met will be reviewed on a case-by-case basis by the QA staff and the appropriate Technical Manager for acceptance. A detailed Quality Investigation Report (QIR) should have been completed and included in the data package to justify any deviation from policy or SOP protocols. Example of a QIR is shown in Figure 15-2.

11.1.2. **Linearity**

11.1.2.1. All calibrations should be linear unless otherwise defined in the specific SOP and allowed by the reference method. Many organic methods may allow or require the use of a quadratic fit for some compounds. Linearity here is defined as a calibration curve that meets the back-calculation criteria presented below, unless the SOP

contains different criteria. Specific protocols outlined in a given SOP will always take precedence over generic policies outlined in this QA Manual.

11.1.2.1.1. Linear Regression

$y = mx + b$

Where:

- y = Response A_x for External Standard or A_x/A_{is} for Internal Standard
- x = Concentration C_x for external standard, or C_x/C_{is} for internal standard
- m = Slope
- b = Intercept

11.1.2.1.2. Linear Regression Statistical Equations

Slope (m) → $m = \frac{[(Swx_i y_i \times Sw) - (Swx_i \times Swy_i)]}{[(Sw \times Swx_i^2) - (Swx_i \times Swx_i)]}$

Intercept (b) → $b = y_{ave} - (m \times (x_{ave}))$

Correlation Coefficient (r) →

$$r = \frac{[(SW \times Swx_i y_i) - (Swx_i - Swy_i)]}{\sqrt{[Sw \times Swx_i^2) - (Swx \times Swx_i)] \times [(Sw \times Swy_i^2) - (Swy_i \times Swy_i)]}}$$

Coefficient of Determination (r^2) → $r^2 = r \times r$

Where:

- n = number of x, y pairs
- x_i = individual values for the independent variable
- y_i = individual values for the dependent variable
- w = weighting factor, for equal or no weighting w = 1
- x_{ave} = average of the x values
- y_{ave} = average of the y values
- S = the sum of all the individual values

11.1.2.1.3. Quadratic Regression Equation

$y = ax^2 + bx + c$

Where:

- y = Response A_x for external standard, or A_x/A_{is} for internal standard
- x = Concentration Cx for external standard, or Cx/Cis for internal standard

11.1.2.1.4. Equation for Concentration

External Standard Equation → $C_x = \frac{(A_x - b)}{m}$

Internal Standard Equation → $C_x = \left(\frac{{}^{A_x}/_{A_{is}} - b}{m}\right) C_{is}$

11.1.2.2. If the method does not specify the acceptance criteria for the linear fit, the laboratory will establish a policy for acceptance criteria of 0.995 for correlation coefficient. The calibration curve is verified using any one of the following:

11.1.2.2.1. Coefficient of Determination (r^2)

$$r^2 = \frac{S(y_i - y_{ave})^2 - [{}^{(n-1)}/_{(n-p)} \times (S(y_i - Y_i)^2]}{S(y_i - y_{ave})^2}$$

Where:

y_i = individual values for each dependent variable
x_i = individual values for each dependent variable
y_{ave} = average of the y values
n = number of pairs of data
p = number of parameters in the polynomial equation (i.e., 3 for third order, 2 for second order

$$Y_i = \frac{\left\{\left[2a \times \left({}^{C_x}/_{C_{is}}\right)^2\right] - b^2 + b + (4ac)\right\}}{4a}$$

S = the sum of all the individual values

11.1.2.2.2. An initial calibration verification standard (ICV's) is immediately run after the curve. The standard is preferably obtained from a 2nd source or different lot if the lot can be demonstrated from the manufacturer as prepared independently from other lots [TNI-EL-V1M4-2009-1.7.1.1.d]. Concentrations that lie in the middle of the curve should have an acceptable recovery of ± 10% of the true value.

11.1.2.2.3. The linear curve will be acceptable if the curve meets the back-calculation criteria, i.e. back calculating the initial calibration standards against the developed model, with an acceptance criteria of ± 10 % recovery of the true value.

11.1.3. **Selection of Quantitation Technique (Organics)**

11.1.3.1. For organic analysis, a decision must be made during the validation process (and detailed in the SOP) as to whether an internal or external quantitation technique will be routinely employed.

11.1.3.2. The internal standard method of quantitation cannot be employed unless all of the following conditions are met:

11.1.3.2.1. The internal standard must be added post-extraction. For Method 525.2, it is added pre-extraction.

11.1.3.2.2. The internal standard must be added quantitatively.

11.1.3.2.3. Any analyte that is a target analyte using the method of interest may not be selected for use as the internal standard.

11.1.3.2.4. The concentration of the internal standard(s) must not exceed the calibration range of the method target analytes. In cases where the target analytes are associated with more than one calibration range (i.e. analytes "1-4" are calibrated from 1 to 10 µg/L, while analyte "5" is calibrated from 10 to 100 µg/L, and analytes "6-10" are calibrated from 2.5 to 25 µg/L), the concentration of the internal standard should be prepared at a level between the highest calibration standard of the highest and lowest absolute calibration range. (e.g. approximately 50 µg/L in the example given).

11.1.3.3. The use of internal standard quantitation is of greatest benefit in those methods subject to a great deal of injection variability, and thus a great deal of variability in the absolute mass injected onto the column(s) employed. The drawback to this technique for GC methods is that any compound that exhibits a similar retention time as the compound used for the internal standard will be identified as the internal standard, leading to erroneous quantitation. For this reason, the internal standard technique is most useful for GC/MS where deuterated analytes not naturally occurring can be detected and quantified.

11.1.4. **Selection of Calibration Method**

11.1.4.1. During the method validation process, a least square regression is initially tried as a calibration method. The responses from each of the calibration standards must then be input into the linear regression equation to determine whether or not the corresponding concentrations meet the acceptance criteria outlined below. If the acceptance criteria cannot be met using a linear regression, then a second order polynomial fit can be used to fit the data, with $r^2 \geq 0.99$ as the acceptance criteria. In the event that neither a simple linear regression nor a second order polynomial fit result in an equation which meets the calibration acceptance criteria, then the calibration range must be broken down into two or more smaller ranges. Each of the subsequent ranges must individually meet all of the requirements for a single

calibration range. If a linear regression works, a single average response factor may be used if the calibration is linear through the origin and it is consistent with the referenced method.

11.1.4.2. As part of the validation process, the specific calibration range and calibration algorithm must be determined and documented in the SOP. Once determined in this manner, the same protocols must be followed each time the method is employed. This will ensure that data reduction is not performed differently on separate data sets or by different analysts.

11.1.5. **Minimum Number of Calibration Levels**

The calibration for linear fits must include a minimum of three initial calibration standards plus a blank unless specified otherwise in the SOP. Polynomial fits must include at least 5 standards. The minimum requirement for a NELAP Lab as per TNI-EL-V1M4-2009-1.7.1.1.j is: a minimum of three (3) standards (one of which is lowest quantitation limits, not including a blank or zero standard), if the reference method does not specify the minimum number of initial calibration standards.

11.1.6. **Selection of Calibration Levels**

11.1.6.1. To avoid weighting a calibration curve to create a better fit than is warranted, three standards must be included per order of magnitude of concentration of the calibration curve. For example 0.1, 0.5, 1.0, 5.0, 10.0 has 3 standards per order of magnitude (0.1, 0.5 and 1.0, and 1.0, 5.0 and 10.0).

11.1.6.2. The lowest calibration standard shall be the lowest concentration for which quantitative data are to be reported. Any data reported below the lower limit of quantitation is considered to have an increased quantitative uncertainty and is reported using either “J” flags or explained in the case narrative [TNI-EL-V1M4-2009-1.7.1.1.f].

11.1.6.3. The highest calibration standard shall be the highest concentration for which quantitative data are to be reported. Any data reported above the highest standard is considered to have an increased quantitative uncertainty and is reported using “E” flags or explained in the case narrative [TNI-EL-V1M4-2009-1.7.1.1.g].

11.1.6.4. Measured concentrations outside the working range are reported as having less certainty and are reported using “E” flags or explained in the case narrative. The lowest calibration standard must be above the limit of detection, usually at MRL level except for ICP that allows zero point and single point calibration. [TNI-EL-V1M4-2009-1.7.1.1.h].

11.1.6.5. A good approach to select calibration levels when the calibration range is expected to span at least one order of magnitude is to set the levels at 1 MRL, 5 MRL, and 10

MRL for a simple 3 point calibration. If more points are desired, then they would follow the same scheme, i.e. 50 MRL, 100 MRL.

11.1.7. **Calibration Analytical Sequence**

11.1.7.1. The calibration must progress from the analysis of the lowest to highest standard unless the instrumentation does not permit it or the method requires calibration from high to low. A blank must be analyzed after the highest calibration standard.

11.1.7.2. If the analysis requires an initial high standard to set the gain a blank must be run before starting with the low calibration standard unless the instrumentation does not permit it.

11.1.8. **Calibration Acceptance Criteria**

For linear fits, in general, the calculated value for standards (using the calibration curve or response factor) must be within 10% of the nominal value for mid-level standards. However, the value determined by the calibration curve for the lowest standard (conc. is at the MRL) must be within ± 50% of the true value or ± 25% of the true value if the lowest standard is >5X & <10X MRL. Accurate quantitation at the MRL level may require use of a second order fit or separation of the curve into multiple linear segments. Mid level standards (conc. > 10X MRL) should be within ± 10% of the true value. Relevant SOPs should be reviewed for the method and laboratory calibration verification specific criteria, which may be different from those stated here.

11.2. CONTINUING INSTRUMENT CALIBRATION

11.2.1. Continuing calibration (CC) is run as required by the method. Refer to specific SOPs to determine the frequency and acceptance criteria of continuing calibration verifications.

11.2.2. The continuing calibration standard must be near the mid-point of the calibration curve unless the method requires rotation of concentration levels.

11.2.3. The calculated value for the continuing calibration standard must be within control limits stated in the specific SOP.

11.2.4. Calibration shall be verified for each batch for each compound, element, or other discrete chemical species, except for multi-component analytes such as Aroclors, Chlordane, or Toxaphene where a representative chemical related substance or mixture can be used.

11.2.5. Instrument calibration verification must be performed:

11.2.5.1. At the beginning and end of each analytical batch (except, if an internal standard is used; only one verification needs to be performed at the beginning of the analytical

batch). As per Standard Methods sections 1020 and 3020, run CCVs every 10 samples.

11.2.5.2. Whenever it is expected that the analytical system may be out of calibration or might not meet the verification acceptance criteria.

11.2.5.3. If the time period for calibration of the most previous calibration has expired, or

11.2.5.4. For analytical systems that contain a calibration verification requirement.

11.2.6. If the method does not specify criteria for the acceptance of a continuing instrument calibration, verification must be established, e.g., relative percent difference.

11.2.7. If the continuing instrument calibration verification results obtained are outside established acceptance criteria, corrective actions must be performed.

11.3. UNACCEPTABLE CONTINUING INSTRUMENT CALIBRATION VERIFICATIONS

11.3.1. If routine corrective action procedures fail to produce a second consecutive (immediate) calibration verification within acceptance criteria, then either the laboratory has to demonstrate acceptable performance after corrective action with two consecutive calibration verifications, or a new initial instrument calibration must be performed.

11.3.2. If the laboratory has not verified calibration, sample analyses may not occur until the analytical system is calibrated or calibration verified. If samples are analyzed using a system on which the calibration has not yet been verified the results shall be flagged.

11.3.3. If these criteria are not met, a second continuing calibration standard must be run (either freshly prepared or a second injection, as appropriate). No individual analyte can fail the CC criteria two consecutive times. If the criteria are still not met, a new initial calibration must be run and the new calibration curve verified. The laboratory qualifies the data with “V” flag if the sample data is associated with failed calibration verification.

11.3.4. As per TNI-EL-V1M4- 2009-1.7.2.e, data associated with an unacceptable calibration verification may be fully useable under the following special conditions:

11.3.4.1. If there was a high bias and there is a failed continuing calibration verification, the lab reports only data associated with samples that are non-detects.

11.3.4.2. If there was a low bias and there is a failed continuing calibration verification, the lab reports only data associated with samples that have a result greater than the maximum regulatory limit/decision level.

Table 11-1 Minimum Calibration Frequency and Acceptance Criteria

Analysis	Method	Calibration Technique	Acceptance Frequency	Criteria
Organohalide Pesticides and PCB products	505	Endrin Breakdown Initial Calibration	Daily beginning and end of analysis	< 20% degradation % RSD ≤20
		Cal Verification Std	beginning and end of analysis	80 – 120 %
		LRB	before start of analysis; each time set of samples extracted or reagents changed	< RL
		LFB	Every 20 samples (all samples extracted within a 24-hr period) points	%R = 70 – 130% Require control charts after 30 data
		MRL checks	Daily	50 – 150% Requires control charts after 30 data points
		LFM IDC, 7 LFBs QCS	Every 10 samples Initial set up, new analyst Quarterly	%R = 65-135 % ≤ 20 % RSD, 70 – 130 % R
Volatile Organics Including DIPE, TAME, ETBE Low level 1,2,3-TCP	524.2	BFB Sensitivity	Every 12 hours of operation	Ion abundance criteria (Table 11-3)
		Initial Calibration (7-pt)	Prior to analysis, or when CC fails	RSD ≤20 % / r≥0.995
		Continuing Calibration	After BFB tune and every 12 hours of operation and at the end of analytical batch (highly recommended by Method)	RF within 30% of the initial calibration
		Surrogate	added to CCV, every sample & all initial calibration stds. (Not required for TCP).	70-130 % Rec
		MS/MSD (upon client request)	Every 20 samples. (Not required for TCP).	70-130 % Rec.
		LCS/LFB	Every 20 samples Every 12 hrs or every 10 samples (TCP)	70-130 % Rec. 80-120 % Rec. (TCP)
		LFB Dup (TCP: can be used in place of Lab Duplicate)	Quarterly	RPD <20%
		Blank	Every 20 samples. Every 10 samples for TCP.	<MRL
		QCS (TCP)	Quarterly (TCP)	%R = 80-120% (TCP)
		MRL checks	Daily	± 50% of the true value
		Lab Duplicate (TCP)	1 per 10 samples (TCP)	% RPD < 20% (TCP)

Analysis	Method	Calibration Technique	Acceptance Frequency	Criteria
Volatile Organics	524.3	BFB Sensitivity	Prior to initial calibration	Refer to Table 1 in 524.3 SOP
		Initial Calibration (7-pt)	At the start of the analytical up or when CCV fails	Cal. Points ≤MRL: ±50% Other cal. Points: ±30%
		ICV/QCS	After initial calibration and quarterly	70-130% Rec
		Continuing Calibration Check	After every 10 field samples	%D for each analyte below MRL: ≤50% Above MRL: ≤30%
		LRB	1 per analytical batch	< ½ MRL
		Matrix Spike/LFSM	1 per analytical batch	70-130% Rec
		LFBD	At least quarterly	70-130% Rec RPD: <20%
		Surrogate	Every sample, QCs, DCCs	70-130% Rec
		Internal Standards	Every sample and calibration standards	±30% of most recent CCC ±50% of average response in ICAL
Semi- Volatiles Organics	525.2	DFTPP Sensitivity	At the beginning of each 12 hours that samples are analyzed	See Table 11-3
		Endrin/DDT Degradation Check	Daily, each 12 hours, before sample run	<20% breakdown
		Initial Calibration	Prior to analysis, when CC fails	< 30% RSD
		Continuing Calibration	Beginning of each 12 hours and every 12 hours that are analyzed and at the end of analytical batch (highly recommended) by Method)	70-130% Rec
		MS	5 % or 1 per sample set Extracted whichever is more frequent	70-130 % Rec
		LCS/ LFB	5 % or 1 per sample set Extracted whichever is more frequent	70-130 % Rec
		Method Blank	1 per sample extraction set	< RL
		Surrogates	added to each sample before extraction	% R =70-130%
		MRL Checks	Analyzed with each extraction batch	50 – 150% Requires control charts after 30 data points
		IS	added to each sample before extraction	area count must not decrease by >50 % from initial calibration and 30% from CCC.

Analysis	Method	Calibration Technique	Acceptance Frequency	Criteria
Trihalomethane /Chloral Hydrate/ Haloacetonitrile /EDB/DBCP	551.1	Initial calibration (Extracted)	Beginning of analysis	< 10 % RSD
		Lab Performance Check	Beginning of analysis	Table 7 of the method
		Endrin Breakdown	Beginning of analysis	< 20 %
		Calibration verification (CCV=LFB)	Before start of analysis, every 10^{th} sample, and after the final sample analysis	% R = 80-120 % for 90 % analytes & 75-125 % for all analytes
		LRB (Lab Reagent Blank)	1 per extraction Batch	< ½ MRL
		LFB (Lab Fortified Blank)	Every 10 samples. (Not Required).	% R = 80-120 % for 90 % analytes & 75-125 % for all analytes
		LFM	every10 samples	80-120 %
		LFM/Duplicate	see sample duplicate	see sample duplicate
		Sample Duplicate	10 % or at least 1 per set, whichever is greater	RPD < 20 for 90 % of analytes, RPD <25% for all analytes
		Surrogate	All samples	80-120 %
		QCS	Quarterly	same as CCV
		IDC, 7 LFBs	Initial set up new analyst	R = 80-120 %, < 15 % RSD
		Stock solutions Verification; Outside Source.	every new lot	< 20% RPD
Volatile Organics	624	BFB Sensitivity	Prior to ICAL and calibration verification, every 12 hours of operation	Ion abundance criteria (Table 11-3)
		Initial Calibration	Prior to analysis, or when CC fails	RF <35 % RSD
		Continuing Calibration (QC Check Std)	After BRB tune, every 12 hours of operation	All analytes' %R must meet % R as specified in Table 5 of Method 624 (See SOP)
		Surrogate	added to CCV, every sample & all initial calibration stds.	%R as specified in SOP
		MS/MSD	Every 20 samples	All analytes' %R must meet % R as specified in Table 5 of Method 624
		MRL check	Daily, prior to sample analysis	50 – 150% Requires control charts after 30 data points
		LCS/LFB	Every 20 samples	All analytes' %R must meet % R as specified in Table 5 of Method 624

Analysis	Method	Calibration Technique	Acceptance Frequency	Criteria
Base Neutrals and Acids	625	DFTPP Sensitivity	Every 12 hours of operation	Ion abundance criteria (Table 11-3)
		Initial Calibration	Prior to analysis, when CC fails	All analytes <35% RSD
		Continuing Calibration (same as MRL Check)	Every 12 hours of operation	All analytes w/in ±20% Of the predicted response
		MS/LFM	Every 20 samples	All analytes' %R must meet % R as specified in Table 6 of the method
		LCS/LFB	Every 20 samples	All analytes' %R must meet % R as specified in Table 6 of the method
HAA	6251B	Calibration curve	each batch	<20%RSD or r ≥ 0.995
		Method Blank	1 per batch of 20 samples or less	< ½ MRL
		LCS/ LFB	1 per sample set Extracted or 20 samples	LCS: 80-120% Mid and High LFB : ±15%
		MRL check	1 per sample set extracted or 20 samples	50-150%
		MS/LFM	1 per 10 sample set extracted	Control Chart Limits updated annually
ICP Metals	200.7/ 6010	ICV	Following calibration	95-105% Rec, <3% RSD
		Method blank	Every 20 samples	< ½ MRL
		MS/MSD	Every 10 samples	70-130%, 75-125% (6010B)
		MRL Check	Beginning and end of the run	50 – 150%
		LCS/LFB	2 per batch of 20 samples	85-115%
ICPMS Metals	200.8/ 6020	Tuning Solution	At the start of QC program or after major maintenance or every 2 weeks	Good Performance: 0.75 amu peak width at 5% peak height Mass calibration: <0.1 amu from unit Mass Instrument stability: 5x run; <5% RSD
		Quality Control Sample(QCS)	Quarterly	90 –110%
		Initial Calibration Verification	Immediately following calibration	95–105% Rec
		Calibration blank	Each batch	< ½ MRL
		Linearity Check 5x CCV/upper limit of Calibration Range	Prior to sample sequence	90-110% Rec
		Replicate Integration	3 replicates	≤ 10% RSD at higher concentrations

Analysis	Method	Calibration Technique	Acceptance Frequency	Criteria
ICPMS Metals (con't.)	200.8/ 6020	Continuing Calibration Verification (CCV)	Every 10 samples	90-110 % Rec
		Minimum Report Limit (MRL), Check/CRDL	Beginning of analysis and end of the sample run	50-150%
		Laboratory Fortified Matrix (LFM)	Every 10 samples	70-130% Rec
		Laboratory Fortified Matrix (LFM) Duplicate	2 in every 20 samples	70-130% Rec 20% RPD
		LCS/LFB	One per batch of 20	85-115% Rec
		Internal Standards (IS)	Spike each sample, standard and blank	60-125% of the response in the calibration blank
		Method Blank	1- per batch of 20-samples	<1/2 MRL or <1/2 CRDL
		Instrument Blank	Prior to Calibration	< ½ MRL
Cr VI (Dissolved)	218.6/ 3500CrB	Initial Calibration	Daily at the beginning of each run	r ≥ 0.999
		IPC(CCV)	1-per 10 samples	95-105 % Rec
		LRB (Lab Reagent Blank)	1-per 10 samples	< ½ MRL
		LFB/QCS	1-per batch of 20 samples	90-110% Rec (external source)
		LFM	1-per 10 samples	90-110% Rec
		LFMD	1-per batch of 20 samples	90-110% Rec (RPD <20%)
		MRL Check	Daily	50 – 150%
		QCS	Quarterly (see LFB)	90-110%
		LDR	Start of program	minimum 7 stds

Automated Wet Chemistry:

Analysis	Method	Calibration Technique	Acceptance Frequency	Criteria
Cyanide Fluoride Nitrate Nitrite Phenolics	335.4, 9012B SM4500F C 353.2, 300.0 300.1 420.1, 420.4	Linear Calibration curve (7-11 pt)	Each batch	r ≥ 0.995 (correlation coefficient)
		Calibration blank	1-per 10 samples	< ½ MRL
		MRL check	Each batch	50 – 150%
		MS/MSD	Every 20 samples (Phenol 420.4 – every 10 samples)	Limits: Fl; 73-124% Phenol, CN, NO3 (353.2); 90-110% NO3, Phenol Low; 80-120%

Analysis	Method	Calibration Technique	Acceptance Frequency	Criteria
Cyanide Fluoride Nitrate Nitrite Phenolics (con't.)	335.4, 9012B SM4500F C 353.2, 300.0 300.1 420.1, 420.4	LCS/LFB	Every 20 samples (Phenol 420.4 – every 10 samples)	Method Limits: Fl; 81-116% CN, NO3, Phenol; 90-110% Phenol Low 80-120%
Residual Chlorine	SM 4500 Cl-G	LCS/LFB	Every 20 samples	85-115%Rec
		MS/LFM	Not Required	20 % RPD
		MRL check	1 per batch of 20 or less	50-150%
		Duplicate	Every 20 samples	<20 % RPD
Anions by IC	300.0/300.1/317	Calibration curve (7-11-pt)	Each batch	r ≥ 0.995 correlation
		Calibration blank	1-per 10 samples	< ½ MRL
		Method Blank	1- per batch of 20-samples	< ½ MRL
		MRL Check	At the beginning of the run	50-150%
		MS/MSD	Every 10 samples (MS); Every 20 samples	80-120 %
		LCS/LFB	Every 20 samples	90-110 %
Total Dissolved Solid	SM2540C	Method Blank	Each time used, 1 per batch	<MRL
		Weight Check	Reweigh till weight difference is <4% or <0.5mg	<4% or <0.5mg difference
		MRL Check	Daily	50-150%
Total Suspended Solids	SM2540D	Method Blank	1 per batch	<MRL
		MRL Check	Each batch	50-150%
Total Solids	SM 2540B	Method Blank	Every 10 samples	<MRL
Total Volatile Solids	160.4	LCS	1 per batch	Within certified range
pH	150.1/SM4500H+B	3 buffers	Each time used	± 0.1 pH unit of true value
Conductivity	120.1/SM 2510B	1 check solution	Each time used	± 1 % of true value
		MRL Check	Daily	50-150%
TOC	SM 5310C	Calibration curve (6-pt)	Every 6 months or until instrument drifts	r ≥ 0.995 correlation
		Blanks	At the beginning of batch and every 10 samples	< 0.250ppm

Analysis	Method	Calibration Technique	Acceptance Frequency	Criteria
TOC (con't.)	SM 5310C	MS/MSD	MS: Every 10 samples MSD: Every 20 samples/batch	80-120%
		LCS/LFB/CCV	At the beginning of batch and every 10 samples	90-110 %
		MRL Check	Daily	50-150%
		LCS1/MRL Check	Every batch	50-150%
		Lab Duplicate	All samples	≤10 % RPD (TOC ≥ 2.0 mg/L) ≤15% RPD (TOC < 2.0 mg/L)
UV 254	SM 5910B	Calibration curve (3-pt) Verification	Prior to analysis of samples	90-110 %
		Blank/UV absorbance @ 254	At the beginning of batch and after every 10 samples	< 0.004 units
		LCS/LFB UV absorbance @ 254 nm	At the beginning of analysis	Agrees within manufacturer's listed range
		MRL Check	Daily	50-150%
		Lab Duplicate	All samples analyzed in duplicate	≤10% RPD ≤15% RPD for low standards

NOTE: 1) Any deviations from the listed criteria are specified in the SOP.
2) Concentrations for all continuing calibrations are in the middle of the linear range.
3) For all other methods not listed in the Quality Manual, see calibration frequency and acceptance criteria in individual SOPs.

Table 11-2 Calibration Procedures

Instruments	Minimum # of Calibration Standards	Calibration Method
TOX	3 points standard (for precision only)	Titration
Anions, Nutrients (Ion Chromatography)		
Nitrate, NO3 Nitrite, NO2 Chloride, Cl2 Sulfate, SO4	11-points 11-points 7- points 10-points	Quadratic Quadratic Quadratic Quadratic
Phenol, Cyanide	5 point	Linear Regression
Fluoride	3 point minimum	Linear Regression (log)
pH	3 point	Slope
Radiation	5-6 points	Efficiency Curve
TOC (TOC Analyzer)	6 Point	Linear Regression
UV 254 (Spectronic 601) Spectrophotometer	3 Point	Efficiency Curve
524.2 (GCMS)	5-7 Points	Linear Regression
HAA (GC)	5 Point	Linear Regression

Note: For all other methods not listed in the Quality Manual, see calibration procedures in individual SOPs.

Table 11-3 Ion Abundance Criteria (Tune Criteria)

(A) BROMOFLUOROBENZENE (BFB) (524.2 and 624)

Mass	Ion Abundance Criteria
50	15 - 40% of mass 95
75	30 - 60% of mass 95 (624) ; 30-80 % mass 95 (524.2)
95	Base peak, 100% relative abundance
96	5 - 9% of mass 95
173	Less than 2% of mass 174
174	Greater than 50% of mass 95
175	5 - 9 % of mass 174
176	Greater than 95%, and less than 101% of mass 174
177	5 - 9% of mass 176

(B) DECAFLUOROTRIPHOSPHINE (DFTPP) (525.2)

Mass	Ion Abundance Criteria
51	10-80% of the Base Peak
68	Less than 2% of mass 69
70	Less than 2% of mass 69
127	10-80% of the base peak
197	Less than 2% of mass 198
198	Base Peak or >50% of 442
199	5 - 9% of mass 198
275	10 - 60% of the base peak
365	Greater than 1% of the base peak
441	Present, but less than mass 443
442	Base Peak or Greater than 50% of mass 198
443	15-24% of mass 442

Table 11-4 Initial Calibration Acceptance Criteria

Anions/Nutrients	Initial calibration value for standards must be within 10% of the nominal value. $r \geq 0.995$
GC	Initial Calibration RF <20% RSD or second order fit, continuing calibration. RF ≤ 20% Difference. Must meet specific method calibration criteria.
GCMS, EPA 524.2	Initial Calibration ≤ 20 % RSD, $r \geq 0.995$
HAAs	Initial Calibration correlation coefficient $r \geq 0.995$, < 20 % RSD
HPLC	Correlation coefficient must be ≥0.995 or 20% RSD
Metals	Initial calibration value for standards must be within 5% of the nominal value.
pH	Values for 4, 7, 10 buffers must be ± 0.1 pH unit of the nominal value
Radiation	Each calibration standard counts must be >10,000.
TOC	Initial calibration value for standards must be within 10% of the nominal value. $r \geq 0.995$
TOX	Initial calibration value for standards must be within 10% of the nominal value. $r \geq 0.995$
UV 254	Initial Calibration value for the standards must be within 10 % of the normal value.

12.0 EQUIPMENT

12.1. ANALYTICAL EQUIPMENT

12.1.1. All equipment is properly maintained, inspected, and cleaned.

12.1.2. Table 12-1 contains a list of the major analytical equipment used during sample preparation and analysis. For Microbiology, pressure cookers are not used for sterilization of growth media [TNI-EL-V1M5-2009-1.7.3.7.b.ii].

12.2. SUPPORT EQUIPMENT

12.2.1. **Balances**

Analysts are responsible for daily calibration verification checks of the analytical balances in the laboratory with Class S weights and annual calibrations of the drying ovens thermometer with an NIST traceable certified thermometer. Documentation of the balance and oven checks is maintained in the appropriate logbook. Reference certified thermometers are calibrated every five years. A yearly thermometer calibration check is done for all other thermometers and all thermometers are labeled showing any necessary correction to achieve true readings. Balances are calibrated annually and Class S-weights are calibrated every 5 years by an outside vendor. Copies of these balance and thermometer records are filed with the QA records for the laboratory. All Class S weights and traceable thermometer standards are used for calibration only and for no other purpose to ensure that the performance as reference standards are always valid.

Balance calibration is verified on the day of use prior to weighing samples, standards or reagents. If balance does not meet the acceptable criteria of ± 0.1 % or other vendor recommended limits, the analyst reports to QA that balance needs service. The instrument is labeled “out of service” until repaired. The Analyst records the problem and identifies corrective action, date of service, and if corrective action resolved the problem.

12.2.2. **Temperature Monitoring**

Refrigerators, incubators, temperature are monitored 2 times daily in at least 4 hour intervals. If the temperature measured is not meeting the acceptance criteria of 4± 2°C, analyst reports to the QA department. QA then monitors the temperature after 2 hours and more often if needed. If non-compliance is still observed, QA calls for service. The instrument is labeled “out of service” until repaired. QA records the problem identified, corrective action, date of service, if called, and if corrective action resolved the problem.

12.2.3. **Pipets**

Eppendorf pipette function verification is done on the day the standards are prepared for pipets used for the preparation of both the primary and secondary standards. Monthly frequency is done for pipets used either for the preparation of either the primary or secondary standards and for Class A pipets used for the preparation of the other set of standards. When used over a range of settings, the pipet is calibrated at the highest and lowest settings. If not meeting the acceptable range of ± 2 % of the set value, the analyst investigates and identifies the problem. The pipet is cleaned if needed and inspected for signs of wear or damages or for residual liquids that may have been sucked in the pipet. After the appropriate Corrective Action, the pipet is again calibrated. Corrective Action taken and problem identified is recorded. If corrective action did not resolve the problem, the analyst documents in the logbook that the pipet is off-line. The pipet is also labeled "out of service" until repaired.

12.2.4. **Microbiology Volumetric Equipment [TNI-EL-V1M5-2009-1.7.3.1.a.iii]**

Volumetric Equipment shall be calibrated as follows:

12.2.4.1. Equipment with movable parts such as automatic dispensers, dispensers/diluters, and mechanical hand pipettes shall be verified for accuracy quarterly.

12.2.4.2. Equipment such as filter funnels, bottles, non-class A glassware and other marked containers shall be calibrated once per lot prior to its use.

12.2.4.3. The volume of the disposable volumetric equipment such as sample bottles, disposable pipettes, and micropipette tips shall be checked once per lot.

12.2.5. **Glassware**

Table 12-2, contains the SOP for glassware cleaning. All class volumetric glassware is dried at room temperature rather than oven baked.

The washing and sterilization procedures for laboratory glassware are tested annually by testing glassware for inhibitory residues as shown in Standard Methods.

12.2.6. **Water Quality File**

The pure water system for EEA was assembled by US Filter in January 2003. It consists of reverse osmosis, mixed bed deionizers, ultraviolet disinfection, filtration, and an organic scavenger side stream return loop. The system is connected to a conductivity meter which signals when the mixed bed resin demineralizers need to be changed.

The quality of laboratory pure water is analyzed monthly for conductivity, pH, chlorine residual, TOC, and standard plate count and annually for water suitability ratio, inhibitory residue (annually, each time new lot of detergent and for new washing procedures), and trace metals (Pb, Cd, Cr, Cu, Ni, and Zn). Table 12-3 lists the

acceptance criteria for these analytes. This data is recorded and submitted to the QA department. These reports are with the QA Department for review and maintained for ten years.

12.2.7. **Out of Service**

All major instruments if off line will be labeled "out of service" until repair.

12.3. PREVENTIVE MAINTENANCE

12.3.1. **Routine Maintenance Activities**

EEA carries maintenance contracts on all major laboratory equipment, under which much of the preventative maintenance is performed. Routine servicing, such as cleaning of rods, source, or detectors, is performed on a regular basis by the analyst. This type of service is performed according to the procedures and at the frequency specified by the manufacturer. Routine maintenance is done when instrument performance starts to degrade as demonstrated by a failure to meet one or more QC criteria, decreased ion sensitivity, degrading peak resolution, lowered response factors, or shifts in calibration curves. Activities that are performed on a routine basis can be found in the Instrument Maintenance Nonmethod14 SOP.

12.3.2. **Documentation**

Instrument maintenance logbooks are maintained for most major instruments. All repairs and any routine or non-routine maintenance activities are recorded in the logbooks. The date of the activity, the person performing it, and the nature of the activity are recorded. Expendable items for all major instruments are kept on hand to minimize downtime.

The following are documented in the instrument logbooks:

12.3.2.1. Name of the item of the equipment

12.3.2.2. Manufacturer's name, type identification and serial number or other unique identification

12.3.2.3. Date received and date placed in service

12.3.2.4. Current location, where appropriate

12.3.2.5. Condition when received (e.g. new, used, reconditioned)

12.3.2.6. Copy of manufacturer's instructions where available

12.3.2.7. Dates and results of calibrations and/or verifications and date of the next calibration and/or verification

12.3.2.8. Details of maintenance plan carried out to date and planned for the future

12.3.2.9. History of any damage, malfunction, modification or repair

12.3.2.10. Records of service calls

12.3.2.11. Calibration status for instrument that are calibrated outside the direct control of the laboratory are checked before use (after an instrument is returned from outside repair) [TNI-EL-V1M2-2009-5.5.9][ISO/IEC 17025:2005(E)-5.5.9].

12.3.3. **Contingency Plans**

12.3.3.1. An effort is made to have a functionally equivalent backup instrument available in case of a catastrophic instrument failure. Maintenance contracts are carried on the major instruments and generally provide for 24-48 hour response for repairs. If necessary, EEA has a list of qualified laboratories to subcontract work to, upon client approval.

12.3.3.2. In the event a holding time expires while the sample is in the custody of EEA, a project manager will call the client to inform them of this situation. Based on subsequent arrangements made between the lab and the client, fees for re-sampling and subsequent analysis may be incurred by the lab.

Table 12-1 Equipment (06/28/13)

Table 12-1 Equipment (06/28/13)

	Vendor	Model	Year Acquired	Detectors	Tests	Serial #
Microbiology	Thermo Scientific	Multiskan FC	2010	Photometer	microcystin, cylindrospermopsin, saxitoxin	357-00450T
	Zeiss		Unknown (this is very old)	Light Microscope	Algae Analysis	#1782
	Molecular Devices	Spectramax L Luminometer	2011		AOC Analysis	LU 03146
	Polytron	1300D	2012	Homogenizer	Microcystin	PF-809-0004-09-01

	Vendor	Model	Year Acquired	Detectors	Tests	Serial #
	Fluid Imaging	Flow Cam	2012	Flow Cytometer	Algae Analysis	5056
Metals	Agilent	7500CS	2011	ICP/MS	200.8	JP93200243
	Cetac	ASX-500 Model 510 Autosampler	2011	ICP/MS Autosampler	200.8	10047ASX
	Nelsab	Merlin M75 Chiller	2011	ICP/MS Chiller	200.8	107043069
	Perkin-Elmer	Elan DRC II	2003	ICP/MS	200.8 & Rare Earth Ele's	Q1280212
	Perkin-Elmer	ELAN 6000	1999	ICP/MS	200.8	3929707
	SeaFast3	Autosampler SC2DX	2009	N/A	Iodide/Iodate	X2DX-HS-TDP-16-100801
	SeaFast3	Nebulizer PC3-F	2009	N/A	Iodide/Iodate	PC3F-100701
	ESI	Autosampler SC2DX	2009	N/A	Speciation	X2DX-HS-TSP-16-100711
	ESI	Nebulizer PC3-F	2009	N/A	Speciation	PC3F-100505
	Optima	4300 DV	2003	ICP	200.7	077N2121801
	Perkin-Elmer	FIMS400	2000	Mercury Analyzer	Mercury	4605
	Environmental Express	Hot Block	2002	Digestion Block	Metals Prep	1763CEC1134
	Environmental Express	Hot Block	2005	Digestion Block	Metals Prep	3703CEC1784
	Environmental Express	Hot Block	2012	Digestion Block	Metals Prep	6974CECW3257

	Vendor	Model	Year Acquired	Detectors	Tests	Serial #
	Thermo	Lindberg/blue WB1130C-1		Mercury Digestion Water Bath	Mercury Prep	R12R-507226-RR
	Agilent	7500CE	2007	IPC/MS	200.8	JP51201349
Rad	Protean 8 channel	MPC9604	1998	Proportional Counter	Gross Alpha/Beta and Ra 228	83023
	Gamma Products Inc	T7500	2006	Proportional Counter	Gross Alpha/Beta and Ra 228	
	Beckman	6500	1993	Liquid Scintillation System	Radon	7067177
	Beckman Coulter	Allegra 6	2003	Benchtop Centrifuge	Ra 228	ALS02M09
	Linberg Blue	HP53025C	2001	Hot Plate	Gross Alpha/Beta	W01K-496436-WK
	Canberra, Inc	Gamma Spec	2010	High Purity Ge Detector	Ra 226 and Ra 228	9771
	Canberra, Inc	Gamma Spec	2013	High Purity Ge Detector	Ra 226 and Ra 228	10466
Ion Chromatography	Metrohm	881 Compact IC w/Conductivity Detector	2009	IC	Fluoride, Ammonia	3170
		887 UV/VIS	2010	IC-Detector UV/VIS	218.6	3146
		818 External Pump	2009	IC-Pump	218.6	10156
		858 Autosampler	2009	IC-Autosampler	All tests	2797
	Dionex	CD20	1998	IC-detector-CD20	300.0	98080693
		GP50	2004	IC-Pump-DX600 series	300.0	0402006
		LC20	1998	IC-Column Cabinet	300.0	98070159

	Vendor	Model	Year Acquired	Detectors	Tests	Serial #
	Dionex	AD25	2001	IC-detector-UV/VIS	218.6	00120138
		IP25	2000	IC-Pump- DX600 series	218.6	00120237
		LC10	1998	IC-Column Cabinet	218.6	98100540
	Dionex	IP25	1998	IC-Pump-DX600 Series	300.1/300.0	98120234
		ED40	2000	IC-Electrochemical Detector	300.1/300.0	96020230
		LC20	2000	IC-Column Cabinet	300.1/300.0	00110243
	Dionex	ICS2000	2005	IC w/Conductivity Detector	300.0	03080229
	Dionex	ICS3000	2006	IC w/Conductivity Detector	314/300.1/317	6030479
		VWD3400	2008	IC-UV/VIS Dectector	317	8004187
		GP40	1998	IC-Pump-DX500 Series	317	98070271
	Dionex	ICS3000	2007	IC w/Conductivity Detector	314/300.0/EDTA/NTA	06120153
	Dionex	ICS3000	2010	IC w/Conductivity Detector	300.0/300.1/317	07050797
		VWD3400	2010	IC-UV/VIS Detector	317	8022929
		GP40	2000	IC-Pump-DX500 Series	317	00120038
	ABSCIEX/ Dionex	API2000	2011	LCMS	331 mod	B5010208H
		Ultimate3000	2011	LCMS Pump	331 mod	8040461
		Ultimate3000	2011	LCMS Solvent Rack	331 mod	8040973

	Vendor	Model	Year Acquired	Detectors	Tests	Serial #
		Ultimate3000	2011	LCMS AutoSampler	331 mod	8040943
	Dionex	ICS 1600	2012	IC w/Conductivity Detector	314	12090982
	Dionex	ICS 1600	2012	IC w/Conductivity Detector	UCMR 3 300.1	12090984
Inorganics	HACH	16500-10	1996	COD Digestor	COD	991000019578, 910404628
	COSA	TOX-100	2007	Coulometric	TOX	A7M-42726
	Sievers	AS-800	2003	UV-Persulfate	TOC, DOC	910404628
	HACH	DR/4000U (DR500)	2003	UV/VIS	UV 254	1225267116
	Mettler	Muli-7 Meter		pH/Ion Meter	pH/ISE, Fluoride, CNDW	1225267116
	HACH	DR/4000V	2002	Spectrophotometer	COD, R-SIO2, etc	0006V0000995
	HACH	DR/4000U	2005	Spectrophotometer	UV 254, COD, R-SiO2, Ferrous, Cr-VI.	9509U0000228
	WTW	OX1730P	2006	DO Meter/Probe	BOD, CBOD, DO	06440156
	Lachat	BD-46	1999	Block Digestor	TKN	1800-712
	Lachat	BD-48	2010	Block Digestor	TKN	1004-00000975
	YSI	YSI-5000	2010	DO Meter/Probe	BOD, CBOD, DO	10A101086
	HACH	DR2010	2010	Spectrophotometer	MBAS, oPO4, S2	99050001332
	OI Analyitcal	390 Autosampler	June 2011	Autosampler	Phenolics Low Level	021102A130

	Vendor	Model	Year Acquired	Detectors	Tests	Serial #
		FS3100		Analyzer Module		114831572
		A515000		Distillation Module		114815572
	Carone	2050-1		Circulator		03301120501288
	Sievers	5310C	2012	UV-Persulfate	TOC	12106054
Misc	Reliance Glass	Midi-Still	1998	Distillation	Cyanide/Phenol/Ammonia	NA
	Olympus	BH-2	1192	Fluorescence Microscope	Protozoan	T2-105170
	Orion	101	1990	Conductivity	Conductance	127
	Hitachi-TEM	600AB	2000	X-Ray	Asbestos	542-50-03

	Type	Model	Year Acquired	Detectors	Tests	ID	Serial #
GC Systems	Varian	3800	2001	Dual ECDs	551.1 SODA	21	3800-02440
	Varian	3800	2001	Dual ECDs	551.1 SODA	22	3800-08107
	Varian	3800	2002	Dual ECDs	505	23	3800-08827
	Agilent	3800	2003	Dual ECDs	HAA – 6251B	24	US10306042
	Agilent	3800	2003	Dual ECDs	515.4	25	US10315085

	Type	Model	Year Acquired	Detectors	Tests	ID	Serial #
	Agilent	3800	2003	Dual ECDs	505	26	US10315084
	Varian	3800	2004	Dual ECDs	505	27	3800-11203
	Agilent	6890N	2005	Dual ECDs	N/A	28	US10427028
	Agilent	6890N	2005	Dual ECDs	551.1	29	US10440020
	Agilent	6890N	2005	Dual ECDs	HAA – 6251B/556	30	US10512068
	Agilent	6890N	2005	Dual ECDs	HAA – 6251B	31	CN10518044
	Agilent	6890N	2005	Dual ECDs	551.1	32	CN10505035
	Varian	3800	2006	Dual ECDs	504.1	33	3800-12789
	Agilent	6890N	2007	Dual ECDs	515.4	34	CN10706031
	Agilent	6890N	2007	Dual ECDs	551.1	35	CN10706032
	Agilent	6890N	2007	Dual ECDs	6251B (HAA)	36	CN10706030
GCMS Systems	HP	5890/5972	1997	VOA - MS	524.2, TCP Low	J	3118A02321
	HP	5890/5972	1995	VOA - MS	524.2, 624, 8260	H	3501A02407
	Varian Ion Trap w/ CI/MS	Saturn	2000	Semivoa - MS	SPME/6040D	ITS2	4654
	Varian Ion Trap	Saturn 2000	2001	Semivoa - MS	521/Nitrosamines, SPME, 522	ITS3	4635-9050
	Agilent	6890/5973	2003	MSD	524.2/624/8260/ 524.3	L	US33246003
	Agilent	5973/6890	2005	MS	524.2/624/8260	N	US4647377
	Agilent	5973/6890	2005	MS	524.2, UCMR3 524.3	P	US44647375
	Varian	CP 3800/4000	2009	GC/MS	Endothal/521	ITS6	4000-0488
	Agilent	6890N	2005	GC	525.2, 526, 527	M	CN10416008
	Agilent	5973 inert	2005	MS	525.2, 526, 527		US40610242

	Type	Model	Year Acquired	Detectors	Tests	ID	Serial #
	Agilent	6890N	2006	GC	EDC4	S	CN10534101
	Agilent	5975N	2006	MS	EDC4		US52420838
	Agilent	6890N	2005	GC	529	R	CN10517080
	Agilent	5973 inert	2005	MS	529	R	US44610770
	Agilent	6890N	2003	GC	525.2, 526, 527	K	CN10331006
	Agilent	5973N	2003	MS		K	US30945838
	Varian	CP-3800	2006	GC	521/Nitrosamines	ITS5	3800-12765
	Varian	4000CIMSM	2006	MS	521/Nitrosamines	ITS5	4000-00291
	Agilent	5975 6850	2007	GCMS GC	524.2, 624, 8260, 524.3	U	US65115374 CN10646018
	Varian	3800 320	2008 2008	GC MS/MS	1613B (Dioxin)	GC-QQQ-1	1204
	Agilent	5975	2011	GCMS	524 TCP LOW	X	US11179701
	Thermo	ISQ	2012	GCMS	524, 524.3	V	ISQ120827
	Thermo	ISQ	2012	GCMS	522	T	ISQ120935
LCMS	Dionex	U3000	2006	HPLC	CLO4, Acrylamide, EDCLC-2	LC-2	2240601
	Waters	Ultima Quattro	2004	MS/MS	CLO4, Acrylamide, EDC, 331	LCMS1	VB125
	Agilent	1200	2007	HPLC	CLO4, Acrylamide, EDC, 331	LC-1	DOOSM9-382M
	AB SCIEX	API 5000	2008	MS/MS	CLO4, Acrylamide, EDC	LCMS4	AG22860808
	Agilent	1200 SL	2007	HPLC	UCMR 3, EDC2, 532-LCMS	LCS-3	
	AB SCIEX	API 4000	2007	MS/MS	UCMR 3, EDC2, 532-LCMS	LCMS2	V20670708
	Thermo	TSQ Vantage	2012	MS/MS	UCMR 3, 537	LCMS5	TQU03178
	Thermo	TSQ Vantage	2012	MS/MS	UCMR 3, 539	LCMS6	TQU02731

	Type	Model	Year Acquired	Detectors	Tests	ID	Serial #
HPLC	Waters	2690	2004	HPLC	549.2, 555	HPLC4	DOOSM9-382M
	Dionex	P580	2001	Fluorescence	531.1, 531.2	P580	1530109
	Waters	2690/2487	1998	UV Detector	532, 549, 555	2	H96SM4168R
	Dionex	P680	2005	Fluorescence	547	P680	1000205

Table 12-2 Glassware Washing Procedures

Refer to the Nonmethod 16 SOP for detailed glassware cleaning procedures.

Table 12-3 Water Quality Parameters

Parameter	Acceptance Criteria
Ammonia	< 0.1 mg/L (monthly check)
Residual Chlorine	< 0.10 mg/L
TOC	< 1 mg/L
pH	5.5 - 7.5
EC	<2 µmhos/cm @ 25oC
	<2 µS (µsiemens/cm)
Trace Metals (Cd, Cr, Cu, Ni, Pb, Zn)	<0.05mg/L each collectively <0.1 mg/L
Bacteriological (HPC) Colony forming units/ml	<500 cfu (TNI < 10000 cfu/ml)
Bacteriological Quality of Reagent Water (Suitability Ratio or Ratio of Growth Rate)	0.8 - 3.00
Student's t	< 2.78 for annual use test
Inhibitory Residue	<15% difference in average count

13.0 DOCUMENT MANAGEMENT/CONTROL OF RECORDS

13.1. ANALYTICAL DOCUMENTATION

A critical dimension of any quality assurance program is the ability to document what is occurring in the laboratory. Accordingly, EEA uses a number of forms to document various aspects of laboratory procedures. A discussion of these forms follows.

13.1.1. Analytical Data and Quality Control Forms

Printed forms are used by analysts to standardize the format of routine analyses. For analyses where forms are not available, the analyst records all required information in a notebook. The forms are designed to minimize calculation errors and provide a summary of all quality control data generated for the run.

Analysts are responsible for maintaining these forms. The QA group spot checks these forms periodically. These forms are actively maintained in hardcopy or electronically for a minimum period of 2 years and then stored electronically or stored in hardcopy offsite.

13.1.2. Chromatograms and Data Processing

Chromatograms and strip chart recordings are assigned unique alpha-numeric codes and backed-up on the server or an external hard drive. Information contained within the code includes; test, date and numerical sequence.

Computer records are stored by internal sample ID and test and therefore can be queried on this information.

Scanned hardcopy outputs of chromatograms and data processing are filed with the analytical data forms. Chromatograms and library searches are stored on magnetic tape and the information is retrievable upon client request.

13.1.3. Inventory Control Logs

Records are maintained on the purchase of laboratory supplies detailing the vendor, purchase order number, date of order, and date of receipt. Bottles of reagents are dated upon received so that the shelf life can be monitored.

13.1.4. Stock Standard Logs

A logbook is maintained for preparation of analytical stock standards for each group. Each log contains the date of fresh stock preparation, the lot number and supplier, the preparer's initials, and the weights used to prepare the stock.

13.1.5. Bacteriological Growth Media Log

Upon receipt of new microbiological media, the date received is noted upon the container. Media supplies are dated not only upon receipt but also when initially opened. A written record of quality control on media, materials, and equipment is logged into the Micro QC book. The record includes the results of the check, the initials of the individual performing the check, and the date. Media prepared in the lab is logged into the Prepared Media Log by the analyst. These records include media lot number, date of preparation, manufacturer and lot number, type and amount of media prepared, sterilization time and temperature, final pH, the analyst's initials, and expiration date.

13.1.6. **Instrument Monitoring and Maintenance Logs**

13.1.6.1. When in use, the operating temperatures of incubators, water baths, hot air ovens, and refrigerators are checked daily and recorded. Adjustments or service calls are made when required. Autoclave sterility checks, using ampules of bacterial spores, are made at least monthly, or whenever a problem is suspected but all items are autoclaved with sterility indicator tape. Records of the maintenance are maintained in equipment logs.

13.1.6.2. A separate maintenance logbook is maintained for each analytical instrument. These logs contain a record of routine maintenance as well as any repair work required during instrument set-up.

13.1.7. **Corrective Action**

The form, presented in Figure 15-2, requires documentation on the determination of the out-of-control event or variance, the diagnostics performed to bring the event back under control, and the manner in which re-establishment of control was demonstrated. A flow chart of QIR process can be found on Figure 15-3. The analyst and their supervisor sign the form electronically in STARLIMS and submit it to the QA department for review. Then, QA distributes the corrective action to the appropriate Project Manager so that the client may be contacted if necessary. The analysts keep hardcopies of original corrective action forms and file them with the appropriate raw data package.

13.2. CONTROL OF RECORDS

Figures 13-1 and 13-2 are examples of worksheets and notebooks used in data reduction.

13.2.1. **General Records**

13.2.1.1. The laboratory’s document control procedure includes identification, collection, indexing, access, filing, storage, maintenance and disposal of quality and technical records. Quality records include reports from internal audits and management reviews as well as records of corrective and preventive actions. Records are in the form of hard copy or electronic media.

13.2.1.2. All records are required to be legible and are stored and retained in such a way that they are readily retrievable in facilities that provide a suitable environment to prevent damage or deterioration and to prevent loss. Records are retained for 5 years held secure and in confidence as per TNI-EL-V1M2-2009-4.13.1.3 and 4.13.3.b (ISO/IEC 17025:2005(E)-4.13.1.3 and 4.13.3.b), 10 years for Massachusetts, Hawaii, and New York samples.

13.2.1.3. The laboratory has implemented procedures to protect and back-up records stored electronically and to prevent unauthorized access to or amendment of these records by setting up level of security and/or designating appropriate personnel responsible for the security of the records.

13.2.1.4. The following information is documented as per TNI-EL-V1M2-2009-4.13.3.

13.2.1.4.1. The records include the identity of personnel involved in sampling, sample receipt, preparation, calibration, or testing.

13.2.1.4.2. 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.

13.2.1.4.3. The record keeping system facilitates the retrieval of all working files and archived records for inspection and verification purposes by setting format for naming electronic files.

13.2.1.4.4. All changes to records are signed or initialed by responsible staff. The reason for the signature or initials is clearly indicated in the records such as “sampled by”, “prepared by”, or “reviewed by”.

13.2.1.4.5. All generated data except those that are generated by automated data collection systems, are recorded directly, promptly and legibly in permanent ink [TNI-EL-V1M2-2009-4.13.3.g].

13.2.1.4.6. Entries in records are not obliterated by methods such as erasures, overwritten files or markings. All corrections to record keeping errors are made by one line marked through the error. The individual making the correction signs (or initials) and date the correction. These criteria also apply to electronically maintained records [TNI-EL-V1M2-2009-4.13.2.3, 4.13.3.g.i (ISO/IEC 17025:2005(E)-4.13.2.3, 4.13.3.g.i). The laboratory keeps correspondence relating to lab activities for specific projects. Documentation includes email correspondence between the Project Manager and client.

13.2.2. **Technical Records**

13.2.2.1. The laboratory retains technical records of original observations, derived data and sufficient information to establish an audit trail, calibration records, staff records and a copy of each test report issued, for a defined period. The record for each environmental test or calibration contains sufficient information to facilitate and to enable the environmental test to be repeated under conditions as close as possible to the original. The records include the identity of personnel responsible for the performance of each environmental test and checking of results.

13.2.2.2. Observations, data and calculations are recorded at the time they are made and are identifiable to the specific task.

13.2.2.3. When mistakes occur in records, each mistake is crossed out, not erased, made illegible or deleted, and the correct value entered alongside. All such alterations to records are initialed and dated by the person making the correction. In the case of records stored electronically, equivalent measures are taken to avoid loss or change of original data. When corrections are due to reasons other than transcription errors, the reason for the correction shall be documented [TNI-EL-V1M2-2009-4.13.3.g.ii].

13.2.2.4. Each report or documents issued shall include the name(s), function(s) and signature(s) or equivalent electronic identification of person(s) authorizing the report or documents, and date of issue. Use of computer password unique to each analyst and level of security prevents loss of original data and change of data.

13.3. DATA STORAGE

13.3.1. EEA maintains electronic report files for at least 10 years. The report files are organized alphabetically by client and contain a copy of the report sent to the client, custody information and scheduling information. Report files also include subcontractor reports. The scannable supporting raw data is scanned within six months and stored electronically in the EEA secured server. The non-scannable data (i.e. hardbound logbooks) are stored in a secured offsite storage for a total of 5 years for all states, except 10 years for Hawaii, Massachusetts, and New York. These files are centrally located and a custodian is assigned to maintain, retrieve, and copy files as needed.

13.3.2. Instrument raw data is stored on each instrument's computer. Data is backed-up to a network server or an external hard drive (Chromeleon is backed up to the network server and GCMS/LCMS is backed up to an external hard drive. If instruments are direct read and transcribed into notebooks, then the notebooks are stored in the lab until they are scanned and filed.

13.3.3. All raw data is organized by instrument or test, then chronologically. Logbooks such as sample custody or balance calibration are organized chronologically.

13.3.4. Electronic data from LIMS is stored on data tapes.

13.4. DOCUMENT CONTROL

13.4.1. Document Control procedures are implemented that allow for adequate documentation and control of specific documents. These procedures use a unique identification system that allows for tracking, training documentation, traceability of official copies and the time period the procedure or document was in force. Documents issued to all personnel in the laboratory as part of the Quality System (QS) shall be reviewed and approved for use to authorized personnel prior to use. The list will identify the current revision status to ensure that invalid or obsolete documents are not used. The document control procedures includes that the authorized editions of documents are accessible by the analysts and invalid or obsolete documents are promptly removed from use. All QS documents such as SOP, QM, logbooks are uniquely identified including the following:

- Date of issue and/or revision ID
- Page numbering
- Total number of pages or markings to signify end of documents.
- Issuing authorities [TNI-EL-V1M2-2009-4.3.3.1][ISO/IEC 17025:2005(E)-4.3.3.1]

13.4.2. To ensure that the QM and SOPs remained controlled documents, the master SOPs and QM (original official version of the SOP and QM) and copies of the SOP and QA Manual will be identified. The cover page of each copy will contain a unique identification indicating that the document is controlled copy ___ of ____ copies, initialed and dated by the Quality Manager or her designee in red ink. This ensures that the analyst is currently using the right update or version.

13.4.3. A SOP/Quality Manual Distribution Form will be prepared for each SOP/QM that will include the SOP/QM ID, control number, individual receiving the SOP/Quality Manual, and date of issue.

13.4.4. Record management system is also implemented for control of laboratory notebooks; instrument logbook; standard logbook; and records for data reduction validation storage and reporting. Laboratory archival system will also be implemented to laboratory books and logbooks.

13.4.5. Notebooks and Logbooks are assigned unique ID numbers for control of laboratory records. Upon completion of the book, the analyst returns the book to QA. A new number is assigned to the newly issued notebook. See Table 13-1 for the laboratory document control system for notebooks and logbooks.

13.4.6. Changes to documents shall be reviewed and approved by the same function that performed the original review unless specifically designated otherwise. The designated personnel shall have access to pertinent background information upon which to base their review and approval. Refer to the Nonmethod 25 SOP for detailed document control procedures.

13.5. DOCUMENT CHANGES TO CONTROLLED DOCUMENTS

13.5.1. All documents and/or changes issued to personnel in the laboratory are reviewed and approved for use by the Lab Director, Technical Director and Quality Manager prior to use. A master list or an equivalent document control procedure identifying the current revision status and distribution of documents in the laboratory are established and are readily available to preclude the use of invalid and/or obsolete documents.

13.5.2. Any changes/alterations to laboratory documents are tracked and properly identified. Amendments are clearly marked, dated and initialed and revised documents are formally re-issued immediately. Any obsolete documents are removed from corresponding binders are archived and stored in a secured place.

13.6. ARCHIVAL SYSTEM

An archival system is implemented for managing and removal of all outdated documentation. Records that are archived are Training Records for personnel no longer with the laboratory and Outdated Quality Manual/SOPs. Only current versions of the Quality Manual/SOPs are retained in the laboratory areas. All outdated versions of the Quality Manual/SOPs are returned to the Quality Manager for archiving. All outdated logbooks/work books are turned in to the supervisor for scanning and archiving. Please see 13.3.1 for more information. Refer to the Nonmethod 30 SOP for archiving procedures.

13.7. STANDARD OPERATING PROCEDURES (SOP)

13.7.1. Laboratories shall maintain SOPs that accurately reflect all phases of current laboratory activities such as assessing data integrity, corrective actions, and all test methods.

13.7.2. When an amendment to the SOP is needed, such as a minor update to the entire procedure, the laboratory will handwrite the update with initials and date of the person who made the change in the original copy of the SOP. Also, when a minor mistake is found in the SOP, the laboratory will strike the section with one line, date and initials of the person who does the change in the original copy of the SOP. For any of these minor updates, the analyst(s), supervisor and QA will be notified and they will be included in the next update of the SOP.

13.7.3. The following format must be used for all final technical SOPs.

13.7.3.1. **Header** - A header must be included in the upper right corner of each page of the SOP. The header must include the SOP reference name or number, the revision number, the date the revision began, page number and total number of pages.

13.7.3.2. **Cover Page -** The SOP cover page consists of a summary of the most recent revision information and the signatures of the Analyst, Technical Manager/Group Supervisor, Quality Manager, and Technical Director/Lab Director stating that they approved the SOP including the date that they read and signed the SOP. The approval, issue, and effective dates are included on the cover page. The effective date is two weeks after the approval date.

13.7.3.3. **Body**

13.7.3.3.1. **Title**

13.7.3.3.2. **Scope and Application** - A brief description of the types of matrices the method is applicable to as well as the regulatory programs that may be supported by the use of the method. This section is also used to indicate any special training or level of ability required to perform the method.

13.7.3.3.3. **Method Summary** - A brief description of the method, simple statement of analytical technique and any pre-treatment steps.

13.7.3.3.4. **Interferences** - This section should include any known interferences, as well as potential interferences, particularly for GC/conventional detector methods. It should also include any interferences that may be present as a result of improper sampling procedures, equipment cleaning or analytical technique must be listed here.

13.7.3.3.5. **Safety Considerations** - Specify any known or suspected carcinogens, mutagens, or teratogens among the standards or reagents used. Indicate that the SDS (safety data sheets) are available and where they are located. Each analyst is required to familiarize him/herself with the contents of the SDS before performing the analysis.

Each SOP includes reference to the Laboratory Chemical Hygiene Plan as per OSHA Standard 29 CFR 1910.1450, Occupational Exposure to Hazardous Chemicals in Laboratories-Final Rule.

13.7.3.3.6. **Instrumentation/Apparatus** - The instrumentation used, including specific columns employed for GC, LC, or GC/MS and whether or not there is a primary and confirmatory column.

13.7.3.3.7. **Reagents and Standards** - The sources of all standards and reagents are listed.

13.7.3.3.8. **Sample Collection, Preservation and Handling** - Indicate bottle type, preservative and volume necessary for analysis. Include holding times for standards.

13.7.3.3.9. **Calibration Procedure** - Detailed preparation instructions for each calibration, LCS or MS/MSD standard should be included. A table should be present to show how daily calibration and control standard solutions are prepared from working stock standards. Calibration frequency should be specified. Expiration information should be included for each type of standard prepared.

13.7.3.3.10. **Analytical Procedure** - Since the purpose of a SOP is to provide clear instruction to avoid loss of key information from one analyst to another, it is critical that this section be detailed enough that any analyst can anticipate and take appropriate corrective action in the event that a problem should arise.

13.7.3.3.11. **Quality Control Requirements** - This section should describe the components, concentrations, frequency, and acceptance criteria for the LCS or MS/MSD samples, as well as any other method specific QC requirements, such as tuning, blanks, or calibration requirements.

13.7.3.3.12. **Calculations** - All relevant calculations should be included, such as how instrument response relates to concentration, the calculation of response factors, etc.

13.7.3.3.13. **Method Performance** - The results of the initial method validation process should be included. The following information should be present:

- Statistically calculated MDLs (40 CFR Part 136 Appendix B),
- MDL spike levels, EEA's MRLs, Accuracy for each compound (mean recovery of each compound determined from analysis of a minimum of 4 replicates spiked at 1-4x MRL (TNI-EL-V1M4-2009-1.6.2.2.a) when method does not specify the spike level for the IDC study), precision data (RSD of the 4 replicates).

13.7.3.3.14. **References** - A list of method references, such as the relevant 500 or 600 series method, the SW-846 methods (including revision number and date), or Standard Methods should be provided.

13.7.3.3.15. **Deviations from Referenced Methodology** - A review of the referenced method is carefully made and EEA will specify any areas in which our method does not conform to referenced method requirements. If any such deviations are noted, an explanation as to what alternative was used and why is described. There are two basic types of method modifications: (1) those that are hardware related and (2) those that are policy or procedural modifications.

13.7.3.3.16. **Method Detection Limit** - Laboratory procedures of conducting MDL studies and a copy of the initial MDL study will be included.

13.7.3.3.17. **Demonstration of Capability** - Laboratory procedures of conducting DOC studies and a copy of the initial DOC study will be included.

13.7.3.3.18. **Definitions** - Definitions will be referred to the Quality Manual since the Quality Manual includes a glossary section that defines all the terms used by the laboratory.

13.7.3.3.19. **Pollution Prevention** - Potential threat of the standards and reagents to the environment is addressed in the SOP.

13.7.3.3.20. **Waste Management** - In addition to the hazardous waste protocol discussed in the SOP, the following references where the information can be find are also included:

- The Lab Hazwaste Management Plan
- The federal hazardous waste management regulations –Resources Conservation and Recovery Act (RCRA)-Title 40 of the Code of Federal Regulations, Parts 260 through 270 (40 CFR 260-270)
- CA Hazardous Waste Control Law (HWCL)-CCR Title 22 where 40 CFR was duplicated into CCR Title 22

13.7.3.3.21. **Revisions** - Revisions are discussed including the dates when revisions are made and the appropriate section numbers where the revisions could be found.

13.7.3.3.22. **Attachments** - A copy of the bench sheet used for the analysis and where applicable, an example chromatogram of the standards, calculations and any other relevant attachments.

Table 13-1 Laboratory Document Control

	Control No.
Instrument Sequence Log Books and Instrument Run Logs	1-200
Maintenance Log Books	201-400
QC Log Books (pH, Micro air monitoring, travel blank, etc.)	401-600
Reagent Prep Books	601-800
Sample Prep/Extraction Books	801-1000
Sample Data Records	1001-1200
Standard Log Books	1201-1400
SOP Books	1401-1600
Support Equipment Log Books (Balance, Pipette, Refrigerator, Incubator, Thermometer, etc)	1601-1800
MSC.	1801-2000
Certification Books	2001-2200
Health and Safety	2401-2600

Figure 13-1 Sample Worksheet

Author: Nina Dude

Date : 1/10/2012

Original Run Filename: OM_1-10-2012_10-18-36PM.OMN created 1/10/2012 10:18:36 PM
Original Run Author's Signature: [Nina Dude]
Current Run Filename: OM_1-10-2012_10-18-36PM.OMN last modified 1/10/2012 11:12:37 PM
Current Run Author's Signature: [Nina Dude]
Description: Default New Run

633883

Sample	Rep.	Cup No.	Channel 1 PHENOL Conc. (mg/L)	Area (Vs)	Detection Time	MDF	
CalStd0.2 ppm	1	S1	0.200	1.35	1/10/2012@10:19:30 PM		
CalStd 0.1 ppm	1	S2	0.100	0.677	1/10/2012@10:21:00 PM		
CalStd 0.05 ppm	1	S3	0.0500	0.345	1/10/2012@10:22:29 PM		
CalStd 0.01 ppm	1	S4	0.0100	0.0846	1/10/2012@10:23:58 PM		
CalStd 0.005 ppm	1	S5	0.00500	0.0387	1/10/2012@10:25:28 PM		
CalBlank	1	S6	0.00	0.00161	1/10/2012@10:26:58 PM		
DQM Test: Minimum Correlation Coefficient							
Result:			0.99995 > 0.99500				
Message			Calibration Passes				
Action			Continue				
ICV 0.2ppm	1	1	0.201	1.35	1/10/2012@10:29:36 PM		101.
Known Conc:			100				
DQM Test: > + Concentration Limit							
Result:			0.201 < 0.220				
Message			Pass				
Action			None				
DQM Test: < - Concentration Limit							
Result:			0.201 > 0.180				
Message			Pass				
Action			None				
Calibration:			Table/Fig. 1				
ICV 0.1ppm	1	2	0.101	0.683	1/10/2012@10:31:06 PM		101.
Known Conc:			0.100				
DQM Test: > + Concentration Limit							
Result:			0.101 < 0.110				
Message			Pass				
Action			None				
DQM Test: < - Concentration Limit							
Result:			0.101 > 0.0900				
Message			Pass				
Action			None				
ICB 0.0 ppm	1	6	-0.00123	2.46e-4	1/10/2012@10:32:35 PM		ND
Known Conc:			0.00				
DQM Test: > + Concentration Limit							
Result:			-0.00123 < 0.0100				
Message			ICB Passes				
Action			None				
MRL 0.005 ppm	1	60	0.00517	0.0431	1/10/2012@10:34:06 PM		103.
Known Conc:			100				
LFB 0.08 ppm	1	7	0.0792	0.538	1/10/2012@10:35:36 PM		99.
Known Conc:			0.0800				
DQM Test: > + Concentration Limit							
Result:			0.0792 < 0.0880				
Message			Pass				
Action			None				
DQM Test: < - Concentration Limit							
Result:			0.0792 > 0.0720				
Message			LFB Passes				
Action			Continue				
MBLANK	1	8	-3.10e-4	0.00642	1/10/2012@10:38:14 PM		ND
Known Conc:			0.00				
DQM Test: > + Concentration Limit							
Result:			-3.10e-4 < 0.0100				
Message			MBLANK Passes				
Action			Continue				
LCS 0.08 ppm	1	9	0.0791	0.538	1/10/2012@10:40:51 PM		99.
Known Conc:			0.0800				
DQM Test: > + Concentration Limit							
Result:			0.0791 < 0.0880				
Message			Pass				
Action			None				
DQM Test: < - Concentration Limit							

Figure 13-2 Example Notebook

Referenced Methods: EPA 525.2

Start Date: 1/31/12 Init:

Comp Date: 1/31/12 Init:

QC'd: Init:

Batch# : 636820

Matrix: Water

Reagent H2O: ***Ultrapure*** mfg/lot LAB

	Inj Vol (uL)	Exp. Date
Solutions:		
IS Soln.		
Surr Soln.		
MRL Spk Soln		
LCS Soln.		
MS Soln.		

Solvent: mfg/lot:

mfg/lot:

mfg/lot:

Salt: mfg/lot:

mfg/lot

Cartridge/Disk:

Preservatives added to QC or unpreserved samples as needed.

Room Temp: Chiller Temp:

Bath Temp: Heater Temp:

	Group	Sample #	Client Code	Sample Source	Test Code	Vi (mL)	Vf (mL)	pH	Cl- ppm	Ext#
1	387096	201201310310	MIKE	Monrovia Tap Water	@ML525					
		Q064162001		LCS1 --						
		Q064162002		LCS2 --						
		Q064162003		MBLK --						
		Q064162004		MRL_CHK --						

Batch Comments:

14.0 DATA REDUCTION, VALIDATION, AND REPORTING

The process of transforming raw analytical data into a finished report involves steps which are generally grouped into the categories of data reduction, data validation, and reporting. It involves mathematical modeling of the standard calibration curves, statistical analysis of the acquired data, calculations to account for preparation steps and dilution, verification of adherence to quality assurance procedures, and the generation of hardcopy output.

14.1. DATA REDUCTION

At EEA the analyst performing an analysis has the primary responsibility for reducing raw data. This process consists of converting raw data values into final, reportable values by comparing individual sample results to those obtained for calibration purposes and then accounting for any dilution or concentration procedures.

The extent to which raw data from the instrument needs to be mathematically processed varies depending on the analysis. For the following methods finished data is directly read from the instrument; pH, conductivity, spectrophotometric/colorimetric measurements (i.e.: Chemical Oxygen Demand (COD), Chromium VI, phenols, phosphorus, Methylene Blue Activated Substances (MBAS, or commonly known as surfactants), odor and presence/absence bacteriological tests. Other methods require mathematical calculations and in some cases, such as for pesticides by GC, qualitative assessment of actual presence.

Below is an outline of the data reduction techniques used by technology.

14.1.1. GC AND GC/MS

A data reduction software system is used to calculate target compound concentrations. These concentrations are calculated by multiplying the average response factor for the compound by the area count as determined by the instrument. Average response factors are determined through linear regression during initial calibration, and may only be used if the correlation criteria have been met. This assumes linearity of the calibration curve through the origin. If linearity is not established then a second order fit (logarithmic regression) may be used to determine response factors. Another alternative is to use single point calibration, which matches the area counts from a single calibration point to the area counts of the sample, upon which a sample concentration is determined. Single point calibration is rarely used.

In all cases data is reduced by the data reduction software. Programs for linear, logarithmic and single point calibrations are available on command. Sample dilution factors are entered into the data reduction software prior to analysis and calculated into the final result.

14.1.2. GC/MS

Reportable results are provided by the data reduction software for GC/MS analyses using linear average response factors, or 2nd order fits, as described, except for diluted samples. For diluted samples the result from the system is multiplied by the dilution factor. Reporting limits are adjusted manually as well.

All regressions and calibration calculations are performed by the system software.

14.1.3. METALS

ICP & ICPMS results are processed and transferred directly into the LIMS system. Dilution and calibration information is entered and processed by the ICP software prior to data transfer.

All other results are reportable directly off the system.

14.1.4. HPLC / IC / SPECTROPHOTOMETRIC / POTENTIOMETRIC

All results are reportable directly off the system software or directly read off instrument. The cell constant for the conductivity meter is 1. All samples and standards are allowed to come to room temperature prior to analysis. Temperature correction is not needed.

14.1.5. MICROBIOLOGY

The ability of an individual analyst to count colonies accurately shall be verified at least once per month, by having two or more analysts count colonies from the same plate on one positive sample. Counts must be within 10% difference to be acceptable [TNI-EL-V1M5-2009-1.7.3.2].

14.2. DATA VALIDATION

Upon completion of each analytical run, the analyst checks the raw data and QC to determine if the run is acceptable for submission. Data are entered into the LIMS either manually or by electronic data upload using files generated by data acquisition programs attached to instruments, which are then imported using the Data Capture Upload program (DCU) in STARLIMS. The data package is submitted to the Supervisor or Peer Analyst for review. The submitted package contains all relevant documentation such as chromatograms, instrument run logs, digestion logs, information about calibration or second source standards and reagents, other printed pages from the instruments, result summary sheets, and/or a checklist.
The Reviewer is responsible for verifying the validity of the data by determining if all quality control parameters have been analyzed and are within method acceptance limits, checking calculations, assessing the acceptability of chromatography, and addressing any inconsistencies in the data with the analyst. Deviations from the method should be

documented by the analyst and/or reviewer. The documentation should explain the deviation, any flags associated with the deviation and the acceptability of the data. The review includes a perusal of the supporting documentation to ensure that the documentation is present and is complete. Before the Reviewer or Validator approves the data in LIMS, data in the package is checked against the LIMS to ensure that there are no transcription errors, retests have been properly initiated, the appropriate flags have been added, and any comments regarding data/sample integrity have been added at the run/sample level.

When all results and calculations for a folder have been approved, an e-mail is sent to the Project Manager stating that the report has been released. The report components are merged and the entire package is reviewed by the project manager on-line. The electronic signature of the project manager is added to the cover page before the report is released to the client.

Any logbooks such as sample preparation, instrument maintenance, calibration, internal custody, and disposal are reviewed by the supervisor/manager of that group. Initials and date of review are written on the final page reviewed. The review will focus on completeness, accuracy, trends, opportunities for improvement, and compliance.

14.3. DATA REVIEW POLICY/CORRELATION OF RESULTS

All analytical data must be reviewed by a peer analyst qualified in that analysis or the group supervisor. Supervisors are ultimately responsible for the quality of reported results. Data review includes the following:

14.3.1. Checking all QC data against the QC criteria.

14.3.2. All the sample calculations must be checked. Samples which are spot checked must be marked by the reviewing analyst.

14.3.3. The analytical run sheet must be signed by the primary analyst and the reviewing peer analyst. Changes to records must be signed and initialed by responsible staff [TNI-EL-V1M2-2009-4.13.2.3][ISO/IEC 17025:2005(E)-4.13.2.3].

14.3.4. All secondary reviewers or Supervisors must check all data sheets. For inorganics and metals they must verify data entry for those samples by checking the database. The secondary reviewer or Supervisor must initial each run sheet they review. For organics, the secondary reviewer or Supervisor must cross check all reports for transcription error from bench sheets.

14.3.5. All Supervisors or designee must validate the data reported into the computer system. The data validation group then reviews and validates the final reports electronically. The reports are then printed and reviewed by the Project Manager.

14.3.6. As part of the annual internal audits, the Quality Manager or QA staff must spot check data sheets to insure that the peer reviews are being performed and that review process is traceable to the peer review.

14.3.7. Correlation of results for different characteristics of a sample (example Total Phosphate ≥ Orthophosphate or TKN ≥ NH3 [TNI-EL-V1M2-2009-5.9.1.e][ISO/IEC 17025:2005(E)-5.9.1.e]

14.4. DATA REPORTING

To meet the TNI report requirement, the laboratory provides the following information in the final test report:

14.4.1. A Title

14.4.2. Name/address of laboratory

14.4.3. Phone number and name of contact person

14.4.4. Unique identification of the certificate or report and unique identification of each page, and the total number of pages

14.4.5. Name and address of client, where appropriate and project name if applicable

14.4.6. Description and unambiguous identification of the tested sample including the client identification code

14.4.7. Identification of results derived from samples that did not meet TNI acceptance requirements such as improper container, holding time, or temperature [TNI-EL-V1M2-2009-5.10.3][ISO/IEC 17025:2005(E)-5.10.3].

14.4.8. Date of receipt of sample, date and time of sample collection, date(s) of performance test, and time of sample preparation and/or analysis if the required holding time for either activity is less than or equal to 72 hours [TNI-EL-V1M2-2009-5.10.2][ISO/IEC 17025:2005(E)-5.10.2].

14.4.9. Identification of the test method used, or unambiguous description of any non-standard method used.

14.4.10. Qualification of numerical results with “E1-E7” flags or appropriate data flags for values outside the working range.

14.4.11. Any deviations from, additions to or exclusions from the test method, and any other information relevant to a specific test, such as environmental conditions including the use of relevant data qualifiers and their meaning.

14.4.12. Measurement, examinations and derived results and identification of any failures (such as failed quality control). Radiochemistry results shall be reported with associated measurement uncertainty [TNI-EL-V1M6-2009-1.7.2.4.b].

14.4.13. Identification whether the data are calculated on dry weight or wet weight, reporting units and when required by method a statement of the estimated uncertainty of the test result.

14.4.14. Signature and title of the person(s) accepting responsibility for the content of the report and date of issue.

14.4.15. Clear identification of all data provided by outside sources (subcontracted laboratories, clients, Non-NELAP accredited work, etc.)

14.4.16. Clear indication of numerical results with values outside of quantitation limits. Test results provided by subcontracted laboratories are identified by subcontractor name or applicable accreditation number.

When the validation steps are completed, and the managers and supervisors have keyed in their initials in the appropriate LIMS field to reflect this, the report number is automatically transferred to an electronic listing in LIMS. Reports on this list are printed out daily. The reports are reviewed for correctness against the data in LIMS and signed off by the project manager prior to being copied for the files and delivery to the client. An example of an analysis report form is shown in Figure 14-1. A sample of a QC Report is shown in Figure 14-3. After the report is issued to the client, the laboratory reports remain unchanged. The report shall not be reproduced except in full, without the written approval of the laboratory [TNI-EL-V1M2-2009-5.10.2] [ISO/IEC 17025:2005(E)-5.10.2]. After issue of report, material amendments to the test report is done in the form of further document or data transfer including the statement "Supplement to test report, group number _____". For EEA revised report, cover page – report # xxxxxx'r'. Comment, report # xxxxxx'r' replaces the original test report. Also, amendments to the formal report must meet all the TNI reporting requirements. The laboratory notifies clients in writing of any event such as the identification of defective measurement or test equipment that casts doubt on the validity of results given in any test report or amendment to a report [TNI-EL-V1M2-2009-4.14.5]. The laboratory also ensures that the TNI reporting requirements are met for test results transmitted by telephone, telex, facsimile or other electronic or electromagnetic means and that all reasonable steps taken to preserve client confidentiality. Final laboratory report includes a statement in the cover page "Laboratory certifies that the test results meet all TNI requirements unless noted in the comments section or the Case Narrative".

If Client requires monthly reports of data that does not include all items listed in 14.4, the laboratory is still required to provide all information in standard TNI report format

required by the Client for use in preparing such regulatory reports [TNI-EL-V1M2-2009-5.10.1] [ISO/IEC 17025:2005(E)-5.10.1].

Copies of all client reports are filed electronically in a centralized server by year and client name. Scanned files are maintained for 5 years, except Massachusetts, Hawaii, and New York clients which are maintained for 10 years.

14.5. ELECTRONIC TRANSMISSION OF RESULTS

In the case of transmission of environmental test results by telephone, facsimile or other electronic means, the laboratory ensures preservation of Client confidentiality by attaching a cover page that includes the following statement:

> "This transmission and/or attachments contain information which is confidential and/or privileged. The information is intended for the addressee only. If you are not the intended recipient, any dissemination, distribution or copying of this communication is prohibited. If you have received this communication in error, please notify and return the original communication to the sender" [TNI-EL-V1M2-2009-5.10.5, 5.4.7.2.b] [ISO/IEC 17025:2005(E)-5.10.5, 5.4.7.2b].

14.6. GOOD AUTOMATED LABORATORY PRACTICES (GALP)

The laboratory assures that all requirements of the TNI and ISO 17025 standards are complied with where computers or automated equipment are used for the capture, processing, manipulation, recording, reporting, storage or retrieval of test data.

Section 8.1 through 8.11 of the EPA document 2185 – GALP is adopted by the laboratory for its computer use even though GALP is not required by TNI standard requirements. The laboratory ensures that the computer software is adequate for use and documented. To protect the integrity of data entry or capture, data storage, data transmission and data processing, the laboratory establishes and implements procedures in compliance to good automated laboratory practices. In addition, appropriate procedures are established for computer and automated equipment to ensure proper functioning and are provided with the environmental and operating conditions necessary to maintain the integrity of calibration and test data. Also the laboratory establishes and implements appropriate procedures for the maintenance of security of data including the prevention of unauthorized access to and the unauthorized amendment of computer records. The laboratory LIMS system provides several levels of security. The first level is the entry of a password to initially log on to the computer, then the person must be designated as a qualified user of STARLIMS. Additionally, the department to which a person is assigned governs accesses to the various functions of the system. The system also provides for read – only access to results to further protect the data from unauthorized modification or deletion. See laboratory GALP SOP for the Implementation of Good Automated Laboratory Practices. Implementation of the GALP includes data point comparison and manual calculations to test LIMS accuracy to be done during the

data package review by the Quality Assurance Unit (QAU) (QM section 16.1.2). LIMS Audit Report form will be completed to document results of the LIMS audit. The laboratory QA group will ensure that all corrective actions are done when deficiencies are observed.

14.7. STATE SPECIFIC REPORTING REQUIREMENTS

Massachusetts – All analytes that are reported to the state of Massachusetts must have a means to distinguish between the analytes for which EEA is certified, and the analytes for which EEA is not certified. To meet this requirement, EEA only sends final reports to the Massachusetts Department of Environmental Protection the analytes for which EEA is certified in Massachusetts. Please see section 3.9 for more information about the certifications that EEA holds.

14.8. MCL NOTIFICATIONS

At approximately 10pm each night the Laboratory's STARLIMS software runs a report called a "Hits Report". This report is sent to all Project Managers. Part of the information in this report is a notification of any hits that exceed MCL levels for all client samples analyzed and approved since the last Hits Report. The Project Manager is responsible for notifying their clients of any exceedance within 24 hours of obtaining valid data. Since the report is sent to all Project Managers, absent PMs will have their client's data checked by their backups in order to ensure that no notifications are missed.

Please see Section 3.10.9 of this QM for the requirements on notification for subcontracting clients.

Figure 14-1 Example Analysis Report Form

eurofins | Eaton Analytical
formerly MWH Laboratories

750 Royal Oaks Drive, Suite 100
Monrovia, California 91016-3629
Tel: (626) 386-1100
Fax: (626) 386-1101
1 800 566 LABS (1 800 566 5227)

Laboratory Report

for

MWH Laboratories
750 Royal Oaks Drive
Suite 100
Monrovia, CA 91106
Attention: Andora Effendi
Fax: (626) 386-1139

Date of Issue
08/21/2012
EUROFINS EATON
ANALYTICAL

NELAP ACCREDITED TNI LABORATORY
01114CA

Report: 405936
Project: WS
Group: WS PT

ABC: Alma.B.Capati
Project Manager

Laboratory certifies that the test results meet all **TNI NELAP** requirements unless noted in the Comments section or the Case Narrative. Following the cover page are Hits Reports, Comments, QC Summary, QC Report and Regulatory Forms. This report shall not be reproduced except in full, without the written approval of the laboratory.

Page 1 of 9 pages

eurofins | Eaton Analytical
formerly MWH Laboratories

STATE CERTIFICATION LIST

State	Certification Number	State	Certification Number
Alabama	41060	Mississippi	Certified
Alaska	CA00006	Montana	Cert 0035
Arizona	AZ0778	Nevada	CA00006-2012-1
Arkansas	Certified	New Hampshire	2959-11
California – NELAP	01114CA	New Jersey	CA 006
California – ELAP	1422	New Mexico	Certified
Colorado	Certified	New York	11320
Connecticut	PH-0107	North Carolina	06701
Delaware	CA 006	North Dakota	R-009
Florida	E871024	Oregon	CA 200003-010
Georgia	947	Pennsylvania	68-565
Guam	11-004r	Rhode Island	01114CA
Hawaii	Certified	South Carolina	87016001
Idaho	Certified	South Dakota	Certified
Illinois	200003	Tennessee	TN02839
Indiana	C-CA-01	Texas	T104704230-11-2
Kansas	E-10268	Utah	Mont-1
Kentucky	90107	Vermont	VT0114
Louisiana	LA110022	Virginia	00210
Maine	CA0006	Washington	C383
Maryland	224	West Virginia	9943 C
Commonwealth of Northern Marianas Is.	MP0004	Wisconsin	998310660
Massachusetts	M-CA006	Wyoming	8TMS-L
Michigan	9906	EPA Region 5	Certified

eurofins | Eaton Analytical
formerly MWH Laboratories

750 Royal Oaks Drive, Suite 100
Monrovia, California 91016-3629
Tel: (626) 386-1100
Fax: (626) 386-1101
1 800 566 LABS (1 800 566 5227)

Laboratory Hits
Report: 406838

MWH Laboratories
Andora Effendi
750 Royal Oaks Drive
Suite 100
Monrovia, CA 91106

Samples Received on:
08/17/2012

Analyzed	Analyte	Sample ID	Result	Federal MCL	Units	MRL
		201208170474	W9 Inorganics			
08/18/2012 19:32	Nitrate + Nitrite as N by RFA		4.2	10	mg/L	0.15
08/18/2012 17:01	Nitrate as N by RFA		4.2		mg/L	0.15

SUMMARY OF POSITIVE DATA ONLY

eurofins | Eaton Analytical
Formerly MWH Laboratories

750 Royal Oaks Drive, Suite 100
Monrovia, California 91016-3629
Tel: (626) 386-1100
Fax: (626) 386-1101
1 800 566 LABS (1 800 566 5227)

Laboratory Data
Report: 406838

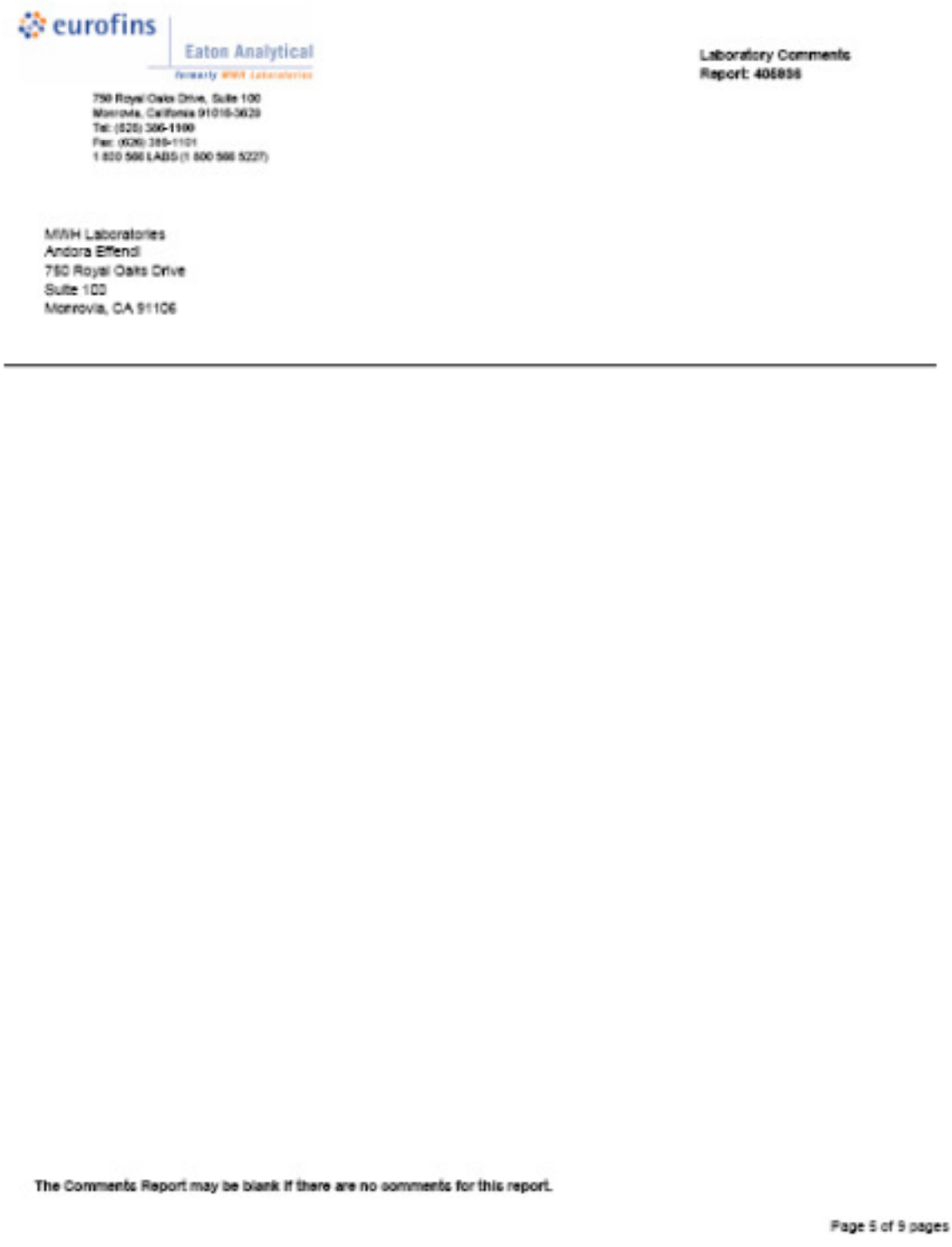
MWH Laboratories
Andora Effendi
750 Royal Oaks Drive
Suite 100
Monrovia, CA 91106

Samples Received on:
08/17/2012

Prepared	Analyzed	QC Ref #	Method	Analyte	Result	Units	MRL	Dilution
WS Inorganics (2012081704 74)						Sampled on 08/17/2012 0000		
Sample Point ID: ERA WS 192 PT_Inorganics								
EPA 353.2 - Nitrate as N by RFA (calc)								
08/19/2012	17:01	667929	(EPA 353.2)	Nitrate as N by RFA	4.2	mg/L	0.15	5
EPA 353.2 - Nitrate + Nitrite as N by RFA								
08/19/2012	19:32	667256	(EPA 353.2)	Nitrate + Nitrite as N by RFA	4.2	mg/L	0.15	5

Rounding on totals after summation.
(C) - indicates calculated results

Figure 14-2 Example Analysis Report Form (Report Comment)



eurofins | Eaton Analytical
formerly MWH Laboratories

750 Royal Oaks Drive, Suite 100
Monrovia, California 91016-3629
Tel: (626) 386-1100
Fax: (626) 386-1101
1 800 566 LABS (1 800 566 5227)

Laboratory Comments
Report: 406856

MWH Laboratories
Andora Effendi
750 Royal Oaks Drive
Suite 100
Monrovia, CA 91106

The Comments Report may be blank if there are no comments for this report.

Page 5 of 9 pages

Figure 14-3 Example QC Report Form

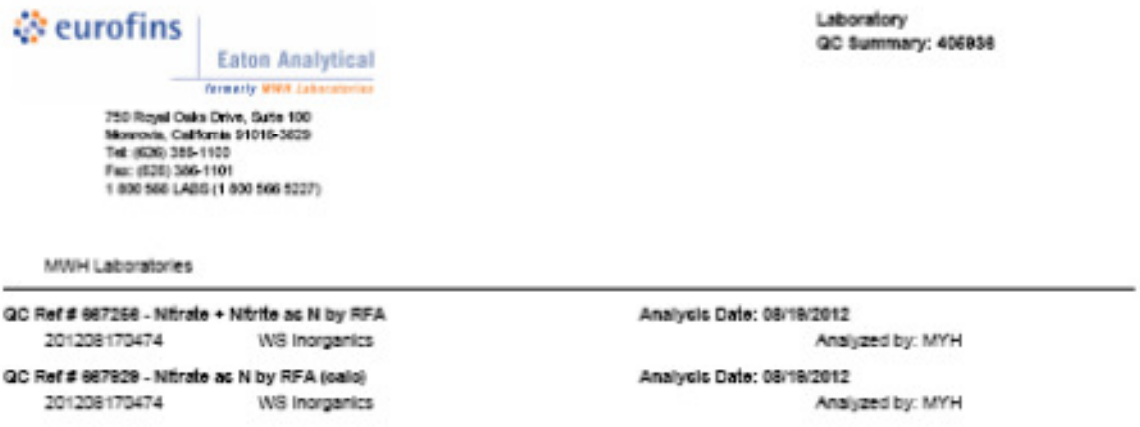
Laboratory QC
Report: 405936

MWH Laboratories

QC Type	Analyte	Native	Spiked	Recovered	Units	Yield (%)	Limits (%)	RPD/Limit (%)	RPD%
QC Ref# 667268 - Nitrate + Nitrite as N by RFA by EPA 353.2						**Analysis Date: 08/19/2012**			
LCS1	Nitrate + Nitrite as N by RFA		1.0	1.06	mg/L	106	(90-110)		
LCS2	Nitrate + Nitrite as N by RFA		1.0	1.05	mg/L	105	(90-110)	20	0.95
MBLK	Nitrate + Nitrite as N by RFA			<0.03	mg/L				
MRL_CHK	Nitrate + Nitrite as N by RFA		0.05	0.0265	mg/L	53	(50-150)		
MS_20120820001G	Nitrate + Nitrite as N by RFA	7.0	1.0	17.1	mg/L	102	(90-110)		
MS_20120820002G	Nitrate + Nitrite as N by RFA	2.1	1.0	13.2	mg/L	110	(90-110)		
MSD_20120820001G	Nitrate + Nitrite as N by RFA	7.0	1.0	14.5	mg/L	75	(90-110)	20	17
MSD_20120820002G	Nitrate + Nitrite as N by RFA	2.1	1.0	12.8	mg/L	107	(90-110)	20	3.1
QC Ref# 667929 - Nitrate as N by RFA (calc) by EPA 353.2						**Analysis Date: 08/19/2012**			
LCS1	Nitrate as N by RFA		1.0	1.06	mg/L	106	(90-110)		
LCS2	Nitrate as N by RFA		1.0	1.05	mg/L	105	(90-110)	20	0.95
MBLK	Nitrate as N by RFA			<0.05	mg/L				
MRL_CHK	Nitrate as N by RFA		0.05	0.0265	mg/L	53	(50-150)		
MS_20120820001S	Nitrate as N by RFA	2.1	1.0	13.2	mg/L	110	(90-110)		
MSD_20120820001S	Nitrate as N by RFA	2.1	1.0	12.8	mg/L	107	(90-110)	20	3.1

Spike recovery is already corrected for native results.
Spikes with exceed Limits and blank detections with positive results are highlighted by Underline.
Criteria for MS and Dup are advisory only, batch control is based on LCS. Criteria for duplicates are advisory only, unless otherwise specified in the method.
RPD not calculated for LCS2 when different exceedance than LCS1 is used.
RPD not calculated for Duplicates when the result is not five times the MRL (Reliable Reporting Level).
(S) - Indicates surrogate compound.
(I) - Indicates internal standard compound.

Figure 14-4 Example QC Report Form (QC Summary)



eurofins | Eaton Analytical
formerly MWH Laboratories

750 Royal Oaks Drive, Suite 100
Monrovia, California 91016-3629
Tel: (626) 386-1100
Fax: (626) 386-1101
1 800 566 LABS (1 800 566 5227)

Laboratory
QC Summary: 406936

MWH Laboratories

QC Ref # 667268 - Nitrate + Nitrite as N by RFA
20120817.0474 WS Inorganics

Analysis Date: 08/18/2012
Analyzed by: MYH

QC Ref # 667829 - Nitrate as N by RFA (calc)
20120817.0474 WS Inorganics

Analysis Date: 08/18/2012
Analyzed by: MYH

15.0 CONTROL OF NON-CONFORMING WORK, CORRECTIVE ACTION, AND PREVENTIVE MEASURES

Corrective actions may be required when there is a failure to meet quality control acceptance criteria, or when internal or external audit samples are not acceptable. Quality control measures for which control limits are established and maintained include: LCS, duplicates, method blanks, surrogate recoveries, MS/MSD, MRLs, calibrations, continuing calibrations and sensitivity checks. Refer to the Nonmethod 28 SOP for Non-conforming work procedures.

15.1. CORRECTIVE ACTION PROCEDURES, BY METHOD

Specific corrective actions on a method-by-method basis can be found in the Table 15-1. This SOP lists the processes and flags used to qualify data for submittal to clients. Corrective action will be initiated as a result of findings from internal or external audits, not acceptable results from performance samples, large variation from split samples and inadequate quality as determined by data validation review.

15.2. CORRECTIVE ACTION PROCEDURES, ROOT CAUSE, PREVENTIVE MEASURES, DATA QUALIFIERS, AND REPORT COMMENTS

15.2.1. Selection and Implementation of Corrective Actions

Failure to meet criteria of the LCS, surrogate spikes, internal standards, continuing calibration standards, holding time exceedance, improperly preserved samples, method blank contamination are QC failures that trigger the generation corrective actions to identify the root cause of the problem. Root causes of the problem are documented in the Quality Investigation Report (QIR).

For instance, when a matrix spike failure occurs during trace metals analysis, the analyst first checks the %RSD for the multiple measurements to see if the %RSD is less than 20%. Then the calibration verification will be checked along the calibration blank, preparation blank, and the second source LCS standard recovery. The standards and reagents preparation and expiration dates are reviewed. Spiking solutions are verified to ensure that here are no errors made in calculations and in spiking. If the MS/MSD recoveries are outside the internal QC limits and all the associated QCs for the batch are acceptable, the RPD for MS/MSD recoveries should be checked. If the RPD is found to be within the 20% criteria, the unacceptable recoveries are annotated in the report as suspect due to matrix effect. If the concentration of the background is much higher than the spiking amount the report will be annotated also. If the RPD is outside the limits, the sample that was spiked is checked visually to see if the sample is homogenous, if the sample is homogenous the batch will be reanalyzed.

15.2.2. Documentation of Corrective Actions

15.2.2.1. All corrective action taken for all QC failures is documented by generating a Quality Investigation Report (QIR). All other corrective action taken is documented on a Corrective Action Report (CAR). See Figure 15-2 for an example QIR.

Additional information is documented about the QC failures in the bench by the analyst.

15.2.2.2. Results are flagged not only for quality control failures where QIRs have been generated but also for all other QC failures that have impact on the data quality of the result. All results are flagged if data is suspect or QC was not acceptable.

15.2.2.3. Data qualifiers are used by the laboratory in reporting analytical results to flag the user about the data. Some of the qualifiers below were requested by a specific client as required in the Project's Quality Assurance Plan to ensure that the Data Quality Objectives of the project are met.

15.2.2.4. Comments on the results are provided to the clients on the final report for QC nonconformance. In addition, any QC data exceeding QC acceptance criteria are underlined to flag the user about the QC failure and its impact to the data quality of the associated samples in the batch.

15.2.2.5. Depending on the significance of nonconformance, the Client is notified by the Project Manager and work recalled if necessary [TNI-EL-V1M2-2009-4.9.1.d] [ISO/IEC 17025:2005(E)-4.9.1.d]. The Client is notified immediately for possible re-sampling.

15.2.2.6. Where the identification of nonconformance or departure casts doubts on the laboratory's compliance with its own policies and procedures, or on its compliance with this Standard, the laboratory shall ensure that the appropriate areas of activity are audited [TNI-EL-V1M2-2009-4.11.5][ISO/IEC 17025:2005(E)-4.11.5].

15.2.3. **Monitoring of Corrective Action**

15.2.3.1. Corrective actions implemented are monitored if corrective actions are effective to remove problem [TNI-EL-V1M2-2009-4.11.4][ISO/IEC 17025:2005(E)-4.11.4].

15.2.3.2. QA monitors CARs and QIRs for trends and notifies the analyst and supervisor of the need to correct the problem and implement corrective action to prevent the problem from reoccurring.

15.2.4. **Preventive Measures**

15.2.4.1. QIRs require the analyst to document preventive measures to ensure that the problems will not re-occur [TNI-EL-V1M2-2009-4.12.1][ISO/IEC 17025:2005(E)-4.12.1].

15.2.4.2. Preventive action, rather than corrective action, aims at minimizing or eliminating inferior data quality or other non-conformance through scheduled maintenance and review, before the non-conformance occurs. Preventive action is generated when circumstances are identified where a quality failure or non-conformance is a possibility or where an opportunity is identified to strengthen the quality system.

15.2.4.3. Preventive action includes, but is not limited to, review of QC data to identify quality trends, regularly scheduled staff quality meetings, annual managerial reviews, running a new LIMS in tandem with the old system to assure at least one working system, and other actions taken to prevent problems.

15.2.4.4. All staff recommends preventive action procedures to the Quality Manager and management is responsible for implementing preventive action.

15.2.4.5. A preventive action record form is generated when an opportunity for improvement arises to record preventive action, record actions generated, and ensure actions are effective. See Figure 15-2 for the Preventive Action Form which is used to document actions required for improvement, name of requestor, responsible personnel to carry out implementation, and estimated completion date.

15.3. ESTABLISHING WARNING/ACTION LIMITS

The incorporation of quality control samples and reference materials into the laboratory quality control program is of little use in maintaining overall analytical quality control unless the laboratory has established acceptance criteria for these samples. Quality control samples falling outside of these criteria serve as flags to signal the production of unacceptable data which must be rerun or reported as suspect data if re-running is not an option due to expired holding times or lack of sample volume.

15.3.1. Approach to Setting Limits

For methods that do not specify acceptance limits, the lab establishes limits through control charts. These limits should be updated once a year. These limits are based upon historical recoveries of LCS samples associated with specific matrices (or where LCS samples are not utilized, they are based on spike recoveries or duplicate limits for matrix specific samples).

For those cases where insufficient historical information exists to set statistically meaningful LCS or matrix specific limits, EEA has set limits based on the expected performance of the analysis until historical limits can be calculated. These limits are then associated with specific control requirements to determine out of control events.

15.3.2. Documentation of Limits

15.3.2.1. Reagent Blanks - Reagent blank values must remain lower than the reported MRL (some methods require ½ or 1/3 MRL) for each analytical procedure. If an analyst notices an increase in the reagent blank which is beginning to approach this limit, the source of contamination must be investigated before further analyses are performed.

15.3.2.2. External Reference Samples - Recoveries on external reference samples must fall within the acceptance limits provided with the true values.

15.3.2.3. Internal and Surrogate Standards - As specified by the methods, internal standards are run with each of the calibration standards and the area counts are recorded on the same form as the response factors. Any standard that has an internal standard area count beyond ±50% of the average internal standard area count for all initial calibration standards must be rerun to meet these criteria. Any sample with an internal standard count beyond ±50% (or as stated in the particular SOP) of the average internal standard counts for the ICAL standards must be rerun. Surrogate standards must meet the recovery limits specified in the analytical method or established historical limits, which are updated periodically.

15.3.2.4. Blind Check Samples - The results of blind check sample analyses must fall within the acceptance criteria provided with the samples.

15.3.3. LCS Control Limits

EEA uses method acceptance limits for LCS limits in water matrix to assess analytical control. All analysts have received a copy of these acceptance limits and must ensure that their LCS sample results fall within the stated acceptable ranges. If specific control limits have not been provided for matrix spikes or duplicates, LCS criteria are used until sufficient data is generated to calculate historical limits for the MS/MSD samples for a particular matrix. Any samples associated with unacceptable LCS samples must be re-run unless other criteria are available to allow acceptance of the data without qualification. If a sample cannot be rerun due to exceeded holding times or lack of sufficient sample volume or weight, then the data must be qualified as estimated when reported to the client.

15.4. CONTROL CHARTS

EEA collects LCS and MS/MSD data in the LIMS computer system for generation of control chart data and limits. Data can be downloaded and plotted on charts to determine trends, which may indicate problems with the analysis, or out of control events.

EEA utilizes a Shewhart mean chart modified to percent recovery to monitor laboratory control sample bias. This procedure is referenced in the EPA Handbook for Analytical

Quality Control in Water and Wastewater Laboratories (EPA-600/4-79-019), March 1979, on pages 6-2 to 6-6. Precision is monitored with control charts, but is compared to absolute limits established by the lab based on method specified limits.

Control charts for LCS and MS data are generated with the LIMS software periodically based on a maximum of 30 data points. The control chart limits are re-calculated at least annually. If analysis parameters are changed significantly or method modifications are performed, control chart limits may be re-calculated more frequently. QA reviews the limits and charts to determine whether any of the data is out-of-control. If the control charts indicate an out-of-control event, appropriate corrective action is immediately taken to bring the analysis back into control. An example of the Shewhart percentage recovery control chart is presented in Figure 15-4.

15.5. PROCEDURES FOR DETERMINING AND REPORTING OUT-OF-CONTROL ANALYSES

15.5.1. Defining an Out-of-Control Analysis

An analysis is out-of-control whenever a quality control sample or parameter falls outside of acceptance limits. Quality control parameters are evaluated for their acceptability on a daily basis according to established acceptance limits and are also monitored with control charts to detect trends in variability, which are indicative of a shift in the methodology due to analytical error.

15.5.1.1. Criteria Used

15.5.1.1.1. Daily Quality Controls

The quality control parameters utilized by EEA were detailed in Section 11.1. All of these controls are evaluated on a daily basis and must pass the criteria detailed in this section. Each analyst is familiar with the criteria for his/her analyses and is responsible for insuring that all quality control parameters on the analytical run are acceptable. An analyst cannot enter his/her data into the laboratory computer until the data is reviewed and approved by an appropriately trained peer or supervisor. In addition, LCS and MS/MSD data are also entered into the computer and linked to specific batches.

LCS and MS/MSD results must fall within given acceptance limits. These limits are provided for water matrix. Reagent blanks must remain below the MRL established for each parameter. External reference samples must fall within the acceptance criteria provided with the true values. Internal and surrogate standards must meet the recoveries specified in the analytical procedure, if historical control chart based information is not available. A new working standard must be checked against the old reference standard to verify its accuracy and must fall within 10% of

its true value. If this agreement is not met, a referee standard must be run. All standards must be traceable to primary standards.

Instrument calibrations must fall within acceptance criteria in order for runs to proceed. Table 11-4 summarizes the instrument's initial calibration acceptance criteria for each analysis.

In addition to monitoring daily QC parameters for acceptability, control charts are utilized and interpreted as described in Section 15.4.

15.5.1.1.2. Approaches to Control Chart Interpretation

The control charts generated by the LIMS System flags the analyst that there is a potential problem whenever seven or more consecutive points fall above or below the mean.

If the above situation is observed, the cause of the shift in mean or increased variability must be investigated, corrected, and documented prior to analyzing any more samples.

15.5.2. Responding to an Out-of-Control Event

It is important to have an operational system within EEA for recognizing out-of-control events as soon as they occur so the appropriate action can be taken to bring the analysis back into control. This will insure that no data gets reported from a period when the analysis was out-of-control.

15.5.2.1. Roles and Responsibilities

The analyst has primary responsibility for verifying that all daily QC parameters fall within the acceptance limits before submitting the data for review. Review at the analyst level enables most errors to be caught immediately and prevents reporting delays. Following the analyst's verification, the data is reviewed by an appropriately trained peer analyst or supervisor. All of the quality control parameters are reviewed for compliance with the acceptance criteria and the calculations on the raw data forms are checked for errors in data manipulation. If the reviewer notices a problem, the analyst is notified immediately and corrective action is taken. All samples associated with unacceptable quality control samples are rerun unless there is insufficient sample, in which case the client is notified by the Client Services group [TNI-EL-V1M2-2009-4.9.1][ISO/IEC 17025:2005(E)-4.9.1]. Every out of control event must be documented by filing a Quality Investigation Report (QIR). See Figures 15-2 and Figure 15-3.

The check of daily QC parameters indicates immediate problems with the data, but trends are only evident on the control charts. Both the analyst and the Group

Supervisor are responsible for reviewing either the data or control charts for pattern to see if any of the out-of-control events summarized in Section 15.5.1 have occurred. If so, the analyst must initiate corrective action before continuing with the analysis.

15.5.2.2. Defining Suspect Samples

Sample data is considered suspect if associated with unacceptable MS/MSD and LCS samples or part of an analytical run that had an unacceptable calibration or an external reference sample was out of an expected range. GC/MS data is considered suspect if the internal or surrogate standards were not recovered within the acceptable range. Sample data is also considered suspect if the reagent blank has substantially increased beyond normal range and exceeds any of the compound MRL's.

15.5.2.3. Ensuring that Suspect Data Are Not Reported

It is the ultimate responsibility of the Group Leader to ensure that suspect data are not reported. The laboratory procedures currently require that analysts may not enter their final data into the computer until their analytical data form and accompanying QC parameters have been reviewed and approved by an appropriately trained peer or supervisor. The QA Group performs periodic system audits to ensure that this procedure is working properly and prepares reports to lab management based on these audits.

15.5.2.4. Corrective Action

15.5.2.4.1. If the calibration fails, the analyst must determine whether the problem lies with the standard, the reagents, or an instrument malfunctions. This is usually determined by reviewing all of the calibration QC parameters and determining which specific parameters do not meet the criteria. For example: 1) the regression statistics and recalculated standards look fine, 2) there was little drift during the run, 3) the peaks appear satisfactory, 4) the reagent blank is low, but 5) the external reference sample was out of range, it is likely that the problem lies with the integrity of the standard used to make up the working standards and a new stock standard should be prepared.

15.5.2.4.2. If calibration appears acceptable but some of the duplicate and spiked samples are unacceptable, the analyst must determine whether there is a matrix problem interfering with the analysis or the preparatory digestion. If all of the unacceptable duplicates and spikes occur on a specific type of matrix, this is good evidence that there is a matrix interference problem. When a preparatory digestion is part of the procedure, the problem can be isolated to the digestion or the instrumental analysis by comparing the LCS, which was carried through the digestion to a LCS sample analyzed without digestion. If a matrix problem is indicated, the analyst must determine the most appropriate procedure for alleviating the interference such as

diluting the sample, using standard additions, performing the analysis at a different wavelength, using a different GC column, or modifying the digestion procedure.

15.5.2.4.3. If an unacceptable result is obtained on a blind check sample, the problem must be isolated. To maintain the blind nature of the samples, the run containing the blind check sample is reviewed by the QA Group to determine if any of the quality control parameters were unacceptable or if the sample was run outside the optimum range of the calibration. If no apparent cause of error is found, a second check sample is submitted to determine whether the error occurred during preparation of the blind check sample.

15.5.2.4.4. If an out-of-control event is indicated by a shift or trend on a control chart, the following diagnostic strategy will be applied:

15.5.2.4.4.1. A shift in the mean of the percentage recovery chart could be caused by incorrect preparation of a standard or a reagent, contamination of the sample, incorrect instrument calibration, instrument component deterioration analyst error, dirty pipettes preventing proper drainage, or other preparatory steps.

15.5.2.4.4.2. A trend of the mean upward could be caused by deterioration of the standard or the reagents or a change in the extraction efficiency

15.5.2.4.4.3. A trend of the mean downward could be caused by concentration of the standard due to evaporation, deterioration of reagents, and a change in the extraction efficiency or instrument component failure

15.5.2.4.4.4. Increased variability could be caused by switching to a different analyst, deviation from the procedure, variable extraction efficiencies

15.5.2.4.4.5. A shift in the mean or increased variability can sometimes be caused by a sample load of an unusual matrix. If this is determined to be the cause of the problem, the analysis will not be considered out-of-control but the situation will be documented.

Figure 15-1 Data Qualifiers

10/24/2012

Data Qualifiers

Revised on 10/24/12 to add Microbiology flags.
Revised on 2/16/10, Based on AZ Data Flag 9/20/07 Rev.3.0 and Attachment A, "Guidance on the Usage of Data Qualifiers"

MWH List

Microbiology:

A1 = Too numerous to count.
A2 = Sample incubation period exceeded method requirement.
A3 = Sample incubation period was shorter than method requirement.
A4 = Target organism detected in associated method blank.
A5 = Incubator/water bath temperature was outside method requirements.
A6 = Target organism not detected in associated positive control.
A7 = Micro sample received without adequate headspace.
A8 = Plate count was outside the method's reporting range. Reported value is estimated.
AA = Insufficient sample received. Sample volume below 100 ml. Data not acceptable for compliance.
AB = Presumptive phase was cloudy without gas in the tube and transferred to confirmed phase. Data not acceptable for compliance.
AC = Sample volume over 100 ml. Excess sample was pipetted out. Data acceptable for compliance.

Method/ calibration blank:

Apply appropriate qualifier to affected analyte in the blank if target analyte is not detected at > RL in the samples. If analytes are detected, then all corresponding analytes for the associated samples should also be qualified.

B1 = Target analyte detected in method blank at or above the method reporting limit.
B2 = Non-target analyte detected in method blank and sample, producing interference.
B3 = Target analyte detected in calibration blank at or above the method reporting limit.
B4 = Target analyte detected in blank at or above method acceptance criteria.
B5 = Target analyte detected in method blank at or above the method reporting limit, but below trigger level or MCL.
B6 = Target analyte detected in calibration blank at or above the method reporting limit, but below trigger level or MCL.
B7 = Target analyte detected in method blank at or above method reporting limit. Concentration found in the sample was 10 times above the concentration found in the method blank. (No TNI Ref, Method Criteria)

BA = Target analyte detected in method blank at or above the laboratory minimum reporting limits (MRL), but analyte not present in the sample. (TNI QM Template 27.2.2.1)

10/24/2012

BE= Target analyte detected in method blank is at or above the method acceptance limits, but below the method reporting limit (MRL).
BF= Target analyte detected in method blank is at or above the method acceptance limits, but below the method reporting limit (MRL) and analyte not present in the sample.
BG = Target analyte detected in method blank (MB) is at or above the method acceptance limits, but below the method reporting limit (MRL). Sample concentration was 10 times above the concentration found in MB. (TNI – M4 #570)

Confirmation:

For methods that require qualitative confirmation. C3 applies to methods that require quantitative confirmation.

C1 = Confirmatory analysis not performed as required by the method.
C3 = Qualitative confirmation performed.
C4 = Confirmatory analysis was past holding time.
C5 = Confirmatory analysis was past holding time. Original result not confirmed.
C8 = Sample RPD between the primary and confirmatory analysis exceeded 40%. Per EPA Method 8000C, the lower value was reported as there was no evidence of chromatographic problems.

Dilution:

If all analytes are reported from the diluted sample, apply qualifier to the entire sample. Otherwise apply qualifier to each analyte that required dilution.

D1 = Sample required dilution due to matrix.
D2 = Sample required dilution due to high concentration of target analyte.
D4 = Minimum Reporting Limit (MRL) adjusted to reflect sample amount received and analyzed.
D5 = Minimum Reporting Limit (MRL) adjusted due to sample dilution; analyte was non-detect in the sample.
D6 = Minimum Reporting Limit (MRL) adjusted due to an automatic 10X dilution performed on this sample for the purpose of reporting traditional drinking water analytes for wastewater requirements.
DA = Sample dilution required due to insufficient sample.

Estimated concentration:

Appropriate qualifier must be used for any analyte result reported outside the calibration range. Affects data reported outside the calibration range or down to the MDL. E8 is only required if additional clarification is necessary.

E1 = Concentration estimated. Analyte exceeded calibration range. Reanalysis not possible due to insufficient sample.
E2 = Concentration estimated. Analyte exceeded calibration range. Reanalysis not performed due to sample matrix.

10/24/2012

E3 = Concentration estimated. Analyte exceeded calibration range. Reanalysis not performed due to holding time requirements.
E4 = Concentration estimated. Analyte was detected below laboratory minimum reporting limit (MRL).
E5 = Concentration estimated. Analyte was detected below laboratory minimum reporting limit (MRL), but not confirmed by alternate analysis.
E6 = Concentration estimated. Internal standard recoveries did not meet method acceptance criteria.
E7 = Concentration estimated. Internal standard recoveries did not meet laboratory acceptance criteria.
E8 = Analyte reported to MDL per project specification. Target analyte was not detected in the sample.
EA = Concentration estimated. Analyte was detected below laboratory minimum reporting limits but above laboratory method detection limits.
EB = Result estimated. Analyte exceeded the highest calibration standard as required by the EPA/SM method
ED = Result estimated. Analyte was detected outside of calibration range as specified by the EPA/SM method.

Hold time:

Qualify samples appropriately when method extraction and/ or analysis holding time have been exceeded.

H1 = Sample analysis performed past holding time. Data not acceptable for regulatory compliance
H2 = Initial analysis within holding time. Reanalysis for the required dilution was past holding time.
H3 = Sample was received and analyzed past holding time. Data not acceptable for regulatory compliance.
H4 = Sample was extracted past required extraction holding time, but analyzed within analysis holding time.
H5 = This test is specified to be performed in the field within 15 minutes of sampling; sample was received and analyzed past the regulatory holding time.
HA= Initial analysis within holding time. Reanalysis was past holding time.
HB = Sample was received within holding time, but holding time exceeded by the Lab. Sample was not analyzed.
HC = Sample was received past holding time and not analyzed.
HD = Sample was extracted past required extraction holding time, but analyzed within analysis holding time. Data not acceptable for regulatory compliance.

BOD/DBOD:

Qualifiers K4, K5, K6 & K8 indicate situations that may impact all results in an analytical run and should be used to qualify all affected samples as well as any affected quality control samples

10/24/2012

when reported. K3 was deleted because if the seed depletion was out, then the situation must be explained in the case narrative.

K1 = The sample dilutions set-up for the BOD/CBOD analysis did not meet the oxygen depletion criteria of at least 2 mg/L. Any reported result is an estimated value.
K2 = The sample dilutions set up for the BOD/CBOD analysis did not meet the criteria of a residual dissolved oxygen of at least 1 mg/L. Any reported result is an estimated value.
K5 = The dilution water D.O. depletion was > 0.2 mg/L.
K6 = Glucose/glutamic acid BOD/CBOD was below method acceptance criteria.
K7 = A discrepancy between the BOD and COD results has been verified by reanalysis of the sample for COD.
K8= Glucose/glutamic acid BOD/CBOD was above method acceptance levels.
KA = The seed depletion was outside the method and laboratory acceptance limits. The reported result is an estimated value.

Laboratory fortified blank/blank spike:

Appropriate qualifier must be applied to the affected analytes in the Laboratory fortified blank/blank spike and to all corresponding analytes in the associated samples.

L1 = The associated blank spike recovery was above laboratory acceptance limits.
L2 = The associated blank spike recovery was below laboratory acceptance limits.
L3 = The associated blank spike recovery was above method acceptance limits.
L4 = The associated blank spike recovery was below method acceptance limits.
LA = The associated blank spike recovery was above laboratory acceptance limits. Analyte is only qualitatively identified.
LB = The associated blank spike recovery was below laboratory acceptance limits. Analyte is only qualitatively identified.
LD = Associated blank spike recovery was within the marginal exceedence limits of the LCS.
LE = MRL Check recovery was above laboratory acceptance limits. (TNI M4 1.7.4.2)
LF = MRL Check recovery was below laboratory acceptance limits.
LG = MRL Check recovery was above method acceptance limits. (TNI M4 1.7.4.2)
LH = MRL Check recovery was below method acceptance limits.
LJ = The associated blank spike recovery was below method acceptance limits. This target analyte exceeded a maximum regulatory limit/decision level. (TNI M4 1.7.4.2)
LK = The associated blank spike recovery was above method acceptance limits. This target analyte was not detected in the sample. (TNI M4 1.7.4.2)

Matrix spike:

Appropriate qualifier must be applied to the affected analytes in the matrix spike and should also be added to all corresponding analytes in the associated spiked sample. If a batch spike recovery is outside of the acceptable range, it is permissible to only flag the sample that was spiked and not the other samples in the batch. As required in the Arizona Adopted Rules A.A.C. R9-14-617.F, clients must always be informed if the batch QC result is unacceptable whether one of their samples was spiked or not. The laboratory can choose how the unacceptable QC is

10/24/2012

reported to the client (e.g., cover letter or flag). The ADEQ policy 0154.000 can be accessed at http://www.azdeq.gov/function/business/download/spike8.pdf

M1 = Matrix spike recovery was high; the associated blank spike recovery was acceptable.
M2 = Matrix spike recovery was low; the associated blank spike recovery was acceptable.
M3 = The spike recovery value is unusable since the analyte concentration in the sample is disproportionate to spike level. The associated blank spike recovery was acceptable. (EPADWM 5th ed)
M4 = The analysis of the spiked sample required a dilution such that the spike recovery calculation does not provide useful information. The associated blank spike recovery was acceptable.
M5 = Analyte concentration was determined by the method of standard addition (MSA).
M6 = Matrix spike recovery was high. Data reported per ADEQ policy 0154.000.
M7 = Matrix spike recovery was low. Data reported per ADEQ policy 0154.000.
MC = Matrix spike recovery was high; the associated blank spike recovery was acceptable. MS/MSD RPD met acceptance criteria
MD = Matrix spike recovery was low; the associated blank spike recovery was acceptable. MS/MSD RPD met acceptance criteria

General:

Use for events that cannot be described by the approved data qualifiers.

N1 = See case narrative.
N2 = See corrective action report.
N4 = The Minimum Reporting Limit (MRL) verification check did not meet the laboratory acceptance limit.
N5 = The Minimum Reporting Limit (MRL) verification check did not meet the method acceptance limit.
N6 = Data suspect due to quality control failure, reported per data user's request.

Sample Quality:

Flag samples with appropriate qualifier when sample quality may be potentially impacted or when method requirements were not met. The ADEQ policy 0154.000 can be accessed at http://www.azdeq.gov/function/business/download/spike8.pdf
The ADEQ policy 0155.000 can be accessed at http://www.azdeq.gov/function/business/download/one_pt3.pdf

Q1 = Sample integrity was not maintained. See case narrative.
Q2 = Sample received with head space.
Q3 = Sample received with improper chemical preservation.
Q4 = Sample received and analyzed without chemical preservation.
Q5 = Sample received with inadequate chemical preservation, but preserved by the laboratory.
Q6 = Sample was received above recommended temperature.
Q7 = Sample inadequately dechlorinated.

10/24/2012

Q8 = Insufficient sample received to meet method QC requirements. Batch QC requirements satisfy ADEQ policies 0154.000 and 0155.000.
Q9 = Insufficient sample received to meet method QC requirements.
Q10 = Sample received in an inappropriate sample container.
Q11 = Sample is heterogeneous. Sample homogeneity could not be readily achieved using routine laboratory practices.
QA = Sample received with incomplete documentation (ID).
QB = Sample received with improper sample label (ISL).
QC = Sample received with signs of damage or contamination (SDC).
QD = Same day sample receipt / sampling time but sample was received with no signs of chilling (c). (SRNC).
QE = Sample was received above method required temperature. Data not acceptable for regulatory compliance.
QF = Sample received without sufficient head space for proper mixing according to the method.

RPD Duplicates:

For use with sample, matrix spike, LFB and LCS duplicates. Qualify all affected analytes. For MS/MSD or sample duplicates qualify only the original source sample.

R1 = RPD/RSD exceeded the method acceptance limit.
R2 = RPD/RSD exceeded the laboratory acceptance limit.
R4 = MS/MSD RPD exceeded the method acceptance limit. Recovery met acceptance criteria.
R5 = MS/MSD RPD exceeded the laboratory acceptance limit. Recovery met acceptance criteria.
R6 = LFB/LFBD RPD exceeded the method acceptance limit. Recovery met acceptance criteria.
R7 = LFB/LFBD RPD exceeded the laboratory acceptance limit. Recovery met acceptance criteria.
R8 = Sample RPD exceeded the method acceptance limit.
R9 = Sample RPD exceeded the laboratory acceptance limit.
RA= MS/MSD RPD exceeded the method acceptance limit. Recovery did not meet acceptance criteria.
RB = MS/MSD RPD exceeded the laboratory acceptance limit. Recovery did not meet acceptance criteria.
RC = Low precision due to analyte concentration close to the MRL.

Surrogate:

Qualify surrogates appropriately when they do not meet criteria. Surrogate failures in quality control samples will most likely require additional narration. S11 & S12 are used to qualify sample surrogates and only in cases where the Laboratory Fortified Blank/LCS has acceptable surrogate recoveries.

10/24/2012

S1 = Surrogate recovery was above laboratory acceptance limits, but within method acceptance limits.
S3 = Surrogate recovery was above laboratory acceptance limits, but within method acceptance limits. No target analytes were detected in the sample.
S4 = Surrogate recovery was above laboratory and method acceptance limits. No target analytes were detected in the sample.
S5 = Surrogate recovery w as below laboratory acceptance limits, but within method acceptance limits.
S6 = Surrogate recovery was below laboratory and method acceptance limits. Re-extraction and/or reanalysis confirms low recovery caused by matrix effect.
S7 = Surrogate recovery was below laboratory and method acceptance limits. Unable to confirm matrix effect.
S8 = The analysis of the sample required a dilution such that the surrogate recovery calculation does not provide any useful information. The associated blank spike recovery was acceptable.
S10 = Surrogate recovery was above laboratory and method acceptance limits.
S11 = Surrogate recovery was high. Data reported per ADEQ policy 0154.000.
S12 = Surrogate recovery was low. Data reported per ADEQ policy 0154.000.
SA = Surrogate recovery was above laboratory and method acceptance limits. Re-extraction and or re-analysis confirms high recovery caused by matrix effect.
SB = Surrogate recovery was above laboratory and method acceptance limits. Unable to confirm matrix effect.
SC = The analysis of the sample required a dilution such that the surrogate concentration was diluted below the laboratory acceptance criteria. The associated blank spike recovery was acceptable.
SD = Surrogate recovery was below laboratory internal limits. Re-extraction and/or reanalysis confirms low recovery caused by matrix effect. No method criteria for surrogate.
SE= Surrogate recovery was below laboratory internal limits. Unable to confirm matrix effect. No method criteria for surrogate.
SF = Surrogate recovery was above laboratory internal limits. Re-extraction and or re-analysis confirms high recovery caused by matrix effect. No method criteria for surrogate.
SG = Surrogate recovery was above laboratory internal limits. Unable to confirm matrix effect. No method criteria for surrogate.

Method/analyte discrepancies:

For use with methods or analytes that are not currently approved under the Environmental Laboratory Licensure Rules.

T4 = Tentatively identified compound. Concentration is estimated and based on the closest internal standard.
T5 = Laboratory not licensed for this parameter.
T6 = The reported result cannot be used for compliance purposes.
T7 = Incubator/Oven temperatures were not monitored as required during all days of use.

Calibration Verification:

10/24/2012

Appropriate qualifier must be applied to all affected analytes in any samples associated with the calibration verification. The ADEQ policy 0155.000 can be accessed at http://www.azdeq.gov/function/business/download/one_pt3.pdf

V1 = CCV recovery was above method acceptance limits. This target analyte was not detected in the sample.
V2 = CCV recovery was above method acceptance limits. This target analyte was detected in the sample. The sample could not be reanalyzed due to insufficient sample.
V3 = CCV recovery was above method acceptance limits. This target analyte was detected in the sample, but the sample was not reanalyzed.
V9 = CCV recovery was below method acceptance limits.
VA = Closing standard recovery was above laboratory limits. Closing standard not required by method.
VB = Closing standard recovery was below laboratory limits. Closing standard not required by method.
VC = CCV is high biased, ND data are reportable as per TNI M4 #402 1.7.2 e ii
VE = CCV is high biased; ND data are reportable per method.
VF = CCV recovery was below method acceptance limits. The sample could not be reanalyzed due to insufficient sample.
VG = CCV recovery was below method acceptance limits. The sample result exceeded a maximum regulatory limit/decision level. (TNI M4 #403)

Internal Standards

IC = CCV Internal Standard recovery was above laboratory and method limits.
ID = CCV Internal Standard recovery was below laboratory and method limits.
IE = Trip Blank Internal Standard recovery was above laboratory and method limits.
IF = Trip Blank Internal Standard recovery was below laboratory and method limits.

Field / trip blank

FA = Target analyte detected in trip blank above the laboratory minimum reporting limit (MRL).

MWH General

NA = The sample was not analyzed
NR = The sample was analyzed but the results not reported due to failure of QC to meet method acceptance limits.

Other States/Clients' Requirements

J = Analyte is positively identified, but tentatively quantified. The reported value is an estimate concentration of the analyte in the sample. The analyte was either detected between MDL and MRL or did not meet any one of the required QC criteria. (MA - CLO4 requirements)

10/24/2012

(San Bernardino J Flag)
JA = Detected, not quantified. Estimated Concentration.

(Metals J Flag)
JB = The reported result is a estimated value (e.g, matrix interference was observed or the analyte was detected at a concentration outside the quantitation range)

(LADWP DNQ Flag)
DN = Detected, not quantified. Estimated Concentration.

Table 15-1 Example Summary of Corrective Action Procedures

Analysis Method	Item	Control Limits	Acceptance Criteria	Corrective Action
Volatile Organics	624	Sensitivity check Ion abundance with BFB	Tune instrument, criteria, see Table 11-3	repeat
		Initial calibration	All analytes < 35% RSD	Re-calibrate instrument
		Continuing calibration (QC Check Standard)	All analytes must meet % R as specified in Table 5 of Method 624	Rerun continuing calibration
		Method blank	<MRL	Determine cause of blank problem, reprep set if necessary
		Spiked samples (MS/MSD)	All analytes must meet % R as specified in Table 5 of Method 624	If LCS is in control, qualify LFM data, reprep set if necessary
		Duplicates (Dup)	RPD < than control limits	Re-prep and reanalyze
		Laboratory control samples (LCS)	All analytes must meet % R as specified in Table 5 of Method 624	Re-analyze batch
		MRL Check	50-150%	MRL check high, flag data. Out low adjust MRL or repeat test.
		Surrogate recovery	% R as specified in SOP	Re-prep and reanalyze
Base/Neutral/Acid Extractable Organics	625 with DFTPP	Sensitivity check Ion abundance with BFB	Tune instrument, criteria, see Table 11-3	repeat
		Initial calibration	<35% RSD	Re-calibrate
		Continuing calibration	RF ± 20%	Rerun continuing calibration, is still out, re-calibrate instrument
		Method blank	<MRL	Investigate problem, reprep set if necessary
		Spiked samples/LFM	All analytes must meet % R as specified in Table 6 of the method	If LCS in control, qualify LFM data, Reprep set if necessary.
		Laboratory control samples (LFB)	All analytes must meet % R as specified in Table 6 of the method	Re-analyze batch
		Surrogate recovery	% R as specified in SOP	Re-prep and reanalyze
Cyanide	335.4/	Initial calibration	r≥0.995	Repeat ICAL

Analysis Method	Item	Control Limits	Acceptance Criteria	Corrective Action
	9012B	MRL Check	50-150%	MRL check high, flag data. Ok to report high biased ND data. Out low adjust MRL or repeat test.
Phenolics	420.1/ 420.4	Calibration blank	<MRL	Investigate problem, re-digest set if necessary
		Continuing calibration	±10% of the expected value	Rerun continuing calibration, is still out, re-calibrate instrument and rerun samples from last CCV.
		Method blank	<MRL	Investigate problem, re-digest set if necessary
		Laboratory control samples (LFB)	% R of analyte within control limits of the method (90-110) for regular phenol and 80%-120% for low level	Re-digest and re-analyze batch
		MRL Check	50-150%	MRL check high, flag data. Ok to report high biased ND data. Out low adjust MRL or repeat test.
		Spiked samples/LFM	%R (90-110) for regular phenol and (80-120) for low level	If LCS in control, qualify LFM data, Reprep set of samples if necessary.
		Duplicates (Dup)	RPD < than control limits	Re-prep and reanalyze
Total Dissolved Solids, TDS	SM 2540C	Balance check	Expected value within 0.01% of balance	Re-calibrate
		MRL Check	50-150%	MRL check high, flag data. Ok to report high biased ND data. Out low adjust MRL or repeat test.
		RPD for reweighing	<4% or <0.5mg difference	Reweigh till weight difference is <4% or <0.5mg
Total Suspended Solids, TSS	SM 2540D	Balance check	expected value within 0.01% of balance	Re-calibrate

Analysis Method	Item	Control Limits	Acceptance Criteria	Corrective Action
		MRL Check	50-150%	MRL check high, flag data. Ok to report high biased ND data. Out low adjust MRL or repeat test.
Total Solids, TS	SM 2540B	Balance check	expected value within 0.01% of balance	Re-calibrate
Total Volatile Residue, TV	160.4	Method blank	<MRL	Investigate root cause of blank problem. Reprep set if necessary.
Total Settleable Solids, TSS	SM 2540F			
pH	SM 4500 H+B/ EPA 150.1 (DW only)	3 buffers	within ±0.1 pH unit of true value	Re-calibrate instrument
		Duplicates	RPD < than control limits	Re-prep duplicates and reanalyze or flag if reported
		Laboratory control samples (LFB)	% R within control limits of the method	Re-analyze batch
Anions: Perchlorate BrO3, ClO2,ClO3, Cl,NO3, NO2, PO4,SO4	300.0/ 300.1/ 314/ 317	Calibration curve	r ≥ 0.995	Rerun calibration
		Continuing calibration Verification,/LCS/LFB	90-110 % Rec	Recalibrate, rerun last
		Spiked samples/LFM	Must meet 80-120 % R	If LFB in control, no action taken
		MRL Check	50-150%	MRL check high, flag data. Ok to report high biased ND data.Out low adjust MRL or repeat test.
		Method Blank	< ½ MRL	Identify and eliminate source of problem. Do not do further sample analysis until contamination problem is resolved.
Anions: Perchlorate BrO3,ClO2,ClO 3,Cl,NO3, NO2, PO4, SO4	300.0/ 300.1/ 314/ 317	Method Blank	< ½ MRL	Repeat sample prep using another source of reagent if contamination is found to be due to the Reagents used.
TOC	SM	Calibration curve	r ≥ 0.995	Rerun calibration

Analysis Method	Item	Control Limits	Acceptance Criteria	Corrective Action
	5310C	Continuing calibration Verification, /LCS/LFB	90-110 % Rec	Recalibrate, rerun last 10 samples between the failing standard and the last standard meeting the acceptance
		MS/LFM	80-120 %	If LFB in control, no action taken
TOC (con't.)	SM 5310C	Method Blank	< 0.250ppm	Identify and eliminate source of problem. Do not do further sample analysis until contamination problem is resolved. Repeat sample prep using another source of reagent if contamination is found to be due to the Reagents used.
		Lab Duplicate	≤ 10% RPD (TOC ≥ 2.0 mg/L)	Reanalyze sample, if it cannot be reanalyzed, flag sample not meeting QC criteria.
			≤ 15% RPD (TOC < 2.0 mg/L)	
		MRL Check	50-150%	MRL check high, flag data. Ok to report high biased ND data.Out low adjust MRL or repeat test.
TOX	SM5320	Initial calibration Curve	r ≥0.995	Repeat ICAL
		Continuing calibration	±10% of the expected value	Rerun continuing calibration, is still out, re-calibrate instrument and rerun last 10 samples.
		Spiked samples/LFM	90-110% Rec	If LCS in control, qualify LFM data, Reprep set of samples if necessary.
		Method blank	< ½ MRL	Investigate problem, re-analyze set of samples if necessary
		Duplicates, (all samples)	RPD 15% (<100 ppb) RPD 10% (≥100 ppb)	Re-analyze to determine if matrix problem

Analysis Method	Item	Control Limits	Acceptance Criteria	Corrective Action
		Laboratory control samples (LFB)	±10% of the true value	Re-analyze batch
Mercury by Cold Vapor AAS	245.1/ 7470A/ 7471A	Initial calibration verification/IPC	± 5% of the expected value	Re-calibrate
Mercury by Cold Vapor AAS (con't.)	245.1/ 7470A/ 7471A	Continuing calibration	±10% of the expected value	Rerun continuing calibration, is still out, re-calibrate instrument and rerun last samples from last Calibration Check
		MRL Check	50-150%	MRL check high, flag data. Out low adjust MRL or repeat test.
		Method Blank	< ½ MRL	Investigate problem, re-digest set of samples if necessary
		Duplicates	RPD < 20%	Re-prep duplicates and re-analyze
		Spiked samples/LFM	70-130%	If LCS in control qualify LFM data, Reprep set of samples if necessary.
		Laboratory control samples (LFB)	85-115%	Re-prep and re-analyze batch
ICP Metals:	200.7/ 6010	Standard validation	± 5% of the expected value	Purchase new concentrates
		Initial calibration verification/IPC	95-105% Rec, RSD < 3%	Rerun calibration standards
		MRL Check	50-150%	MRL check high, flag data. Out low adjust MRL or repeat test.
ICPMS Metals	200.8	Calibration blank	<1/2 MRL	Investigate problem, re-run blank
		Continuing calibration verification	±10% of the expected value	Rerun standards, is still out, re-calibrate instrument and rerun samples from last CCV.
		Method blank	< ½ MRL	Investigate problem, re-digest set if necessary

Analysis Method	Item	Control Limits	Acceptance Criteria	Corrective Action
		Spiked samples/MS	70-130%	If LCS in control qualify LCS data, Reprep set of samples if necessary.
		Laboratory control samples (LCS)	85-115%	Re-prep and re-analyze batch
ICPMS Metals (cont.)	200.8	MRL Check	50-150%	MRL check high, flag data. Ok to report high biased ND data. Out low adjust MRL or repeat test.
Cr VI (Dissolved)	218.6	Initial Calibration	r≥ 0.999	Identify problem and rerun ICAL
		IPC (CCV)	95-105%	Perform another IPC. If failed again, recalibrate and reanalyze previous 10 samples
		LRB	< ½ MRL	Correct source of contamination and reanalyze sample.
		LFB/QCS (external source)	90-110 %	Procedure is out of control, identify source of problem and resolve before continuing analysis
		LFM	90-110%	If failed but LFB passed, problem is matrix related Flag unspiked sample as "suspect matrix"
		LFMD	90-110%/20% RPD	If failed but LFB passed, Problem is matrix related Flag unspiked sample
		QCS LDR	90-110% minimum 7stds	See LFB Start of Program
		MRL Check	50-150%	MRL check high, flag data. Ok to report high biased ND data. Out low adjust MRL or repeat test.

Analysis Method	Item	Control Limits	Acceptance Criteria	Corrective Action
HAAs	6251 B	Initial Calibration Curve	RSD< 20% r ≥ 0.995	If r < 0.995, use second order fit as calibration curve. Check for error if % RSD exceeds 30 %.
HAAs (con't.)	6251 B	Method blank	< ½ MRL	Identify and eliminate source of problem. Do not do further sample analysis until contamination problem is resolved. Repeat sample prep using another source of reagent if contamination is found to be due to the Reagents used.
		Laboratory control samples LCS/LFB/CCV)	LCS: 80-120% Rec Low ± 50% High ± 15%	If primary column results fail, use results from secondary. If both fail, re-analyze. If repeat fails, re-extract.
		LFM	Control chart limits updated annually	If LFB is in control, no action taken
		Surrogate recovery	70-130 % Rec	Re-analyze the samples
		MRL Check	50-150%	MRL check high, flag data. Ok to report high biased ND data. Out low adjust MRL or repeat test.
UV 254	SM 5910B	Calibration curve	90-110 % Rec.	Rerun Calibration
		Method blank	< 0.004 units	Identify and eliminate source of problem. Do not do further sample analysis until contamination problem is resolved. Repeat sample prep using another source of reagent if contamination is found to be due to the Reagents used.
		CCV(Low)	For 0.5mg/L, absorbance value between 0.008 to 0.010	Rerun continuing calibration, is still out,

Analysis Method	Item	Control Limits	Acceptance Criteria	Corrective Action
		CCV (Mid/High Verification)	For 6.0 mg/L, absorbance value between 0.079 to 0.097; 0.779 to 0.953 (for 60mg/L)	re-calibrate instrument and rerun last 10 samples between the failing standard and the last standard meeting the acceptance criteria.
UV 254 (con't.)	SM 5910B	LCS/LFB	Value within manufacturer's listed range	
		Lab Duplicate	≤ 10 % RPD ≤ 15% RPD for low standard	Reanalyze sample. If cannot be reanalyzed, flag not meeting QC criteria.
		MRL Check	50-150%	MRL check high, flag data. Ok to report high biased ND data. Out low adjust MRL or repeat test.
Residual Chlorine	SM 4500 Cl-G	LCS/LFB	85-115 %	Rerun standard. Prepare new standard, if needed.
		MRL Check	50-150%	MRL check high, flag data. Ok to report high biased ND data. Out low adjust MRL or repeat test.
		Duplicate	<20 % RPD	Reanalyze sample.
Organohalide Pesticides and PCB	505	Instrument Performance	CCV 70-130% Recovery	Determine the cause and eliminate the problem; if necessary, generate a new curve or set of cal factors to verify the decreased response before searching for problem source.
		Endrin breakdown	< 20 % degradation	Perform routine maintenance. Consistent breakdown suggests breakdown occurrence in instrument system; methodology is in control, correct for potential background concentration.

Analysis Method	Item	Control Limits	Acceptance Criteria	Corrective Action
		IDC	%R = 70-130% RSD ≤ 20 %	Source of problem identified and resolved before continuing analysis.
Organohalide Pesticides and PCB (con't.)	505	LFB	%R = 70-130% (need control charts after 30 data points per lab performance)	Source of problem identified and resolved before continuing analysis.
		Initial Calibration Curve	% RSD ≤ 20	Repeat test using a fresh cal std. If results still not agree, generate a new calibration curve.
		Continuing Calibration verification Standard	70-130 %	Reanalyze sample extracts for the suspected field sample analytes after acceptable cal is restored.
		LRB	< MRL	Determine source of contamination and eliminate interference before processing sample.
		LFM	% R = 65-135%	If lab performance is shown to be in control, problem is matrix-related, not system-related. Label result suspect/matrix to inform data user the results are suspect due to matrix effects.
		LFMD	not required 20 % RPD (initial guidance)	
		QCS	70 – 130 %	Done quarterly. Source of problem identified and resolved.
		MRL Check	50-150%	MRL check high, flag data. Ok to report high biased ND data. Out low adjust MRL or repeat test.
Volatiles, DIPE TAME, ETBE	524.2	Sensitivity check Ion abundance with BFB	Tune instrument, criteria, see Table 11-3 by GCMS	Retune Instrument. Ionizer may need to be cleaned before criteria can be met.

Analysis Method	Item	Control Limits	Acceptance Criteria	Corrective Action
		Initial calibration	≤ 20% RSD, r ≥ 0.995	Re-calibrate instrument. Prepare new standard and analyze.
Volatiles, DIPE TAME, ETBE (con't.)	524.2	Continuing calibration (QC Check Standard)	70-130%	Rerun continuing calibration. prepare new CCV std and re-analyze.
			80-120% (TCP)	
		Lab blank	< MRL	Reanalyze. If blank cannot be reanalyzed, flag associated data when samples have hits > MRL.
			< MRL (TCP)	(TCP: source of contamination investigated and measures taken to correct, minimize, or eliminate problem)
		Lab Duplicates (Dup)	< 20 % RPD	Re-prep and reanalyze
		Laboratory control samples (LCS/LFB)	70-130%	Re-analyze batch
			80-120% (TCP)	Problem resolved before additional samples may be reliably analyzed
		Surrogate recovery	80-120 % (initial demonstration of capability, IDOC) 70-130 % (CCV, samples)	Re-prep and reanalyze
		MRL Check	50-150%	MRL check high, flag data. Ok to report high biased ND data. Out low adjust MRL or repeat test.
Volatiles	524.3	BFB Sensitivity	Refer to Table 1 in 524.3 SOP	Optimize the instrument and reanalyze BFB tune
		Initial Calibration	Cal. Point ≤MRL: ±50% Other cal. points: ±30%	Re-calibrate instrument. Check for leaks and conduct repair and regular maintenance
		ICV/QCS	70-130% Rec	Re-run ICV, if out-of-control, correct the problem source then repeat ICAL

Analysis Method	Item	Control Limits	Acceptance Criteria	Corrective Action
		CCC	%D for each analyte below MRL: ≤50% Above MRL: ≤30%	Re-run CCC. If out-of-control, conduct maintenance and recalibrate
Volatiles (con't.)	524.3	LRB	< ½ MRL	Identify and eliminate affected batch, re-analyze associated samples
		Matrix Spike/LFSM LFSMD	70-130% Rec	If matrix effects are observed or suspected to be causing low recoveries and the LFSMD confirms this, analyze a lab fortified matrix sample for that matrix.
		LFBD	10-130% Rec RPD: <20%	If out of control, re-run LFBD and associated samples. Otherwise recalibrate.
		Surrogate	70-130% Rec	Check and optimize the instrument. Re-analyze out of control samples.
		Internal Standards (IS)	±30% of most recent CCC ±50% of average response in ICAL	Check and optimize the instrument. Re-analyze out of control samples.
Trihalomethanes /Chloral Hydrate/ Haloacetonitrile	551.1	Initial calibration curve (Extracted)	< 10 % RSD	recalibrate if fails criteria

Analysis Method	Item	Control Limits	Acceptance Criteria	Corrective Action
		Lab Performance Check	Table 7 of the method	Failed LPC, reevaluate the instrument system, if performance Criteria not met, install new column, correct column flows
		Endrin Breakdown	< 20 %	Perform routine maintenance In the injection port; replace injection port sleeve & all Associated seals & septa.
		Calibration Verification (CCV=LFB) (2 different conc. levels) (MLFB & HLFB)	% R = 80-120 % for 90 % of analytes	Reanalyze CCV. If failed again recalibrate & the previous samples reanalyzed or analytes out of acceptable range should be reported suspect to the data user.
Trihalomethanes /Chloral Hydrate/ Haloacetonitrile (con't.)	551.1	LRB	< ½ MRL	Determine source of contamination & eliminate the interference before processing samples
		LFB/CCV	% R = 80-120% for 90 % of analytes	Reanalyze CCV. If failed again recalibrate & the previous samples reanalyzed or analytes out of acceptable range should be reported suspect to the data user.
			75-125 % for all analytes	
		LFM	80-120%	When analyte recovery fails LFM criteria, a bias is concluded & analyte for that matrix is reported to the data user
		LFM/Duplicate	See Sample Duplicate	
		Sample Duplicate	RPD <20 for 90% of analytes, RPD <25% for all analytes	If failing, repeat analyses. Upon repeated failure, sampling must be repeated or analyte out of control must be reported as suspect to the data user.

Analysis Method	Item	Control Limits	Acceptance Criteria	Corrective Action
		Surrogate	80-120 % Recovery	Deviations in surrogate recovery may indicate a problem: Renalyze extract if extraction upon reanalysis, recovery is failing extract fresh sample. If not, data for all analytes from the sample should be reported as suspect.
		CCV Surrogate	80-120% Recovery	Recalibrate if fails criteria
		Sample Peak	Within the linear range of calibration curve	Dilute final extract and reanalyze

Note: Refer to individual SOPs for detailed corrective action procedures for all methods.

Figure 15-2 Preventive Action Record Form

Preventive Action Record
(ISO/IEC 17025 Section 4.12)

Date:

Log Number:

Regulatory Reference or Requirements:

Name of Requester:

Responsible Personnel

Department/Method:

Audit By:

Source: (check all that apply):

Annual Management Review___ Analysis of data ___ PT results ___

Trend and Risk Analysis ___ Other (explain)_____

Opportunity for Improvement:

Preventive Action:

Prevention:

PAR Completion Due Date:

Responsible for Preventive Action (Print Name and Signature):

QA Review Signature:

Follow-up of Preventive Action (2 to 3 months):

Date:

Status (open/closed):

Additional/Revised Preventive Action Needed:

New Due Date:

Date of Preventive Action Implementation:

Closing Date

Effectiveness of Preventive Action Taken:

Signature of Responsible Personnel as to the Completion of the PAR:

QA Signature:

Figure 15-3 Quality Investigation Report (QIR) Flow Chart

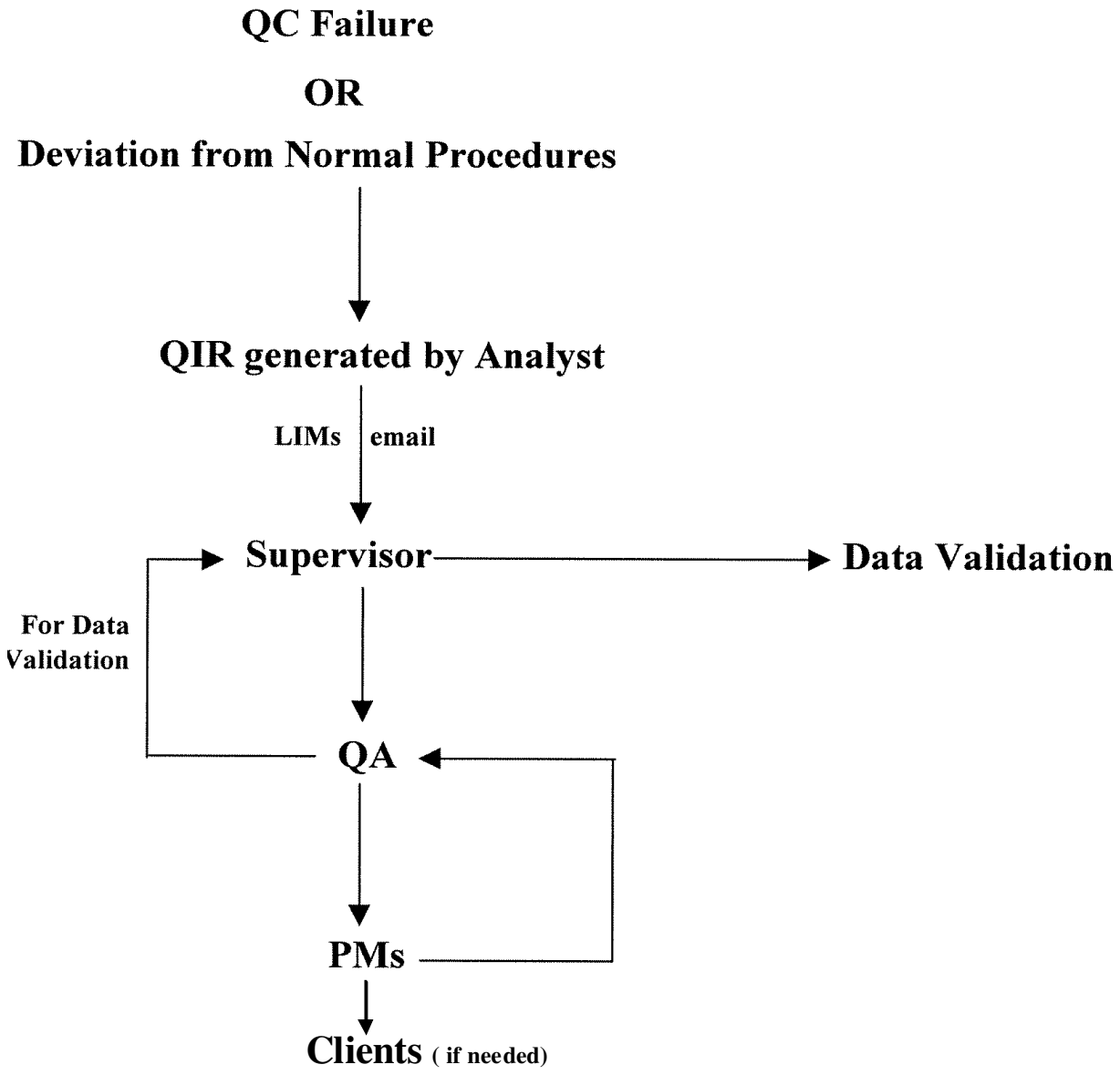
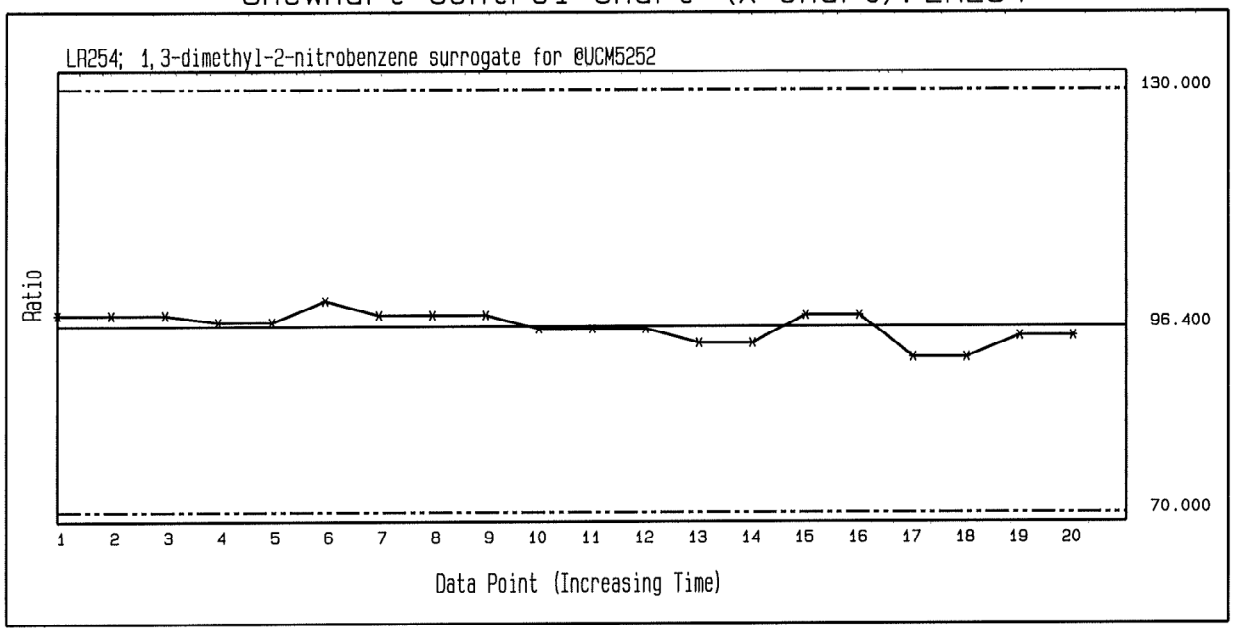


Figure 15-4 Example Surrogate Control Chart

mi_qcocc-A-3.1
06-jul-2009

Page: 1

Shewhart Control Chart (X-chart): LR254



Data Set Consisting of 20 Points
Inclusive Date Range: 12-JUN-2009 thru 30-JUN-2009
Last Updated: 31-JAN-2001 By: JDC

Historical		Calculated	
Average:	100.000	Average:	96.400
Plus Three Sigma:	130.000	Plus Three Sigma:	102.886
Minus Three Sigma:	70.000	Minus Three Sigma:	89.914

Data Point	Date	Actual	Found	Ratio	Component Lot ID#
1	12-jun-2009 15:59:00	100	98	98.000	
2	16-jun-2009 14:57:00	100	98	98.000	
3	16-jun-2009 14:57:00	100	98	98.000	
4	16-jun-2009 15:21:00	100	97	97.000	
5	16-jun-2009 15:21:00	100	97	97.000	
6	22-jun-2009 16:16:00	97.000	97.000	100.000	
7	22-jun-2009 16:40:00	100	98	98.000	
8	22-jun-2009 16:40:00	100	98	98.000	
9	22-jun-2009 16:40:00	100	98	98.000	
10	22-jun-2009 17:03:00	100	96	96.000	
11	22-jun-2009 17:03:00	100	96	96.000	
12	22-jun-2009 17:03:00	100	96	96.000	
13	24-jun-2009 13:11:00	100	94	94.000	
14	24-jun-2009 13:11:00	100	94	94.000	
15	24-jun-2009 13:34:00	100	98	98.000	
16	24-jun-2009 13:34:00	100	98	98.000	
17	30-jun-2009 11:30:00	100	92	92.000	
18	30-jun-2009 11:30:00	100	92	92.000	
19	30-jun-2009 11:53:00	100	95	95.000	
20	30-jun-2009 11:53:00	100	95	95.000	

End of Data

16.0 PERFORMANCE AND SYSTEM AUDITS/MANAGEMENT REVIEW

The Quality Manager at EEA is not directly involved in the production of analytical data. The QA department is responsible for an ongoing program of internal system audits and performance evaluation samples, and for coordinating all external audits and PT samples. In addition, the QA department is responsible for maintaining state and agency certifications.

16.1. INTERNAL AUDITS

The audits are carried out by the Quality Manager or designee(s) who will be independent of the activity to be audited. Also, to develop a proactive program for the detection of improper, unethical or illegal actions, the Quality Manager or designee, during the internal audit procedure, includes the auditing of any improper, unethical or illegal action committed by the analyst or supervisor.

16.1.1. **Annual and Periodical Internal Audits**

16.1.1.1. The laboratory Quality Assurance Group conducts an annual lab internal audit to verify that its operations continue to comply with the requirements of the laboratory's quality system as per TNI-EL-V1M2-2009-4.14.1 and ISO/IEC 17025:2005(E)-4.14.1.

16.1.1.2. The laboratory, in accordance with a predetermined schedule and procedure, conducts internal audits, at least annually, of the activities to verify that the operations continue to comply with the requirements of the quality systems of TNI and ISO 17025 standards. The internal audit program addresses all elements of the quality system, including environmental testing and/or calibration activities. The Quality Manager plans and organizes audits as required by the schedule and requested by management. Such audits are carried out by the Quality Manager and trained QA staff who are independent of the activity to be audited. Personnel are trained not to audit their own activities except when it can be demonstrated that an effective audit will be carried out [TNI-EL-V1M2-2009-4.14.1][ISO/IEC 17025:2005(E)-4.14.1].

16.1.1.3. When audit findings cast doubt on the effectiveness of the operations or on the correctness or validity of the laboratory's environmental test or calibration results, the laboratory takes timely corrective action, and notifies the clients in writing when the investigations show that the laboratory results are affected. The laboratory notifies the client promptly, in writing of any event such as the identification of defective measuring or test equipment that casts doubt on the validity of the results given in any test report or test certificate or amendment to a report or certificate [TNI-EL-V1M2-2009-4.14.2][ISO/IEC 17025:2005(E)-4.14.2].

16.1.1.4. The area of activity audited, the audit findings, and corrective actions that arise from them are recorded [TNI-EL-V1M2-2009-4.14.3][ISO/IEC 17025:2005(E)-4.14.3].

The laboratory management ensures that these actions are discharge within the agreed time frame as indicated in the audit finding documentation. Corrective actions are required within 30-days after findings have been reported to the Technical Manager.

16.1.1.5. Follow up audit activities of the laboratory are conducted in 2 to 3 months to verify and record the implementation and effectiveness of the corrective action taken [TNI-EL-V1M2-2009-4.14.4][ISO/IEC 17025:2005(E)-4.14.4].

16.1.2. **Data Package Reviews**

16.1.2.1. As part of the annual internal audit, data package review is conducted annually by the Quality Manager or designee. At the start of the audit program, PT results obtained by using the drinking water, wastewater, hazardous waste methods are evaluated in order to have an objective assessment on the quality of the data generated by the lab. Annually several analytical methods i.e. at least one representative technology method from Wet Chem, Metals, Rad, GC, HPLC, GCMS, Asbestos and Microbiology are selected either from PT or client data reports for data package reviews. The laboratory ensures that at the end of the year, a representative method from each TNI list of technology for drinking water, wastewater, and hazardous waste analysis have been reviewed. Compliance with all required QC is evaluated. A data package review checklist is used to serve as guidelines during the data package review. A report on the results of the data package review is submitted to the supervisors and the Lab Director after the data package review for corrective actions.

16.1.2.2. In addition, a response to the findings and appropriate corrective action is implemented by the supervisors to ensure continuous compliance to all method requirements. Also, to develop a proactive program for the detection of improper, unethical or illegal actions, the Quality Manager or designee during the data package review includes the detection of any potential improper, unethical or illegal action by any of the lab personnel. The data integrity checklist from Arizona is used as a guideline for reviewing data packages.

16.2. EXTERNAL AUDITS

16.2.1. External System audits are performed by outside agencies such as the California Department of Public Health (at least every 2 and 1/2 years for TNI accreditation) and by other state agencies where EEA is certified.

16.2.2. External audits are also conducted by the State of Arizona every 1-2 years, and Wisconsin every three (3) years. All other TNI states recognize CA-DOH on-site assessment in accordance to TNI secondary accreditation program. All corrective action reports audit findings and audit responses are retained by the laboratory for a minimum of 5 years (TNI) and 10-years (Massachusetts, Hawaii and New York).

16.3. PERFORMANCE AUDITS

PT samples are used to provide a direct evaluation of the ability of the analytical systems to generate data that is consistent with the laboratories' stated objectives for accuracy and precision. EEA analyzes internal PT samples as part of the ongoing QA program, while external PT samples are analyzed as part of the certification and approval process for various state and federal agencies, as well as for other organizations.

16.3.1. **Internal Proficiency Testing Samples/Internal Check Sample Program**

Internal PT Program is conducted as part of the corrective action process for any PT reported as unacceptable and evaluated by the PT provider as "check for error" or did not pass the PT provider's warning limits. Internal QC samples are also provided as needed as part of the analyst's initial demonstration of capability. The QA group maintains a logbook of all blind PT samples for traceability of the true and reported values. A LIMS report is generated for each QC sample logged in the LIMS system. Problem areas are reviewed as soon as they surface; the probable cause is determined as expeditiously as possible and corrective action implemented. If a severe problem with the analysis is evident, the analysis is halted until the cause is found and corrected.

16.3.2. **External Proficiency Testing (PT) Samples**

16.3.2.1. External Proficiency Testing (PT) samples are analyzed twice a year as part of the NELAP accreditation and approval process for various state and federal agencies.

16.3.2.2. Blind PT samples are procured from NIST/TNI/ISO 17025 Approved PT Providers to include the following samples:

- Semi-annual Drinking Water PT Samples (WS series) Organic and Inorganic Samples, Coliform Microbe, HPC, and source water E.Coli
- Radiochemistry Gross Alpha, Beta , Radium 228 and Uranium PT samples
- Annual NPDES/DMR PT sample as required by EPA.
- Semi-annual Asbestos PT Samples
- Semi-annual Wastewater PT Samples (WP series)/NPDES Organic and Inorganic PT samples/Hazardous Waste (Aqueous matrices)

16.3.3. **Proficiency Testing Protocol**

16.3.3.1. **Frequency**

16.3.3.1.1. The laboratory participates in the PT program of a NIST approved PT provider twice in each calendar year.

16.3.3.1.2. The laboratory enrolls and participates in a proficiency-testing program (PT) for each analyte or interdependent analyte group using all routine drinking water methods. When new analytes are added to the certification, 2 successful PT studies

must be performed at least "30 (for MA)" calendar days apart from closing date of one study to the shipment of another study for the same field of proficiency testing, at least 15 calendar days between the analysis dates of successive PT samples for the same accreditation, and will be completed within 18 months from the date the additional groups are added on the Laboratory Application. [TNI-EL-V1M1-2009-4.1.3].

16.3.3.2. **Laboratory Handling**

16.3.3.2.1. As per TNI Standard Volume 1, Module 1, PT samples are managed, analyzed and reported in the same manner as real routine samples by utilizing the same staff, methods as used for routine analysis of that analyte, procedures, equipment, facilities, and frequency of analysis.

16.3.3.2.2. The laboratory follows the proficiency testing provider's instructions for preparing the proficiency-testing sample dilution (as needed) and analyzes the proficiency-testing sample as if it were a client sample.

16.3.3.2.3. The laboratory complies with the following prohibitions:

- Performing multiple analyses (replicates, duplicates) which are not normally performed in the course of analysis of routine samples;
- Performing increased frequency of quality control samples or initial and continuing calibrations which are not normally performed in the course of analysis of routine samples;
- Averaging the results of multiple analyses for reporting when not specifically required by the method; or
- Permitting anyone other than bona fide laboratory employees who perform the analyses on a day-to-day basis for the certified laboratory to participate in the generation of data or reporting of results.

16.3.3.2.4. The laboratory does not:

- Discuss the results of a proficiency testing audit with any other laboratory until after the deadline for receipt of results by the proficiency testing provider;
- Attempt to obtain the assigned value of any proficiency testing sample from the proficiency testing provider.
- Send proficiency testing samples or portions of samples to another laboratory to be tested; or
- Knowingly receive a proficiency-testing sample from another laboratory for analysis and fail to notify the department of the receipt of the other laboratory's sample within five business days of discovery.

16.3.3.2.5. The laboratory maintains a copy of all proficiency testing records, including analytical worksheets. The proficiency testing records include a copy of the authorized proficiency testing provider report forms used by the laboratory to record proficiency testing results.

16.3.3.2.6. The laboratory participates in Client/State sponsored PT programs. The director of the laboratory or representatives of the laboratory provides, if needed, an attestation statement stating that the laboratory followed the proficiency testing provider's instructions for preparing the proficiency testing sample and analyzed the proficiency testing sample as if it were a client sample.

16.3.3.3. **Not Acceptable PT Results**

16.3.3.3.1. If the laboratory fails a PT sample, a corrective action plan is submitted to CA DPH and other states requiring corrective action, such as Nevada, Washington, Mariana Islands, Guam, Virginia, Maryland, West Virginia, and Massachusetts, within 30-days after receipt of PT report.

16.3.3.3.2. Corrective Action Reports are generated when non-acceptable results are reported. Data reported by the laboratory not within the warning limits and flagged as “check for error” are also investigated to determine the root cause of the problems. Internal PT samples are provided to the analyst to determine if corrective action implemented was effective to resolve the problem. Acceptable results of the internal PT samples help the analyst to determine if the analysis is in control after the implementation of the corrective action.

16.3.3.3.3. Make-up PT or supplemental PT samples are also analyzed when the laboratory fails to maintain a record of passing two out of the most recent three PT studies and wishes to re-establish its history of successful performance. Analysis dates of make up PT studies must be at least 15 calendar days between analysis date. Since some states, such as Massachusetts requires at least 30-days apart, thus the Lab adopts the “30-days apart” requirement for Make-up samples from the closing date of one study to the shipment date of another study.

16.3.3.4. **Reporting**

16.3.3.4.1. The laboratory analyzes and reports the results of the proficiency-testing test by the deadline set by the proficiency-testing provider.

16.3.3.4.2. When the PT falls below the range of the analytical method, the laboratory reports “<MRL” and does not perform special procedures to determine the low level result [TNI-EL-V1M1-2009-5.2.1].

16.3.3.4.3. The laboratory reports the results of the proficiency testing test by the procedure specified by the proficiency-testing provider.

16.3.3.4.4. The laboratory notifies the approving states such as Wisconsin of the authorized proficiency testing program or programs in which it has enrolled for each analyte or interdependent analyte group.

16.3.3.4.5. The laboratory directs the proficiency-testing provider to send, either in hard copy or electronically, a copy of each evaluation of the laboratory's proficiency testing audit results to the state requiring the PT results. The laboratory allows the proficiency-testing provider to release all information necessary for the state to assess the laboratory's compliance to PT requirements.

16.3.3.5. **Remedial PT**

16.3.3.5.1. The certified laboratory participates in only one remedial proficiency-testing audit for an analyte or independent analyte group in any 12-month period to obtain or upgrade approval under this section, as per Massachusetts's PT requirements.

16.3.3.5.2. The laboratory directs the proficiency-testing provider to send, either in hard copy or electronically, a copy of each evaluation of the certified laboratory's remedial proficiency testing results to California, and all other NELAP and other non-NELAP states. The laboratory allows the proficiency-testing provider to release all information necessary for the state to assess the certified laboratory's compliance with this rule.

16.4. SYSTEM AUDITS AND MANAGEMENT REVIEW

In order to insure that the Quality Assurance program at the laboratory maintains a high profile, there are several mechanisms in place which insure that QA information is routinely conveyed to laboratory management. This includes an annual QA report, reports on internal and external PE samples, and verbal transmittal of QA information to the Laboratory Director and group supervisors during a weekly staff meeting.

16.4.1. System Audits

System audits are performed both by external agencies, and by the laboratory Quality Assurance Group. The focus of these audits is the overall analytical "system", from login to delivery of the finished reports. The purpose of the audits is to document compliance with specified methodology contained in the SOPs.

All audit and review findings and any corrective actions that arise from them shall be documented. The laboratory shall ensure that these actions are discharged within the agreed time frame.

16.4.2. Management Review

16.4.2.1. The Quality Manager prepares and submits an annual QA/QC report for the Laboratory Director and Technical Directors. This report describes all the quality assurance activities conducted during the year, including proficiency test sample results (both internal and external), holding time exceedances, de-briefing from external and internal systems audits, and a summary of all out of control events that required corrective action/preventive measures and the effectiveness of the initiated corrective action. Whenever any such quality assurance information impacts a specific analytical project, the events are immediately related to the Client Services Group, who is responsible for informing the client.

16.4.2.2. The annual QC report includes the outcome of recent internal audits, assessments by external bodies, the results of inter-laboratory comparisons of proficiency tests and corrective actions. The annual QC report also include a discussion of the lab certifications, the laboratory SOPs generated for the year including SOP updates, control charts, acceptance limits updates, Quality Manual updates and data review results.

16.4.2.3. The Laboratory Director and Technical Directors perform an annual managerial review of the laboratory quality system and its testing and calibration activities to ensure its continuing suitability and effectiveness. Any necessary changes or improvements in the quality system and laboratory operations are introduced during the annual managerial review. Thus, the Laboratory Director, Technical Directors, Quality Manager, and Technical Managers review the annual QC report, provide an overall assessment of all the QC activities stated in the annual QC report, and introduce any necessary changes or improvements in the quality system and laboratory operations. The annual managerial review also takes into account changes in the volume and type of work undertaken for the previous year and feedback from clients, complaints and other relevant factors, such as resources and staff training [TNI-EL-V1M2-2009-4.15.1][ISO/IEC 17025:2005(E)-4.15.1].

16.4.2.4. The QA Group conducts performance audits of the laboratory and also maintains a program of blind proficiency testing samples. Results of these blind performance samples are scored according to the methods criteria. In addition a debriefing to group leaders and the Laboratory Director is prepared by the QA group following each set of PT samples. Evaluations of any failures on external PT samples are prepared by Group Supervisors and summarized by the Quality Assurance Group for the certifying agencies, with copies conveyed to the Laboratory Director.

16.5. IMPROVEMENT

As per TNI-EL-V1M2-2009-4.10 (ISO/IEC 17025:2005(E)-4.10), improvement in the overall effectiveness of the laboratory management system is a result of the implementation of the various aspects of the laboratory's management system: quality policy (Section 3.2 – "Quality Policy") and objectives (Section 5.0 – "Quality Assurance Objectives"), internal audit practices (Section 16.1 – "Internal Audits"), the review and

analysis of data (Section 6.0 – "Quality of Test Results"), the corrective and preventive action process (Section 15.0 – "Control of Non-Conforming Work, Corrective Action, and Preventive Measures"), and the annual management review of the quality management system (Section 16.4.2 – "Management Review") where the various aspects of the management/quality are summarized, and evaluated and plans for improvement are developed. See Figure 15-2 for the Preventive Action Form which is used to document actions required for improvement, name of requestor, responsible personnel to carry out implementation, and estimated completion date.

APPENDIX I

Arizona Certification and Approval

Arizona Department of Health Services
Office of Laboratory Licensure, Certification & Training
250 North 17th Avenue, Phoenix, AZ 85007

Tuesday, February 12 2013

AZ License: **AZ0778**

Lab Director: **Mr. Ed Wilson**

Lab Name: Eurofins Eaton Analytical, Inc.
Phone: (626) 386-1100
Fax: (626) 386-1139

Program **HW**

Parameter	EPA Method	Billing Code	Cert Date
Edb And Dbcp By Microextraction And Gc	EPA 8011	OC4	11/12/96
Purge And Trap For Aqueous Samples	EPA 5030C	PREP2	12/05/06
Titanium	EPA 6010B	MTL3	11/10/05
Total Metals	EPA 3010A	PREP1	11/04/10
Tox	EPA 9020B	MISC2	04/26/99
Vocs By Gc/Ms	EPA 8260B	OC8	10/18/99

Total Licensed Parameters in this Program: 6

Program **SDW**

Parameter	EPA Method	Billing Code	Cert Date
Acetanilide Degradation Products - Additional	EPA 535 (1.1)	OC37	08/20/09
Acetanilide Parent Compound - Additional	EPA 525.2 (2.0)	OC10	08/20/09
Alkalinity	SM 2320B	NIA1	04/06/96
Aluminum	EPA 200.7	MTL3	09/30/96
Aluminum	EPA 200.8	MTL1	11/17/95
Antimony	EPA 200.8	MTL1	12/19/94
Arsenic	EPA 200.8	MTL1	12/19/94
Asbestos	EPA 100.2	MISC27	06/03/03
Barium	EPA 200.7	MTL3	11/24/93
Barium	EPA 200.8	MTL1	12/21/94
Beryllium	EPA 200.7	MTL3	01/10/94
Beryllium	EPA 200.8	MTL1	11/17/95
Bromate	EPA 300.1	NIIIA1	06/05/01
Bromate	EPA 317.0	NIIIA2	11/06/06
Bromide	EPA 300.0	NIIIA1	04/20/03
Bromide	EPA 300.1	NIIIA1	11/16/01
Cadmium	EPA 200.7	MTL3	11/24/93
Cadmium	EPA 200.8	MTL1	12/21/94
Calcium	EPA 200.7	MTL3	09/26/94
Carbamates By Hplc/Post Column	EPA 531.2 (1.0)	OC4	08/14/03
Carbamates By Hplc/Post Column - Additional	EPA 531.2 (1.0)	OC10	08/14/03
Carbon, Dissolved Organic	SM 5310C	MISC1	10/23/08
Carbon, Total Organic	SM 5310C	MISC1	03/24/99
Chloramine	SM 4500 CL-G	NIA15	09/22/03
Chlorate	EPA 300.1	NIIIA1	05/29/12
Chloride	EPA 300.0	NIIIA1	11/24/93
Chlorinated Acids And Dalapon By Gc-Ecd	EPA 515.4 (1.0)	OC4	08/14/03
Chlorinated Acids And Dalapon By Gc-Ecd	EPA 515.4 (1.0)	OC10	10/23/08
Additional Chlorine	SM 4500-CL G	NIA2	04/06/96

Arizona Department of Health Services
Office of Laboratory Licensure, Certification & Training
250 North 17th Avenue, Phoenix, AZ 85007

Tuesday, February 12 2013

AZ License: **AZ0778** **Lab Name: Eurofins Eaton Analytical, Inc.**

Program **SDW**

Parameter	EPA Method	Billing Code	Cert Date
Chlorine Dioxide	SM 4500-CL02 D	NIA15	11/16/01
Chlorite	EPA 300.0	NIIIA1	03/24/99
Chlorite	EPA 300.1	NIIIA1	03/05/01
Chlorite	EPA 317.0	NIA15	10/23/08
Chromium Total	EPA 200.7	MTL3	11/24/93
Chromium Total	EPA 200.8	MTL1	11/17/95
Chromium, Hexavalent	EPA 218.7	MISC20	05/22/12
Colbalt	EPA 200.8	MTL1	05/22/12
Color	SM 2120B	NIA4	07/20/97
Copper	EPA 200.7	MTL3	11/24/93
Copper	EPA 200.8	MTL1	12/19/94
Corrosivity	SM 2330B	NIA2	08/16/93
Cyanide	EPA 335.4	MISC2	07/15/96
Cyanide	SM 4500 CN B	PREP1	04/06/96
Cyanide	SM 4500 CN C	PREP2	04/06/96
Cyanide	SM 4500 CN F	MISC2	04/06/96
Cyanide Amenable To Chlorination	SM 4500-CN G	MISC2	12/11/02
Dbp By Micro-Liquid Extraction/Gc-Ecd	EPA 6251B	OC4	12/14/98
Dbp, Solvents And Pesticides	EPA 551.1 (1.0)	OC4	10/25/04
Dbp,Solvents & Pesticides - Additional	EPA 551.1 (1.0)	OC10	10/23/08
Determination Of 1,4-Dioxane (Gc/Ms) (Sim)	EPA 522	OC8	05/22/12
Determination Of Hormones Liquid Chrom Electrospray (Lc Esi Ms/Ms)	EPA 539	OC8	05/22/12
Determination Of Selected Perfluorinated Alkyl Acids (Lc/Ms/Ms)	EPA 537	OC8	05/22/12
Dioxin	EPA 1613B	OC17	05/07/10
Diquat	EPA 549.2 (1.0)	OC4	02/20/01
Diquat And Paraquat - Additional	EPA 549.2 (1.0)	OC10	02/02/01
Edb/Dbcp	EPA 504.1 (1.1)	OC4	11/12/96
Endothall	EPA 548.1 (1.0)	OC4	12/21/94
Explosives And Related Compounds - Additional	EPA 529	OC8	08/20/09
Fecal Coliform	SM 9221E	MIC1	12/11/02
Fluoride	EPA 300.0	NIIIA1	05/15/12
Fluoride	SM 4500-F C	NIB9	07/15/96
Glyphosate	EPA 547 (7/90)	OC4	11/24/93
Gross Alpha	EPA 900	RADIO	01/10/94
Gross Beta	EPA 900	RADIO	10/27/03
Haloacetic Acids & Dalapon	EPA 552.3 (1.0)	OC4	05/20/10
Hardness	EPA 200.7, CA&MG	MTL3	10/25/04
Hardness	SM 2340B	MTL3	11/24/93
Heterotrophic Plate Count	SM 9215B	MIC3	09/02/03
Hydrogen Ion (Ph)	EPA 150.1	NIA2	08/16/93

Arizona Department of Health Services
Office of Laboratory Licensure, Certification & Training
250 North 17th Avenue, Phoenix, AZ 85007

Tuesday, February 12 2013

AZ License: AZ0778 **Lab Name: Eurofins Eaton Analytical, Inc.**

Program SDW

Parameter	EPA Method	Billing Code	Cert Date
Hydrogen Ion (Ph)	SM 4500-H B	NIA2	11/30/97
Iron	EPA 200.7	MTL3	11/24/93
Lead	EPA 200.8	MTL1	12/19/94
Magnesium	EPA 200.7	MTL3	09/26/94
Manganese	EPA 200.7	MTL3	11/24/93
Manganese	EPA 200.8	MTL1	12/19/94
Mercury	EPA 245.1	MTL5	08/16/93
Molybdenum	EPA 200.8	MTL1	05/22/12
Nickel	EPA 200.7	MTL3	01/10/94
Nickel	EPA 200.8	MTL1	12/19/94
Nitrate	EPA 300.0	NIIIA1	11/24/93
Nitrate	EPA 353.2	NIB1	04/02/98
Nitrite	EPA 300.0	NIIIA1	01/10/94
Nitrite	EPA 353.2	NIIB4	12/11/02
Nitrosamines By Ms/Ms - Additional	EPA 521	OC37	08/20/09
Odor	SM 2150B	NIA4	10/29/03
Organics By Gc/Ms	EPA 525.2 (2.0)	OC8	02/22/06
Organics By Gc/Ms - Additional	EPA 525.2 (2.0)	OC10	12/05/06
Orthophosphate	EPA 365.1	NIIB4	11/17/95
Orthophosphate	SM 4500-P E	NIIB4	04/06/96
Orthophosphate	SM 4500-P F	NIIB4	03/20/08
Perchlorate	EPA 314.0	NIB1	03/30/01
Perchlorate	EPA 331.0	NIIIA1	10/23/08
Pesticides & Flame Retardants By Gc/Ms - Additional	EPA 527	OC8	08/20/09
Pesticides And Pcbs By Gc	EPA 505 (2.1)	OC8	04/03/03
Pesticides And Pcbs By Gc - Additional	EPA 505 (2.1)	OC10	10/23/08
Radium 226	GAMMARAY HPGE GE(LI)	RADIO	10/28/11
Radium 228	GAMMARAY HPGE GE(LI)	RADIO	10/28/11
Residue, Filterable (Tds)	SM 2540C	NIA2	04/06/96
Selenium	EPA 200.8	MTL1	12/19/94
Silica	EPA 200.7	MTL3	11/17/95
Silica	SM 4500-SI D	MISC2	11/08/02
Silica	SM 4500-SIO2C	MISC2	03/24/08
Silver	EPA 200.7	MTL3	11/24/93
Silver	EPA 200.8	MTL1	11/17/95
Sodium	EPA 200.7	MTL3	09/26/94
Specific Conductance	SM 2510B	NIA2	04/06/96
Strontium	EPA 200.7	MTL3	11/24/93
Strontium	EPA 200.8	MTL1	05/22/12

Arizona Department of Health Services
Office of Laboratory Licensure, Certification & Training
250 North 17th Avenue, Phoenix, AZ 85007

Tuesday, February 12 2013

AZ License: AZ0778 **Lab Name: Eurofins Eaton Analytical, Inc.**

Program SDW

Parameter	EPA Method	Billing Code	Cert Date
Sulfate	EPA 300.0	NIIIA1	11/24/93
Surfactant (Mbas)	SM 5540C	NIIA1	07/15/96
Thallium	EPA 200.8	MTL1	12/19/94
Total Coliforms & E. Coli By Colilert	SM 9223B	MIC3	04/02/98
Total Coliforms By Mtf	SM 9221B & C	MIC1	12/23/97
Turbidity, Ntu: Nephelometric	EPA 180.1	NIA2	02/10/98
Uranium	EPA 200.8	MTL1	09/08/04
Uv Absorbing Organic Constituents	SM 5910B	NIB1	07/10/99
Vanadium	EPA 200.8	MTL1	05/22/12
Vocs By Gc/Ms	EPA 524.2 (4.1)	OC8	01/15/03
Vocs By Gc/Ms	EPA 524.3 (1.0)	OC8	10/28/11
Vocs By Gc/Ms-Additional	EPA 524.2 (4.1)	OC10	10/23/08
Vocs By Gc/Ms-Additional	EPA 524.3 (1.0)	OC10	10/28/11
Zinc	EPA 200.7	MTL3	11/24/93
Zinc	EPA 200.8	MTL1	12/19/94

Total Licensed Parameters in this Program: 123

Program WW

Parameter	EPA Method	Billing Code	Cert Date
Alkalinity, Total	SM 2320B	NIA1	04/02/98
Aluminum	EPA 200.7	MTL3	04/02/98
Aluminum	EPA 200.8	MTL1	04/02/98
Ammonia	EPA 350.1	NIIB1	12/23/97
Antimony	EPA 200.7	MTL3	04/02/98
Antimony	EPA 200.8	MTL1	04/02/98
Arsenic	EPA 200.8	MTL1	04/02/98
Barium	EPA 200.7	MTL3	04/02/98
Barium	EPA 200.8	MTL1	04/02/98
Base/Neutrals And Acids Excluding Pesticides	EPA 625	OC8	05/09/94
Beryllium	EPA 200.7	MTL3	04/02/98
Beryllium	EPA 200.8	MTL1	04/02/98
Biochemical Oxygen Demand	SM 5210B	DEM1	11/30/97
Boron	EPA 200.7	MTL3	04/02/98
Bromide	EPA 300.0	NIIIA1	04/02/98
Cadmium	EPA 200.7	MTL3	04/02/98
Cadmium	EPA 200.8	MTL1	04/02/98
Calcium	EPA 200.7	MTL3	04/02/98
Carbon, Total Organic (Toc)	SM 5310C	MISC1	04/02/98
Chemical Oxygen Demand	EPA 410.4	DEM3	12/23/97
Chemical Oxygen Demand	SM 5220D	DEM3	10/27/03
Chloride	EPA 300.0	NIIIA1	04/02/98

Arizona Department of Health Services
Office of Laboratory Licensure, Certification & Training
250 North 17th Avenue, Phoenix, AZ 85007

Tuesday, February 12 2013

AZ License: **AZ0778** **Lab Name: Eurofins Eaton Analytical, Inc.**

Program **WW**

Parameter	EPA Method	Billing Code	Cert Date
Chlorine Residual Total	SM 4500-CL G	NIA2	04/02/98
Chlorine Total Residual	HACH 8167	NIA2	10/23/08
Chlorine, Free	HACH 8021	NIA2	10/23/08
Chromium Total	EPA 200.7	MTL3	04/02/98
Chromium Total	EPA 200.8	MTL1	04/02/98
Chromium, Hexavalent	EPA 218.6, R 3.3	MTL1	11/20/07
Chromium, Hexavalent	SM 3500-CR D	MTL8	02/07/98
Cobalt	EPA 200.7	MTL3	04/02/98
Cobalt	EPA 200.8	MTL1	04/02/98
Color	SM 2120B	NIA4	07/20/97
Copper	EPA 200.7	MTL3	04/02/98
Copper	EPA 200.8	MTL1	04/02/98
Cyanide Amenable To Chlorination	SM 4500-CN G	MISC2	10/16/07
Cyanide, Total	EPA 335.4	MISC2	10/16/07
Cyanide, Total	SM 4500CN-F	MISC2	10/16/07
Fluoride	EPA 300.0	NIIIA1	10/20/09
Fluoride	SM 4500-F B	NIB3	12/23/97
Fluoride	SM 4500-F C	NIB3	12/23/97
Gross Alpha	EPA 900	RADIO	10/18/99
Gross Beta	EPA 900	RADIO	10/18/99
Hardness	EPA 200.7	MTL3	10/23/08
Hardness	SM 2340B	MTL8	04/02/98
Hydrogen Ion (Ph)	SM 4500-H B	NIA2	03/10/98
Iron	EPA 200.7	MTL3	04/02/98
Kjeldahl Nitrogen	EPA 351.2	NIIB4	11/30/97
Lead	EPA 200.8	MTL1	04/02/98
Magnesium	EPA 200.7	MTL3	04/02/98
Manganese	EPA 200.7	MTL3	04/02/98
Manganese	EPA 200.8	MTL1	04/02/98
Mercury	EPA 245.1	MTL5	04/02/98
Molybdenum	EPA 200.7	MTL3	04/02/98
Molybdenum	EPA 200.8	MTL1	04/02/98
Nickel	EPA 200.7	MTL3	04/02/98
Nickel	EPA 200.8	MTL1	04/02/98
Nitrate	EPA 300.0	NIIIA1	04/02/98
Nitrate-Nitrite (As N)	EPA 353.2	NIB1	12/23/97
Nitrite	EPA 353.2	NIIB4	10/16/07
Nitrite (As N)	EPA 300.0	NIIIA1	04/02/98
Orthophosphate	EPA 365.1	NIIB4	03/20/08
Orthophosphate	HACH 8048	NIIB1	12/05/06
Orthophosphate	SM 4500-P E	NIIB4	11/20/07

Arizona Department of Health Services
Office of Laboratory Licensure, Certification & Training
250 North 17th Avenue, Phoenix, AZ 85007

Tuesday, February 12 2013

AZ License: AZ0778 **Lab Name: Eurofins Eaton Analytical, Inc.**

Program WW

Parameter	EPA Method	Billing Code	Cert Date
Orthophosphate	SM 4500-P F	NIIB4	03/20/08
Oxygen, Dissolved	SM 4500-O G	NIA12	10/25/04
Phenols	EPA 420.1	MISC5	12/11/02
Phosphorus, Total	EPA 365.1	NIIB4	04/26/99
Phosphorus, Total	SM 4500-P E	NIIB4	10/25/04
Phosphorus, Total	SM 4500-P F	NIIB4	01/16/99
Potassium	EPA 200.7	MTL3	04/02/98
Purgeables	EPA 624	OC8	05/09/94
Residue Nonfilterable	SM 2540D	NIIA1	11/30/97
Residue Total	SM 2540B	NIIA1	12/05/06
Residue Volatile	EPA 160.4	NIIA1	10/27/03
Residue, Filterable	SM 2540C	NIA2	12/23/97
Residue, Settleable Solids	SM 2540F	NIIA1	12/11/02
Selenium	EPA 200.8	MTL1	04/02/98
Semivolatile Organic By Gc/Ms	EPA 1625B	OC8	11/06/01
Silica, Dissolved	EPA 200.7	MTL3	09/02/03
Silica, Dissolved	SM 4500-SI D	MISC2	11/08/02
Silica, Dissolved	SM 4500-SIO2C	MISC2	10/23/08
Silver	EPA 200.7	MTL3	04/02/98
Silver	EPA 200.8	MTL1	04/02/98
Sodium	EPA 200.7	MTL3	04/02/98
Specific Conductance	EPA 120.1	NIA2	12/11/02
Specific Conductance	SM 2510B	NIA2	12/23/97
Strontium	EPA 200.7	MTL3	11/17/95
Sulfate	EPA 300.0	NIIIA1	04/02/98
Sulfide	SM 4500-S D	MISC2	12/05/06
Surfactants (Mbas)	SM 5540C	NIIA1	07/10/99
Thallium	EPA 200.7	MTL3	04/02/98
Thallium	EPA 200.8	MTL1	04/02/98
Tin	EPA 200.7	MTL3	10/18/99
Tin	EPA 200.8	MTL1	10/23/08
Titanium	EPA 200.7	MTL3	10/23/08
Titanium	EPA 200.8	MTL1	10/23/08
Total Coliforms By Mtf	SM 9221B	MIC1	04/02/98
Turbidity, Ntu	EPA 180.1	NIA2	02/08/98
Turbidity, Ntu	SM 2130B	NIA2	02/08/98
Vanadium	EPA 200.7	MTL3	04/02/98
Vanadium	EPA 200.8	MTL1	04/02/98
Zinc	EPA 200.7	MTL3	04/02/98
Zinc	EPA 200.8	MTL1	04/02/98

Arizona Department of Health Services
Office of Laboratory Licensure, Certification & Training
250 North 17th Avenue, Phoenix, AZ 85007

Page: 7

Tuesday, February 12 2013

AZ License: AZ0778 **Lab Name: Eurofins Eaton Analytical, Inc.**

Program	**WW**			
	Parameter	EPA Method	Billing Code	Cert Date
Total Licensed Parameters in this Program:	103			

Instruments	**Quantity**	**Date**
GAS CHROMATOGRAPH	17	08/07/12
GAS CHROMATOGRAPH/MASS SPECTROMETER	11	08/07/12
ION CHROMATOGRAPH	10	08/07/12
HIGH PERFORMANCE LIQUID CHROMATOGRAPH	3	08/07/12
INDUCTIVELY COUPLED PLASMA/MASS SPECTROMETER	3	08/07/12
TOTAL ORGANIC ANALYZER	2	08/07/12
AUTOMATED AUTOANALYZER	2	08/07/12
HIGH PERFORMANCE LIQUID CHROMATOGRAPH/MASS SPE	1	08/07/12
MERCURY ANALYZER	1	08/07/12
TRANSMISSION ELECTRON MICROSCOPE	1	08/07/12
GAS CHROMATOGRAPH/MASS SPECTROMETER-HI RESOLUT	1	08/07/12
GAS FLOW PROPORTIONAL COUNTER	1	08/07/12
COUNTERS FOR RADIOACTIVITY	1	12/13/12
INDUCTIVELY COUPLED PLASMA SPECTROMETER	1	08/07/12

Softwares
TURBOCHROM - GC
PERKIN ELMER - ICP
PERKIN ELMER - AA
CHROMELEON (DIONEX) - IC
CHEMSTATION - GC/MS
VARIAN-MS-WORKSTATION - GC/MS
TOX-10E MITSUBISHI CHEMICAL CORPORATION
PIC MDS
CHROMELEON GC
CHROMELEON HPLC
PERKIN ELMER FLOW INJECTION SYSTEM
FIMS 400

Arizona Department of Health Services

ENVIRONMENTAL LABORATORY LICENSE

Issued to:

Laboratory Director: **Ed Wilson**
Owner/Representative: **Ed Wilson**

Eurofins Eaton Analytical, Inc.
AZ0778

is in compliance with Environmental Laboratory's applicable standards for the State of Arizona and maintains on file a List of Parameters for which the laboratory is certified to perform analysis.

PERIOD OF LICENSURE FROM: 12/15/2012 TO: 12/14/2013

Steven D. Baker, Chief
Office of Laboratory Licensure & Certification
Bureau of State Laboratory Services

APPENDIX II

Glossary
EEA Vendor List

Appendix II: Glossary

Acceptance Criteria: Specified limits placed on characteristics of an item, process, or service defined in requirement documents.

Acceptance Criteria: Specified limits placed on characteristics of an item, process, or service defined in requirement documents.

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.

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 that are due to sampling and analytical operations; a data quality indicator.

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.

Assessment: The evaluation process used to measure or establish the performance, effectiveness, and conformance of an organization and/or its systems to defined criteria (to the standards and requirements of laboratory accreditation).

Audit: A systematic and independent examination of facilities, equipment, personnel, training, procedures, record-keeping, data validation, data management, and reporting aspects of a system to determine whether QA/QC and technical activities are being conducted as planned and whether these activities will effectively achieve quality objectives.

Batch: Environmental samples that 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 (1) to twenty (20) environmental samples of the same quality systems 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 twenty-four (24) hours. An **analytical batch** is composed of prepared environmental samples (extracts, digestates or concentrates) which are analyzed together as a group. An analytical batch can include prepared samples originating from various quality system matrices and can exceed twenty (20) samples.

Bias: The systematic or persistent distortion of a measurement process, which causes errors in one direction (i.e., the expected sample measurement is different from the sample's true value).

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.

Calibration: A set of operations that establish, under specified conditions, the relationship between values of quantities indicated by a measuring instrument or measuring system, or values represented by a material measure or a reference material, and the corresponding values realized by standards.

Calibration Blank (CB): A volume of reagent water fortified with the same matrix as the calibration standards, but without the analytes, internal standards, or surrogates analytes.

Calibration Curve: The mathematical relationship between the known values, such as concentrations, of a series of calibration standards and their instrument response.

Calibration Standard (CAL): A solution prepared from the primary dilution standard solution and stock standard solutions of the internal standards and surrogate analytes. The CAL solutions are used to calibrate the instrument response with respect to analyte concentration.

Chain of Custody Form: Record that documents the possession of the samples from the time of collection to receipt in the laboratory. This record generally includes: the number and types of containers; the mode of collection; the collector; time of collection; preservation; and requested analyses. See also Legal Chain of Custody Protocols.

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.

Data Reduction: The process of transforming the number of data items by arithmetic or statistical calculation, standard curves, and concentration factors, and collating them into a more useful form.

Demonstration of Capability: A procedure to establish the ability of the analyst to generate analytical results of acceptable accuracy and precision.

Dissolved Analyte: The concentration of analyte in an aqueous sample that will pass through a 0.45 µm membrane filter assembly prior to sample acidification (Section 11.1).

Dissolved Phosphorus (P-D): All of the phosphorus present in the filtrate of a sample filtered through a phosphorus-free filter of 0.45 micron pore size and measured by the persulfate digestion procedure.

Dissolved Orthophosphate (P-D ortho): As measured by the direct colorimetric analysis procedure.

Dissolved Hydrolyzable Phosphorus (P-D, hydro): As measured by the sulfuric acid hydrolysis procedure and minus predetermined dissolved orthophosphates.

Dissolved Organic Phosphorus (P-D, org): As measured by the persulfate digestion procedure, and minus dissolved hydrolysable phosphorus and orthophosphate

Estimated Detection Limit (EDL): Defined as either the MDL or a level of compound in a sample yielding a peak in the final extract with a signal to noise (S/N) ratio of approximately five, whichever is greater.

External Standard (ES): A pure analyte(s) that is measured in an experiment separate from the experiment used to measure the analyte(s) in the sample. The signal observed for a known quantity of the pure external standard(s) is used to calibrate the instrument response for the corresponding analyte(s). The instrument response is used to calculate the concentrations of the analyte(s) in the sample.

Field Duplicates (FD1 and FD2): Two separate samples collected at the same time and place under identical circumstances and treated exactly the same throughout field and laboratory procedures. Analyses of FD1 and FD2 give a measure of the precision associated with sample collection, preservation and storage, as well as with laboratory procedures. Since laboratory duplicates cannot be analyzed, the collection and analysis of field duplicates for this method is critical.

Field Reagent Blank (FRB): An aliquot of reagent water or other blank matrix that is placed in a sample container in the laboratory and treated as a sample in all respects, including shipment to the sampling site, exposure to sampling site conditions, storage, preservation, and all analytical procedures. The purpose of the FRB is to determine if method analytes or other interferences are present in the field environment.

Finding: An assessment conclusion referenced to a laboratory accreditation standard and supported by objective evidence that identifies a deviation from a laboratory accreditation standard requirement.

Holding Times: The maximum time that can elapse between two (2) specified activities.

Instrument Performance Check Solution (IPC): A solution of one or more method analytes surrogates, internal standards, or other test substances used to evaluate the performance of the instrument system with respect to a defined set of criteria.

Instrument Detection Limit (IDL): The concentration equivalent to the analyte signal which is equal to three times the standard deviation of a series of 10 replicate measurements of the calibration blank signal at the same wavelength (Table 1.)

Internal Standard: Pure analyte(s) added to a sample, extract, or standard solution in known amount(s) and used to measure the relative responses of other method analytes that are

components of the same sample or solution, and as a reference for evaluating and controlling the precision and bias of the applied analytical method. The internal standard must be an analyte that is not a sample component.

Laboratory Reagent Blank (LRB): An aliquot of reagent water or other blank matrices that are treated exactly as a sample including exposure to all glassware, equipment, solvents, reagents, internal standards, and surrogates that are used with other samples. The LRB is used to determine if method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus.

Linear Calibration Range (LCR): The concentration range over which the instrument response is linear.

Laboratory Duplicates (LD1 and LD2): Two aliquots of the same sample taken in the laboratory and analyzed separately with identical procedures. Analyses of LD1 and LD2 indicates precision associated with laboratory procedures, but not with sample collection, preservation, or storage procedures.

Laboratory Fortified Blank (LFB): 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 and taken through all sample preparation and analytical steps of the procedure unless otherwise noted in a reference method. 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.

Laboratory Fortified Sample Matrix (LFM): An aliquot of an environmental sample to which known quantities of the method analytes are added in the laboratory. The LFM is analyzed exactly like a sample, and its purpose is to determine whether the sample matrix contributes bias to the analytical results. The background concentrations of the analytes in the sample matrix must be determined in a separate aliquot and the measured values in the LFM corrected for background concentrations.

Linear Dynamic Range (LDR): The concentration range over which the instrument response to an analyte is linear.

Laboratory Performance Check Solution (LPC): A solution of selected method analytes, surrogate(s), internal standard(s), or other test substances used to evaluate the performance of the instrument system with respect to a defined set of method criteria.

Limit of Detection (LOD): The estimated minimum amount of analyte in a given matrix that an analytical process can reliably detect, but not necessarily quantitated as an exact value. The LOD may be expressed as:

LOD = 3.3 * SD / S

where:
SD = the standard deviation of the response
S = the slope of the calibration curve

Limit of Quantitation (LOQ): Also known as Minimum Reporting Level (MRL). The minimum levels, concentrations, or quantities of an analyte that can be quantitatively determined with suitable precision and accuracy.

Linear Calibration Range (LCR): The concentration range over which the instrument response is linear.

Material Safety Data Sheet (MSDS): Written information provided by vendors concerning a chemical's toxicity, health hazards, physical properties, fire, and reactivity data including storage, spill, and handling precautions.

Method Detection Limit (MDL): The minimum concentration of an analyte that can be identified, measured, and reported with 99% confidence that the analyte concentration is greater than zero (Section 9.2.4 and Table 4.). Procedural Standard Calibration - A calibration method where aqueous calibration standards are prepared and processed (e.g., purged, extracted, and/or derivatized) in exactly the same manner as a sample. All steps in the process from addition of sampling preservatives through instrumental analyses are included in the calibration. Using procedural standard calibration compensates for any inefficiencies in the processing procedure.

Matrix: The substrate of a test sample.

Matrix Duplicate: A replicate matrix prepared in the laboratory and analyzed to obtain a measure
of precision.

Matrix Spike : A sample prepared, taken through all sample preparation and analytical steps of the procedure unless otherwise noted in a referenced method, by adding a known amount of target analyte to a specified amount of sample for which an independent test result 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 Spike Duplicate (spiked sample or fortified sample duplicate): A replicate matrix spike prepared in the laboratory and analyzed to obtain a measure of the precision of the recovery for each analyte.

Method: A body of procedures and techniques for performing an activity (e.g., sampling, chemical analysis, quantification), systematically presented in the order in which they are to be executed.

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.

Minimum Reporting Level (MRL): Also known as Limit of Quantitation (LOQ). The lowest amount of analyte in a sample that can be quantitatively determined with suitable precision and accuracy.

National Institute of Standards and Technology (NIST): A federal agency of the US Department of Commerce's Technology Administration that is designed as the United States national metrology institute (NMI).

Plasma Solution: A solution that is used to determine the optimum height above the work coil for viewing the plasma (Sections 7.15 and 10.2.3).

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.

Preservation: Any conditions under which a sample must be kept in order to maintain chemical and/or biological integrity prior to analysis.

Primary Calibration Standard (PCAL): A suspension prepared from the primary dilution stock standard suspension. The PCAL suspensions are used to calibrate the instrument response with respect to analyte concentration.

Primary Dilution Standard Solution (PDS): A solution of several analytes prepared in the laboratory from stock standard solutions and diluted as needed to prepare calibration solutions and other needed analyte solutions. The following forms, when sufficient amounts of phosphorus are present in the sample to warrant such consideration, may be calculated:

Insoluble Phosphorus (P-I) = (P) - (P-D).

Insoluble Orthophosphate (P-I, ortho) = (P, ortho) - (P-D, ortho).

Insoluble Hydrolyzable Phosphorus (P-I, hydro) = (P, hydro) - (P-D, hydro).

Insoluble Organic Phosphorus (P-I, org) = (P, org) - (P-D, org).

All phosphorus forms shall be reported as P, mg/L, to the third place.

Pro-forma: Forms

Procedural Standard Calibration: A calibration method where aqueous calibration standards are prepared and processed (e.g., purged, extracted, and/or derivatized) in exactly the same

manner as a sample. All steps in the process from addition of sampling preservatives through instrumental analyses are included in the calibration. Using procedural standard calibration compensates for any inefficiency in the processing procedure.
Procedure: A specified way to carry out an activity or process. Procedures can be documented or not.

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.

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.

Proficiency Test Sample (PT): A sample, the composition of which is unknown to the laboratory and is provided to test whether the laboratory can produce analytical results within the specified acceptance criteria.

Protocol: A detailed written procedure for field and/or laboratory operation (e.g., sampling, analysis) which must be strictly followed.

Quality Assurance: An integrated system of management activities involving planning, implementation, assessment, reporting, and quality improvement to ensure that a process, item, or service is of the type and quality needed and expected by the client.

Quality Control: The overall system of technical activities that measures the attributes and performance of a process, item, or service against defined standards to verify that they meet the stated requirements established by the customer; operational techniques and activities that are used to fulfill requirements for quality; also the system of activities and checks used to ensure that measurement systems are maintained within prescribed limits, providing protection against "out of control" conditions and ensuring that the results are of acceptable quality.

Quality Control Sample (QCS): A solution of method analytes of known concentrations which is used to fortify an aliquot of LRB or sample matrix. The QCS is obtained from a source external to the laboratory and different from the source of calibration standards. It is used to check either laboratory or instrument performance.

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.

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 quality assurance (QA) and quality control (QC) activities.

Raw Data: The documentation generated during sampling and analysis. This documentation includes, but is not limited to, field notes, electronic data, magnetic tapes, untabulated sample results, QC sample results, print outs of chromatograms, instrument outputs, and handwritten records.

Reference Material: Material or substance one or more of whose property values are sufficiently homogeneous and well established to be used for the calibration of an apparatus, the assessment of a measurement method, or for assigning values to materials.

Reference Standard: Standard used for the calibration of working measurement standards in a given organization or at a given location.

Sampling: Activity related to obtaining a representative sample of the object of conformity assessment, according to a procedure.

Secondary Calibration Standards (SCAL): Commercially prepared, stabilized sealed liquid or gel turbidity standards calibrated against properly prepared and diluted formazin or styrene divinylbenzene polymers.

Selectivity: The ability to analyze, distinguish, and determine a specific analyte or parameter from another component that may be a potential interferent or that may behave similarly to the target analyte or parameter within the measurement system.

Sensitivity: The capability of a method or instrument to discriminate between measurement responses representing different levels (e.g., concentrations) of a variable of interest.

Standard: The document describing the elements of laboratory accreditation that has been developed and established within the consensus principles of standard setting and meets the approval requirements of standard adoption organizations procedures and policies.

Standard Operating Procedures (SOPs): A written document that details the method for an operation, analysis, or action, with thoroughly prescribed techniques and steps. SOPs are officially approved as the methods for performing certain routine or repetitive tasks.

Stock Standard Suspension (SSS): A concentrated suspension containing the analyte prepared in the laboratory using assayed reference materials or purchased from a reputable commercial source. Stock standard suspension is used to prepare calibration suspensions and other needed suspensions.

Solid Sample: For the purpose of this method, a sample taken from material classified as either soil, sediment or sludge.

Spectral Interference Check (SIC) Solution: A solution of selected method analytes of higher concentrations, which is used to evaluate the procedural routine for correcting known interelement spectral interferences with respect to a defined set of method criteria.

Standard Addition: The addition of a known amount of analyte to the sample in order to determine the relative response of the detector to an analyte within the sample matrix. The relative response is then used to assess either an operative matrix effect or the sample analyte concentration.

Stock Standard Solution (SSS): A concentrated solution containing one or more method analytes prepared in the laboratory using assayed reference materials or purchased from a reputable commercial source

Surrogate Analyte (SA): A pure analyte(s), which is extremely unlikely to be found in any sample, and which is added to a sample aliquot in known amount(s) before extraction or other processing and is measured with the same procedures used to measure other sample components. The purpose of the SA is to monitor method performance with each sample.

Technology: A specific arrangement of analytical instruments, detection systems, and/or preparation techniques.

Total Recoverable Analyte: The concentration of analyte determined either by "direct analysis" of an unfiltered acid preserved drinking water sample with turbidity of <1 NTU , or by analysis of the solution extract of a solid sample or an unfiltered aqueous sample following digestion by refluxing with hot dilute mineral acid(s) as specified in the method.

Total Phosphorus (P): All of the phosphorus present in the sample regardless of forms, as measured by the persulfate digestion procedure.

Total Orthophosphate (P-ortho): Inorganic phosphorus [(PO)] in the 4 -3 sample as measured by the direct colorimetric analysis procedure.

Total Hydrolyzable Phosphorus (P-hydro): Phosphorus in the sample as measured by the sulfuric acid hydrolysis procedure and minus predetermined orthophosphates. This hydrolyzable phosphorus includes polyphosphates [(P O) , (P O) , etc.] plus some organic 2 7 3 10-4 –5 phosphorus.

Total Organic Phosphorus (P-org): Phosphorus (inorganic plus oxidizable organic) in the sample as measured by the persulfate digestion procedure, and minus hydrolyzable phosphorus and orthophosphate.

Traceability: The ability to trace the history, application, or location of an entity by means of

recorded identifications. In a calibration sense, traceability relates measuring equipment to national or international standards, primary standards, basic physical constants or properties, or reference materials. In a data collection sense, it relates calculations and data generated throughout the project back to the requirements for the quality of the project.

Tuning Solution: A solution which is used to determine acceptable instrument performance prior to calibration and sample analyses.

Water Sample: For the purpose of this method, a sample taken from one of the following sources: drinking, surface, ground, storm runoff, industrial or domestic wastewater.

Verification: Confirmation by examination and objective evidence that specified requirements have been met.

Appendix II: EEA's Vendor List

Supplier	Address	Used by	Intended Use
Abraxis	54 Steamwhistle Dr Warminster, PA 18974	Microbiology	Cylindrospermopsin, Saxitoxin Supplies
Absolute Standards, Inc.	P. O. Box 5585 Hamden, Ct. 06518-0585	GCMS Lab, Inorganic Lab, HPLC/LCMS Lab	Standards
AccuStandard	125 Market Street New Haven, Ct. 06513	GCMS Lab, GC. HPLC/LCMS Lab	Standards
AF Murphy Die & Machine Co.	430 Hancock St Quincy, MA 02171	Inorganic Lab	Radiochemistry Planchetts
Agilent Technologies	Chemical Analysis Group 2850 Centerville Rd. Wilmington, De. 19808	GCMS Lab, GC Lab	Supplies, instrument maintenance, repair, technical support
Altech Associates, Inc.	P.O. Box 23 Deerfield, IL 60015	Inorganic Lab	Chemicals
American Type Culture Collection	12301 Parklawn Lane Rockville, Me. 20852	Microbiology Lab	Bacterial Controls
Anchem Scientific	104 Marty Dr. Suite 3 Buffalo, MN 55313	Inorganic Lab	
Beckman Instruments, Inc.	2500 Harbor Blvd., E-20-D Fullerton, Ca. 92634	Inorganic Lab	Instrument maintenance, repair, technical support
Biomerieux Industry	595 Anglum Rd Hazelwood, MO 63042	Microbiology Lab	BactID Supplies
Canberra Industries, Inc.	800 Research Parkway Meriden, Connecticut 06450	Radiochemistry Lab	Gamma Spectrometer Instrument Tech Support
Chem Service, Inc.	660 Tower Lane P. O. Box 310 West Chester, Pa. 19380	GC Lab	Reagents, supplies
Cole Parmer Instrument Co.	Dept CH 10464 Palatine, IL 60055	Inorganic Lab	Supplies
Cosa Instruments Corporation	84G Horseblock Road Yaphank, NY 11980	Inorganic Lab	Supplies
CPI International	P. O. Box 1290 Suisun City, Ca. 94585-1290	Inorganic Lab	Standards, Reagents
Crescent Chemical Co., Inc.	1324 Motor Parkway Hauppauge, NY 11788	Inorganic Lab	Standards, Reagents
Dionex Corporation	1228 Titan Way Sunnyvale, Ca. 94088-3603	Inorganic Lab, HPLC Lab, GC	Instrument maintenance, repair, technical support

Appendix II: EEA's Vendor List

Supplier	Address	Used by	Intended Use
Envirologix	500 Riverside Industrial Parkway Portland, Maine 04103-1486	Microbiology	Asbestos Supplies
Environmental Express LTD	490 Wando Park Blvd. P. O. Box 669 Mt. Pleasant, SC. 29464	Inorganic Lab	Standards, reagents, supplies
Environmental Resource Associates	6000 West 54th Avenue Arvada, CO 80002	Inorganic Lab	Standards
Fisher Scientific	Dept. LA21160 Pasadena, CA 91185	Inorganic Lab	Chemicals, Supplies
Full Spectrum Analytics, Inc.	5635 West Las Positas Blvd. #403 Pleasanton, Ca. 94588	GCMS Lab, GC Lab, Inorganic Lab	Instrument maintenance, repair, technical support
GE Analytical	13256 Collections Center Dr. Chicago, IL 60693	Inorganic Lab (TOC Instrument)	
GI Plastek	5 Wickers Drive Wolfeboro, NH 03894-4323	Radiochemistry Lab	Ra228 discs and plates
Glass Expansion Inc.	4 Barlows Landing, Unit #2 Pocasset, MA 02559	Inorganic Lab	Supplies
Greenwater Labs	205 Zeagler Drive Suite 302 Palatka, FL 32177	Microbiology	Toxin Standards
Hach Company	P. O. Box 389 Denver, Co. 80539	GC Lab, Inorganic Lab	Reagents, supplies
High Purity Standards	P.O. Box 41727 Charleston, SC 29423	Inorganic Lab	Standards
IDEXX Distribution Corporation	6100 E. Shellby Dr. Memphis, Tn. 38141-7602	Microbiology Lab	Microbiological media
Inorganic Ventures	195 Lehigh Ave. Ste 4 Lakewood, NJ 08701	Inorganic Lab	Supplies, Standards
Isotope Products Laboratories	1800 North Keystone Street Burbank, Ca. 91504	Inorganic Lab	Standards
Lab Safety Supply - WI	P.I. Box 5004 Janesville, WI 53547	Inorganic Lab, Health and Safety Department	Safety equipment
Lachat Instruments	5566 Collections Center Dr Chicago, IL 60693	Inorganic Lab	Supplies
Man-Tech Associates Inc.	600 Main St. Tonawanda, NY 14150	Inorganic Lab	Supplies
McBain Instruments	9601 Variel Ave. Chatsworth, Ca. 91311-4914	Microbiology Lab	Instrument maintenance, repair

Appendix II: EEA's Vendor List

Supplier	Address	Used by	Intended Use
Metrohm USA	6555 Pelican Creek Circle Riverview FL, 33578	Inorganic Lab	IC instrument
Miele Professional	9 Independence Way Princeton, NJ 08540	Dishwashing	Supplies
NSI Solutions	7212 ACC Blvd Raleigh NC, 27617	Inorganic Lab	Standards
National Research Council Canada	1200 Montreal Rd M-58 Ottawa, Ontario KIA 0R6 Canada	Inorganic Lab	CRMs
OI Analytical	P. O. Box 9010 151 Graham Road College Station, Tx. 77842-0440	GCMS Lab	Instrument maintenance, repair, technical support, supplies, chemicals
Perkin Elmer	761 Main Ave. Norwalk, Ct. 06859-0001	Inorganic Lab	Instrument maintenance, repair, technical support
Phenomenex	411 Madrid Avenue Torrance, CA 90501	HPLC/LCMS Lab	Supplies
Pickering Laboratories, Inc	1280 Space Park Way Mountain View, CA 94043	HPLC/LCMS Lab	Instrument supplies
Precision Glassblowing	14775 E. Hinsdale Ave. Centennial, CO 80112	Inorganic Lab, Microbiology	Supplies
Protean Instrument Corporation	P. O. Box 1008 260 Grand Street Lenoir City, Tn. 37771-1008	Inorganic Lab	Instrument maintenance, repair, technical support
Protocol Analytical Supplies, Inc.	472 Lincoln Blvd. Middlesex, NJ 08846	GCMS Lab	Standards
Restek Corporation	Penn Eagle Industrial Park 110 Benner Circle Bellefonte, Pa. 16823-8812	GC Lab, HPLC Lab, GCMS Lab	Reagents, supplies
Scientific Instrument, SIS	1027 Old York Road Ringoes, NJ 08551-1039	GCMS Lab	Supplies
SCP Science	348 Route 11 Champlain, NY 12919	Inorganic Lab	Standards, Supplies
SEAL Analytical, Inc	1492 Mequon Road Mequon, WI 53092	Inorganic Lab	Supplies
Sigma_Aldrich, Inc.	P. O. Box 952968 St. Louis, Mo. 63195-2968	Inorganic Lab, GCMS Lab, GC	Standards, Reagents, supplies

Appendix II: EEA's Vendor List

Supplier	Address	Used by	Intended Use
Spectrum Laboratories, Inc. dba	755 Jersey Ave. New Brunswick, NJ 08901	Inorganic Lab	Supplies
STS, Inc	541N Main St. 104-353 Corona, CA 92880	Microbiology	Autoclave Maintenance and Supplies
Ted Pella	4595 Mountain Lakes Blvd Redding, CA 96003-1448	Microbiology	Asbestos Supplies
Tekmar Company	7143 East Kemper Road Cincinnnati, Oh. 45249	GCMS Lab	Instrument maintenance, repair, technical support, supplies, chemicals
T.G. Scientific Glass	23041 La Cadena Dr. Laguna Hills, CA 92653	GCMS Lab	Supplies
Davis Inotek	5730 Ayala Ave. Irwindale, Ca. 91703	Quality Assurance Department	Calibration of reference thermometers
Thermo Optek Corporation	Service Operations Drawer CS 100623 Atlanta, Ga. 30384-0623	Inorganic Lab, GCMS Lab	Instrument maintenance, repair, technical support
Ultra Scientific	250 Smith Street North Kingstown, RI 02852-7723	Inorganic Lab, GCMS Lab, GC Lab, HPLC Lab, QA Department	Standards, supplies, reagents
Varian	Chromatography Systems 2700 Mitchell Drive Walnut Creek, Ca. 94598	GC Lab, GCMS Lab	Instrument maintenance, repair, technical support, supplies, chemicals
VWR Scientific Products Corporation*	P. O. Box 640169 Pittsburgh, Pa. 15264-0169	Inorganic Lab, GCMS Lab, GC Lab, QA Department, Microbiology, HPLC/LCMS Lab	Standards, reagents, supplies, standard thermometers
Waters Corporation	34 Maple Street Milford, MA 01757	HPLC/LCMS Lab	Instrument Supplies
Watson Brothers, Inc.	1235 South Victory Blvd. Burbank, Ca. 91502	Quality Assurance Department	Maintenance and calibration of the laboratory's balances and S class weights
WestAir Gases and Equipment		All Labs	Reagents, Supplies

*VWR supplies EEA with reagents, standards and supplies from many companies, including but not limited to the following:
JT Baker, Mallinckrodt, Difco, Becton Dickinson, Ricca, Gelman, J & W Scientific, Ultra Scientific, EM Science

Supplier	**Address**	**Used by**	**Intended Use**
Post Security		Facilities Management	Fire alarm panel maintenance
Iron Mountain	P.O. Box 65017 Charlotte, NC 28265-0017	All Departments	Archiving and off-site data storage
MOE Plumbing		Facilities Management	Building maintenance
Post Alarm		Facilities Management	Building security, escorts
Viking Refrigeration	1770 East Cypress Covina, CA 91724	Facilities Management	Refrigerator maintenance
DuraCold	1551 S. Primrose Lane Monrovia, CA 91016	Facilities Management, Sample Control Department	Walk-in coolers, storage refrigerator maintenance
Westway Electrical Systems		Facilities Management	Building maintenance

ATTACHMENT A2

TESTAMERICA QUALITY ASSURANCE MANUALS AND SOPS

Title: Particle Size Analysis (ASTM D 2217 and D422-63)

Approval Signatures:

William S. Cicero
Laboratory Director

Christopher G. Callahan
Department Manager

Kirstin L. McCracken
Quality Assurance Manager

Bryce E. Stearns
Technical Director

Dan W. Helfrich
Health & Safety Coordinator

Approval Date: March 17, 2010

Facility Distribution No. Electronic **Distributed To: Facility Intranet**

The controlled copy of this SOP is the PDF copy of the SOP that is posted to the laboratory's SOP Directory. Printed copies of this SOP or electronic copies of this SOP distributed outside the facility are considered uncontrolled.

SOP REVIEW FORM

SOP Number	Revision	Effective Date:	Title
GR-GT-006	5	03/17/10	Particle Size Analysis

Review Statement:

My signature signifies that I reviewed and compared the above referenced SOP against current bench practice.

			Revision Needed	
Date	Reviewer	Job Title	Yes[1]	No
04/03/12	Christopher Calloway	Dept Manager		X
04/03/12	Eric Lee	Analyst		X

[1] *List the section for revision and the the reason using the revision summary page and attach to this cover sheet.*

QA Use Only:

X The SOP was reviewed and does not require revision. Attach this form to the SOP.

____ The SOP Revision will be made with a Change in Progress Attachment (CIPA).

____ The SOP Revision will be released as a new version of the SOP.

____ The SOP Revision requires method validation or demonstration of capability

____ The SOP Revision does not reqire method validation or demonatration of capability.

____ The SOP revison affects other SOPs that must now also be revised (List SOPs)

Kristin Daigle
QA Signature

4/3/2012
Date

Comments:

1.0 Scope and Application

This SOP describes the laboratory procedure for the determination of particle size distribution in soils.

2.0 Summary of Method

A portion of sample is soaked in a dispersing agent then partitioned into separate portions, material retained on a #10 sieve and material passing the #10 sieve. The material retained on the #10 sieve is dried to constant weight then passed through a large size sieve stack; the material retained on each sieve is measured and recorded. Material passing the #10 sieve is subject to hydrometer analysis then passed through a small size sieve stack, the material retained on each sieve is measured and recorded. All measurements, large and small sieves and hydrometer readings and the hygroscopic moisture are used to establish the particle size distribution of the sample.

This SOP is based on the following reference methods:

- ASTM Standard D 2217 – 85 (Rapproved 1998) "Standard Practice for Wet Preparation of Soil Samples for Particle-Size Analysis and Determination of Soil Constants", ASTM International, West Conshohocken, PA 2003, DOI: 10.1520/C0033-03, www.astm.org

- ASTM Standard D 422-63 (Rapproved 2007) "Standard Test Method for Particle-Size Analysis of Soils", ASTM International, West Conshohocken, PA 2003, DOI: 10.1520/C0033-03, www.astm.org

NOTE: ASTM D2217 was method was withdrawn without replacement by ASTM in 2007. A withdrawn standard is an ASTM standard that has been discontinued by the ASTM Sponsoring Committee responsible for the standard.

If the laboratory has modified the procedure from the reference method(s) a list of modifications will be provided in Section 16.0.

3.0 Definitions

Not Applicable

4.0 Interferences

Not Applicable

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001) 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 coats and closed-toe, nonabsorbent shoes are a minimum.

5.1 Specific Safety Concerns or Requirements

None

5.2 Primary Materials Used

Not Applicable

6.0 Equipment and Supplies

Catalog numbers listed in this SOP are subject to change at the discretion of the vendor. Analysts are cautioned to be sure equipment used meets the specification of this SOP.

- Top-Loading Balance, capable of weight measurement to 0.01 g
- Mechanical Stirring Device and Dispersion Cup
- Thermometer: Accurate to 0.5°C
- Mortar and Rubber Tipped Pestle
- Sedimentation Cylinder(s) 1000 mL
- Hydrometer: ASTM 151H in specification E 100.
- Sieves, of the following size(s): Gilson Company, Inc. or equivalent
  3.0” (75.00 mm)
  2.0” (50.00 mm)
  1.5” (37.50 mm)
  1.0” (25.00 mm)
  3/4" (19.00 mm)
  3/8” (9.50 mm)
  # 4 (4.75 mm)
  #10 (2.00 mm)
  #20 (850.0 um)
  #40 (425 um)
  #60 (250.0 um)
  #80 (180.0 um)
  #100 (150.0 um)
  #200 (75.0 um)
- Drying Oven with temperature range of 60-110°C
- Stainless Steel Spatulas & Spoons
- Metal & Bristle Brushes
- Ro-Tap Sieve Shaker, W. S. Tyler or equivalent.
- Timing Device with second hand and capable of counting up to 25 hours

7.0 Reagents and Standards

- Reverse Osmosis (RO) water: In-House System
- Sodium Hexametaphosphate: ELE International or equivalent.

Sodium Hexametaphosphate Solution: Add 120 g of sodium hexametaphosphate and 2940 g of reagent water to a 1-gallon bottle. Add a stir rod to the container and place on a stir plate. Mix the solution until it is homogeneous. Assign an expiration date of 30 days from the date made

unless the parent reagent expires sooner in which case use the earliest expiration date. Store the prepared solution at ambient temperature.

8.0 Sample Collection, Preservation, Shipment and Storage

The laboratory does not perform sample collection so these procedures are not included in this SOP. Sampling requirements may be found in the published reference method.

Listed below are minimum sample size, preservation and holding time requirements:

Matrix	Sample Container	Minimum Sample Size	Preservation	Holding Time	Reference
Solid	Glass Jar w/ Teflon Lid	500 g	None	None	ASTM D422-63

Unless otherwise specified by client or regulatory program, after analysis, samples and extracts are retained for a minimum of 30 days after provision of the project report and then disposed of in accordance with applicable regulations.

9.0 Quality Control

Not Applicable

10.0 Procedure

10.1 Equipment Calibration

Check the calibration of the balance on each day of use prior to use using at least 2 Class S weights that bracket the range of use. Record in the logbook designated for this purpose.

Check the temperature of the drying oven(s) each day of use, prior to use. Record in the logbook designated for this purpose.

NOTE: The QA Manager or her designee checks the calibration of liquid in glass thermometers annually against a NIST-traceable thermometer following the procedures given in laboratory SOP BR-QA-004. Electronic / digital thermometers that are battery-operated are checked quarterly using the same procedure.

Calibrate the hydrometers every two years following the procedure given in BR-GT-008.

Calibrate the sieves 6 months following the procedure given in BR-GT-008.

Calibrate the Ro-Tap sieve shaker every 12 months following the procedure given in BR-GT-008.

10.2 Hygroscopic Moisture Determination

Label an aluminum pan with the Lab ID for each sample. Tare the balance, weigh each pan and record the weight measurement in the spreadsheet.

Mix the sample with a stainless steel spatula. Measure at least 10-15 g of each sample into the labeled aluminum pan and record the weight of sample in the spreadsheet.

Place the pan + sample in an oven maintained at a temperature of 110ºC and dry the sample for at least 16 hours. Reweigh each pan and record the weight measurement in the spreadsheet.

Percent solids are calculated using the equation given in Section 11.0.

10.3 Sample Preparation

Use the calculated percent solids and the sample characteristic for each sample to determine the amount needed for analysis using Table 2. For example, if the calculated percent solids for a sample are 50% and the sample characteristic is sand, use 200 g for analysis. If there is an insufficient amount of sample available, initiate a nonconformance memo (NCM) and contact the PM for further instruction.

Place a 1000 mL plastic beaker on the balance and tare the balance. Weight the amount of sample for analysis and record the weight in the bench sheet.

Add 125 mL of sodium hexametaphosphate solution to each beaker. Stir to mix and soak the sample in this solution for 16 hours

10.4 Sample Partition

Rinse the sample slurry into a dispersion cup using reagent water. Fill the dispersion cup ½ full with reagent water and place the cup on the blender to mix for one minute.

NOTE: Some samples may not be amendable to using the blender examples include but not limited to large gravel, sands, or organic material. If the sample is not amenable, initiate a NCM to notify the PM of the anomaly and proceed to the next step without blending the sample.

Place a #10 sieve on a 1000 mL graduated cylinder. Pour the sample through the sieve. Rinse the dispersion cup with reagent water and pour the rinse through the sieve. Repeat until transfer is complete. Bring the volume in the graduated cylinder to 1000 mL with reagent water. Cover the cylinder with a rubber stopper and equilibrate the sample to ambient temperature in preparation for hydrometer analysis.

Label a medium size aluminum dish with the sample's LAB ID then transfer the sample material that was retained on the #10 sieve to the dish. Place the aluminum dish in the drying oven set at 110 ± 5° C and dry the sample material for at least 16 hours or until constant weight. Set aside for sieve analysis.

10.5 Hydrometer Analysis

Prepare a hydrometer rinse bath by adding 1000 mL of reagent water to a 1000 mL graduated cylinder

Record the hydrometer ID and start time on the worksheet. Set the timer for the elapsed time and perform each task as listed in Table 1: Hydrometer Reading Table.

To shake the cylinder, rotate the flask up and down for one minute approximating at least 60 turns. One turn down and one turn up equals two turns.

To take a hydrometer reading, gently insert the hydrometer into the graduated cylinder and wait ~ 20 seconds. Read the hydrometer from the top of the meniscus to the nearest 0.0005. Enter the reading on the worksheet. After each reading, clean the hydrometer by twisting and dropping the hydrometer into the hydrometer rinse bath.

Insert a temperature probe into the cylinder to the same depth used for the hydrometer reading. Read the temperature to the nearest 0.5°C and enter the temperature measurement on the worksheet. Rinse the temperature probe in the hydrometer rinse bath.

Repeat the above process taking hydrometer readings every 2, 5, 15, 30, 60, 240 and 1440 minutes as per Table 1 then proceed to small sieve analysis.

10.6 Sieve Analysis

Inspect the sample material in the aluminum pan and record a description of the non-soil material (e.g.- sticks, grass, wood, plastic), hardness of material and shape of material in the worksheet.

Hardness qualifiers include hard, soft or brittle. Shape qualifiers include well rounded, rounded, subrounded, subangular, and angular.

Large Sieves

Weigh the 3/4", 3/8", #4 and #10 sieves and enter the weight measurements in the worksheet as the tare weight.

Stack the sieves then transfer the sample material from the aluminum dish to the sieve stack. If the sample material is less than 30 g, manually shake the sieve stack for 2 minutes. If the sample material is greater than 30 g, place the sieve stack into the Ro-tap machine and shake the sieve stack for 10 minutes.

Weigh each sieve and record these measurements in the worksheet.

Small Sieves

Transfer the sample from the graduated cylinder to a #200 wet wash sieve. Wash the sample through the #200 sieve until the water runs clear then transfer the material retained on the sieve to a 250 mL glass beaker labeled with the sample's LAB ID.

Place the beaker in the drying oven and dry at a temperature of 110ºC for at least 16 hours. After 16 hours, remove the beaker from the oven and allow it to cool.

Gently mix the dried contents of the beaker with a rubber-tipped pestle to break any soil aggregates that may have formed during the drying stage.

Tare the balance and weigh the sieve stack sized between #20 and #200 and record the tare weights.

Transfer the sample to the sieve stack and ensure complete transfer. Use hair or wire brushes to clean the beaker. Place the sieve stack on the Rotap machine and shake for ten minutes.

Weigh each sieve and record these measurements in the worksheet.

11.0 Calculations / Data Reduction

11.1 Calculations

Sample Used (SU): Dry Preparation

$$SU = (pan + dry\ sample\ -\ pan) - (pan + non\ -\ soil\ material\ -\ pan) \otimes HMCF$$

Where:
HMCF = Hygroscopic moisture correction factor.

Sieve Analysis (Percent Finer = PF)

Large Sieves:

3 inch: PF = 100-100 (Sieve and Sample (3 inch) - Sieve (3 inch))/SU*

2 inch: PF = PF (3 inch) - 100(Sieve and Sample (2 inch) - Sieve (2 inch))/SU and so on through the #10 Sieve.*

Small Sieves:

#20: PF = PF(#10) - 100(mass passing #10/sample mass (Hyd))*(sieve and sample (#20) - sieve(#20))/sample used*

#40: PF = PF (#20) - 100(mass passing #10/sample mass (Hyd))*(sieve and sample (#40) - sieve (#40))/sample used and so on up through #10 sieve.*

Hydrometer Analysis

Particle size, Micron

1000*sqrt [930*viscosity/980*(SG-1))*(effective depth/time)]

Viscosity at sample temperature, poises
Effective Depth, cm = 16.29-264.5*(actual Hydrometer reading - 1) above equation for effective depth based on equation found with table 2 in method, in which 16.29 = 0.5*(14.0-67.0/27.8)+10.5 and 264.5 = (10.5-2.3)/0.031
Time, minutes = Time of hydrometer reading from beginning of sedimentation
Sqrt - square root
SG - Specific Gravity of soil
Viscosity - is the resistance of a liquid to flow
Percent Finer (PF):

PF = Constant*(actual hydrometer reading - hydrometer correction factor - 1)

Where:
Constant = (100,000/W)*SG/(SG-1)
W = (Total sample used *sample used for hydrometer analysis*HMCF)/Amount of total sample

passing #10 sieve
Hydrometer Correction = slope*sample temperature + Intercept
Slope = ((low temp. reading -1)-(high temp. reading -1)/(low temp. - high temp.))
Intercept = (low temp. reading -1) - (low temp. * slope)

11.2 Data Reduction

11.2.1 Primary Data Review

Review project documents such as the Project Plan (PP), Project Memo or any other document/process used to communicate project requirements to ensure those project requirements were met. If project requirements were not met, immediately notify the project manager (PM) to determine an appropriate course of action.

Upload the batch information into LIMS and complete the batch editor and worksheet. Initiate NCMs for any anomalies observed during the preparation process. Set the status of the batch to 1st level review.

11.2.2 Secondary Data Review

Review project documents such as the Project Plan (PP), Project Memo or any other document/process used to communicate project requirements and verify those project requirements were met. If project requirements were not met, immediately notify the project manager (PM) to determine an appropriate course of action.

Check the batch editor and worksheet to verify the batch is complete and any outages are documented with an NCM along with the results of any corrective actions taken. Set the status of the batch to second level review.

11.2.3 Lab Complete

Review the batch, run QC checker as appropriate and set the status to lab complete.

11.2.4 Data Reporting

Sample results are reported from the laboratory's LIMS system using the formatter specified by the Project Manager.

11.2.5 Data Archival

Data are stored in the laboratory's LIMS system.

12.0 Method Performance

Not Applicable

13.0 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."

14.0 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 BR-EH-001. The following waste streams are produced when this method is carried out.

- Solid Waste-Satellite Container: Solid Waste 5 Gallon Plastic Bucket (inside fume hood)
- Liquid Waste- 55 gallon poly drum

15.0 References / Cross-References

- ASTM Standard D 2217 – 85 (Reapproved 1998) "Standard Practice for Wet Preparation of Soil Samples for Particle-Size Analysis and Determination of Soil Constants", ASTM International, West Conshohocken, PA 2003, DOI: 10.1520/C0033-03, www.astm.org

- ASTM Standard D 422-63 (Rapproved 2007) "Standard Test Method for Particle-Size Analysis of Soils", ASTM International, West Conshohocken, PA 2003, DOI: 10.1520/C0033-03, www.astm.org

16.0 Method Modifications

D2217: The laboratory performs sample portioning after soaking the solution in the dispersing agent because the dispersion agent helps break up aggregates associated with clay and sediments.

D422: The laboratory does not always use the recommended amount of sample for analysis because sufficient sample volume is not always received.

17.0 Attachments

- Table 1: Hydrometer Reading Table (For up to 12 Sedimentation Cylinders)
- Table 2: Percent Solids Table for Weight Determination for D422.

18.0 Revision History

BR-GT-006, Revision 6:

- Title Page: Updated approval signatures
- All Sections: Removed references to dry preparation by ASTM D421; Added procedure for wet preparation.
- Attachments: Inserted Percent Solids Table

Table 1: Hydrometer Reading Table (For up to 12 Sedimentation Cylinders)

Elapsed Time (hr:min)	Task	Cyl. No.	Actual Time (min)	Elapsed Time (hr:min)	Task	Cyl. No.	Actual Time (min)
0:00	Shake	1		1:01	Read	10	5
0:01	**Place**	**1**		**1:02**	Shake	11	
0:01	**Shake**	**2**		**1:03**	Place	11	
0:02	Place	2		1:04	Read	9	15
0:03	Read	1	2	1:05	Read	11	2
0:04	Read	2	2	1:06	Read	7	31
0:06	Read	1	5	1:07	Read	3	58
0:07	Read	2	5	1:08	Read	11	5
0:08	Shake	3		**1:09**	Shake	12	
0:09	**Place**	**3**		**1:10**	Place	12	
0:09	**Shake**	**4**		1:11	Read	10	15
0:10	Place	4		1:12	Read	12	2
0:11	Read	3	2	1:13	Read	4	63
0:12	Read	4	2	1:14	Read	8	32
0:14	Read	3	5	1:15	Read	12	5
0:15	Read	4	5	1:18	Read	11	15
0:16	Read	1	15	1:19	Read	9	30
0:17	Read	2	15	1:21	Read	5	60
0:20	Shake	5		1:25	Read	12	15
0:21	Place	5		1:26	Read	10	30
0:23	Read	5	2	1:27	Read	6	59
0:24	Read	3	15	1:33	Read	11	30
0:25	Read	4	15	1:34	Read	7	59
0:26	Read	5	5	1:41	Read	12	31
0:27	Shake	6		1:42	Read	8	60
0:28	Place	6		1:52	Read	9	63
0:30	Read	6	2	1:53	Read	10	57
0:31	Read	1	30	2:06	Read	11	63
0:32	Read	2	30	2:07	Read	12	57
0:33	Read	6	5	4:17	Read	1	256
0:34	Shake	7		4:18	Read	2	256
0:35	Place	7		4:19	Read	3	250
0:36	Read	5	15	4:20	Read	4	250
0:37	Read	7	2	4:21	Read	5	240
0:38	Read	3	29	4:22	Read	6	234
0:39	Read	4	29	5:00	Read	7	265
0:40	Read	7	5	5:01	Read	8	259
0:41	Shake	8		5:02	Read	9	253
0:42	Place	8		5:03	Read	10	247
0:43	Read	6	15	5:04	Read	11	241
0:44	Read	8	2	5:05	Read	12	235
0:47	Read	8	5	24:01	Read	1	1440
0:48	Shake	9		24:02	Read	2	1440
0:49	Place	9		24:03	Read	3	1434
0:50	Read	7	15	24:04	Read	4	1434
0:51	Read	9	2	24:05	Read	5	1424
0:52	Read	5	31	24:06	Read	6	1418
0:54	Read	9	5	24:07	Read	7	1412
0:55	Shake	10		24:08	Read	8	1406

0:56	Place	10		24:09	Read	9	1400
0:57	Read	8	15	24:10	Read	10	1394
0:58	Read	10	2	24:11	Read	11	1388
0:59	Read	6	31	24:12	Read	12	1382
1:00	**Read**	**1**	**59**				
1:00	**Read**	**2**	**58**				

Source: Laboratory Prepared Reference Document

Table 2: Percent Solids Table for Weight Determination for D422.

Percent Solid Table

Quantities of sample (in grams) to be utilized in Wet method version of ASTM D854 and D422

% Sol	Spec Grav	Hydrometer Slt/Cl	Slt/Snd	Snd	Snd/Gr
	25	**50**	**75**	**100**	**200**
1	2500	5000	7500	10000	20000
2	1250	2500	3750	5000	10000
3	833	1667	2500	3333	6667
4	625	1250	1875	2500	5000
5	500	1000	1500	2000	4000
6	417	833	1250	1667	3333
7	357	714	1071	1429	2857
8	313	625	938	1250	2500
9	278	556	833	1111	2222
10	250	500	750	1000	2000
11	227	455	682	909	1818
12	208	417	625	833	1667
13	192	385	577	769	1538
14	179	357	536	714	1429
15	167	333	500	667	1333
16	156	313	469	625	1250
17	147	294	441	588	1176
18	139	278	417	556	1111
19	132	263	395	526	1053
20	125	250	375	500	1000
21	119	238	357	476	952
22	114	227	341	455	909
23	109	217	326	435	870
24	104	208	313	417	833
25	100	200	300	400	800
26	96	192	288	385	769
27	93	185	278	370	741
28	89	179	268	357	714
29	86	172	259	345	690
30	83	167	250	333	667
31	81	161	242	323	645
32	78	156	234	313	625
33	76	152	227	303	606
34	74	147	221	294	588
35	71	143	214	286	571
36	69	139	208	278	556
37	68	135	203	270	541
38	66	132	197	263	526
39	64	128	192	256	513
40	63	125	188	250	500
41	61	122	183	244	488
42	60	119	179	238	476
43	58	116	174	233	465
44	57	114	170	227	455
45	56	111	167	222	444
46	54	109	163	217	435
47	53	106	160	213	426
48	52	104	156	208	417
49	51	102	153	204	408
50	50	100	150	200	400

% Sol	Spec Grav	Hydrometer Slt/Cl	Slt/Snd	Snd	Snd/Gr
	25	**50**	**75**	**100**	**200**
51	49	98	147	196	392
52	48	96	144	192	385
53	47	94	142	189	377
54	46	93	139	185	370
55	45	91	136	182	364
56	45	89	134	179	357
57	44	88	132	175	351
58	43	86	129	172	345
59	42	85	127	169	339
60	42	83	125	167	333
61	41	82	123	164	328
62	40	81	121	161	323
63	40	79	119	159	317
64	39	78	117	156	313
65	38	77	115	154	308
66	38	76	114	152	303
67	37	75	112	149	299
68	37	74	110	147	294
69	36	72	109	145	290
70	36	71	107	143	286
71	35	70	106	141	282
72	35	69	104	139	278
73	34	68	103	137	274
74	34	68	101	135	270
75	33	67	100	133	267
76	33	66	99	132	263
77	32	65	97	130	260
78	32	64	96	128	256
79	32	63	95	127	253
80	31	63	94	125	250
81	31	62	93	123	247
82	30	61	91	122	244
83	30	60	90	120	241
84	30	60	89	119	238
85	29	59	88	118	235
86	29	58	87	116	233
87	29	57	86	115	230
88	28	57	85	114	227
89	28	56	84	112	225
90	28	56	83	111	222
91	27	55	82	110	220
92	27	54	82	109	217
93	27	54	81	108	215
94	27	53	80	106	213
95	26	53	79	105	211
96	26	52	78	104	208
97	26	52	77	103	206
98	26	51	77	102	204
99	25	51	76	101	202
100	25	50	75	100	200

Quality Assurance Manual

TestAmerica Burlington
30 Community Drive, Suite 11
South Burlington, VT 05403
Phone: (802) 660-1990
Fax: (802) 660-1919

www.testamericainc.com

Facility Distribution No. Electronic **Distributed To: Facility Server**

The controlled copy of this document is the PDF copy of the document posted to the laboratory's controlled distribution SOP Directory. Printed copies of this document or electronic copies of this document distributed outside the facility are considered uncontrolled.

Quality Assurance Manual
Approval Signatures

Laboratory Director – Kirstin L. Daigle	February 4, 2013 Date
Quality Assurance Manager – Sara S. Goff	February 4, 2013 Date
Technology Manager – Bradley W. Chirgwin	February 4, 2013 Date

SECTION 2. TABLE OF CONTENTS

LIST OF TABLES

LIST OF FIGURES

LIST OF APPENDICES

REFERENCED CORPORATE SOPs AND POLICIES

SOP / Policy Reference	Title
CA-Q-S-001	Solvent and Acid Lot Testing and Approval
CA-Q-S-002	Acceptable Manual Integration Practices
CA-Q-S-004	Method Compliance & Data Authenticity Audits
CA-Q-S-006	Detection Limits
CA-Q-S-008	Management Systems Review
CW-Q-S-001	Corporate Document Control and Archiving
CW-Q-S-002	Writing a Standard Operating Procedure (SOPs)
CW-L-S-002	Internal Investigation of Potential Data Discrepancies and Determination for Data Recall
CA-L-S-002	Subcontracting Procedures
CW-L-P-004	Ethics Policy
CA-L-P-002	Contract Compliance Policy
CW-F-P-002	Authorization Matrix
CW-F-P-004	Procurement and Contracts Policy
CA-C-S-001	Work Sharing Process
CA-T-P-001	Qualified Products List
CW-F-S-007	Controlled Purchases Policy
CW-F-S-018	Vendor Selection
CA-Q-M-002	Corporate Quality Management Plan
CW-E-M-001	Corporate Environmental Health & Safety Manual

REFERENCED LABORATORY SOPs

SOP Reference	Title
BR-QA-003	Document Control
BR-QA-004	Complaint Resolution
BR-QA-011	Employee Training and Demonstration of Proficiency
BR-QA-005	Detection Limits, Limit of Detection and Limit of Quantitation
BR-QA-006	Manual Integration
BR-QA-020	Sample Homogenization and Subsampling
BR-SM-001	Sample Management

SECTION 3. INTRODUCTION, SCOPE AND APPLICABILITY

3.1 Introduction and Compliance References

TestAmerica Burlington'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 NELAC Institute (TNI) Standard, dated 2009, Volume 1 Modules 2 and 4, and ISO/IEC Guide 17025:2005(E). 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 3. 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 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.*
- *U.S. Department of Defense, Quality Systems Manual for Environmental Laboratories, Version 4.2, October 2010.*
- Federal Register, 40 CFR Parts 136, 141, 172, 173, 178, 179 and 261.
- *Manual for the Certification of Laboratories Analyzing Drinking Water (EPA 815-R-05-004, January 2005) (DW labs only)*
- APHA, *Standard Methods for the Examination of Water and Wastewater,* 18th Edition, 19th, 20th, 21st, and on-line Editions.
- *U.S. Department of Energy* Order 414.1B, *Quality Assurance*, Approved April 29, 2004.
- *U.S. Department of Energy* Order 414.1C, *Quality Assurance*, June 17, 2005.
- *U.S. Department of Energy*, *Quality Systems for Analytical Services*, Revision 3.6, November 2010.
- U.S. Department of Defense, *Air Force Center for Environmental Excellence Quality Assurance Project Plan (QAPP),* Version 4.0.02, May 2006.
- Nuclear Regulatory Commission (NRC) Quality Assurance Requirements.
- Marine Protection, Research, and Sanctuaries Act (MPRSA).
- Toxic Substances Control Act (TSCA).

3.2 Terms and Definitions

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 2 for the Glossary/Acronyms.

3.3 Scope / Fields of Testing

The laboratory analyzes a broad range of environmental and industrial samples every month. Sample matrices vary among air, drinking water, effluent water, groundwater, hazardous waste, sludge, soils, sediments, tissue and other biological matrices. 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 processes, reviewing results, servicing clients and tracking samples through the laboratory. The technical and service requirements of all analytical requests 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 performed by the laboratory can be found on the company's data portal, Total Access, or from a representative of the laboratory. The approach of this manual is to define the minimum level of quality assurance and quality control necessary to meet these 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 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

The template on which this manual is based is reviewed annually by Corporate Quality Management Personnel to assure that it remains in compliance with Section 3.1. The manual itself 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 and approved by the senior laboratory management staff according to the laboratory's Document Control procedure (SOP No. BR-QA-003).

SECTION 4. MANAGEMENT REQUIREMENTS

4.1 Overview

TestAmerica Burlington 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 etc.). The laboratory operational and support staff work under the direction of the Laboratory Director. The organizational structure for both Corporate & TestAmerica Burlington is presented in Figure 4-1.

4.2 Roles and Responsibilities

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 briefly define each role in its relationship to the Quality Assurance Program.

4.2.1 Additional Requirements for Laboratories

The responsibility for quality resides 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 TestAmerica's Burlington laboratory.

4.2.2 Quality Assurance (QA) Manager or Designee

The QA Manager has responsibility and authority to ensure the continuous implementation of the quality system.

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 (e.g., 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 QA staff to accomplish specific responsibilities, which include, but are not limited to:

- Serves as the focal point for QA/QC in the laboratory.
- Having functions independent from laboratory operations for which he/she has quality assurance oversight.
- Maintaining and updating the QAM.
- Monitoring and evaluating laboratory certifications; scheduling proficiency testing samples.

- Monitoring and communicating regulatory changes that may affect the laboratory to management.
- Training and advising the laboratory staff on quality assurance/quality control procedures that are pertinent to their daily activities.
- Have documented training and/or experience in QA/QC procedures and the laboratory's Quality System.
- 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 shall be investigated following procedures outlined in Section 12 and if deemed necessary may be temporarily suspended during the investigation.
- Objectively monitor standards of performance in quality control and quality assurance without outside (e.g., managerial) influence.
- Coordinating of document control of SOPs, MDLs, control limits, and miscellaneous forms and information.
- 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.
- Captains the QA team to enable communication and to distribute duties and responsibilities.
- Ensuring Communication & 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 ISO 17025. (where applicable)

4.2.3 Technology Manager (AKA Technical Director) & Department Manager (DM)

The Technology Manager report(s) directly to the Laboratory Director. The Technology Manager along with the Laboratory Director, the QA Manager and each Department Manager is accountable for compliance with the ISO 17025 Standard. The Technology Manager works with QA and the Department Managers to solve day to day technical issues, provide technical training and guidance to laboratory staff, project managers, and clients, and assists with method development and validation.

The Department Managers report to the Laboratory Directory. The DMs maintain overall responsibility for a defined portion of the laboratory. These responsibilities include but are not limited to:

- Day-to-day supervision of laboratory operations for the appropriate field of accreditation and reporting of results. Working with the QA Manager to coordinate preparation of test method SOPs and performs subsequent analyst training and interpretation of the SOPs for implementation and unusual project samples.
- Reviews and approves proposals from marketing, in accordance with the established procedure for the review of requests and contracts.
- Monitoring the validity of the analyses performed and data generated in the laboratory.
- 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.
- Enhancing efficiency and improving quality through technical advances and improved LIMS utilization. Capital forecasting and instrument life cycle planning for second generation methods and instruments as well as asset inventory management.
- Working with the QA Manager to scheduling all QA/QC-related requirements for compliance, e.g., MDLs, etc.
- Captains department personnel to communicate quality, technical, personnel, and instrumental issues for a consistent team approach.

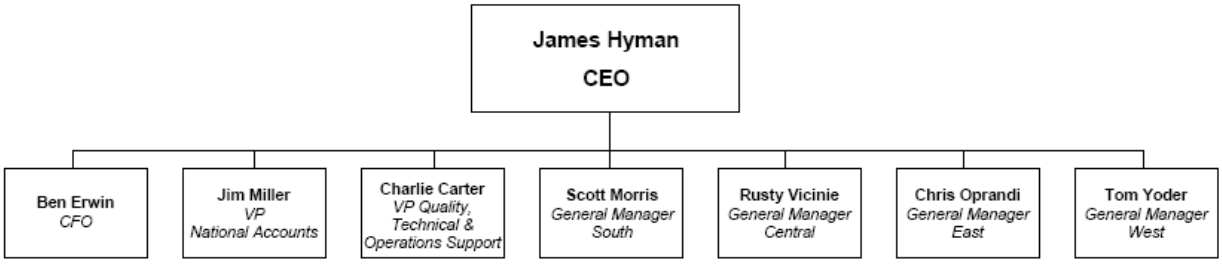
4.3 Deputies

The following table defines who assumes the responsibilities of key personnel in their absence:

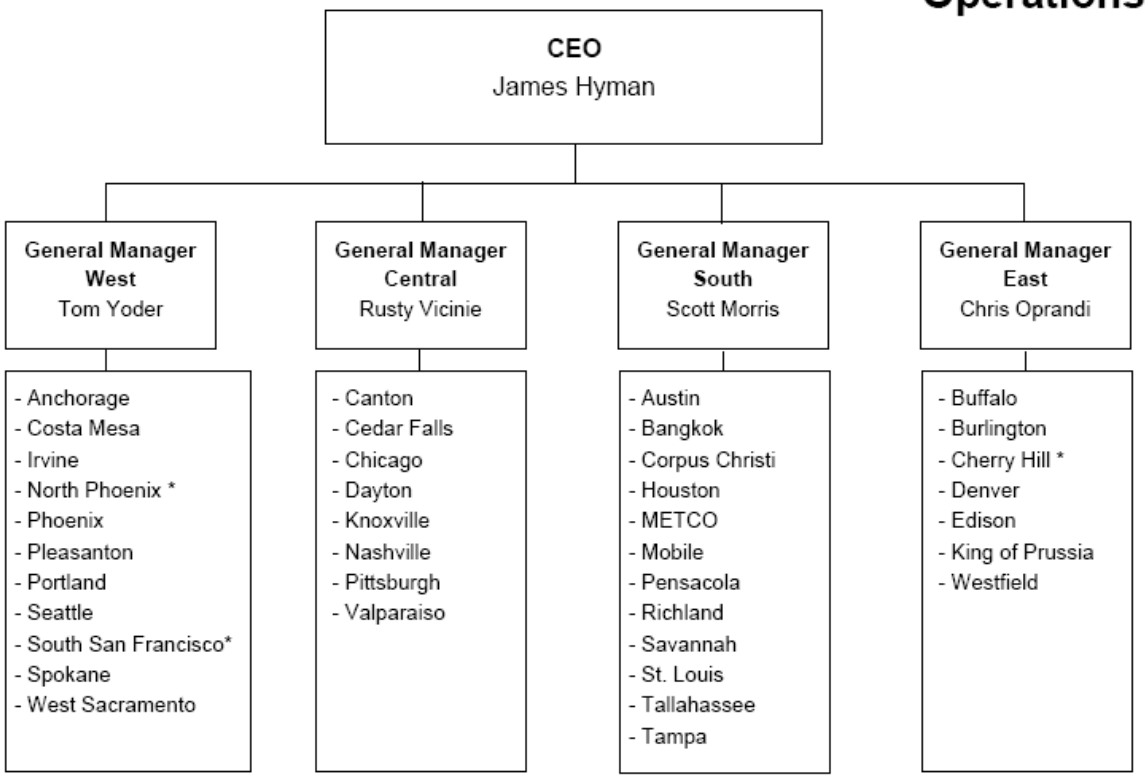
Key Personnel	Deputy
Kirstin L. Daigle Laboratory Director	Brad Chirgwin, Technology Manager Sara S. Goff, QA Manager
Sara S. Goff QA Manager	Kirstin L. Daigle, Laboratory Director Brad Chirgwin, Technology Manager Bonnie Morgan, QA Assistant
Brad Chirgwin Technology Manager	Kirstin L. Daigle, Laboratory Director Sara S. Goff, QA Manager
Dan E. Helfrich EHS Coordinator	Kirstin L. Daigle, Laboratory Director

Figure 4-1. Corporate and Laboratory Organization Charts

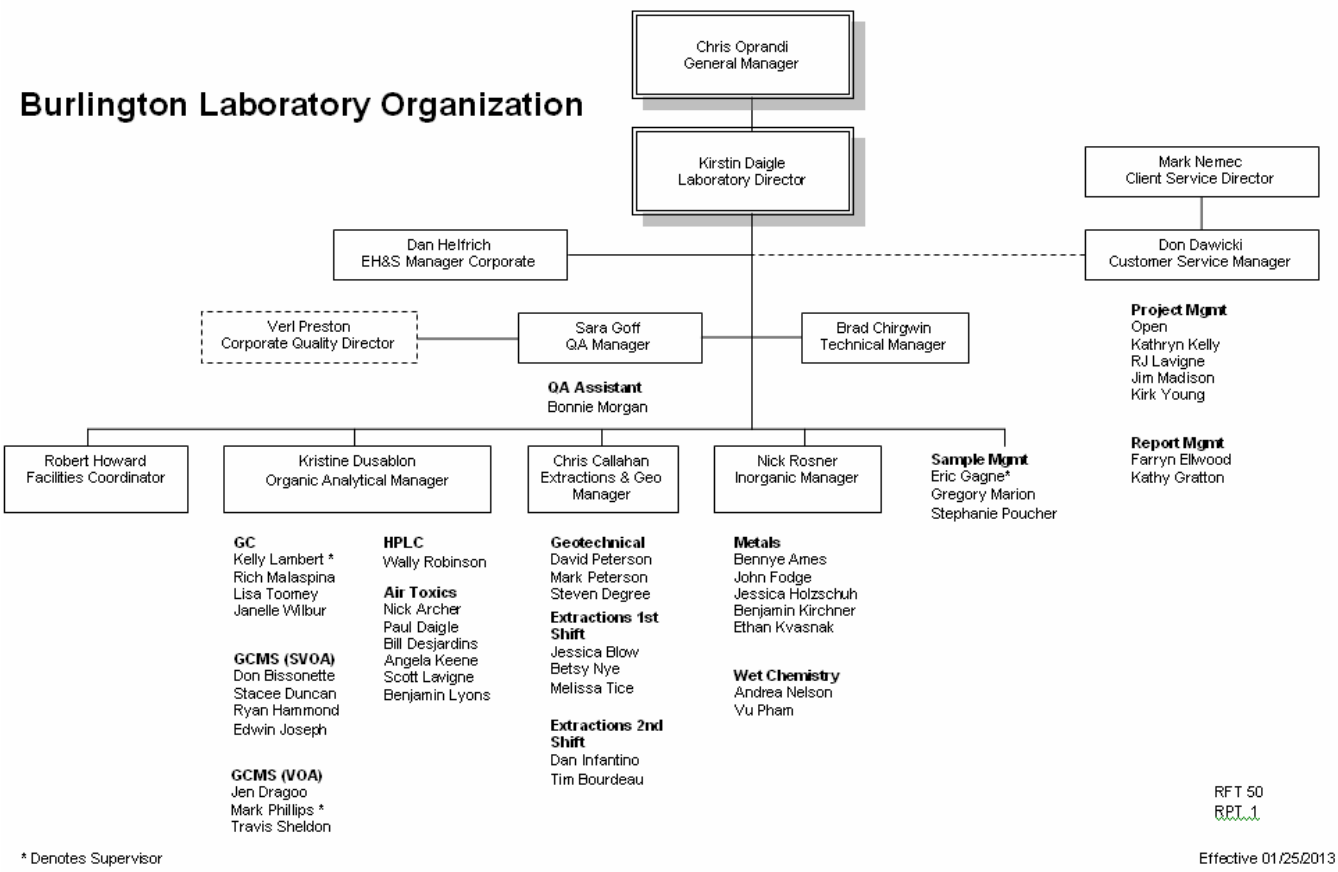
Executive Committee



Operations



* Note: EMLab P&K microlabs report to these facilities.



SECTION 5. QUALITY SYSTEM

5.1 Quality Policy Statement

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.
- To comply with the ISO/IEC 17025:2005(E) International Standard, the 2009 TNI Standard 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

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. CW-L-P-004) and Employee Ethics Statements.
- Ethics and Compliance Officers (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. CW-L-S-002)
- Procedures and guidance for recalling data if necessary (Corporate SOP No. CW-L-S-002).
- Effective external and internal monitoring system that includes procedures for internal audits (Section 15).
- Produce results, which are accurate and include QA/QC information that meets client pre-defined Data Quality Objectives (DQOs).
- 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 Documentation

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
- Laboratory QA/QC Policy Memorandums

5.3.1 Order of Precedence

In the event of a conflict or discrepancy between policies, the order of precedence is as follows:

- 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

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 their project QAPPs. The laboratory will provide support to the client for developing the sections of the QAPP that concern laboratory activities. 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 it is finalized.

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.

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.

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 as specified for a particular project, is 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 (Detection Limit, Limit of Detection) or quantified (Limit of Quantiation or Reporting Limit).

5.5 Criteria for Quality Indicators

The laboratory limits used for quality control are stored in the LIMS database (TALS) and may also be published in laboratory SOPs. Limits for accuracy and precision are laboratory generated or 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. The laboratory procedure for establishment of control limits is described in laboratory SOP BR-QA-013.

5.6 Statistical Quality Control

Statistically-derived precision and accuracy limits are required by selected methods (such as SW-846) and by program. The laboratory routinely utilizes statistically-derived limits to evaluate method performance and determine when corrective action is appropriate. If a method requires the generation of limits from historical data the lab develops such limits from data stored in the LIMS database following the procedure specified in laboratory SOP BR-QA-013.

For each job analysts are instructed to use the current limits that are entered as reference data data in the Laboratory Information Management System (LIMS) On occasion, a client requests contract-specified limits for a specific project in which case project specific limits are entered into each LIMS job by the PM handling the project.

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

The laboratory's procedures for the creation of control charts are described in laboratory SOP BR-QA-013. Control charts are created from data stored in the LIMS. The charts are evaluated by QA or technical staff to determine if limits need to be updated or to assess the need for corrective actions to improve method performance.

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 16). These metrics are used to drive continuous improvement in the laboratory's Quality System.

SECTION 6. DOCUMENT CONTROL

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
- Laboratory Standard Operating Procedures (SOP)
- Laboratory Policies
- Work Instructions and Forms
- Corporate Policies and Procedures distributed outside the intranet

Corporate Quality 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. The laboratory's internal document control procedure is defined in SOP BR-QA-003.

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 records for 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 are kept by the QA department. Raw analytical data consists of bound logbooks, instrument printouts, any other notes, magnetic media, electronic data and final reports are retained electronically, by each analytical section or by the QA department.

6.2 Document Approval and Issue

The pertinent elements of a document control system for each document include a unique document title and number, pagination, the total number of pages of the item or an 'end of document' page, the effective date, revision number and the laboratory's name. The QA personnel are responsible for the maintenance of this system.

Controlled documents are authorized by the QA Department. To develop a new document, the department manager or any employee with approval from the department manager submits an draft of the form to the QA Department for approval before use. Upon approval QA personnel add the identifying version information to the document and retains a copy of the document as the official document on file. The document is then provided to all applicable operational units (may include electronic access) 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 annually and revised as appropriate. Changes to documents occur when a procedural change warrants.

6.3 Procedures for Document Control Policy

For changes to the QA Manual, refer to SOP BR-QA-003. Uncontrolled copies must not be used within the laboratory. Previous revisions are stored by the QA department. The current revision is located in the public controlled document folder accessible to all employees.

For changes to SOPs, refer to SOP No. CW-Q-S-002, Writing a Standard Operating Procedure SOP.

Forms, worksheets, work instructions and information are organized by the QA department in accordance with the procedures specified in laboratory SOP BR-QA-003.

6.4 Obsolete Documents

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 according to SOP BR-QA-003.

SECTION 7. SERVICE TO THE CLIENT

7.1 Overview

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 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

Work requests are reviewed by appropriate personnel 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 Sales Directors, 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. Appropriate personnel include but are not limited to:

- Legal & Contracts Director
- General Manager
- Laboratory Director
- Laboratory Project Manager
- Laboratory Technology Manager
- Laboratory Department Manager
- Laboratory Customer Service Manager
- Information Technology Manager
- Account Executives
- Laboratory and/or Corporate Quality Managers
- Laboratory and/or Corporate Environmental Health and Safety Managers/Directors

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 Laboratory Director reviews the formal laboratory quote and makes final acceptance for their facility. The Project Manager, Sales Director, Legal Contracts Director, Account Executive or Proposal Coordinator then submits the final proposal to the client. The Legal & Contracts Director and facility Customer Service Manager maintains copies of all signed contracts.

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. Records of review are organized and kept by the designated project manager (PM).

The contract will be distributed to and maintained by the appropriate sales/marketing personnel and the Account Executive. A copy of the contract and formal quote will be filed with the laboratory PM.

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. These records are retained by the laboratory PM.

7.3.1 Project-Specific Quality Planning

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. 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.

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 available 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 laboratory staff.

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

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 15 and 25).

Note: ISO/IEC 17025 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 25. Special services are also available and provided upon request. These services include:

- Reasonable access for our clients or their representatives to the relevant 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

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 non-conformances in either sample receipt or sample analysis. Project management will maintain ongoing client communication throughout the entire client project.

The Technology Manager and the Quality Assurance Manager are available to discuss any technical questions or concerns that the client may have.

7.6 Reporting

The laboratory works with our clients to produce any special communication reports required by the contract.

7.7 Client Surveys

The laboratory assesses both positive and negative client feedback. The results are used to improve overall laboratory quality and client service. TestAmerica's Sales and Marketing teams periodically develops lab and client specific surveys to assess client satisfaction.

SECTION 8. SUBCONTRACTING OF TESTS

8.1 Overview

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 (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 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.

Project Managers (PMs), Customer Service Managers (CSM), or Account Executives (AE) 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 (e.g, USDA) or contracts (e.g, certain USACE projects) may require notification prior to placing such work**.**

8.2 Qualifying and Monitoring Subcontractors

Whenever a PM, Account Executive (AE) or Customer Service Manager 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 is available on the TestAmerica intranet site. Supporting documentation is maintained by corporate offices and by the TestAmerica laboratory originally requesting approval of the subcontract lab.
- 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. (Corporate SOP No. CA-C-S-001, Work Sharing Process).

When the potential sub-contract laboratory has not been previously approved, management staff 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 and notify the finance group for JD Edwards.

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 are posted using the Vendor Performance Report.
- Information shall 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 Laboratory 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 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 and in the project folder. An example form that may be used for documentation is provided as Figure 8-1. 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 TestAmerica Chain of Custody (COC). A copy of the original COC sent by the client must also be included with all samples workshared within TestAmerica. Client CoCs are only forwarded to external subcontractors when samples are shipped directly from the project site to the subcontractor lab. Under routine circumstances, client CoCs are not provided to external subcontractors.

Through communication with the subcontracted laboratory, the PM monitors the status of the subcontracted analyses, facilitates 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; however, this decision & justification must be documented in the project files, and the 'Purchase Order Terms And Conditions For Subcontracted Laboratory Services' must be sent with the samples and Chain-of-Custody. In the event this provision is utilized, the laboratory (e.g., PM) will be required to verify and document the applicable accreditations of the subcontractor. All other quality and accreditation requirements will still be applicable, but the subcontractor need not have signed a subcontract with TestAmerica at this time. The comprehensive approval process must then be initiated within 30 calendar days of subcontracting.

Figure 8-1.

Example - Subcontracted Sample Form

Date/Time: _____

Subcontracted Laboratory Information:

- Subcontractor's Name: _____
- Subcontractor Point of Contact: _____
- Subcontractor's Address: _____
- Subcontractor's Phone: _____
- Analyte/Method: _____
- Certified for State of Origin: _____
- NELAC Certified: Yes_____No_____
- USDA Permit (__Domestic __ Foreign) Yes_____No_____
- ISO 17025 Certified: Yes_____No_____
- CLP-like Required: Yes_____No_____
  (Full doc required)
- Requested Sample Due Date: _____
  (Must be put on COC)
- Client POC Approval on-file to Subcontract Samples to Sub Laboratory: Yes_____No_____

Project Manager: _____

Laboratory Sample # Range: _____
(Only of Subcontracted Samples)

Laboratory Project Number (Billing Control #): _____

All subcontracted samples are to be sent via bonded carrier and Priority Overnight. Please attach tracking number below and maintain these records in the project files.

PM Signature_____**Date**_____

SECTION 9. PURCHASING SERVICES AND SUPPLIES

9.1 Overview

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 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 (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

Purchasing guidelines for equipment and reagents must meet 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.

9.3.2 Receiving

It is the responsibility of the manager that placed the order to receive the shipment. It is the responsibility of the manager or their designee who ordered the materials to document the date

materials where received. Once the ordered reagents or materials are received 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 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

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, analytical reagent grade will 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 and solvents unless noted otherwise by the manufacturer or by the reference source method. Chemicals/solvents should not be used past the manufacturer's or SOPs expiration date unless 'verified' (refer to item 3 listed below).

- An expiration date **cannot** be extended if the dry chemical/solvent is discolored or appears otherwise physically degraded, the dry chemical/solvent must be discarded.
- Expiration dates can be extended if the dry chemical/solvent 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/solvent is used for the preparation of standards, the expiration dates can be extended 6 months if the dry chemical/solvent is compared to an unexpired independent source in performing the method and the performance of the dry chemical/solvent is found to be satisfactory. The comparison must show that the dry chemical/solvent meets CCV limits. The comparison studies are maintained in each laboratory section.

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 120 psig for Helium, 100 psig for liquid Argon and 30 psig for Nitrogen or the tank must be replaced. To prevent a tank from going to dryness, close observation of the tank gauge must take place as pressure decreases towards the minimum 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- µmhom/cm (or specific resistivity of greater than 1.0 megohm-cm) at 25^{o}C. The specific conductivity is checked and recorded daily. If the water's specific conductivity is greater than the specified limit, the Facility Manager and appropriate Technical Managers 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 bottleware used for sampling must be certified clean and the certificates must be maintained. If uncertified sampling bottleware is 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 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 Technology Manager 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.

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 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.

Upon receipt of a new or used piece of equipment, an identification name is assigned and added to the equipment list. IT must also be notified so that they can 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 19). For software, its operation must be deemed reliable and evidence of instrument verification must be retained by IT or the QA Department. Software certificates supplied by the vendors are kept by IT. The manufacturer's operation manual is retained at the bench.

9.5 Services

Service to analytical instruments (except analytical balances) is performed on an as needed basis. Routine preventative maintenance is discussed in Section 20. The need for service is determined by analysts and/or Technical Managers. If an external contractor is selected to perform service, the service providers that perform the services are approved by the Technical Manager.

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.

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.

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 and/or the Technology Director are consulted with vendor

and product selection that have an impact on quality.

SECTION 10. COMPLAINTS

10.1 Overview

The laboratory considers an effective client complaint handling processes 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 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 12 (Corrective Actions) and is documented following laboratory SOP BR-QA-004.

10.2 External Complaints

An employee that receives a complaint initiates the complaint resolution process by first documenting the complaint according to laboratory SOP BR-QA-004.

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 likelihood 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.

10.3 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 12. In addition, Corporate Management, Sales and Marketing and IT may initiate a complaint by contacting the laboratory or through the corrective action system described in Section 12.

10.4 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 16).

SECTION 11. CONTROL OF NON-CONFORMING WORK

11.1 Overview

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 12).

Due to the frequently unique nature of environmental samples, sometimes departures from documented policies and procedures are needed. Any modifications to the routine procedure are documented in the project record and described in the case narrative submitted 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. Any project specific modifications to the procedure are documented in the project record.

11.2 Responsibilities and Authorities

TestAmerica's Corporate SOP entitled *Internal Investigation of Potential Data Discrepancies and Determination for Data Recall* (SOP No. CW-L-S-002) 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.

The Laboratory Director, a Technology or Department Manager or a member of the QA team may 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 documented. 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, and the Technical Managers. 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.

11.3 Evaluation of Significance and Actions Taken

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 & Recall Procedure (SOP No. CW-L-S-002) 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 in lieu of the data recall determination form contained in TestAmerica's Corporate SOP No. CW-L-S-002.

11.4 Prevention of NonConforming Work

If it is determined that the nonconforming work could recur, further corrective actions must be made following the laboratory's corrective action system. Periodically as defined by the laboratory's preventive action schedule, 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.

11.5 Method Suspension / Restriction (Stop Work Procedures)

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 11.2, Paragraph 5.

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 12 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 (e.g., 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, Technology Manager, QA Manager) can devise a start-up plan to cover all steps from client notification through compliance and release of reports. Project Management and the Directors 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.

SECTION 12. CORRECTIVE ACTION

12.1 Overview

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 Reports (NCR) and Corrective Action Reports (CAR) (refer to Figure 12-1).

12.2 General

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.

12.2.1 Non-Conformance Report (NCR) - is used to document the following types of corrective actions:

- Deviations from an established procedure or SOP
- QC outside of limits (non-matrix related)
- Isolated reporting / calculation errors
- Client complaints
- Discrepancies in materials / goods received vs. manufacturer packing slips.

12.2.2 Corrective Action Report (CAR) - is used to document the following types of corrective actions:

- Questionable trends that are found in the review of NCRs.
- Issues found while reviewing NCRs that warrant further investigation.
- Failed or unacceptable PT results.
- Corrective actions that cross multiple departments in the laboratory.
- Systematic reporting / calculation errors
- Client complaints
- Data recall investigations
- Identified poor process or method performance trends
- Excessive revised reports

This will provide background documentation to enable root cause analysis and preventive action.

12.3 Closed Loop Corrective Action Process

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.

12.3.1 Cause Analysis

- Upon discovery of a non-conformance event, the event must be defined and documented. An NCM or CAR must be initiated, someone is assigned to investigate the issue and the event is investigated for cause. Table 12-1 provides 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 Technology Manager and/or Department Manager, Laboratory Director, or QA Manager (or QA designee) is consulted.

12.3.2 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 or CAR is used for this documentation.

12.3.3 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.

To perform root cause analysis, systematically analyze and document the root causes of the more significant problems reported then 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; for example, 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.

12.3.4 Monitoring of the Corrective Actions

- The Technology Manager and/or Department Manager 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. Technical Managers are accountable to the Laboratory Director to ensure final acceptable resolution is achieved and documented appropriately.
- Corrective actions are tracked by the QA department.
- The QA Manager reviews NCMs and CARs monthly for trends. Highlights are included in the QA monthly report (refer to Section 16). 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.

12.3.5 Follow-up Audits

- 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.
- 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 15.1.4, Special Audits.)

12.4 Technical Corrective Actions

In addition to providing acceptance criteria and specific protocols for technical corrective actions in the method SOPs, 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 11). The documentation of these procedures is through the use of an NCM or CAR.

Table 12-1 includes examples of general technical corrective actions. For specific criteria and corrective actions, refer to specific method SOPs.

Table 12-1 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 SOPs, Work Instructions, QAM Sections 19 and 20. All corrective actions are reviewed monthly, at a minimum, 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 an NCM and appropriate corrective action (e.g., reanalysis) is taken and documented**.**

12.5 Basic Corrections

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 12-1.
Example - Corrective Action Report

CORRECTIVE ACTION REPORT (CAR)			Tracking Number:
Initiated By:		Assigned To:	
Initiation Date:		CC:	
Due Date:			
Section 1: Describe Problem & Attach Supporting Documentation As Needed			
Corrective Action Prompted By:			
Recurring NCR	Internal Audit	External Audit	Complaint / Other
Section 2: Root Cause Analysis			
Section 3: Describe Actions Required to Correct & Prevent Problem			
Section 4: QA Review and Close Out			
Action Taken Was:	Acceptable	Not Acceptable	Other
Comments:			
Close Out Date:		Closed By:	
Section 5: Follow Up (From Close-Out Date)			
Time Frame:	Performed By:	Date:	Is action taken preventing recurrence?
1 Month			
3 Month			
6 Month			
Comments:			

FQA018:03.29.07:2
TestAmerica Burlington

Table 12-1. Example – General Corrective Action Procedures

QC Activity *(Individual Responsible for Initiation/Assessment)*	Acceptance Criteria	Recommended Corrective Action
Initial Instrument Blank (Analyst)	See details in Method SOP	- Prepare another blank. - If same response, determine cause of contamination: reagents, environment, instrument equipment failure, etc.
Initial Calibration Standards (Analyst)	See details in Method SOP	- Reanalyze standards. - If still unacceptable, remake standards and recalibrate instrument.
Independent Calibration Verification (Second Source) (Analyst)	% Recovery within limits in TALS	- Remake and reanalyze standard. - If still unacceptable, then remake calibration standards or use new primary standards and recalibrate instrument.
Continuing Calibration Standards (Analyst)	- See details in Method SOP	- Reanalyze standard. - If still unacceptable, then recalibrate and rerun affected samples.
Matrix Spike / Matrix Spike Duplicate (MS/MSD) (Analyst)	% Recovery within limits in TALS	- If the acceptance criteria for duplicates or matrix spikes are not met because of matrix interferences, the acceptance of the analytical batch is determined by the validity of the LCS. - If the LCS is within acceptable limits the batch is acceptable. - The results of the duplicates, matrix spikes and the LCS are reported with the data set. - For matrix spike or duplicate results outside criteria the data for that sample shall be reported with qualifiers.

QC Activity *(Individual Responsible for Initiation/Assessment)*	Acceptance Criteria	Recommended Corrective Action
Laboratory Control Sample (LCS) (Analyst)	% Recovery within limits in TALS	- Batch must be re-prepared and re-analyzed. This includes any allowable marginal exceedance. When not using marginal exceedances, the following exceptions apply: 1) when the acceptance criteria for the positive control are exceeded high (i.e., high bias) and there are associated samples that are non-detects, then those non-detects may be reported with data qualifying codes; 2) when the acceptance criteria for the positive control are exceeded low (i.e., low bias), those sample results may be reported if they exceed a maximum regulatory limit/decision level with data qualifying codes. **Note:** If there is insufficient sample or the holding time cannot be met, contact client and report with flags.
Surrogates (Analyst)	- % Recovery within limits in TALS.	- Individual sample must be repeated. Place comment in LIMS. - Surrogate results outside criteria shall be reported with qualifiers.
Method Blank (MB) (Analyst)	< Reporting Limit or as specified by regulatory program.	- Reanalyze blank. - If still positive, determine source of contamination. If necessary, reprocess (i.e. digest or extract) entire sample batch. Report blank results. - Qualify the result(s) if the concentration of a targeted analyte in the MB is at or above the reporting limit AND is > 1/10 of the amount measured in the sample.
Proficiency Testing (PT) Samples (QA Manager, Department Manager(s)	- 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.

QC Activity *(Individual Responsible for Initiation/Assessment)*	Acceptance Criteria	Recommended Corrective Action
Internal / External Audits (QA Manager, Technology Manager and Department Manager(s), 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.
Reporting / Calculation Errors (Depends on issue – possible individuals include: Analysts, Data Reviewers, Project Managers, Technical Managers, QA Manager, Corporate QA, Corporate Management)	- SOP CW-L-S-002Internal Investigation of Potential Data Discrepancies and Determination for Data Recall.	- Corrective action is determined by type of error. Follow the procedures in SOP CW-L-S-002 or your lab's CA SOP.
Client Complaints (Project Managers, Lab Director/Manager, Sales and Marketing)	-	- 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 16 for an example) (QA Manager, Lab Director)	- QAM, SOPs.	- Corrective action is determined by the type of issue. For example, CARs for the month are reviewed and possible trends are investigated.
Health and Safety Violation (Safety Officer, Lab Director)	- Environmental Health and Safety (EHS) Manual.	- Non-conformance is investigated and corrected through CAR system.

SECTION 13. PREVENTIVE ACTION / IMPROVEMENT

13.1 Overview

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 and continuous process of improvement activities 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 client satisfaction can be improved through continuous improvements to laboratory systems.

Opportunities for improvement may be discovered during management reviews, the monthly QA Metrics Report, evaluation of internal or external audits, results & evaluation of proficiency testing (PT) performance, data analysis & review processing operations, client complaints, staff observation, etc.

The monthly Management Systems Metrics Report shows performance indicators in all areas of the laboratory and 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 in evaluating the management and quality system performance on an ongoing basis and provide a tool for identifying areas for improvement.

The laboratory's corrective action process 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.

13.1.1 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.

13.1.2 Any Preventive Actions undertaken or attempted shall be taken into account during the annual Management Systems Review (Section 16). A highly detailed report is not required; however, a summary of successes and failures within the preventive action program is sufficient to provide management with a measurement for evaluation.

13.2 Management of Change

The Management of Change process is designed to manage significant events and changes that occur within the laboratory such as the addition of new equipment or personnel. Procedures for minimization of potential risks inherent with a new event or change are described in various laboratory standard operating procedures.

SECTION 14. CONTROL OF RECORDS

The laboratory maintains a records management 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.

14.1 Overview

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 14-1. Quality records are maintained by the QA department. Records are of two types; 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 by each laboratory section.

Table 14-1. Record Index[1]

	Record Types [1]:	Retention Time:
Technical Records	- Raw Data - Logbooks[2] - Standards - Certificates - Analytical Records - MDLs/IDLs/DOCs - 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 Types [1]:	Retention Time:
QA Records	- Internal & External Audits/Responses - Certifications - Corrective/Preventive Actions - Management Reviews - 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 and Amendments - Correspondence - QAPP - SAP - Telephone Logbooks - Lab Reports	5 Years from analytical report issue*
Administrative Records	Finance and Accounting	10 years
	EH&S Manual, Permits	7 years
	Disposal Records	Indefinitely
	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 14-2.

14.1.1 All records are stored and retained in such a way that they are secure and readily retrievable at the laboratory facility or an offsite location that provides a suitable environment to prevent damage or deterioration and to prevent loss**.** 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 and shall be documented with an access log**.** 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 14-2 have lengthier retention requirements and are subject to the requirements in Section 14.1.3.

14.1.2 Programs with Longer Retention Requirements

Some regulatory programs have longer record retention requirements than the standard record retention time. These are detailed in Table 14-2 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 14-2. Example: Special Record Retention Requirements

Program	[1]Retention Requirement
Drinking Water – All States	**5 years (project records)** **10 years - Radiochemistry (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.

14.1.3 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 19.14.1 for more information.

14.1.4 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. 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.

- 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 purpose. Instrument data is stored by instrument. Run logs are maintained for each instrument. 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 12 and 19. 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.

- Also refer to Section 19.14.1 ‘Computer and Electronic Data Related Requirements’**.**

14.2 Technical and Analytical Records

14.2.1 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. 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.

14.2.2 Observations, data and calculations are recorded real-time and are identifiable to the specific task.

14.2.3 Changes to hardcopy records shall follow the procedures outlined in Section 12 and 19. 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:

- laboratory sample ID code**;**
- Date of analysis; 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 or on a benchsheet.

- Instrumentation identification and instrument operating conditions/parameters.
- analysis type;
- all manual calculations and manual integrations;
- analyst's or operator's initials/signature;
- sample preparation
- 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.

14.3 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

14.3.1 Sample Handling Records

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.

14.4 Administrative Records

The laboratory also maintains the administrative records in either electronic or hard copy form. Refer to Table 14-1.

14.5 Records Management, Storage and Disposal

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 upon request.

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.

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**.**

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. The procedures for document are described in laboratory SOP BR-QA-003.

14.5.1 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.

14.5.2 Records Disposal

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 14-1 and 14-2).

Electronic copies of records must be destroyed by erasure or physically damaging off-line storage media so no records can be read.

If a third party records management company is hired to dispose of records, a "Certificate of Destruction" is required.

SECTION 15. AUDITS

15.1 Internal Audits

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 Auditing, SOP No. CA-Q-S-004. The types and frequency of routine internal audits are described in Table 15-1. Special or ad hoc assessments may be conducted as needed under the direction of the QA staff.

Table 15-1. Types of Internal Audits and Frequency

Description	Performed by	Frequency
Quality Systems Audits	QA Department, QA approved designee, or Corporate QA	All areas of the laboratory annually
Method Audits	Joint responsibility: a) QA Manager or designee b) Technology Manager or Designee (Refer to CA-Q-S-004)	Methods Audits Frequency: 50% of methods annually 100% of methods annually (DoD Labs)
Special	QA Department or Designee	Surveillance or spot checks performed as needed, e.g., to confirm corrective actions from other audits.
Performance Testing	Analysts	Two successful per year for each TNI field of proficiency testing or as dictated by regulatory requirements

15.1.1 Annual Quality Systems Audit

An annual quality systems audit is required to ensure compliance to analytical methods and SOPs, TestAmerica's Data Integrity and Ethics Policies, the TNI quality systems requirements, 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 for effectiveness & sustainability. The audit is divided into sections for each operating or support area of the lab, and each section is comprehensive for a given area. The area audits may be performed on a rotating schedule throughout the year to ensure adequate coverage of all areas. This schedule may change as situations in the laboratory warrant.

15.1.2 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, electronic audit miner programs (e.g., MintMiner and Chrom AuditMiner) used to identify unusual manipulations of the data deserving closer scrutiny. QA technical audits will include all methods within a two-year period.

15.1.3 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 Quality Assurance Manager or qualified designee at least every two years.

15.1.4 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.

15.1.5 Performance Testing

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: water, soil, air.

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.

15.2 External Audits

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. When requested, 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.

15.2.1 Confidential Business Information (CBI) Considerations

During on-site audits, 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 2009 TNI standards.

15.3 Audit Findings

Audit findings are documented in audit reports and tracked by the QA department. The laboratory's corrective action responses for internal and external audits include action plans and date for completion. If a completion date cannot be met, a new a completion date must be set and agreed to by the QA Manager.

Developing and implementing corrective actions to findings is the responsibility of the Department Manager 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.

SECTION 16. MANAGEMENT REVIEWS

16.1 Quality Assurance Report

A comprehensive QA Report shall be prepared each month by the laboratory's QA Department and forwarded to the Laboratory Director, 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.

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.

16.2 Annual Management Review

The senior lab management team (Laboratory Director, Technology Manager, Department Manager and QA 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 goals, & objectives and action items that feed into the laboratory planning system. 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 (Corporate SOP No. CA-Q-S-008 & Work Instruction No. CA-Q-WI-020) uses information generated during the preceding year to assess the "big picture" by ensuring that routine actions taken and reviewed on a monthly basis are not components of larger systematic concerns. The monthly review 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 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 management. 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.

16.3 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. CW-L-S-002). 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.

SECTION 17. PERSONNEL

17.1 Overview

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 organization chart in Figure 4-1.

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.

17.2 Education and Experience Requirements for Technical Personnel

The laboratory makes every effort to hire analytical staffs that possess 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. 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.

As a general rule for analytical staff:

Specialty	Education	Experience
Extractions, Digestions, some electrode methods (pH, DO, Redox, etc.), or Titrimetric and Gravimetric Analyses	H.S. Diploma	On the job training (OJT)
GFAA, CVAA, FLAA, Single component or short list Chromatography (e.g., Fuels, 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

Specialty	Education	Experience
ICP, ICPMS, Long List or complex chromatography (e.g., Pesticides, PCB, Herbicides, 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
Technology Manager / Department Manager– **General**	Bachelors Degree in an applied science or engineering. The Technology Manager must also have 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 peer or supervisors 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.

17.3 Training

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	All
Ethics – Comprehensive Refresher	Annually	All
Initial Demonstration of Capability (DOC)	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 19.

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 19).
- 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.

Further details of the laboratory's training program are described in the Laboratory Training SOP BR-QA-011.

17.4 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 (Policy No. CW-L-P-004) and an Ethics Statement. All initial and annual training is documented by signature on the signed Ethics Statement 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
- How and when to report ethical/data integrity issues. Confidential reporting.
- Recordkeeping
- 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 18. ACCOMMODATIONS AND ENVIRONMENTAL CONDITIONS

18.1 Overview

The laboratory is a 22,000 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 preparation, volatile organic sample analysis, non-volatile organic sample analysis, inorganic sample analysis, and administrative functions.

18.2 Environment

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.

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.

Environmental conditions of the facility housing the computer network and LIMS are regulated to protect against raw data loss.

18.3 Work Areas

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.

18.4 Floor Plan

A floor plan can be found in Appendix 1.

18.5 Building Security

Building cards are distributed to 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. Signs are posted in the laboratory designating employee only areas - "Authorized employees beyond this point".

SECTION 19. TEST METHODS AND METHOD VALIDATION

19.1 Overview

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.

19.2 Standard Operating Procedures (SOPS)

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.

- All SOPs contain a revision number, effective date, and appropriate approval signatures. Controlled copies are available to all staff.
- Procedures for writing an SOP are incorporated by reference to TestAmerica's Corporate SOP entitled 'Writing a Standard Operating Procedure', No. CW-Q-S-002.
- SOPs are reviewed at a minimum of every 2 years except for SOPs for Drinking Water and DoD SOPs which are reviewed annually. Whenever necessary, SOPs may be revised to ensure continuing suitability and compliance with applicable requirements.

19.3 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.

19.4 Selection of Methods

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), 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.

19.4.1 Sources of Methods

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.

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:

- *Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air, US EPA, January 1996.*
- *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.*
- *Standard Methods for the Examination of Water and Wastewater, 18th/19th /20th/ 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.*
- *National Status and Trends Program, National Oceanographic and Atmospheric Administration, Volume I-IV, 1985-1994.*
- *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.

19.4.2 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. 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.

A demonstration of capability (DOC) is performed whenever there is a change in instrument type (e.g., new instrumentation), method or personnel (e.g., analyst hasn't performed the test within the last 12 months).

The initial demonstration of capability must be thoroughly documented and approved by the Technical Manager and QA Manager prior to independently analyzing client samples. All associated documentation must be retained in accordance with the laboratories archiving procedures.

The laboratory must have an approved SOP, demonstrate satisfactory performance, and conduct an MDL 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 quantitation limit (QL), must be at or above the lowest non-zero standard in the calibration curve and must be reliably determined. Project RLs are client specified reporting levels which may be higher than the QL. Results reported below the QL must be qualified as estimated values. Also see Section 19.6.1.3, Relationship of Limit of Detection (LOD) to Quantitation Limit (QL).
- 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: *Reporting Limit based on the low standard of the calibration curve.*

19.4.3 Initial Demonstration of Capability (IDOC) Procedures

19.4.3.1 The spiking standard used should be prepared independently from those used in instrument calibration.

19.4.3.2 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.

19.4.3.3 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 days).

19.4.3.4 Using all of the results, calculate the mean recovery in the appropriate reporting units and the standard deviations for each parameter of interest.

19.4.3.5 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.

19.4.3.6 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.

19.4.3.7 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 19.4.3.3 above.
- Beginning with 19.4.3.3 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 19.4.3.1 above.

Note: Results of successive LCS analyses can be used to fulfill the DOC requirement.

A certification statement (refer to Figure 19-1 as an example) shall be used to document the completion of each initial demonstration of capability. A copy of the certification is archived in the analyst's training folder.

19.5 Laboratory Developed Methods and Non-Standard Methods

Any new method developed by the laboratory must be fully defined in an SOP 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.

19.6 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.

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.

19.6.1 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.

19.6.1.1 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.

19.6.1.2 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.

19.6.1.3 Relationship of Limit of Detection (LOD) to the Quantitation Limit (QL)

An important characteristic of expression of sensitivity is the difference in the LOD and the QL. The LOD is the minimum level at which the presence of an analyte can be reliably concluded. The QL is the minimum concentration of analyte that can be quantitatively determined with acceptable 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 or LOD) and below the QL. 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 QL, 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.

19.6.1.4 Determination of Interferences

A determination that the method is free from interferences in a blank matrix is performed.

19.6.1.5 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 highest acceptable calibration concentration. The lower quantitation limit or QL cannot be lower than the lowest non-zero calibration level, and can be constrained by required levels of bias and precision.

19.6.1.6 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.

19.6.1.7 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.

19.6.1.8 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.

19.7 Method Detection Limits (MDL) / Limits of Detection (LOD)

Method detection limits (MDL) 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. MDL is also sometimes referred to as Limit of Detection (LOD). The MDL 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 MDL is determined for each analyte 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. Generally, 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. 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.

Refer to the laboratory SOP No. BR-QA-005 for details on the laboratory's MDL process, including detection limit procedures specific to the CLP SOWs for ISM and SOM.

19.8 Instrument Detection Limits (IDL)

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.

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.

If IDL is > than the MDL, it may be used as the reported MDL.

19.9 Verification of Detection and Reporting Limits

Once the MDL is determined, it must be verified on each instrument used for the given method. TestAmerica defines the DoD QSM Detection Limit (DL) as being equal to the MDL. TestAmerica also defines the DoD QSM Limit of Detection (LOD) as being equal to the lowest concentration standard that successfully verifies the MDL, also referred to as the MDLV standard. MDL and MDLV standards are extracted/digested and analyzed through the entire analytical process. The MDL and MDLV determinations do 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 MDLV standard is not successful, then the laboratory will redevelop their MDL or perform and pass two consecutive MDLVs at a higher concentration and set the LOD at the higher concentration. Initial and quarterly verification is required for all methods listed in the laboratory's DoD ELAP Scope of Accreditation. Refer to the laboratory SOP BR-QA-005 Method Detection Limits (MDLs/DLs) for further details.

The laboratory quantitation limit is equivalent to the DoD Limit of Quantitation (LOQ), which is at a concentration equal to or greater than the lowest non-zero calibration standard. The DoD QSM requires the laboratory to perform an initial characterization of the bias and precision at the LOQ and quarterly LOQ verifications thereafter. If the quarterly verification results are not consistent with three-standard deviation confidence limits established initially, then the bias and precision will be reevaluated and clients contacted for any on-going projects. For DoD projects, TestAmerica makes a distinction between the Reporting Limit (RL) and the LOQ. The RL is a level at or above the LOQ that is used for specific project reporting purposes, as agreed to between the laboratory and the client. The RL cannot be lower than the LOQ concentration, but may be higher.

19.10 Retention Time Windows

Most organic analyses and some inorganic analyses use chromatography techniques for qualitative and quantitative determinations. For every chromatography analysis or as specific in the reference method, 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. Complete details are available in the laboratory SOPs.

19.11 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.

19.12 Estimation of Uncertainty of Measurement

19.12.1 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.

19.12.2 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.

19.12.3 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 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.

19.12.4 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.

19.12.5 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.

19.13 Sample Reanalysis Guidelines

Because there is a certain level of uncertainty with any analytical measurement, a sample re-preparation (where appropriate) and subsequent analysis (hereafter referred to as '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 or Contractual Terms & Conditions for reanalysis protocols may supersede 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 $\pm$ 1 reporting limit for samples $\leq$ 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 and may reanalyze the sample a third time for confirmation if sufficient sample is available.

- 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.

19.14 Control of Data

The laboratory has policies and procedures in place to ensure the authenticity, integrity, and accuracy of the analytical data generated by the laboratory.

19.14.1 Computer and Electronic Data Related Requirements

The three basic objectives of our computer security procedures and policies are shown below. More detail is outlined in corporate IT procedure and policies. The laboratory is currently running TALS which is a custom in-house developed LIMS system 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 an SQL database which is an industry standard relational database platform. It is referred to as Database for the remainder of this section.

19.14.1.1 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. Cells containing calculations must be lock-protected and controlled.
- Instrument hardware and software adjustments are safeguarded through maintenance logs, audit trails and controlled access.

19.14.1.2 **Ensure Information Availability:** Protection against loss of information or service is ensured 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.

19.14.1.3 **Maintain Confidentiality:** Ensure data confidentiality through physical access controls such as password protextion or website access approval when electronically transmitting data.

19.14.2 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.

Manual integration of peaks will be documented and reviewed and the raw data will be flagged in accordance with the TestAmerica Corporate SOP No. CA-Q-S-002, *Acceptable Manual Integration Practices* and laboratory SOP BR-QA-006.

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.

19.14.2.1 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. 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.

19.14.2.2 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**%.** Units are defined in each lab SOP.

19.14.2.3 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 the number of significant figured programmed in the LIMS formatter selected by the PM.

19.14.2.4 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. LIMS has a defined significant figure criterion for each analyte.

19.14.2.5 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 reviewing the quantitation report, and removing unrequested or poor spectrally-matched compounds. The analyst prints a copy of what has been entered to check for errors. This printout and the instrument's printout of calibrations, concentrations, retention times, chromatograms, and mass spectra, if applicable, are retained with the data file. The data file is stored in a monthly folder on the instrument computer; periodically, this file is transferred to the server and, eventually, to a tape file.

19.14.3 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 12.
- 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 QA Manager at the facility. The QA department controls all worksheets following the procedures in Section 6.

19.14.4 Review / Verification Procedures

Review procedures are out lined in several SOP BR-QA-019 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 has an SOP for manual integration, BR-QA-005. The general review concepts are discussed below, more specific information can be found in the SOPs.

19.14.4.1 The data review process at the laboratory starts at the Sample Control level. Sample Control personnel review chain-of-custody forms and input the sample information and required analyses into a computer LIMS. The Sample Control Supervisor reviews the transaction of the chain-of-custody forms and the inputted information. The Project Managers perform final review of the chain-of-custody forms and inputted information.

19.14.4.2 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 LIMS and add data qualifiers if applicable. To ensure data compliance, a different analyst performs a second level of review. Second level review is accomplished by checking reported results against raw data and evaluating the results for accuracy. During the second level review, blank runs, QA/QC check results, initial and continuing calibration results, laboratory control samples, sample data, qualifiers and spike information are

evaluated. Where calibration is not required on a daily basis, secondary review of the initial calibration results may be conducted at the time of calibration. Approximately 15% of all sample data from manual methods and from automated methods, all GC/MS spectra and all manual integrations are reviewed. Manual integrations are also electronically reviewed utilizing auditing software to help ensure compliance to ethics and manual integration policies. Issues that deem further review include the following:

- 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

19.14.4.3 Unacceptable analytical results may require reanalysis of the samples. Any problems are brought to the attention of the Department Manager, Project Manager, QA Manager or Technology Manager, as necessary. Corrective action is initiated whenever necessary.

19.14.4.4 The results are then entered or directly transferred into the computer database and a report is prepared for the client.

19.14.4.5 As a final review prior to the release of the report, the Project Manager reviews the report for completeness. This review and approval ensures that client requirements have been met and that the final report has been properly completed. The process includes, but is not limited to, verifying that chemical relationships are evaluated, COC is followed, cover letters/ narratives are present, flags are appropriate, and project specific requirements are met.

19.14.4.6 Any project that requires a data package is subject to a tertiary data review for transcription errors and acceptable quality control requirements. The Project Manager then signs the final report. The accounting personnel also check the report for any clerical or invoicing errors. When complete, the report is sent out to the client.

19.14.5 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 for our internal SOP BR-QA-006.

19.14.5.1 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.

19.14.5.2 Analysts shall not increase or decrease peak areas 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.

19.14.5.3 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.

19.14.5.4 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 19-1. Example - Demonstration of Capability Documentation

Analyst Demonstration of Capability

TestAmerica Burlington

Michelle Tam

10/12/2011

Preparation Method(s):	3010A
Analytical Method(s):	6020
Matrix:	Water
Method Description:	Metals (ICP/MS)
Preparation SOP No:	BR-ME-009R16
Analytical SOP No:	BR-ME-003R7

We, the undersigned, CERTIFY that:

1. The analyst identified above, using the cited test method with the specifications in the cited SOP, which is in use at this facility for the analysis of samples under the laboratory's Quality Assurance Plan, has completed the Demonstration of Capability(DOC).
2. The test method(s) was performed by the analyst identified on this certificate.
3. A copy of test method(s) and laboratory SOPs are available for all personnel on-site. **These documents have been reviewed by the analyst as part of this DOC.**
4. The data associated with the demonstration of capability are true, accurate, complete and self-explanatory.
5. All raw data necessary to reconstruct and validate these analyses have been retained at the facility. The associated information is organized and available for review.

Michelle Tam

Analyst	**Signature**	**Date** 10/12/11
Technical Director	**Signature**	**Date** 10/12/11
Quality Assurance Officer	**Signature**	**Date** 10/13/11

Page 1 of 7

SECTION 20. EQUIPMENT and CALIBRATIONS

20.1 Overview

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. A list of laboratory instrumentation is presented in Table 20-1.

Equipment is only operated by authorized and trained personnel. Manufacturers instructions for equipment use are readily accessible to all appropriate laboratory personnel.

20.2 Preventive Maintenance

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.

Routine preventive maintenance procedures and frequency, such as 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.

Table 20-2 lists examples of scheduled routine maintenance. It is the responsibility of each Department Manager 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.

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.

- 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.
- 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.

- 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.

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.

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.

If an instrument is sent out for service or transferred to another facility, it must be recalibrated and verified (including new initial MDL study) prior to return to lab operations.

20.3 Support Equipment

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.

20.3.1 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.

20.3.2 pH, Conductivity, and Turbidity Meters

The pH meters used in the laboratory are accurate to ± 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.

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.

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.

20.3.3 Thermometers

All thermometers are calibrated on an annual basis with a NIST-traceable thermometer. IR thermometers, digital probes and thermocouples are calibrated quarterly**.**

The mercury/digital 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 1 degree (0.5 degree or less increments are required for drinking water microbiological laboratories), and have ranges applicable to 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 support equipment logbooks.

20.3.4 Refrigerators/Freezer Units, Waterbaths, Ovens and Incubators

The temperatures of all refrigerator units and freezers used for sample and standard storage are monitored each day.

Ovens, waterbaths 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 > 0ºC and ≤ 6 ºC.

Specific temperature settings/ranges for other refrigerators, ovens waterbaths, and incubators can be found in method specific SOPs.

All of this information is documented in logbooks designated for this purpose.

20.3.5 Autopipettors, Dilutors, and Syringes

Mechanical volumetric dispensing devices including burettes (except Class A Glassware and Glass microliter syringes) are given unique identification numbers and the delivery volumes are verified gravimetrically, at a minimum, on a quarterly basis.

For those dispensers that are not used for analytical measurements, a label is 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.

20.4 Instrument Calibrations

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 12).

Note: Instruments are calibrated initially and as needed after that and at least annually. Except isotopic dilution methods do not require annual calibration.

20.4.1 Calibration Standards

Calibration standards are prepared using the procedures indicated in the Reagents and Standards section of the determinative method SOP. If a reference method does not specify

the number of calibration standards, a minimum of 3 calibration points (exception being ICP and ICP/MS methods) will be used.

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.

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 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 must be reported as having less certainty, e.g., defined qualifiers or flags (additional information may be included in the case narrative). The exception to these rules is ICP methods or other methods where the referenced method does not specify two or more standards.

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, such as air analysis where no other source or lot is available, a standard made by a different analyst at a different time or a different preparation would be considered a second source. This verification occurs immediately after the calibration curve has been analyzed, and before the analysis of any samples.

20.4.1.1 Calibration Verification

The calibration relationship established during the initial calibration must be verified initially and at least daily as specified in the laboratory method SOPs in accordance with the referenced analytical methods and in the 2009 TNI Standard. 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 verification 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 2009 TNI Std. EL-V1M4 Sec. 1.7.2.

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).

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.

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.

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).

If the results of a CCV are outside the established acceptance criteria and analysis of a second consecutive (and immediate) CCV fails to produce results within acceptance criteria, corrective action shall be performed. Once corrective actions have been completed & documented, the laboratory shall demonstrate acceptable instrument / method performance by analyzing two consecutive CCVs, or a new initial instrument calibration shall be performed.

Sample analyses and reporting of data may not occur or continue until the analytical system is calibrated or calibration verified. However, data associated with an unacceptable calibration verification may be fully useable based upon discussion and approval of the client:

a). when the acceptance criteria for the CCV are exceeded high (i.e., high bias) and the associated samples within the batch are non-detects, then those non-detects may be reported with a footnote or case narrative explaining the high bias. Otherwise the samples affected by the unacceptable CCV shall be re-analyzed after a new calibration curve has been established, evaluated and accepted; or

b). when the acceptance criteria for the CCV 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 CCV shall be re-analyzed after a new calibration curve has been established, evaluated and accepted.

Samples reported by the 2 conditions identified above will be appropriately flagged.

20.4.1.2 Verification of Linear and Non-Linear Calibrations

Calibration verification for 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. (These calculations are available in the laboratory method SOPs. 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.

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.

20.5 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.

20.6 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.

Table 20-1. Example: Instrumentation List

Instrument Type	Manufacturer	Model Number	Serial Number	Year Put into Service	Condition When Received
Analytical Balance	Mettler	AT200	113081164	UNKNOWN	UNKNOWN
Analytical Balance	Mettler	ML204	1123452701	2010	NEW
Analytical Balance	Metler	ML204	1123452699	2010	NEW
Analytical Balance	Sartorius	XM1000P	40090006	UNKNOWN	UNKNOWN
Automated Distillation Apparatus	Westco	Easy Dist	1090	2002	NEW
Automated Distillation Apparatus	Westco	Easy Dist	1091	2002	NEW
COD Reactor	HACH	45600-00	11000022452	UNKNOWN	UNKNOWN
Conductivity Meter	Oakton	CON110	234045	2001	UNKNOWN
Conductivity Meter	Oakton	CON110	297661	2004	NEW
CVAA	Leeman (CV3)	HydraAA112-0064-1	2031	2003	NEW
CVAA	Leeman (CV4)	HydraAA112-0064-1	8015	2008	NEW
GC/ECD/ECD	Agilent (7424)	6890N	US10332093	2003	NEW
GC/ECD/ECD	Agilent (3283)	6890N	US10805001	2008	NEW
GC/ECD/ECD	Hewlett-Packard	6890	US00028263	UNKNOWN	UNKNOWN
GC/ECD/ECD	Hewlett-Packard (2618)Screen	5890II	3203A41055	1987	UNKNOWN
GC/ECD/ECD	Agilent (7227)	6890N	CN10602095	2006	NEW
GC/ECD/ECD	Agilent (0825)	6890N	US10202136	2002	NEW
GC/ECD/ECD	Agilent (1031)	7890A	CN10301031	2010	NEW
GC/ECD/ECD	Agilent (5253)	6890N	CN10723008	2007	NEW
GC/ECD/ECD	Agilent (0911)	6890N	US10230082	2002	NEW
GC/ECD/ECD	Agilent (5005)	6890N	CN10615005	2009	USED
GC/FID/ECD	Hewlett-Packard (Screen)	5890	GC 2415A01109	UNKNOWN	UNKNOWN
GC/FID/FID	Hewlett-Packard (3012)	5890II	3235A45259	1984	UNKNOWN
GC/FID/FID/TCD	Varian (CP3800)	CP-3800	S/N 10328	2003	NEW
GC/FID/TCD	Varian (2866)	VR-3600	2866	1998	UNKNOWN
GC/FPD/FPD	Hewlett-Packard (2860)	5890II	2950A27078	1990	UNKNOWN
GC/MS	Hewlett-Packard (N)	5890II / 5971	3203A40979	1998	NEW
GC/MS	Hewlett Packard (V)	5890II / 5972	3336A61485	1998	NEW
GC/MS	Agilent (B)	6890N/ 5973	CN10317006	2003	NEW
GC/MS	Agilent (C)	6890N / 5973	CN10424016	UNKNOWN	NEW
GC/MS	Agilent (G)	6890N / 5973	CN10437065	UNKNOWN	USED
GC/MS	Agilent (E)	6890N / 5973	CN10453004	2005	NEW
GC/MS	Agilent (F)	6890N/ 5973	CN10531065	2005	NEW
GC/MS	Agilent (S)	7890A/5975	CN10211095	2010	NEW
GC/MS	Agilent (T)	7890A/5975	CN10211037	2010	USED
GC/MS	Hewlett-Packard (L)	5890II / 5971	3203A40982	1998	NEW
GC/MS	Agilent (D)	6890N / 5973	CN10439015	2004	NEW
GC/MS	Hewlett-Packard (P)	5890II / 5971	3203A40985	1992	USED
GC/MS	Hewlett-Packard (Q)	5890II / 5971	3203A40983	1992	NEW
GC/MS	Hewlett-Packard (R)	5890II / 5971	3203A40984	1992	NEW
GC/MS	Hewlett-Packard (U)	5890II Plus/ 5972	3336A61535	1997	NEW
GC/MS	Agilent (H)	6890N / 5975	CN10608102	2006	NEW
GC/MS	Agilent (Z)	6890A/ 5973	US00036343	2000	NEW
GC/MS	Agilent (J)	6890N / 5973	CN10430052	2009	USED
GC/FID	Hewlett-Packard (6453-K) Screen	5890 II	3203A41768	UNKNOWN	UNKNOWN
GPC	J2 Scientific (I)	Autoinject 110	02D-1030-2.1	2002	NEW
GPC	J2 Scientific (H)	Autoinject 110	02D-1031-2.1	2001	NEW
GPC	J2 Scientific (J)	AccuPrep	03G1076-3.0	2003	NEW
HPLC/UV	Dionex (1488)	P680	1680407	1991	UNKNOWN
HPLC/UV/PDA	Waters (1208)	600	60004790RP	1988	NEW
Hydrogen Generator	Parker Hannafin	H2-800	h2-800081C	2006	NEW
Hydrogen Generator	Parker Hannafin	H2-800	h2-800099C	2006	NEW
ICP-MS	Thermo Elemental (2)	X7	X0288	2003	NEW
ICP-OES	Thermo Electron Corp (7)	iCAP 6000	ICP20063302	2006	NEW
LC/MS/MS	Waters (1111)	Acquity/Quattro micro	QAA929	2005	NEW
HPLC	Waters (3062)	616	MX5NM6829M	UNKNOWN	NEW
Particle Size Analyzer	Malvern	MasterSizer 2000	MAL101709/MAL110286	UNKNOWN	UNKNOWN

Instrument Type	Manufacturer	Model Number	Serial Number	Year Put into Service	Condition When Received
pH Meter	Denver Instruments	UB-5	UB503B365	UNKNOWN	UNKNOWN
Soxtherm	Gerhardt (SOXA)	SE3AS306A	4012396	UNKNOWN	UNKNOWN
Soxtherm	Gerhardt (SOXB)	SE3AS306A	4022047	UNKNOWN	UNKNOWN
Soxtherm	Gerhardt (SOXC)	SE3AS306A	4022046	UNKNOWN	UNKNOWN
Soxtherm	Gerhardt (SOXD)	SE3AS306A	4022045	UNKNOWN	UNKNOWN
Soxtherm	Gerhardt (SOXE)	SE3AS306A	4022030	UNKNOWN	UNKNOWN
Soxtherm	Gerhardt (SOXF)	SE3AS306A	4012397	UNKNOWN	UNKNOWN
Soxtherm	Gerhardt (SOXG)	SE-416	4051586	2012	USED
Soxtherm	Gerhardt (SOXH)	SE-416	4031739	2012	USED
Soxtherm	Gerhardt (SOXI)	SE-416	4031738	2012	USED
Soxtherm	Gerhardt (SOXJ)	SE-416	4031737	2012	USED
TKN Digestion System	Aim Lab	AIM600 Block	5048A23014	2011	NEW
Elemental Analyzer (TOC)	Carlo Erba	NA 1500	220465	1991	UNKNOWN
Elemental Analyzer (TOC)	Carlo Erba	EA1108	249146	1991	NEW
Nitrogen/Protein Analyzer	Costech	4010	231009973	2005	UNKNOWN
TOC Analyzer	Shimadzu	TOC-V CPH	H51314800321AE	2011	NEW
Turbidimeter	HF Scientific	Micro 100	208463	2001	UNKNOWN
Spectrophotometer	Genesys	Spectronic 20	3SGB029021	1999	UNKNOWN
Spectrophotometer	Genesys	Spectronic 20	3SGE165024	2002	UNKNOWN
Flow Injection Analyzer	Lachat	QuikChem 8000	A83000-2167	2000	UNKNOWN

Table 20-2. Example: Schedule of Routine Maintenance

Instrument	Procedure	Frequency
Leeman Mercury Analyzer	Check Peristaltic Pump tubing	As required
	Lubricate Autosampler rods	Monthly
	Clean Autosampler	Weekly
	Check and fill Rinse Vessel	As required
	Check and fill Stannous Chloride	As required
	Check Waste Vessel	Daily
	Empty Waste Vessel	As required
ICP	Check Peristaltic Pump tubing	As required
	Clean Torch	Daily
	Replace Torch	As required
	Check and fill Rinse Vessel	As required
	Check and fill IS Vessel	As required
	Fill Standards Cup	Daily
	Check Waste Vessel	Daily
	Empty Waste Vessel	As required
	Check and clean Cones	As required
	Perform Auto Peak Adjustment	As required
ICP MS	Check Peristaltic Pump tubing	As required
	Clean Torch	As required
	Check and fill Rinse Vessel	As required
	Check and fill IS Vessel	As required
	Fill standards cup	Daily
	Check Waste Vessel	Daily
	Empty Waste Vessel	As required
	Check and clean Cones	As required
UV-Vis Spectrophotometer	Clean ambient flow cell	As required
	Wavelength verification check	As required
	Clean Cuvette with Cuvette Cleaning Solution	As required
Hewlett Packard GC/MS (VOA)	Clean Injection Port and Liner	As required
	Change Septa	As required
	Cut 2-3 inches from GC Column	As required
	Fill Autosampler rinse vials	As required
	Clean Purge and Trap mount and purge vessel	As required
	Check Purge Flow	As required

Instrument	Procedure	Frequency
Hewlett Packard GC/MS (SVOA)	Clean Injection Port and Liner	Daily
	Change Septa	Daily
	Replace or clip Guard Column	Daily
	Replace or clip Analytical Column	Daily
	Fill Autosampler rinse vials	Daily
Hewlett Packard GC/MS (Air)	Check GC / Entech Column Interface	As required
	Check Nitrogen Tank Volume	As required
	Check Nitrogen Valves Software and Valves	As required
	Cut 2-3 inches from GC Column	As required
Gas Chromatograph	Replace Septa	As required
	Clean and replace Injection Port Liner	As required
	Replace or clip Guard Column	As required
	Replace or clip Analytical Column	As required
	Bake, Re-foil, Refurbish Detector	As required
Zero Air Generator	Change pre-filter cartridge	Annually
	Replace catalyst module	Indicator Light Blinks
	Check Indicator Beads in Moisture Filters	Daily
	Bake and Refill Mol Sieve Dry Rite Beads	As required
Hydrogen Generator	Fill Water Reservoir	Daily
	Replace Water in Water Reservoir	Semi-Annually
	Replace Ionic Bags in Water Reservoir	Semi-Annually
HPLC	Change Transfer Lines	As required
	Replace Guard Column	As required
	Replace Analytical Column	As required
	Replace or clean Pump Head Check Valves	As required
	Change Plunger Seals	As required
	Change Suppressor	As required
	Change Eluent Generator Cartridge and CR-ATC	As required
LC/MS/MS	Replace Guard Column	As required
	Replace Analytical Column	As required
	Replace or clean Pump Head Check Valves	As required
	Change Plunger Seals	As required
	Change In Line Filter	As required
	Clean or Change Sample Cone	As required
	Clean Source	As required
Balances	Class “1” traceable weight check	Daily, when used
	Clean pan and check if level	Daily
	Field service	Annually
Latchat	Change Tubing	As required
	Replace Bulb	As required
Conductivity Meter	Calibrate	Daily
Turbidimeter	Calibrate	As required
	Check light bulb	Daily, when used
Drying Ovens	Temperature monitoring	Daily
	Temperature adjustments	As required
Refrigerators/ Freezers	Temperature monitoring	Daily
	Temperature adjustment	As required
	Defrosting/cleaning	As required
pH/Specific Ion Meter	Calibrate	Daily
	Clean electrode	As required
Centrifuge	Check brushes and bearings	Every 6 months or as needed

Instrument	Procedure	Frequency
Water baths	Temperature monitoring Water replaced	Daily, when used Monthly or as needed

SECTION 21. MEASUREMENT TRACEABILITY

21.1 Overview

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 20.3). With the exception of Class A Glassware and Glass microliter syringes quarterly accuracy checks are performed for all mechanical volumetric devices. Wherever possible, subsidiary or peripheral equipment is checked against standard equipment or standards that are traceable to national or international standards. Class A Glassware and Glass microliter syringes should be routinely inspected for chips, acid etching or deformity (e.g., bent needle). If the Class A glassware or syringe is suspect, the accuracy of the glassware will be assessed prior to use.

21.2 NIST-Traceable Weights and Thermometers

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.

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 mercury thermometers are calibrated annually against a traceable reference thermometer. Temperature readings of ovens, refrigerators, and incubators are checked on each day of use.

21.3 Reference Standards / Materials

Reference standards/materials, where commercially available, are traceable to certified reference materials. Commercially prepared standard materials are purchased from vendors 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. The receipt of all reference standards must be documented. Reference standards are labeled with a unique Standard Identification Number and expiration date. All documentation received with the reference standard is retained as a QC record and references the Standard Identification Number.

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, such as air analysis 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 the Corporate Environmental Health & Safety Manual or laboratory SOPs. For safety requirements, please refer to method SOPs and the laboratory Environmental Health and Safety Manual.

Standards and reference materials shall not be used after their expiration dates unless their reliability is verified by the laboratory and their use is approved by the Quality Assurance Manager. The laboratory must have documented contingency procedures for re-verifying expired standards.

21.4 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 each lab section. 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 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.

21.4.1 All standards, reagents, and reference materials must be labeled in an unambiguous manner. Standards are logged into the laboratory's LIMS system, and are assigned a unique identification number. The following information is typically recorded in the electronic database within the LIMS.

- Standard ID
- Description of Standard
- Preparer's name
- Final volume
- Solvent type and lot number
- Preparation Date
- Expiration Date
- Parent standard ID (if applicable)
- Parent Standard Analyte Concentration (if applicable)
- Parent Standard Amount used (if applicable)
- Component Analytes
- Final concentration of each analyte
- Comment box (text field)

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.

21.4.2 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
- Special Health/Safety warnings if applicable

Records must also be maintained of the date of receipt for commercially purchased items or date of preparation for laboratory prepared items. Special Health/Safety warnings must also be available to the analyst. This information is maintained on the company's intranet and in test method SOPs.

21.4.3 In addition, the following information may be helpful:

- 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 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.

SECTION 22. SAMPLING

22.1 Overview

The laboratory does not provide sampling services. The laboratory's responsibility in the sample collection process lies in supplying the sampler with the necessary coolers, reagent water, sample containers, preservatives, sample labels, custody seals, COC forms, ice, and packing materials required to properly preserve, pack, and ship samples to the laboratory

22.2 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.

22.2.1 Preservatives

Upon request, preservatives are provided to the client in pre-cleaned sampling containers. In some cases containers may be purchased pre-preserved from the container supplier. Whether prepared by the laboratory or bought pre-preserved, 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

22.3 Definition of Holding Time

The date and time of sampling documented on the 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 programs that determine holding time

compliance based on the date and specific time of analysis compared to the time of sampling regardless of how long the holding time is.

22.4 Sampling Containers, Preservation Requirements, Holding Times

The preservation and holding time criteria specified in the laboratory SOPs are derived from the source documents for the methods. If method required holding times 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.

22.5 Sample Aliquots / Subsampling

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 subsample or aliquot of the sample provided for analysis.

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.

Guidelines on taking sample aliquots & subsampling are located in test method SOPs.

SECTION 23. HANDLING OF SAMPLES

Sample management procedures at the laboratory ensure that sample integrity and custody are maintained and documented from sampling/receipt through disposal.

23.1 Chain of Custody (COC)

The COC form is the written documented history of any sample and is 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 23-1.

23.1.1 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 23-1). This form should include 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
- The container description
- The total number of each type of container
- 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.

When the sampling personnel deliver the samples directly to TestAmerica personnel, 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 personnel. 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. When sampling personnel deliver the samples through a common carrier (Fed-Ex, UPS), the CoC relinquished date/time is completed by the field personnel and samples are released to the carrier. Samples are only considered to be received by lab when personnel at the fixed laboratory facility 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 receipt from the courier is stored in log-in by date; it lists all receipts each date.

23.1.2 Legal / Evidentiary Chain-of-Custody

If the client requests legal COC sample management personnel will initiate an internal COC for laboratory use by analysts and a sample disposal record.

23.2 Sample Receipt

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.

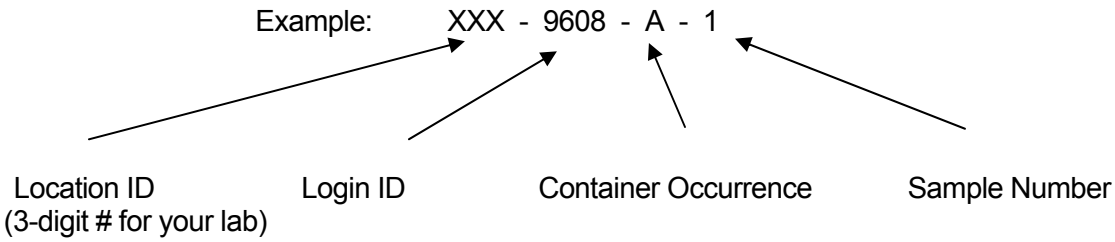
23.2.1 Laboratory Receipt

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*]* and brought to the immediate attention of 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.

23.2.1.1 Unique Sample Identification

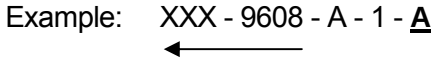
All samples that are processed through the laboratory receive a unique sample identification to ensure that there can be no confusion regarding the identity of such samples at anytime. This system includes identification for all samples, subsamples and subsequent extracts and/or digestates.

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 <location> Laboratory (Location XXX). 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.



Secondary Container Occurrence

Example: 220-9608-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.

23.3 Sample Acceptance Policy

The laboratory has a written sample acceptance policy (Figure 23-2) that clearly outlines the circumstances under which samples shall be accepted or rejected. These include:

- a complete COC;
- 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);
- 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.

23.3.1 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.

23.3.2 Any deviations from these checks 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 are not met, the laboratory shall either:

- Retain all correspondence and/or records of communications with the client regarding the disposition of rejected samples, or

- Fully document any decision to proceed with sample analysis that does not meet sample acceptance criteria.

Once sample acceptance is verified, the samples are logged into the LIMS according to laboratory SOP BR-SM-001.

23.4 Sample Storage

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, freezers or protected locations suitable for the sample matrix. 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 weekly.

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 until disposal.

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.

23.5 Hazardous Samples and Foreign Soils

To minimize exposure to personnel and to avoid potential accidents, hazardous and foreign soil samples are stored in an isolated area designated for hazardous waste only. For any sample that is known to be hazardous at the time of receipt or, if after completion of analysis the result exceeds the acceptable regulatory levels, a Hazardous Sample Notice must be completed by the analyst. This form may be completed by Sample Control, Project Managers, or analysts and must be attached to the report. The sample itself is clearly marked with a red stamp, stamped on the sample label reading “HAZARDOUS” or “FOREIGN SOIL” and placed in a colored and/or marked bag to easily identify the sample. The date, log number, lab sample number, and the result or brief description of the hazard are all written on the Hazardous & Foreign Soil Sample Notice. A copy of the form must be included with the original COC and Work Order and the original must be given to the Sample Control Custodian. Analysts will notify Sample Control of any sample determined to be hazardous after completion of analysis by completing a Hazardous Sample Notice. All hazardous samples are either returned to the client or disposed of appropriately through a hazardous waste disposal firm that lab-packs all hazardous samples and removes them from the laboratory.

23.6 Sample Shipping

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 is enclosed for those samples requiring water/solid volatile organic analyses (see Note). The chain-of-custody form is signed by the sample control technician 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.

23.7 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: BR-EH-001) All procedures in the laboratory Environmental, Health and Safety Manual are followed during disposal. Samples are normally maintained in the laboratory no longer than two 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, 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 should be completed.

Figure 23-1. Example: Chain of Custody (COC)

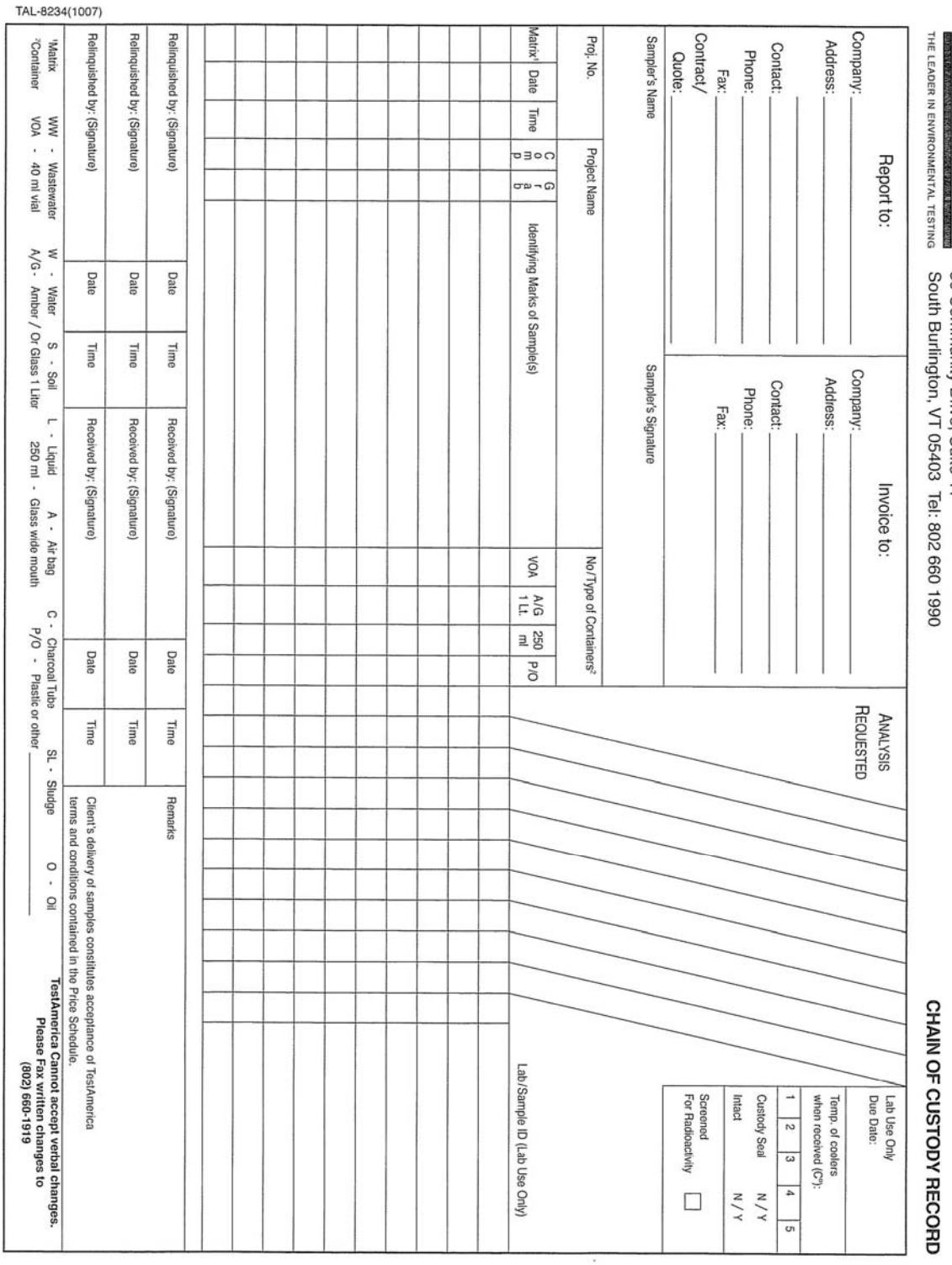


Figure 23-2. Example: Sample Acceptance Policy

The receipt of samples is acknowledged on the chain of custody (COC) form with the signature and date/time of the sample custodian. The condition of samples upon receipt is documented on checklists designated for this purpose. Any deficiencies identified during sample receipt are recorded and communicated to the laboratory project manager (PM), who will contact the client and fully document any decision to proceed with analysis in the project record. Consultation with the client should be immediate and timely (next business day or as specified in the project plan). Correspondence records and/or records of conversations concerning the decision to proceed with analysis and/or the disposition of rejected samples is maintained in the project record, and should be maintained in association with the sample receipt checklist. All data associated with samples that did not meet the sample acceptance criteria must be qualified with a Non-Conformance Report (NCR) and/or noted in the project narrative that accompanies the final test report.

Sample receipt is considered deficient when the following conditions are observed:

- Shipping cooler and/or samples are received outside the temperature specification
- Sample bottles are received broken or leaking
- Samples are received beyond holding time
- Samples are received without the appropriate preservation
- Samples are not received in appropriate containers
- Chain of Custody does not match the samples received
- Chain of Custody was not received or is incomplete*
- Custody seals are broken
- Evidence of tampering with the cooler and/or samples
- Headspace in 40mL or 22 mL VOA vials
- Seepage of extraneous water or other material into the samples
- Inadequate sample volume
- Illegible, impermanent ink, or non-unique sample labeling
- One or more coolers missing from a multi parcel shipment
- Shipping container is damaged

**Complete documentation shall include sample identification, the location date/time of collection, collector's name, preservation type, sample type and any special remarks concerning the sample.*

Figure 23-3. Example: Cooler Receipt Form

TestAmerica Burlington
SAMPLE RECEIPT & LOG IN CHECKLIST

Client:	**Date Received:**	**Job #:**
Project #:	**Time Received:**	
PM:	**Received By:**	**Login#:**
Login Date:	**# Coolers Received:**	
	Samples Delivered By:	**ICOC Required? Y/N**
Initials:	⊐ Shipping Service	*If "Y", attach copy(s) of ICOC*
Signature:	⊐ Courier	
	⊐ Hand	

Receipt Info	**YES**	**NO**	**NA**	**COMMENTS**
There is ***no*** evidence to indicate tampering				
Custody seals are present and intact				
Custody seal numbers are present				
If yes, list custody seal numbers:				

IR Gun ID: **Correction Factor:** ° C

Thermal Preservation Type: ⊐ Wet Ice ⊐ Blue Ice ⊐ None ⊐ Other (specify)
Packing Material: ⊐ Bubble Wrap ⊐ Cardboard ⊐ Corrugated Paper ⊐ Shredded Paper ⊐ Styrofoam ⊐ Vermiculite ⊐ None

Cooler 1:	°C	Cooler 6	°C	Cooler 11	°C	Cooler 16	°C
Cooler 2:	°C	Cooler 7	°C	Cooler 12	°C	Cooler 17	°C
Cooler 3:	°C	Cooler 8	°C	Cooler 13	°C	Cooler 18	°C
Cooler 4:	°C	Cooler 9	°C	Cooler 14	°C	Cooler 19	°C
Cooler 5	°C	Cooler 10	°C	Cooler 15	°C	Cooler 20	°C

Unless otherwise documented, the recorded temperature readings are adjusted readings to account for the CF of the IR Gun
EPA Criteria: 0-6°C, except for air and geo samples which should be at ambient temperature and tissue samples, which may be frozen.
Some clients require thermal preservation criteria of 2-4°C or other such criteria. The PM must notify SM when alternate criteria is specified.
Comments:

Report Management-*Workshare (TALS Labs)*	**YES**	**NA**	**Initials**	**Date**
Workshare (TALS Labs)-Shipping and Receiving Documents scanned and attached to job.				
Email notification sent to job contact				
Report Management-*Login Review*	**YES**	**NA**	**Initials**	**Date**
Shipping and Receiving Documents: Scanned and attached to deliverable job folders.				
Project Manager-*Login Review*	**YES**		**Initials**	**Date**
Login Review Performed				
EDD Questions	**YES**	**NA**	**Initials**	**Date**
Applicable login/job questions answered				
Report Management- *Preliminary Deliverables*	**YES**	**NA**	**Initials**	**Date**
Lab Documents-Scanned and attached to appropriate job deliverables folder.				
Subcontract Data (if applicable)- Non-TALS Labs, 3rd Party Labs Scanned and attached to appropriate job deliverables folders				
Narrative-NCMs added, if present				
Preliminary Reports-run and assembled				
EDD(s)-run and created				
Project Manager	**YES**		**Initials**	**Date**
Final review and release completed				
Report Management-*Final Deliverables*	**YES**	**NA**	**Initials**	**Date**
Reports-Hardcopy printed (if requested), CD(s) burned				
Invoice-Approved in Invoice Desktop and printed (if hardcopy requested)				
Email-Report(s), EDD(s), and invoice (as requested)				
Delivery confirmed for report(s), EDD(s) and Invoice in TALS				

BR-FSR002:09.19.11:4
TestAmerica

SECTION 24. ASSURING THE QUALITY OF TEST RESULTS

24.1 Overview

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 20, 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.

24.2 Controls

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.

24.3 Negative Controls

Table 24-1. Example – Negative Controls

Control Type	Details
Method Blank (MB)	are used to assess preparation and analysis for possible contamination during the preparation and processing steps.
	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.
	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. The method blank goes through all of the steps of the process (including as necessary: filtration, clean-ups, etc.).
	Reanalyze or qualify associated sample results when the concentration of a targeted analyte in the blank is at or above the reporting limit as established by the method or by regulation, AND is greater than 1/10 of the amount measured in the sample.
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.
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.

Table 24-1. Example – Negative Controls

Control Type	Details
Trip Blank [1]	are required to be submitted by the client with each shipment of samples requiring aqueous and solid volatiles analyses (or as specified in the client's project plan). 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.
Field Blanks [1]	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)
Equipment Blanks [1]	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. (TNI)
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

[1] When known, these field QC samples 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.

24.4 Positive Controls

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) (Matrix spikes are not applicable to air) 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.

24.4.1 Method Performance Control - Laboratory Control Sample (LCS)

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.

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.

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.).

The specific frequency of use for LCS during the analytical sequence is defined in the specific standard operating procedure for each analysis. It is generally 1 for each batch of samples; not to exceed 20 environmental samples.

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.

- For methods that have 1-10 target analytes, spike all components.
- For methods that include 11-20 target analytes, spike at least 10 or 80%, whichever is greater.
- For methods with more than 20 target analytes, spike at least 16 components.
- Exception: Due to analyte incompatibility in pesticides, Toxaphene and Chlordane are only spiked at client request based on specific project needs.
- 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.

24.5 Sample Matrix Controls

Table 24-2. Sample Matrix Control

Control Type	Details	
Matrix Spikes (MS)	Use	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;

Table 24-2. Sample Matrix Control

Control Type	Details	
	Typical Frequency [1]	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.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. Refer to the method SOP for complete details
	Description	essentially a sample fortified with a known amount of the test analyte(s).
Surrogate	Use	Measures method performance to sample matrix (organics only).
	Typical Frequency [1]	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. 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.
	Description	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.
Duplicates[2]	Use	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.
	Typical Frequency [1]	Duplicate samples are usually analyzed with methods that do not require matrix spike analysis.
	Description	Performed by analyzing two aliquots of the same field sample independently or an additional LCS.
Internal Standards	Use	Are spiked into all environmental and quality control samples (including the initial calibration standards) to monitor the qualitative aspect of organic and some inorganic analytical measurements.
	Typical Frequency [1]	All organic and ICP methods as required by the analytical method.
	Description	Used to correct for matrix effects and to help troubleshoot variability in analytical response and are assessed after data acquisition. Possible sources of poor internal standard response are sample matrix, poor analytical technique or instrument performance.

[1] See the specific analytical SOP for type and frequency of sample matrix control samples.

[2] 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.

24.6 Acceptance Criteria (Control Limits)

As mandated by the test method and regulation, each individual analyte in the LCS, MS, or Surrogate Spike is evaluated against the control limits 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 control limits. When this occurs, the regulatory or project limits will supersede the laboratory's in-house limits.

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.

Once control limits have been established, they are verified, reviewed, and updated when necessary unless the method requires more frequent updating. Control limits are established per method (as opposed to per instrument) regardless of the number of instruments utilized.

Laboratory generated % Recovery acceptance (control) limits are generally established by taking ± 3 Standard Deviations (99% confidence level) from the average recovery of a minimum of 20-30 data points (more points are preferred).

- 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).

- 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 laboratory control limits are not consistent with DQOs, then alternatives must be considered, such as method improvements or use of an alternate analytical method.

- 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.

24.6.1 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.

24.6.2 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 12) is also initiated if an LCS exceeds the acceptance limits. Sample results may be qualified and reported without reanalysis if:

- The analyte results are below the reporting limit and the LCS is above the upper control limit.

- If the analytical results are above the relevant regulatory limit and the LCS is below the lower control limit.

24.6.3 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 the lab's method SOPs and in Section 12.

24.6.4 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).

24.7 Additonal Procedures to Assure Quality Control

The laboratory has written and approved method SOPs to assure the accuracy of the test method including calibration (see Section 20), use of certified reference materials (see Section 21) and use of PT samples (see Section 15).

A discussion regarding MDLs, Limit of Detection (LOD) and Limit of Quantitation (LOQ) can be found in Section 19.

- Use of formulae to reduce data is discussed in the method SOPs and in Section 20.
- Selection of appropriate reagents and standards is included in Section 9 and 21.
- A discussion on selectivity of the test is included in Section 5.
- Constant and consistent test conditions are discussed in Section 18.
- The laboratories sample acceptance policy is included in Section 23.

SECTION 25. REPORTING RESULTS

25.1 Overview

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 conflict between client requests and laboratory ethics or regulatory requirements, the laboratory's ethical and legal requirements are paramount, and the laboratory will work with the client during project set up to develop an acceptable solution. Refer to Section 7.

The format of each report type is specific to the client or regulatory program and is therefore not included in the QAM.

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 19.

25.2 Test Reports

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 is printed on laboratory letterhead, reviewed, and signed by the appropriate project manager. At a minimum, the standard laboratory report shall contain the following information:

25.2.1 A report title (e.g. Analytical Report For Samples) with a "sample results" column header.

25.2.2 Each report cover page printed on company letterhead, which includes the laboratory name, address and telephone number.

25.2.3 A unique identification of the report 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.

25.2.4 A copy of the chain of custody (COC).

- Any COCs involved with Subcontracting are included.

25.2.5 The name and address of client and a project name/number, if applicable.

25.2.6 Client project manager or other contact

25.2.7 Description and unambiguous identification of the tested sample(s) including the client identification code.

25.2.8 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.

25.2.9 Date reported or date of revision, if applicable.

25.2.10 Method of analysis including method code (EPA, Standard Methods, etc).

25.2.11 Practical quantitation limits or reporting limit.

25.2.12 Method detection limits (if requested)

25.2.13 Definition of Data qualifiers and reporting acronyms (e.g. ND).

25.2.14 Sample results.

25.2.15 QC data consisting of method blank, surrogate, LCS, and MS/MSD recoveries and control limits.

25.2.16 Condition of samples at receipt including temperature. This may be accomplished in a narrative or by attaching sample login sheets (Refer to Sec. 25.2.4 – Item 3 regarding additional addenda).

25.2.17 A statement to the effect that the results relate only to the items tested and the sample as received by the laboratory.

25.2.18 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.

25.2.19 When NELAC accreditation is required, the lab shall certify that the test results meet all requirements of TNI Standard or provide reasons and/or justification if they do not.

25.2.20 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.

25.2.21 When soil samples are analyzed, a specific identification as to whether soils are reported on a "wet weight" or "dry weight" basis.

25.2.22 Appropriate laboratory certification number for the state of origin of the sample, if applicable.

25.2.23 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., partial report, or how your lab identifies it). A complete report must be sent once all of the work has been completed.

25.2.24 Any non-TestAmerica subcontracted analysis results are provided as a separate report on the official letterhead of the subcontractor. All TestAmerica subcontracting is clearly identified on the report as to which laboratory performed a specific analysis.

25.2.28 A clear statement notifying the client that non-accredited tests were performed and directing the client to the laboratory's accreditation certificates of approval shall be provided when non-accredited tests are included in the report.

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.

25.3 Reporting Level or Report Type

The laboratory routinely offers four levels of quality control reporting.

- Level I is a report with the features described in Section 25.2 above except QC summary information is not included.
- Level II is a Level I report plus QC summary information.
- Level III contains all the information supplied in Level II, but presented on CLP-like summary forms, and relevant calibration information. No raw data is provided.
- Level IV is the same as Level III with the addition of all raw supporting data.

The format of each report type is specific to the client or regulatory program and is therefore not included in the QAM. The reporting specifications for CLP contract samples must comply with the specifications for CSF organization, preparation and review as specified in the SOW. Procedures for preparation of the CSF are provided in laboratory SOP BR-RM-001.

25.3.1 Electronic Data Deliverables (EDDs)

EDDs are routinely offered as part of TestAmerica's services. TestAmerica Burlington offers a variety of EDD formats including Environmental Restoration Information Management System (ERPIMS), New Agency Standard (NAS), Format A, Excel, Dbase, GISKEY, and Text Files.

EDD specifications are submitted to the IT department by the PM for review and undergo the contract review process. 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. The validation of the code is retained by the IT staff coding the EDD.

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.

25.4 Supplemental Information for Test

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.

Numeric results with values outside of the calibration range, either high or low are qualified as 'estimated'.

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 sample acceptance requirements such as improper container, holding time, or temperature.

Where applicable, a statement on the estimated uncertainty of measurements; information on uncertainty is needed when a client's instructions so require.

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.

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.

25.5 Environmental Testing Obtained From Subcontractors

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).

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 TestAmerica are reported to the client on the subcontract laboratory's original report stationary and the report includes any accompanying documentation.

25.6 Client Confidentiality

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.

25.6.1 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:

This material is intended only for the use of the individual(s) or entity to whom it is addressed, and may contain information that is privileged and confidential. If you are not the intended recipient, or the employee or agent responsible for delivering this material to the intended recipient, you are hereby notified that any dissemination, distribution or copying of this communication is strictly prohibited. If you have received this communication in error, please notify us immediately by telephone at the 1-800-765-0980 (or for e-mails: please notify us immediately by e-mail or by phone (1-800-765-0980) and delete this material from any computer).

25.7 Format of Reports

The format of reports is designed to accommodate each type of environmental test carried out and to minimize the possibility of misunderstanding or misuse.

25.8 Amendments to Test Reports

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 12).

The revised report is retained on the Archive data server, as is the original report. The revised report will have the word "revised" or "amended" next to the date rather than the word "reported".

When the report is re-issued, a notation of "report re-issue "is placed on the cover/signature page of the report *or at the top of the narrative page* with a brief explanation of reason for the re-issue and a reference back to the last final report generated. *For Example*: *Report was revised on 11/3/08 to include toluene in sample NQA1504 per client's request. This final report replaces the final report generated on 10/27/08 at 10:47am.*

25.9 Policies on Client Requests for Amendments

25.9.1 Policy on Data Omissions or Reporting Limit Increases

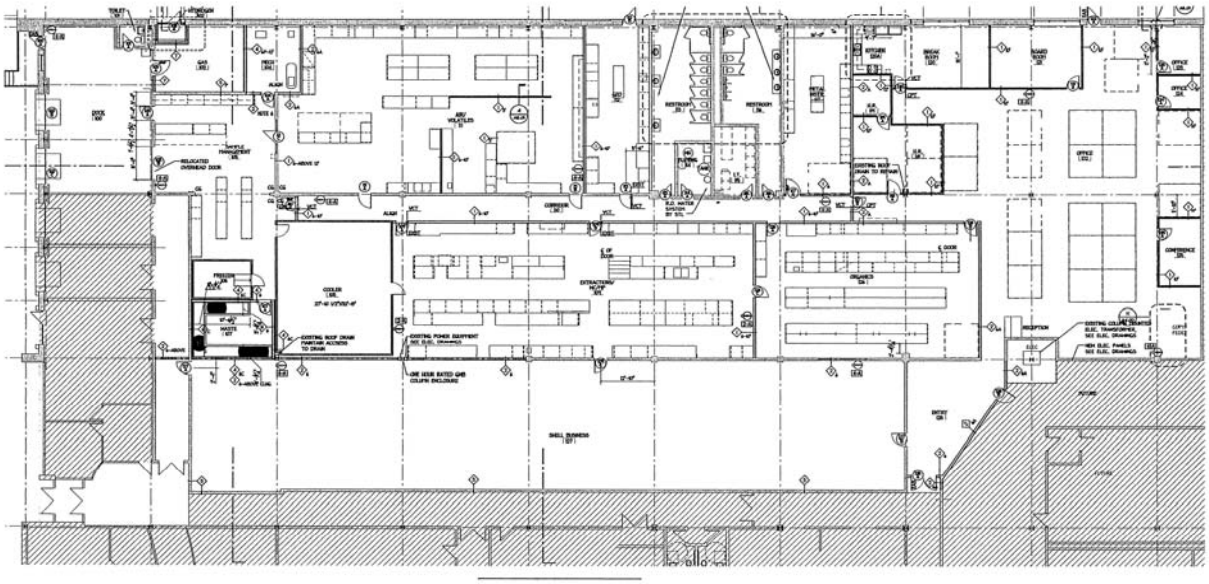
Fundamentally, our policy is simply to not omit previously reported results (including data qualifiers) or to not raise reporting limits and report sample results as ND. This policy has few exceptions. Exceptions are:

- 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.

25.9.2 Multiple Reports

TestAmerica does not issue multiple reports for the same work order 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. Laboratory Floor Plan



Appendix 2. Glossary/Acronyms (EL-V1M2 Sec. 3.1)

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.

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.

Analytical Uncertainty: A subset of Measurement Uncertainty that includes all laboratory activities performed as part of the analysis. (TNI)

Assessment: The evaluation process used to measure or establish the performance, effectiveness, and conformance of an organization and/or its systems to defined criteria (to the standards and requirements of laboratory accreditation). (TNI)

Audit: A systematic and independent examination of facilities, equipment, personnel, training, procedures, record-keeping, data validation, data management, and reporting aspects of a system to determine whether QA/QC and technical activities are being conducted as planned and whether these activities will effectively achieve quality objectives. (TNI)

Batch: Environmental samples that 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 (1) to twenty (20) environmental samples of the same quality systems 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 twenty-four (24) hours. An **analytical batch** is composed of prepared environmental samples (extracts, digestates or concentrates) which are analyzed together as a group. An analytical batch can include prepared samples originating from various quality system matrices and can exceed twenty (20) samples. (TNI)

Bias: The systematic or persistent distortion of a measurement process, which causes errors in one direction (i.e., the expected sample measurement is different from the sample's true value). (TNI)

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)

Calibration: A set of operations that establish, under specified conditions, the relationship between values of quantities indicated by a measuring instrument or measuring system, or values represented by a material measure or a reference material, and the corresponding values realized by standards. (TNI)

1) In calibration of support equipment the values realized by standards are established through the use of reference standards that are traceable to the International System of Units (SI).

2) In calibration according to methods, the values realized by standards are typically established through the use of Reference Materials that are either purchased by the laboratory with a certificate of analysis or purity, or prepared by the laboratory using support equipment that has been calibrated or verified to meet specifications.

Calibration Curve: The mathematical ~~r~~elationship between the known values, such as concentrations, of a series of calibration standards and their instrument response. (TNI)

Calibration Standard: A substance or reference material used to calibrate an instrument (QAMS)

Certified Reference Material (CRM): A reference material~~,~~ accompanied by a certificate, having a value, measurement uncertainty, and stated metrological traceability chain to a national metrology institute. (TNI)

Chain of Custody (COC) Form: Record that documents the possession of the samples from the time of collection to receipt in the laboratory. This record generally includes: the number and types of containers; the mode of collection; the collector; time of collection; preservation; and requested analyses. (TNI)

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.

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. 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. (TNI)

Conformance: An affirmative indication or judgment 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).

Data Reduction: The process of transforming the number of data items by arithmetic or statistical calculations, standard curves, and concentration factors, and collation into a more useable form. (TNI)

Deficiency: An unauthorized deviation from acceptable procedures or practices, or a defect in an item. (ASQC)

Demonstration of Capability: A procedure to establish the ability of the analyst to generate analytical results of acceptable accuracy and precision. (TNI)

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)

Equipment Blank: Sample of analyte-free media which has been used to rinse common sampling equipment to check effectiveness of decontamination procedures.
External Standard Calibration: Calibrations for methods that do not utilize internal standards to compensate for changes in instrument conditions.

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 of Accreditation: hose matrix, technology/method, and analyte combinations for which the NELAP accreditation body offers accreditation.

Holding Times: The maximum time that samples may be held prior to analyses and still be considered valid or not compromised. (40 CFR Part 136)

Internal Standard: A known amount of standard added to a test portion of a sample as a reference for evaluating and controlling the precision and bias of the applied analytical test method. (TNI)

Internal Standard Calibration: Calibrations for methods that utilize internal standards to compensate for changes in instrument conditions.

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.

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 of the procedure unless otherwise noted in a reference method. 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.

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(s) of Detection (LOD) [a.k.a., Method Detection Limit (MDL)]: A laboratory's estimate of the minimum amount of an analyte in a given matrix that an analytical process can reliably detect in their facility. (TNI)

LOD Verification [a.k.a., MDL Verification]: A processed QC sample in the matrix of interest, spiked with the analyte at no more than 3X the LOD for single analyte tests and 4X the LOD for multiple analyte tests and processed through the entire analytical procedure.

Limit(s) of Quantitation (LOQ) [a.k.a., Reporting Limit]: The minimum levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be reported with a specified degree of confidence. (TNI)

(QS) 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 or Saline/Estuarine. 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 & Emissions: 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. (TNI)

Matrix Spike (spiked sample or fortified sample): A sample prepared, taken through all sample preparation and analytical steps of the procedure unless otherwise noted in a referenced method, by adding a known amount of target analyte to a specified amount of sample for which an independent test

result 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 Spike Duplicate (spiked sample or fortified sample duplicate): A replicate matrix spike prepared and analyzed to obtain a measure of the precision of the recovery for each analyte.

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.

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)

Negative Control: Measures taken to ensure that a test, its components, or the environment do not cause undesired effects, or produce incorrect test results.

Non-conformance: An indication, judgment, or state of not having met the requirements of the relevant specifications, contract, or regulation.

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.

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.

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. (TNI)

Preservation: Any conditions under which a sample must be kept in order to maintain chemical and/or biological integrity prior to analysis. (TNI)

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. (TNI)

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. (TNI)

Proficiency Test Sample (PT): A sample, the composition of which is unknown to the laboratory and is provided to test whether the aboratory can produce analytical results within specified acceptance criteria. (TNI)

Quality Assurance: An integrated system of management activities involving planning, implementation, assessment, reporting and quality improvement to ensure that a process, item, ~~product~~ or service is of the type of quality needed and expected by the client. (TNI)

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 that measures the attributes and performance of a process, item, or service against defined standards to verify that they meet the stated requirements established by the customer; operational techniques and activities that are used to fulfill requirements for quality; also the system of activities and checks used to ensure that measurement systems are maintained within prescribed limits, providing protection against "out of control" conditions and ensuring that the results are of acceptable quality. (TNI)

Quality Control Sample: A sample used to assess the performance of all or a portion of the measurement system. One of any number of samples, such as Certified Reference Materials, a quality system matrix fortified by spiking, or actual samples fortified by spiking, intended to demonstrate that a measurement system or activity is in control. (TNI)

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. (TNI)

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 activities. (TNI)

Raw Data: The documentation generated during sampling and analysis. This documentation includes, but is not limited to, field notes, electronic data, magnetic tapes, untabulated sample results, QC sample results, print outs of chromatograms, instrument outputs, and handwritten records. (TNI)

Record Retention: The systematic collection, indexing and storing of documented information under secure conditions.

Reference Material: Material or substance one or more properties of which are sufficiently homogeneous and well established to be used for the calibration of an apparatus, the assessment of a measurement method, or for assigning values to materials. (TNI)

Reference Standard: Standard used for the calibration of working measurement standards in a given organization or a given location. (TNI)

Sampling: Activity related to obtaining a representative sample of the object of conformity assessment, according to a procedure.

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: The ability to analyze, distinguish, and determine a specific analyte or parameter from another component that may be a potential interferent or that may behave similarly to the target analyte or parameter within the measurement system. (TNI)

Sensitivity: The capability of a method or instrument to discriminate between measurement responses representing different levels (e.g., concentrations) of a variable of interest. (TNI)

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.

Standard: The document describing the elements of laboratory accreditation that has been developed and established within the consensus principles of standard setting and meets the approval requirements of standard adoption organizations procedures and policies. (TNI)

Standard Operating Procedures (SOPs): A written document which details the method for an operation, analysis, or action, with thoroughly prescribed techniques and steps. SOPs are officially approved as the methods for performing certain routine or repetitive tasks. (TNI)

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)

Technology: A specific arrangement of analytical instruments, detection systems, and/or preparation techniques.

Traceability: The ability to trace the history, application, or location of an entity by means of recorded identifications. In a calibration sense, traceability relates measuring equipment to national or international standards, primary standards, basic physical constants or properties, or reference materials. In a data collection sense, it relates calculations and data generated throughout the project back to the requirements for the quality of the project. (TNI)

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
DOC – Demonstration of Capability
DQO – Data Quality Objectives
DUP - 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
ICP/MS – ICP/Mass Spectrometry
ICV – Initial Calibration Verification
IDL – Instrument Detection Limit
IH – Industrial Hygiene
IS – Internal Standard
LCS – Laboratory Control Sample
LCSD – Laboratory Control Sample Duplicate
LIMS – Laboratory Information Management System
LOD – Limit of Detection
LOQ – Limit of Quantitation
MDL – Method Detection Limit
MDLV – MDL Verification Check Standard
MRL – Method Reporting Limit Check Standard
MS – Matrix Spike
MSD – Matrix Spike Duplicate
MSDS - Material Safety Data Sheet
NELAC - National Environmental Laboratory Accreditation Conference
NELAP - National Environmental Laboratory Accreditation Program
PT – Performance Testing
TNI – The NELAC Institute
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 – Standard Deviation
SOP – Standard Operating Procedure
TAT – Turn-Around-Time
VOA – Volatiles
VOC – Volatile Organic Compound

Appendix 3. Laboratory Certifications, Accreditations, Validations

TestAmerica Burlington maintains accreditation, certifications and approvals with numerous state and national entities. At the time of this QA Manual revision, the laboratory has accreditation/certification/licensing with the following organizations:

Lab ID	Program	Program Type	Authority
NA	Delaware DNREC		Delaware
ADE-1492	DoD ELAP	DoD	L-A-B
200610	NELAC	Secondary AB	New Hampshire
VT972	NELAC	Primary AB	New Jersey
10391	NELAC	Secondary AB	New York
68-00489	NELAC	Secondary AB	Pennsylvania
E87467	NELAC	Secondary AB	Florida
176292	NELAC	Secondary AB	Louisiana
PH-0751	State Program		Connecticut
VT00008	State Program		Maine
050-999-436	State Program		Minnesota
LAO00298	State Program		Rhode Island
VT-4000	State Program		Vermont
P330-11-00093	USDA		USDA

The certificates and parameter lists are available upon request from a laboratory representative. A complete list of analytical capabilities may be found on the company's web site, the laboratory's public server or from a representative of the laboratory.

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

TestAmerica Denver

BUSINESS CONFIDENTIAL INFORMATION
DO NOT COPY
DO NOT DISTRIBUTE

Electronic Document Cover

The electronic copy of this document, as maintained on the TestAmerica Denver computer network, is the controlled copy. Any printed copy becomes uncontrolled, and all work performed should ultimately reference the controlled electronic version.

Any printed or electronic copy of this document that is distributed external to TestAmerica Denver becomes uncontrolled. To arrange for automatic updates to this document, contact TestAmerica Denver.

TestAmerica Laboratories, Inc.
TestAmerica Denver
4955 Yarrow Street
Arvada, CO 80002

Phone: 303-736-0100
Fax: 303-431-7171

Electronic Copy Only

Title: Quality Assurance Program

Approvals (Signature/Date):

Margaret S. Sleevi 7/15/13
Margaret S. Sleevi Date
Quality Assurance Manager

William S. Cicero 7/15/13
William S. Cicero Date
Laboratory Director

Facility Distribution No. _____ **Distributed To:_____**

1.0 PURPOSE

This policy describes TestAmerica Denver's program of routine analytical quality control (QC) activities. The objective is to generate QC data that demonstrate that the analytical process is in control and that the data meet client and method requirements. The policy outlines QC requirements for a variety of regulatory programs, with the stipulation that lacking specific direction from our clients, TestAmerica Denver will default to routine RCRA program QC requirements. TestAmerica Denver Policy DV-QA-024P, Requirements for Federal Programs, should be consulted for quality control activities specific to analyses performed under programs for the Department of Defense (DoD), Airforce Center for Environmental Excellence (AFCEE), and the Department of Energy (DOE).

2.0 SCOPE

This policy is to be enforced and followed throughout the laboratory.

QUALITY POLICY STATEMENT

The management of TestAmerica and TestAmerica Denver are committed to providing data of known quality to its clients by adhering to approved methodologies, regulatory requirements and the QA/QC protocols described in this manual. In addition, management is committed to compliance with the NELAC Institute (TNI) standards, International ANS/ISO/IEC Standard 17025 Guide 17025 (2005) and the various accreditation & certification programs listed in Appendix 6. Management is also committed to continually improving the effectiveness of the management system.

In all aspects of the laboratory and business operations, management is dedicated in maintaining the highest ethical standards. Training on ethical and legal responsibilities is provided annually and each employee signs off annually on the policy as a condition of employment.

It is TestAmerica's Policy to continually improve systems and provide support to quality improvement efforts in laboratory, administrative and managerial activities. The company recognizes that the implementation of a quality assurance program requires management's commitment and support as well as the involvement of the entire staff.

TestAmerica Denver strives to provide clients with the highest level of professionalism and the best service practices in the industry.

Every staff member at TestAmerica Denver 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.

3.0 SAFETY

3.1 There are no specific safety hazards associated with this SOP.

3.2 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 Acceptance Criteria - The specified limits placed on characteristics of an item, process, or service defined in requirement documents.

4.2 Accuracy - The degree of agreement between an observed value and an accepted reference value.

4.3 Batch - As defined by NELAC, a batch consists of environmental samples that 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 samples of the same matrix, meeting the aforementioned criteria and with a maximum time between the start of processing of the first and last sample in the batch to be 24 hours. Field samples prepared at different dilutions are not duplicates and must be counted as separate samples toward the batch count of 20. An analytical batch is composed of prepared samples (e.g., extracts, digestates, or concentrates) that are analyzed together as a group. For QC purposes, if the number of samples in a group is greater than 20, then each group of 20 samples or less will all be handled as a separate batch.

4.4 QC Batch - The QC batch is a set of up to 20 field samples plus associated laboratory QC samples that are similar in composition (matrix) and that are processed within the same time period using the same reagents and standard lots.

4.5 Calibration - A set of operations that establish, under specified conditions, the relationship between values of quantities indicated by a measuring instrument or measuring system, or values represented by a material measure or a reference material, and the corresponding values realized by standards.

4.6 Corrective Action - The action taken to eliminate the causes of an existing nonconformity, defect, of other undesirable situation in order to prevent recurrence.

4.7 Instrument/Calibration Blank - The instrument blank is prepared using the same solvents and reagents (e.g., hexane, methylene chloride, or reagent water) used to dilute the prepared sample extracts or digests. Unlike the method blank, it is analyzed without being subject to the preparation steps of the analytical procedure. It is used to monitor laboratory or reagent contamination introduced at the instrumental analysis phase of work. For procedures without a separate preparation step, an instrument blank is equivalent to the method blank, and serves the same purpose.

4.8 Laboratory Control Sample (LCS) - The LCS consists of a well-characterized matrix (e.g., reagent water or Ottawa sand) that is known to be free of analytes of interest, and that is spiked with known and verified concentrations of representative analytes. Alternate matrices (e.g., glass beads) may be used for

soil analyses when Ottawa sand is not appropriate. As part of a QC batch, it accompanies the samples through all steps of the analytical process. The LCS is used to evaluate the performance of the total analytical system, including all preparation and analysis steps, independent of possible interference effects due to sample matrix.

4.9 Limit of Detection (LOD) - An estimate of the amount of a substance that an analytical process can reliably detect. An LOD is analyte-matrix-specific and may be laboratory-specific.

4.10 Limit of Quantitation (LOQ) - The minimum levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be reported with a specified degree of confidence.

4.11 Duplicate Control Sample (DCS) - A duplicate laboratory control sample (LCSD or DCS) may be prepared at the request of the client. It is required for some projects, particularly when insufficient sample volume is received to prepare and analyze an MS/MSD pair. LCS/LCSD pairs provide information regarding the precision of the measurement process.

4.12 Matrix Spike (MS) and Matrix Spike Duplicate (MSD)

Matrix Spike - A matrix spike (MS) is a replicate aliquot of one field sample in the QC batch that is spiked with known amounts of target analytes. An MS is spiked with the same analytes at the same concentrations that are added to the LCS. As part of the QC batch, it accompanies the field samples through all steps of the analytical process. Matrix spike data are meaningful only for the sample in which they are prepared and possibly for samples from the same site. The information obtained from MS data are sample/matrix specific and would not normally be used to determine the validity of the entire batch. However, a number of regulatory entities require matrix spikes in each batch, and so it remains a general TestAmerica Denver QC requirement.

4.12.1 Matrix Spike Duplicate - A matrix spike duplicate (MSD) consists of an additional aliquot of the same sample used to prepare the MS. This aliquot is spiked and processed exactly as is the MS.

4.12.2 The MS and MSD results are used to determine the effect of the sample matrix on the precision and accuracy of analytical results. Due to the potential variability of the matrix of each sample, the MS and MSD results may not have immediate bearing on any samples except the one spiked.

4.13 Measurement System - A test method, as implemented at a particular laboratory, and which includes the equipment and reagents used to perform the test and the analyst(s)

4.14 Method Blank (MB) - The method blank (MB) consists of a well-characterized matrix (e.g., reagent water or Ottawa sand) that is similar to the associated samples and is known to be free of the analytes of interest. The MB is prepared using the same method and reagents used for the samples. Specifically, reagents are added to the method blank in the same volumes or proportions as used in

sample processing. As part of a QC batch, it accompanies the samples through all steps of the analytical procedure. The method blank is used to assess the level of contamination introduced to a batch of samples as a result of processing in the laboratory.

4.15 Method Detection Limit (MDL) - One way to establish a Limit of Detection (LOD), defined as 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.

4.16 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, whether absolute or relative terms.

4.17 Sample Duplicate - A sample duplicate is a second aliquot of an environmental sample, taken from the same sample container when possible, that is processed with the first aliquot of that sample. That is, sample duplicates, using the same size aliquot from the sample container for each replicate, are processed as independent samples within the same QC batch. The sample and duplicate results are compared to determine the effect of the sample matrix on the precision of the analytical process. As with the MS/MSD results, the sample duplicate precision results are not necessarily representative of the precision for other samples in the batch. Different sized aliquots of the same sample in the same batch are not considered duplicates and each counts toward the batch count of 20.

4.18 Spike - A known mass of target analyte added to a blank sample or sub-sample; used to determine recovery efficiency or for other quality purposes.

4.19 Surrogates - Surrogates are organic compounds similar in chemical behavior to the target analytes, but that are not normally found in environmental samples. Surrogate compounds are chosen to reflect the chemistries of the targeted analytes of the method. Surrogates are added to all samples, standards, and blanks in a batch prior to sample preparation/extraction. Surrogates provide a measure of the recovery of analytes for every sample matrix and are used to monitor the effects of both the matrix and the analytical process on accuracy.

4.20 Uncertainty - a parameter associated with the result of a measurement that characterizes the dispersion of the values that could reasonably be attributed to the analytical result. The parameter associated with most analytical results for reporting uncertainty will be the relative standard deviation derived from the control limits.

4.21 Working Range - The difference between the Limit of Quantitation and the upper limit of measurement system calibration.

5.0 PROCEDURE

5.1 Assessments of QC data relative to established control limits determine the acceptability of sample test results. Whenever control criteria are not met, the

data must be evaluated to determine appropriate corrective action. Corrective action decisions, particularly whether or not to reanalyze samples, should be done in consultation with the client to the extent possible when operating under project-specific QA plans.

5.2 TestAmerica Denver's standard QC program shall be communicated to the client prior to acceptance of work. Alternative QC procedures may be required depending on the clients' special project requirements. In the event that alternative QC procedures are not specified by our clients, the standard QC protocols specified in this policy must be followed to ensure the generation of legally and scientifically defensible analytical data.

5.3 Quality control requirements specific to the Department of Defense (DoD), Airforce Center for Environmental Excellence (AFCEE), and the Department of Energy (DOE) are described in a separate TestAmerica Denver policy, DV-QA-024P, Requirements for Federal Programs. When performing analyses for DoD, AFCEE, or DOE projects, DV-QA-024P shall be consulted to ensure that program-specific requirements are met.

5.4 TestAmerica Denver's QC program applies to the following:

5.4.1 RCRA and SW-846 Projects - All routine analytical projects performed using SW-846 methods must comply with the requirements described in TestAmerica Denver's Quality Assurance Manual (QAM) and this policy. The Quality Control sections of analytical standard operating procedures (SOPs) referencing SW-846 methods must be consistent with the requirements in this policy.

5.4.2 CWA and 40 CFR Part 136 Projects - Any analytical work conducted in support of an NPDES permit or other Clean Water Act (CWA) compliance activities, must meet applicable quality control specifications as summarized in the QAM. The quality control requirements listed in the QAM define the minimum requirements that must be given in laboratory analytical SOPs.

5.4.3 Safe Drinking Water Act (SDWA) Projects – Any analytical work conducted in support of SDWA compliance activities must meet the quality control specifications shown in TestAmerica Denver Policy DV-QA-020P, "Quality Control for Drinking Water Programs."

5.4.4 Method specific QC requirements that are more restrictive than the requirements defined in this policy take precedence. For example, a method that requires the MS/MSD be performed every 10 samples sets the number of samples in the QC batch to 10, not 20.

5.5 Other Programs or Projects with Clearly Defined QC Requirements

5.5.1 The differences between TestAmerica Denver's standard QC program and special project requirements must be specified in project documents. These documents may include Quality Assurance Project Plans (QAPjPs), Quality Assurance Program Plans (QAPPs), Sampling and Analysis Plans

(SAPs), project-specific Quality Assurance Summaries (QASs), SOPs, contracts, protocols, or other approved documents.

5.5.2 Documents describing special project requirements must be reviewed and approved by appropriate QA and operations staff.

5.5.3 If the special project requirements appear to result in modifications that contradict federal or state regulatory requirements, the variance must be noted in writing and communicated to the client. A record of this communication must be retained as a permanent part of the project file.

5.5.4 Any special client's project requirements must be communicated to TestAmerica Denver's analysts in advance of releasing samples for analysis, and the work must be clearly differentiated in the analytical documentation, otherwise this policy's requirements will be followed.

5.6 QC for RCRA Projects and Projects without defined QC requirements

NOTE: Analytical SOPs must include a quality control section that addresses these general QC requirements, unless method-specific requirements exist. As relevant, specific method QC requirements are given precedence to these general requirements and must be included in the SOP.

5.6.1 Method Proficiency

5.6.1.1 The proficiency of a method is defined by its precision, bias (accuracy), limit of quantitation, limit of detection, and working range.

5.6.1.2 The limit of quantitation (LOQ) is established at the time of calibration and is typically defined as the lowest level standard that is used in the method calibration. Alternatively, the LOQ may be defined in relation to an established lower limit of detection (LOD).

5.6.1.3 The working range is established by the highest level standard used in the measurement system calibration.

5.6.1.4 The method detection limit (MDL), which is a measure of the LOD of the measurement system, must be initially determined in accordance with Policy DV-QA-005P. The MDL must be verified annually for most commercial projects, and quarterly for Department of Defense (DoD) projects and Texas TRRP projects.

5.6.1.5 Prior to using a method for actual samples and at any time there is a change in instrument type, personnel, or test method, a NELAC-compliant demonstration of capability (DOC) must be performed by the analyst(s) who will be performing the method in accordance with SOP DV-QA-0024. The analyst must analyze spiked control samples and achieve recoveries within prescribed acceptance criteria. For methods where spiked samples are not appropriate,

the DOC consists of demonstration of a properly performed QC batch. Analysts performing a method must demonstrate their continued proficiency annually.

5.6.1.6 Evaluation of LCS data over the long term establishes the precision and bias of the analytical method free of any matrix interference. Control charts are reviewed by the group leaders on a quarterly basis. Limits can also be trended in real time by the analyst when they are the sole person responsible for the test. Limits are reviewed annually against method and project requirements. They are updated by the QA Department when necessary. The "Save Log" feature in TALS can be used to document review. Recording the review in TALS supersedes the use of the Control Limits review form (see Attachment 2), and indicates the items on this review form were addressed.

5.6.1.7 Evaluation of MS and MSD data over the long term establishes the precision and bias of the analytical method in a variety of sample matrices. Control charts are reviewed in real time by the group leaders on a quarterly basis. Limits are reviewed annually against method and project requirements. They are updated by the QA Department when necessary. The "Save Log" feature in TALS can be used to document review. Recording the review in TALS supersedes the use of the Control Limits review form (see Attachment 2), and indicates the items on this review form were addressed.

5.6.2 Batch QC Elements and Batch Processing

5.6.2.1 A QC batch is designed to allow assessment of the quality, in terms of accuracy and precision, of the analytical results obtained for a group of up to 20 field samples. With some exceptions as described in Sections 5.6.3.6 through 5.6.2.8 below, the minimum QC elements for each QC batch consist of the following:

- one method blank (MB),
- one laboratory control sample (LCS),
- one matrix spike (MS), and
- one matrix spike duplicate (MSD).

5.6.2.2 The identity of each QC batch must be documented and traceable, i.e., each batch of field samples must be clearly associated with the applicable QC samples.

5.6.2.3 To the extent possible, samples that require a preparation step should be analyzed together with their associated QC samples. If the samples in a given QC batch require separate analytical runs, the minimum batch QC in each run is an acceptable MB or instrument/calibration blank. To the extent possible, the QC

samples should not be analyzed independently of the field samples on a different instrument.

5.6.2.4 For analytical procedures that do not include a separate extraction or digestion (e.g., volatile organic analysis by purge and trap), the QC batch must be analyzed sequentially using the same instrument and instrument configuration within the same calibration event. That is, the same calibration curve, calibration factors, or response factors must be in effect throughout the analysis.

5.6.2.5 Field QC samples (e.g., trip blanks, equipment rinsates, and field duplicates) count as individual samples, therefore, they add to the QC batch count. Samples that require simple reanalysis (e.g., dilutions to adjust a sample extract to the working range of the instrument), as opposed to re-extraction or digestion and reanalysis, do not count as additional samples in the QC batch. For procedures without a separate preparation, a reanalysis within the same calibration event (as defined in Section 5.6.2.4) does not add to the batch count.

5.6.2.6 MS/MSD pairs are not the only acceptable means of demonstrating precision.

5.6.2.6.1 As requested by clients or required by some methods, batch precision may also be demonstrated through the analysis of sample duplicates. However, the client should be advised that a sample duplicate is less likely to provide usable precision statistics depending on the likelihood of finding concentrations above reporting limits.

5.6.2.6.2 A duplicate LCS (LCSD or DCS) may be used to demonstrate method batch precision independent of the client's matrix. LCSDs are prepared at the client's request, and can be used when the client has not supplied sufficient sample to prepare an MS and MSD, or sample duplicate.

5.6.2.6.3 On-going monitoring of LCS results can be used to determine long-term precision and accuracy for a method independent of matrix effects.

5.6.2.7 Some methods, including isotope-dilution methods, pH, and ignitability, for example, do not use all of the QC elements listed in Section 5.6.2.1. Method exceptions to these requirements are listed in the laboratory's analytical SOPs.

5.6.2.8 Deviations from these QC elements must be noted either in project planning documents (QAPPs, QAPjPs, SAPs, SOWs, QAS, or equivalent) or in a nonconformance memo (see SOP DV-QA-0031 for details).

5.6.3 Data Evaluation and Corrective Action

5.6.3.1 General Guidelines

5.6.3.1.1 Any QC component that fails acceptance criteria is considered an out-of-control event. All out-of-control events must be documented and the associated data evaluated. Depending on the specific circumstances, evaluation can lead to a variety of actions. The following sections and the flowcharts (Figures 1-4) describe the appropriate corrective actions for the most common QC failures. However, it is not possible to address all possible data evaluation scenarios in this policy. The guiding principle for all evaluations is that the data and corrective action decisions must be defensible using TestAmerica Denver policies, procedures, or scientific evidence, and justified in the project records.

5.6.3.1.2 If reanalysis for QC failures is conducted and the second analysis confirms a QC problem that is outside of the laboratory's control, further testing is not necessary. The problem must be documented and the data properly qualified in the analytical report.

5.6.3.1.3 QC failures that are not corrected by reanalysis are documented in TestAmerica Denver's electronic nonconformance system, as described in SOP DV-QA-0031.

5.6.3.1.4 QC failures due to sample matrix interferences (particularly sample duplicate, and sample surrogate failures) are documented through the use of the electronic nonconformance system. The laboratory does not control on MS/MSD recoveries or RPDs so these data are flagged for QC failures. In either case, matrix QC failures must be communicated to the laboratory project manager, and significant matrix QC failures must be discussed in the final report case narrative.

5.6.3.1.5 When ongoing, systematic problems are identified, work must stop until it can be demonstrated that the system is in control again.

5.6.3.2 Method Blank (MB) Evaluation (also see Figure 1)

5.6.3.2.1 Method Blank Acceptance Criteria

When appropriate for the specific analytical method, the results of the method blank shall be one of the QC measures used to assess batch acceptance. SW-846 guidance is to have no detectable contaminants in the

method blank, i.e., the method blank result must be less than or equal to the MDL for each target analyte. However, this may not be practically achievable in a laboratory setting, and method blank contamination between the MDL and the laboratory's reporting limit may not have an adverse affect on data quality. Each method blank must be critically evaluated as to the nature of the interference and the effect on the analysis of each sample in the batch.

TestAmerica Denver policy is that the method blank is acceptable as long as all analytes of interest are less than one-half the laboratory's reporting limit (RL) for organic, metals, and some wet chemistry analyses, unless otherwise specified by specific projects or clients or listed here.

For the following list of wet chemistry analytes, a method blank is acceptable if the analytes of interest are less than the reporting limit:

Alkalinity
BOD
Cl^{1-} – colorimetric
COD
HEM
HEM SGT
Low level phenol
Phenols by distillation
TDS
TSS

When the method blank result is above the acceptance limit, the results for the associated samples may be accepted with qualification if the method blank meets one of the following criteria, unless otherwise prescribed by project-specific requirements:

- The concentration of the analyte of concern in the method blank is less than or equal to 10% (1/10) of the regulatory limit for that analyte, or
- The concentration of the analyte of concern in the method blank is less than or equal to 10% (1/10) of the measured concentration of that analyte in the sample, or
- The same analyte was not detected above the MDL in the associated samples (and therefore the apparent contamination in the blank did not represent corresponding elevated values in the samples).

NOTE: Positive method blank results slightly below the reporting limit should still be evaluated by the analyst for potential impact on sample results at or near the reporting limit.

The following criteria shall apply to DoD work unless project data quality objectives (DQOs) specify otherwise:

- All samples should be reprocessed if contamination is greater than one-half of the quantitation limit (the quantitation limit is equivalent to the laboratory's standard reporting limit), unless
- Action levels are specified and contamination is less than 10% of the regulatory limit.

5.6.3.2.2 Corrective Action for Method Blank Failure

If the method blank does not meet the acceptance criteria, the source of contamination must be investigated and measures taken to correct, minimize, or eliminate the problem. Samples associated with the contaminated blank shall be reprocessed for analysis or, under the following circumstances, may be reported as qualified (qualifier flags or narrative comments):

- MB contamination is at a level less than the one-half the reporting limit (or less that the reporting limit for some Wet Chemistry tests listed in Section 5.6.3.2.1) with sample results at levels near the RL, and based on the analyst's judgment, the data may be flagged, or
- Analyte concentrations in samples are greater than 10 times blank contamination, or
- The contaminant is a common laboratory contaminant (see the table below) and the MB concentration is less than 5 times the RL for organics or less than 2 times the RL for metals. Note that some programs do not recognize common laboratory contaminants.

Common Laboratory Contaminants

Analyte	Method
Methylene Chloride	Volatile Organics (GC or GC/MS)
Acetone	Volatile Organics (GC or GC/MS)
2-Butanone	Volatile Organics (GC or GC/MS)
Phthalate Esters	Semi-Volatile Organics (GC or GC/MS)
Copper	Metals (ICP or GFAA)

Analyte	Method
Zinc	Metals (ICP or GFAA)
Iron	Metals (ICP or GFAA)
Lead	Metals (Trace ICP or GFAA)

5.6.3.3 Laboratory Control Samples (LCS) Evaluation (also see Figure 2)

5.6.3.3.1 LCS Acceptance Criteria

The LCS recovery for the control analytes must be within established control limits. Unless otherwise specified in a reference method or project requirements, the control limits are set at ± 3 standard deviations around the mean of the historical data. An LCS that is determined to be within acceptance criteria effectively demonstrates that the analytical system is in control and validates system performance for the samples in the associated batch.

If there are a large number of analytes in the LCS, as is the case for many organic analyses, then NELAC allows a specified number of results to fall beyond the LCS control limit (3 standard deviations), but within the marginal exceedance (ME) limits, which are set at ± 4 standard deviations around the mean of historical data (marginal exceedance limits are posted in the Outlook public folders under Public Folders\All Public Folders\Analytical\Denver\NELAC-Marginal Exceedance Tables. The number of marginal exceedances is based on the number of analytes in the LCS, as shown in the following: table:

# of Analytes in LCS	# of Allowed Marginal Exceedances
> 90	5
71 – 90	4
51 – 70	3
31 – 50	2
11 – 30	1
< 11	0

If more analytes exceed the LCS control limits than is allowed, or if any analyte exceeds the ME limits, the LCS fails and corrective action is necessary. Marginal exceedances must be random. If the same analyte repeatedly fails the LCS control limits, it is an indication of a systematic problem. The source of the error must be identified and corrective action taken.

The percent recovery is calculated as follows:

$$\text{LCS Percent Recovery} = \frac{\text{measured value}}{\text{expected value}} \times 100\%$$

5.6.3.3.2 Corrective Action for LCS Failure

Samples analyzed along with an LCS that is determined to be "out of control" are considered suspect and the samples must be reprocessed and reanalyzed, or the data reported with appropriate data qualification. If the LCS result does not fall within statistical control limits, check calculations, check instrument performance, reanalyze the LCS, and if still outside of control limits, re-prepare and reanalyze all samples in the QC batch.

It is acceptable to report the data if the LCS recovery is out high and any analyte of concern was not detected in any of the samples.

In the case of volatile analyses, if the LCS fails, a new LCS may be re-prepared and reanalyzed within the same tune period.

In the case where all target requested analytes are within control, but some other LCS compounds are out of control, the LCS may still be considered acceptable for reporting.

See Figure 2 for additional detail.

5.6.3.4 Duplicate Laboratory Control Samples (LCS/LCSD or DCS) Evaluation (also see Figure 2)

5.6.3.4.1 LCS/LCSD Acceptance Criteria

The recovery for each analyte in the LCS and LCSD must be within established control limits as described in Section 5.3.3.3.1. The equation used to calculate LCSD recovery is the same as the equation for LCS recovery. If either LCS fails, this must be described in the final report if samples are not reanalyzed.

The LCS precision is calculated as the relative percent difference (RPD) between the LCS and LCSD and must not exceed the established limit. Unless otherwise specified in the reference method or in project requirements, the limit is set at the mean of the historical RPD data plus three standard deviations. The RPD between the LCS and LCSD is calculated as follows:

$$RPD = \left[\frac{|LCS - LCSD|}{\frac{(LCS + LCSD)}{2}} \right] \times 100\%$$

Where:

LCS = measured concentration for the LCS

LCSD = measured concentration for the duplicate LCS

5.6.3.4.2 Corrective Action for LCS/LCSD Recovery (Accuracy) Failure

See Section 5.3.3.3.2 for corrective actions for LCS recovery failures.

NOTE: If either the LCS or the LCSD spike fails and the batch cannot be reanalyzed, the failure must be documented and noted in the final report.

5.6.3.4.3 Corrective Action for LCS/LCSD Precision Failure

Because the LCS/LCSD precision limits are based on the standard deviation of data collected over time and include long-term precision, it would be unusual to fail precision limits while meeting accuracy limits. If this occurs with any frequency, control limits should be reevaluated. For any single precision failure, check calculations; verify, if possible, that the LCS and LCSD were spiked correctly; check instrument performance; and if the RPD is out of control but both accuracy recoveries are within acceptance criteria, prepare an NCM, and qualify the reported results.

5.6.3.5 Surrogate Evaluation (also see Figure 3)

5.6.3.5.1 Acceptance Criteria

Surrogate recovery must be within established control limits. Unless otherwise specified in a reference method or project requirements, the control limits are set at ± 3 standard deviations around the mean of the historical data. Method QC (MB, LCS, and/or LCSD) results are not acceptable unless the surrogate recoveries for those QC samples are within control limits. If MS/MSD, duplicate, or field samples require dilutions beyond the threshold stated in the analytical SOPs, routine surrogate control limits do not apply and recoveries are not evaluated. This should be noted in the final report. The surrogate recovery is calculated as follows:

$$\text{Surrogate Percent Recovery} = \frac{\text{measured value}}{\text{expected value}} \times 100\%$$

5.6.3.5.2 Corrective Action for Surrogate Failure

Corrective action must be considered for any surrogate failure. Analysts and data reviewers must review specific project instructions to be certain that the required actions are taken. Lacking instructions to the contrary, the following guidelines apply:

- Check calculations and instrument performance.
- Failed Surrogates in QC Samples: Evaluate the surrogate results together with the QC sample results for all QC samples in the batch to determine whether associated samples should be re-prepared and reanalyzed. Refer to Figures 1-4 for details. For example, consistent surrogate failures in all the QC samples in a batch indicate a method failure. Surrogate failures in only one QC sample in a batch may indicate a problem with that one sample only, especially if surrogate recoveries fall within limits for all other samples in the batch. Document the failure and evaluation in the final report.
- Failed Surrogates in Field Samples: Refer to Figure 3.

5.6.3.6 Matrix Spike and Matrix Spike Duplicates (MS/MSD) Evaluation (also see Figure 4)

5.6.3.6.1 MS/MSD Acceptance Criteria

The MS and MSD recoveries for control analytes should be within established control limits, which are either mandated in the published methods or regulatory programs, or are set at ± 3 standard deviations around the mean of historical data. In addition, the relative percent difference (RPD) between the MS and MSD results should be less than or equal to the established upper control limit. If MS or MSD samples require dilutions beyond the threshold stated in the analytical SOPs, routine control limits do not apply and recoveries are not evaluated, but this should be noted in the final report. The RPD between the MS and MSD is calculated the same way as the RPD between the LCS and LCSD, as shown in Section 5.3.3.4.1. The MS and MSD recoveries are calculated using observed concentrations (except as noted below):

$$\text{MS or MSD \%Recovery} = \left(\frac{SSR - SR}{SA} \right) \times 100\%$$

Where:

SSR = observed concentration in spiked sample

SR = observed concentration in unspiked sample

SA = concentration of spike added to sample

NOTES:

1. If the sample result is ND, then SR = 0 when no values are reported below RL.
2. If the sample result is reported as a value less than the RL, then SR = the reported value.
3. CLP forms software uses observed recovery, not concentrations.

5.6.3.6.2 Corrective Action for MS/MSD Recovery (Accuracy) Failure

As noted previously, matrix spike data are meaningful only for the sample in which they are prepared and possibly for samples from the same site. The information obtained from MS data are sample/matrix specific and are not normally used to determine the validity of the entire batch. If the MS and/or MSD recovery falls outside of the established control limits, the LCS recovery must be within control limits in order to accept results for the associated samples. The following corrective actions are required for MS/MSD recovery failures:

- Check calculation and instrument performance;
- Verify, if possible, that the MS and MSD were spiked correctly;
- Consider objective evidence of matrix interference (e.g., heterogeneous sample, interfering compounds seen on chromatograms, or interference demonstrated by prior analyses); and
- Flag the data and the PM shall note it in the final report;
- See Figure 4 for additional detail.

NOTE: Some client programs require reanalysis to confirm matrix interferences. Check special project

requirements for this corrective action.

5.6.3.6.3 Corrective Action for MS/MSD Precision Failure

For any single precision failure, check calculations; verify, if possible, that the MS and MSD were spiked correctly; check instrument performance; consider objective evidence of matrix interference or sample inhomogeneity; and flag the data.

5.6.3.7 Sample Duplicate

5.6.3.7.1 Sample Duplicate Acceptance Criteria

The RPD between the sample and its duplicate must be within established control limits. The RPD between the sample and its duplicate is calculated the same way as the RPD between the LCS and its duplicate, as shown in Section 5.3.3.4.1.

5.6.3.7.2 Corrective Action for Duplicate Failure

For any single precision failure, check calculations and instrument performance. Document the QC failure in an NCM and note it on the final report.

5.6.4 Reporting Uncertainty with Measurements

It is the responsibility of the project manager to notify the appropriate laboratory personnel whenever the uncertainty for a given analyte is to be reported. It is the responsibility of the laboratory personnel to calculate and report the uncertainty for each analyte requested in accordance with the procedures in this section

NOTE: The laboratory does not have an automated reporting mechanism for reporting the uncertainty associated with each measurement. Reporting this information would require project-specific arrangements to accommodate manual calculation and manual reporting.

5.6.4.1 Procedure

Determine the average and standard deviation of a minimum of twenty recovery results. Calculate the relative standard deviation (RSD) as follows:

$$RSD = SD / X_{avg}$$

The average percent recovery (Xavg) and the standard deviation (SD) can be derived from the control limits (at the 99% confidence interval):

$$X_{avg} = (UCL + LCL)/2$$

Where: UCL=upper control limit, LCL= lower control limit

Calculate the uncertainty (U(X)) associated with an analytical result as follows:

$$U(X) = C + (2 \times RSD \times C)$$

Where C = Sample Concentration

5.6.4.2 Example Calculation

The analytical result for phenol is 120 ug/L. The control limits for phenol are 33-122%. The average recovery for phenol is 77.5% with a standard deviation of 14.8%. The average percent recovery and the standard deviation can be derived from the control limits (at the 99% confidence interval).

1) Calculate the average percent recovery:

$$X_{avg} = (122+33)/2 = 77.5$$

2) Calculate the Standard Deviation:

SD = 14.8

Standard Deviation

$$S.D. = \sqrt{\frac{\sum_{s=1}^{m}\sum_{i=1}^{n}(y_{is} - M)^2}{(n_y - 1)}}$$

where:

s = series number

i = point number in series s

m = number of series for point y in chart

n = number of points in each series

Y_{is} = data value of series s and the ith point

N_y = total number of data values in all series

M = arithmetic mean

3) Calculate the RSD: 14.8 / 77.5 = 0.19

4) Calculate the uncertainty of the analytical result

U (x) = 120 + (2 x 0.19 x 120) = 165.6

U (x) = 120 – (2 x 0.19 x 120) = 74.4

5) Report the analytical result as 120ug/L with an uncertainty range of 74.4 ug/L to 165.6 ug/L at the 95% confidence interval.

5.6.5 Establishing QC Acceptance Limits

5.6.5.1 Initial Control Limits

5.6.5.1.1 For new procedures, published method limits can be used until sufficient QC data are acquired (a minimum of 20 to 30 data points recommended). However, the published limits may not be appropriate if they are based on a single-operator or single-laboratory study. In this case, the QA Manager may establish default limits until enough data are collected to calculate statistical limits.

5.6.5.1.2 Limits are trended quarterly by the individual group leaders or can be done in real time by the analyst when they are the sole person responsible for the test. Established control limits are reviewed annually by the QA Department and reset as needed. If the recalculated limits are consistent with the historical limits, the historical limits may remain unchanged.

5.6.5.2 TALS Control Chart Program

Evaluating control charts is an important first step in considering new control limits. Control charts are generated by TALS Control Chart program. Only QA personnel who are familiar with the organization of TestAmerica Denver's spike lists are authorized to set control limits. The program collects a specified set of QC data, calculates the mean and three standard deviation control limits, compares those limits to the existing limits in the LIMS, and generates an I-type control chart (ref. ASTM D 6299). This control chart is a plot of results, in chronological order, to which existing control limits and a centerline have been added. The control chart aids in the examination of the data to be sure that it is representative and appropriate for use in setting new limits. See Attachment 1 for complete details. Some specific requirements include the following:

5.6.5.2.1 Select QC Type Options

QC Type	Description
LCS/LCSD	Used to establish LCS control limits.
LCS/LCSD Surrogates	Used to establish surrogate control limits for LCS controls.
MS/MSD	Used to set matrix-specific control limits.
All Surrogates	This option will produce a pooled set of LCS/LCSD, MB, and MS/MSD. There is an option to include or not include client samples. In addition you can pull charts for just client samples.

5.6.5.2.2 Representative Time Period

The appropriate time period depends on the frequency with which the test is performed and the frequency of other events, such as calibrations and standards preparation. A minimum of three months is desirable to capture data from multiple instruments, multiple instrument tunes, multiple calibrations, and multiple standard preparations. For infrequent tests, it may be necessary to collect nine months or more of data. However, collecting more than 100 data points is normally unnecessary, makes the control charts hard to read, and results in abnormally tight control limits.

5.6.5.2.3 Grubbs' Test for Outliers

The Control Limits program automatically runs the Grubbs' test for outliers using a 5% level of significance, i.e., the risk of falsely rejecting a data point. The initial assumption is made that the data are normally distributed. The Grubbs' test detects one outlier at a time, eliminates that outlier, and repeats the test until all outliers are eliminated. The test should not be used for sample sizes of six or less.

The test is defined for the hypothesis H_0, there are no outliers in the data set, and H_a, there is at least one outlier in the data set. The test statistic "G" is calculated as the ratio of the difference between the suspect point and the mean value to the calculated standard deviation, as follows:

$$G = \frac{\max \left| Y_i - \overline{Y} \right|}{s}$$

Where:

Y_i = the point being considered for rejection

$\overline{Y}$ = the mean value of the data set

s = the standard deviation

The hypothesis of no outliers, and consequently the suspect point, is rejected if

$$G > \frac{(N-1)}{\sqrt{N}} \sqrt{\frac{t^2_{(\alpha/(2N),N-2)}}{N-2+t^2_{(\alpha/(2N),N-2)}}}$$

Where:

N = number of points

$t_{(\alpha/(2N),N-2)}$ = the critical value of the t-distribution with (N-2)/2 degrees of freedom and a significance level of $\alpha/(2N)$.

Tables for critical values of t are given in John Taylor, Quality Assurance of Chemical Measurements, Lewis Publishers; 1987. For a complete discussion of the Grubbs' Test for Outliers also see NIST/SEMATECH e-Handbook of Statistical Methods, Section 4.3.5.1.7: http://www.itl.nist.gov/div898/handbook/eda/section3/eda35h.htm.

5.6.5.3 Examine and Investigate Collected Data

Assuming that an adequate amount of data are collected, the next step involves determining that the data set is representative of the laboratory's performance, and therefore provides a useful prediction of future performance. A key part of the process is examining the data for bias, discontinuities, and/or trends. Ideally, if conditions are constant over the time period selected and existing limits are appropriate, the data will be evenly distributed around the centerline, with very few points outside control limits (i.e., less than 1 point in 100 should lie beyond the 3 standard deviation control limits). The reasons for deviations from the ideal should be investigated to be sure that the collected data are appropriate. Specific conditions requiring further investigation include data sets with no outliers, data with significant bias relative to existing limits, excessive number of outliers, discontinuous patterns, and upward or downward sloping trends (see Attachment 1).

5.6.5.4 Selecting New Control Limits

Generally control limits are based on the following statistics for the historical data:

Accuracy:	mean recovery
Precision:	standard deviation
Control Limits:	mean recovery $\pm$ 3 standard deviations

The limits cannot be wider than method or program requirements. If the calculated control limits are tighter than the method calibration verification criterion (e.g., CCV acceptance limits for ICP are $\pm$ 10% of expected value), then the new limits are set to the mean value $\pm$ calibration criterion.

5.6.5.5 Communicating and Implementing New Control Limits

The laboratory groups prepare a Control Limit review form after reviewing the control limit data. The supervisor must review the control charts and associated data and sign the review form to

confirm that the data selected are representative of current performance. The memo and the control chart data are sent to the QA group for further review and establishment of new limits (if necessary). The QA department and the group supervisor will confirm a date that the instrument data systems and TALS will be updated.

5.6.6 Reporting QC Data

QC data that are routinely reported with sample results include the LCS, method blank, and surrogate standards. Client reporting format requirements are negotiable and documented as part of the project records. Ultimately, all reporting decisions should accommodate the client's requirements.

6.0 RESPONSIBILITIES

6.1 Successful implementation of this QC program requires that it is clearly understood by all TestAmerica staff. Training based on this policy will be conducted periodically and provided to new personnel as appropriate for their functions.

6.2 Project Managers

6.2.1 The laboratory project managers (PMs) serve as a liaison between the clients and the laboratory staff to ensure that requirements are properly communicated in writing to both parties.

6.2.2 The PM communicates any QC problems to clients and documents decisions made with clients.

6.3 Analytical Groups

6.3.1 The analytical groups are responsible for the initial evaluation of control limits, frequently in conjunction with data review software and/or senior analysts or supervisors.

6.3.2 Analytical groups shall review control chart data and notify QA when limits need to be updated as needed.

6.4 QA Group

6.4.1 The QA manager can establish default control limits until enough data points are collected to calculate statistical limits.

6.4.2 The QA staff shall pull statistical limits when the analytical groups ask for updates to the control limits.

6.4.3 After coordinating a date and time with the analytical groups, the QA staff will update the control limits in the TestAmerica Denver LIMS system.

7.0 REFERENCES / CROSS-REFERENCES

7.1 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, USEPA SW-846, 3rd Edition, with promulgated updates, Chapter One, Quality Control, Revision 1, July 1992.

7.2 2003 NELAC Standard, EPA/600/R-04/003, June 5, 2003, Appendix D, Quality Systems.

7.3 2009 TNI Standard, Management and Technical Requirements for Laboratories Performing Environmental Analysis, Volume 1, The NELAC Institute.

7.4 A2LA Guidance for the Estimation of Uncertainty for testing" Thomas Adams, July 2002 (from the A2LA website)

7.5 ASTM D 6299, Standard Practice for Applying Statistical Quality Assurance and Control Charting Techniques to Evaluate Analytical Measurement System Performance, Volume 5.03.

7.6 John Taylor, Quality Assurance of Chemical Measurements, Lewis Publishers; 1987.

7.7 NIST/SEMATECH e-Handbook of Statistical Methods, http://www.itl.nist.gov/div898/handbook/eda/section3/eda35h.htm, April 1, 2012.

8.0 ATTACHMENTS

Figure 1 : Method Blank Evaluation
Figure 2 : LCS/LCSD Evaluation
Figure 3 : Surrogate Evaluation
Figure 4 : Matrix Spike/Matrix Spike Duplicate Evaluation
Attachment 1: Guidelines for QA Staff in Setting Control Limits
Attachment 2: Example Control Limits Review Form

9.0 REVISION HISTORY

- Revision 8, dated 31 May 2013
    - Revised section 4.3 to reflect current practice
    - Clarified section 4.4 regarding differentiating what samples count toward the batch count.
    - Added section 5.1.4 to clarify method QC requirements take precedence over this policy.
    - Revised 5.6.1.5 to reflect use of DOCs for tests without an LCS.
    - Revised section 5.6.3.2.1 to clarify method blank acceptance criteria for wet chemistry methods.
    - Updated Section 5.6.3.2 DoD requirements to match QSM 4.2
    - Corrected reference to public folders in section 5.6.3.3.1
    - Updated sections 5.6.3.6.2, 5.6.3.6.3 and Figure 4 to reflect policy change to flag MS/MSD results rather than write NCM
    - Revised Sections 5.6.4.1 and 5.6.5.1.2 for clarity

- Revised Figures to reflect changes to policy
- Relabeled Figure 5 as Attachment 2 and updated form
- Added references cited in text of SOP
- Formatting and editorial changes throughout

- Revision 7.3, dated 06 July 2012
  - Removed criteria that the LOQ to be at least three times the LOD.
  - Updated sections 5.3.1.6 and 5.3.1.7 to meet current procedures.
  - Added requirement for limits to be reviewed annually by QA to section 5.3.1.6 and 5.3.1.7.
  - Updated Figures 2,3 and 4.
  - Removed section 5.3.3.5.2.2 which pertained to DoD requirements for surrogate failures. Can be found in the DoD specific SOP.
  - Updated surrogate section 5.3.5.2.1 to meet current LIMS.
  - Updated section 5.3.5.1.2 for new procedure.
- Revision 7.2, dated 01 March 2011
  - Updated references to TALS.
- Revision 7.1, dated 26 August 2009
  - Added a Quality Policy Statement under section 2.0.
- Revision 7, dated 16 February 2009
  - Incorporated Attachment 1 QC for RCRA Projects and Projects without defined QC Requirements into the policy.
  - Changed Attachment 2 Guidelines for QA Staff in Setting Control Limits to Attachment 1.
  - Added the review of control limits every 10-20 LCS data points and 20-30 MS/MSD data points requirement.
  - Changed control chart review responsibility from the QA Department to laboratory groups.
- Previous Revisions
  - Reformatted to new TestAmerica format and renumbered under new TestAmerica scheme. SOP was currently numbered as QA-003
  - Changes From the Previous Version of the Policy
  - Changed references to reflect “TestAmerica” name.
  - Added section for general Measurement Uncertainty Calculations to Attachment 2.

Figure 1. Method Blank Evaluation

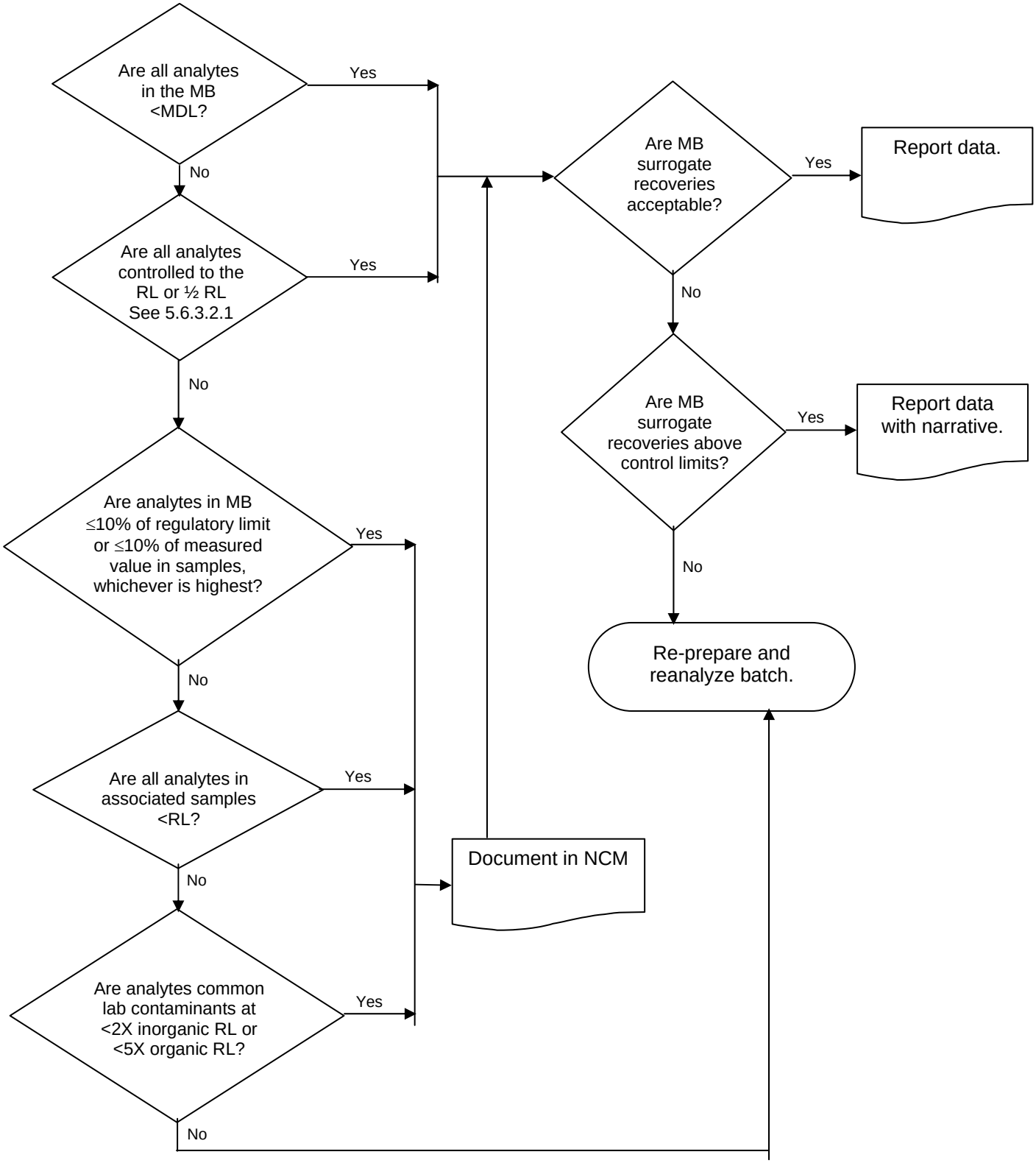


Figure 2

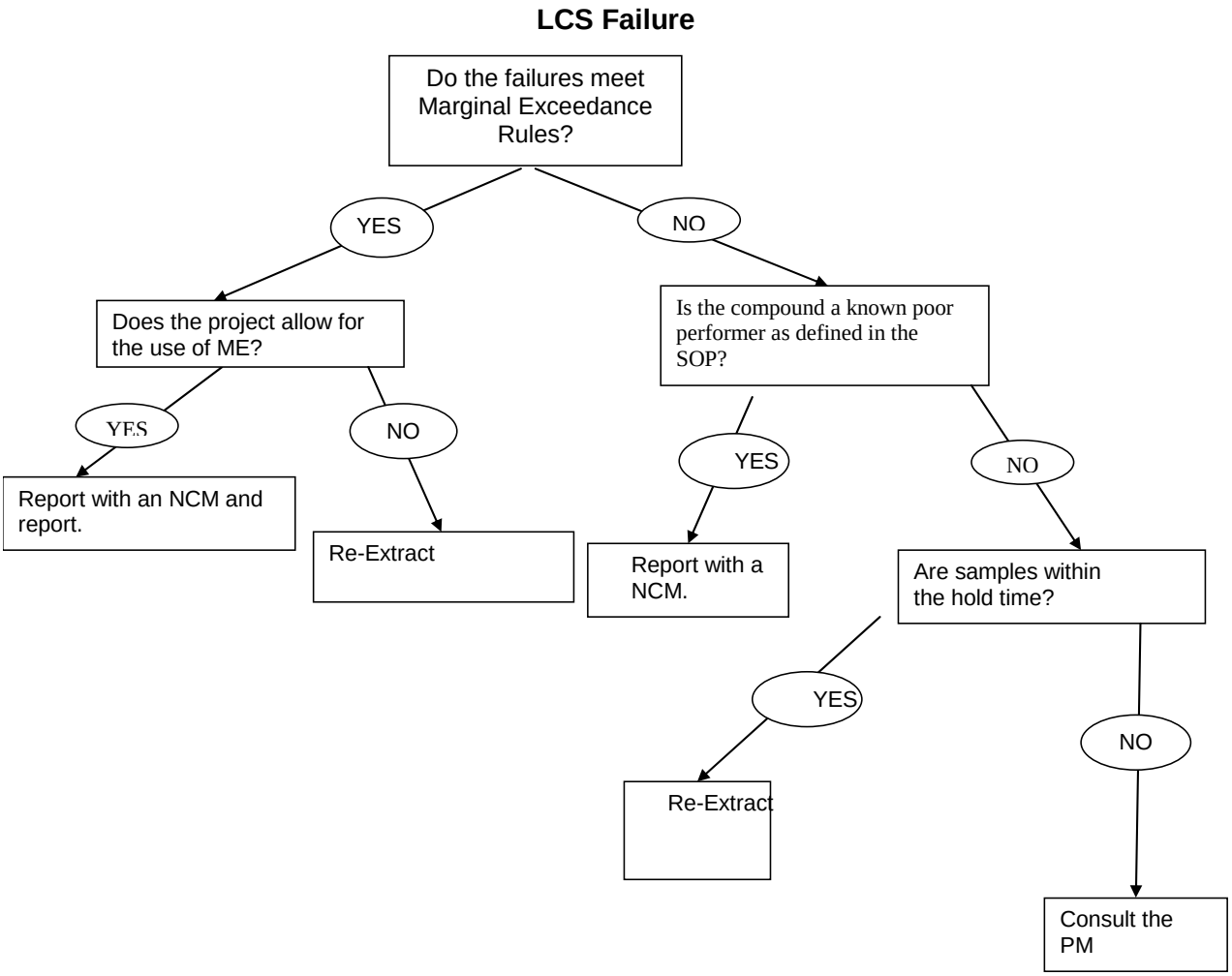

LCS Failure
Do the failures meet Marginal Exceedance Rules?
YES
NO
Does the project allow for the use of ME?
Is the compound a known poor performer as defined in the SOP?
YES
NO
YES
NO
Report with an NCM and report.
Re-Extract
Report with a NCM.
Are samples within the hold time?
YES
NO
Re-Extract
Consult the PM

Figure 3

Are samples within the hold time?

Surrogate Failures

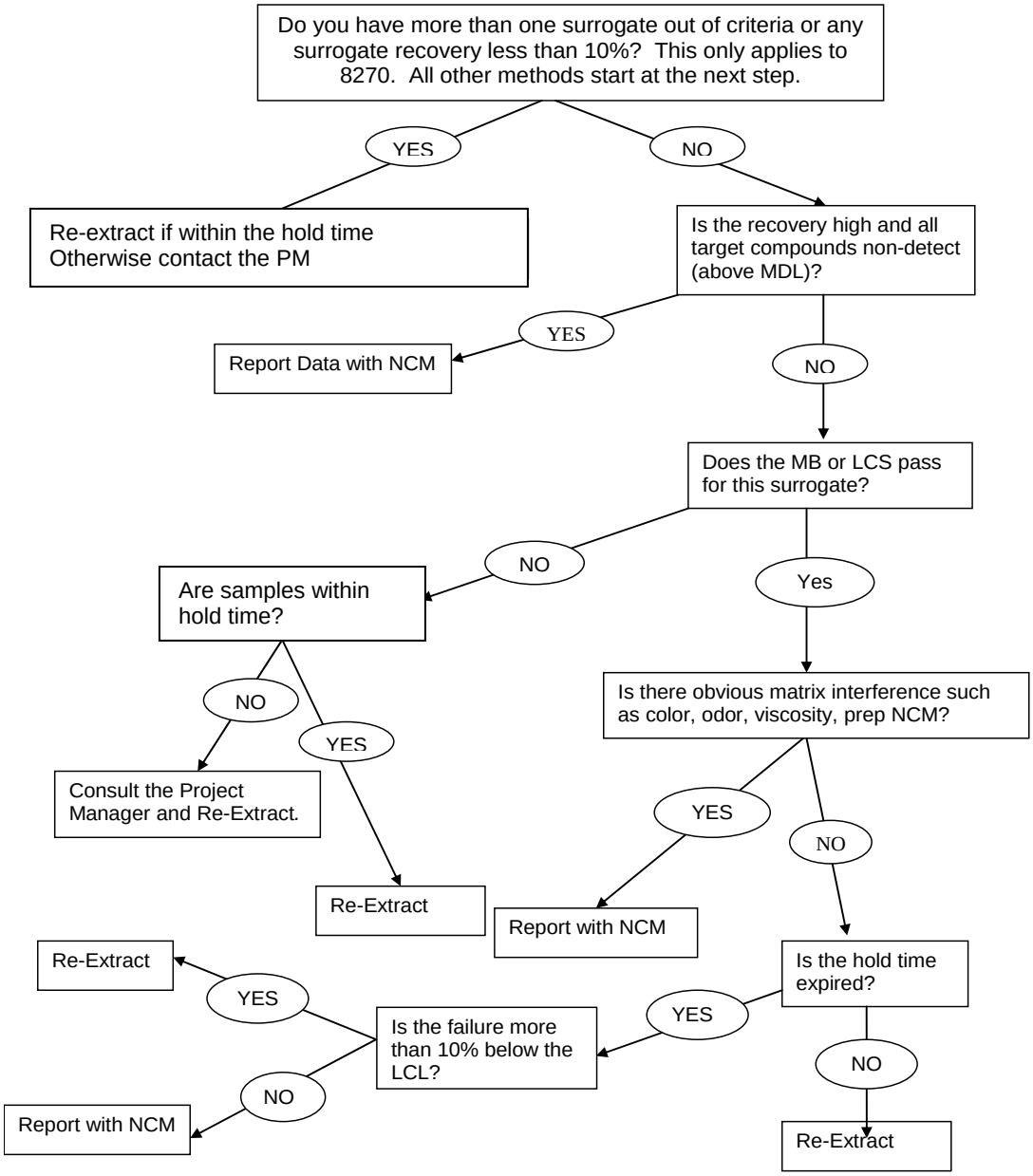
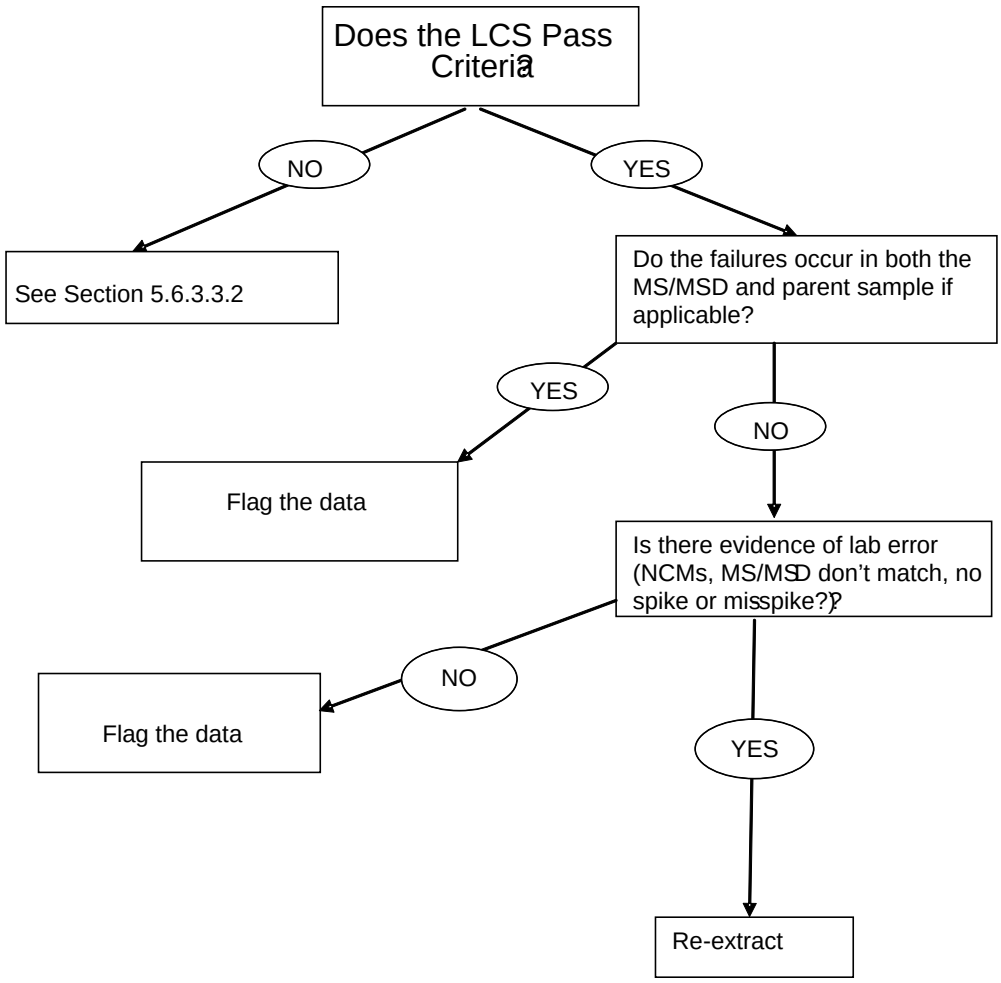


Figure 4

MS/MSD Failure



ATTACHMENT 1

Guidelines for QA Staff in Setting Control Limits

TestAmerica Denver's QC Policy (DV-QA-003P) requires control limits to be evaluated and recalculated every six months, or when necessary. Evaluating control charts is an important first step in considering new control limits. This is done using the Control Chart program in TALS. The program collects a specified set of QC data; performs a Grubbs' test for outliers; calculates the mean and standard deviation for the data; calculates the three-standard-deviation control limits; compares those limits to the ones active in TALS; and generates an I-type control chart (ref. ASTM D 6299). An I-type control chart is a plot of results in chronological order to which the existing control limits and a center line have been added. The control chart aids in the evaluation of the data to ensure that the data are representative and appropriate for use in setting new control limits.

NOTE: This attachment is written with the assumption that the user is well trained in the use of the TestAmerica Denver LIMS, i.e., TALS.

1.0 Running the Control Limits Program

The Control Chart program collects data from a database used for reporting purposes.

NOTE: The control data used by the Control Chart program is limited to the QC data that are uploaded. In many cases, failed QC data are not uploaded. Consequently the control limits calculated by the Control Chart program are most likely artificially tight.

1.1 Select Control Chart under the Analyst desk top.

If a chart has already been set up you can select the method then search "Find Chart" to pull up the desired chart. If a new chart needs to be started choose "New."

1.2 Select your method.

1.3 Select a sub list.

1.4 Select your QC Type (i.e. LCS, MDLS, etc.). If you select LCS the program will ask you if you want the QC linked to the LCSD. Select yes.

1.5 You will be asked to only include analytes in the normal spike. Select yes unless you want to add surrogates.

1.6 You will then be asked if the method includes a Prep Method. Select yes if it does and add the prep method you are charting and okay. If the method includes a cleanup method it can be added at this time. Otherwise select no to continue.

1.7 Select the Container Matrix for Prep and hit the Select button.

1.8 Select the Limit Group and hit the Select button.

1.9 At this point the program will notify you that you can make additional changes. Select OK and make any changes before saving the control chart. You can make any to items that were selected in the setup. Additional things that can be modified include adding or removing compounds, define analyst, batches, date ranges or equipment.

1.10 When completed save the chart the select View Chart and then Collect Data. The chart can be printed after it has been generated.

2.0 Calculating Marginal Exceedance Limits

The Control Chart Program does not calculate the 4-standard deviation marginal exceedance limits that are used when there are a large number of analytes in the LCS. As explained in Section 5.3.1 of Attachment 1, if there are a large number of analytes in the LCS, then NELAC allows a specified number of results to fall beyond the LCS control limits ($\pm$ 3 standard deviations), but within the marginal exceedance (ME) limits, which are set at $\pm$ 4 standard deviations around the mean of historical data.

NOTE: When calculating 4-standard deviation limits, it is possible to calculate a negative lower control limit. To prevent this, the lower control limit must always be ≥ 1.

After using the Control Chart program to collect data and calculate limits, the control data are exported to a verified spreadsheet tool that calculates the 4-standard deviation limits for marginal exceedances.

3.0 Calculating RPD Limits

The Control Chart Program also calculates limits for the relative percent difference (RPD) between LCS and MS duplicates. When there are insufficient LCS or MS duplicate data, these calculated limits may not be appropriate. An alternate approach has been developed to estimate the RPD limit using the precision data for the LCS or MS percent recovery data.

The assumption is made that the standard deviation of the recovery data is representative of the one-sigma analytical uncertainty. The difference between an LCS or MS and its duplicate is calculated as follows:

$$Diff = S - SD$$

Where S is the sample (LCS or MS) result and SD is the duplicate (LCSD or MSD) result.

The propagated uncertainty at the 99% confidence level of the difference between an LCS or MS and its duplicate is calculated as follows:

$$U_{Diff} = 1.96 \times \sqrt{(U_S)^2 + (U_{SD})^2}$$

Where:

US = Uncertainty of the sample result, estimated by the standard deviation of a set of control data.

USD = Uncertainty of the duplicate result, estimate by the standard deviation of a set of control data.

1.96 = Student's t value for alpha =0.05 (95% confidence) and 29 degrees of freedom (for the typical data set of 30).

Since the sample result and its duplicate come from the same data population, US equals USD, and the equation can be rewritten as follows:

$$U_{Diff} = 1.96\sqrt{2s^2}$$

Where s is the standard deviation of the data set.

For example, the mean percent recovery of a set of LCS data is 100%; the standard deviation is 10%; and the control limits are set at ± three standard deviations, or 70 to 130%. Using the equation for the propagated uncertainty of the difference, the RPD limits for duplicates would be set at 28%.

Although this is not a rigorous statistical treatment of the data, the resulting RPD limit is a reasonable estimate of the expected precision for duplicate sample results given the demonstrated precision of the percent recovery data. Data from the TALS database are exported to a verified spreadsheet tool that calculates both the RPD limit and the 4-standard deviation limits for marginal exceedances.

4.0 Evaluating and Investigating Collected Data

Assuming that an adequate amount of data are collected, the next step involves determining whether the data set is representative of the laboratory's performance, and therefore provides a useful prediction of future performance. A key part of the process is examining the data for bias, discontinuities, and/or trends. Ideally, if conditions are constant over the time period selected and existing limits are appropriate, the data will be evenly distributed around the centerline, with less than one in 100 points beyond the control limits. The following are very general guidelines for assessing the representativeness of a data set that does not follow the ideal pattern.

4.1 No Outlier Data

If there are no outlier data and little or no data outside the 2-standard-deviation warning limits, then one of the following is true:

4.1.1 Insufficient data have been collected, which can be tested by generating charts using a longer time period.

4.1.2 Outlier data are being censored (not entered into TALS). Check with the analysts to verify this. Analysts should be told that omitting outliers (not blunders, but statistical outliers) is essential to avoid generating even tighter limits.

4.1.3 Existing limits are much too wide (possibly because the performance of the analytical system has significantly improved) and should be changed immediately (see section on Establishing New Control Limits below).

4.2 Bias Relative to Existing Limits

If there are a significant majority of QC results falling on one side of the centerline, then consider the following:

4.2.1 The procedure, equipment, or calibration may have changed. Verify the accuracy of the SOP with analysts.

4.2.2 The analyst's skill level may have changed. Check with the supervisor to find out if new analysts might account for the bias.

4.2.3 Equipment may have been changed. Check with supervisor.

4.2.4 The standards used for calibration, including those used for internal standards, may have changed or may have been incorrectly prepared.

4.2.5 The time since the limits were last set may be longer than six months, and an update to the control limits is overdue.

4.3 Excessive Number of Outliers

If significantly more than 1 point in 100 is outside the control limits, the following should be considered:

4.3.1 The variability in the analytical system may have changed significantly, either as the result of a specific event, or degradation in the instrumentation.

4.3.2 The existing limits may not be statistically based. Review control limits records to check the basis of the old limits.

4.3.3 Audit the method to verify the accuracy of the SOP, competency of the analysts, and reliability of the instrumentation.

4.3.4 Consult with the supervisor.

4.3.5 Compare laboratory's performance to other laboratories. The Tri-Agency QSM limits is one source of limits by competent laboratories, other TestAmerica laboratories is another, and method limits have to be considered as well. Limits should not be widened if the laboratory's performance is not consistent with other labs.

4.4 Discontinuous Pattern

If the data appear to run for a period at one mean recovery and then suddenly jump to a different level, then the following should be considered:

4.4.1 The accuracy of the analytical system experienced a statistically significant change, and most likely there is an event that caused that change, such as recalibration, change in instrument or instrument settings, change in calibration standards, change in methodology, or a change in analyst.

4.4.2 Consult with the supervisor.

4.4.3 Unless the discontinuity is characteristic of the method somehow, a decision will usually need to be made as to which mode of operation is the best predictor of future performance. A selective time period may be used for calculating representative limits.

4.5 Upward or Downward Sloping Pattern

An upward or downward trend is typically indicative of an unstable instrument or progressive changes in background or contamination levels. Such trends are early warning that the analytical system will soon be out of control. When a trend is detected, investigate as follows:

4.5.1 Consult with the supervisor, and have the supervisor consider the condition of standards and maintenance of equipment.

4.5.2 Review the SOP and check the proficiency of the analysts.

4.5.3 Reliable control limits cannot be set using data during an unstable period. Maintaining the old limits until a stable period is documented is probably the best course.

5.0 Establishing New Control Limits

Having collected sufficient data and determined that the data are representative, the next step is to establish the new limits. Control limits are set at ± 3 standard deviations around the mean of the collected data with the following exceptions:

5.1 If the calculated 3-standard-deviation limits are tighter than the method calibration verification criteria (e.g., CCV acceptance limits for ICP are ± 10% of the expected value), then the new limits are set to the mean value ± the calibration acceptance limits.

5.2 If the calculated limits are wider than method or program requirements, then the laboratory's performance should be reconsidered. If the limits are marginally wider, inspect the control chart to estimate the frequency of failures using the program limits. If, based on the control chart, the failure rate is predicted to be low (less than 2% is normally acceptable), then program limits might be adopted. Otherwise the laboratory will need to either not offer the test or request a variance.

5.3 If the lower control limit is very low, e.g., less than 10%, there is a concern about accepting data that is not quantitatively reliable. Inspect the control chart data to predict the failure rate if the lower control limit is elevated to 10%. If, based on the control chart, the failure rate is predicted to be low, then the lower control limit might be elevated to 10%.

5.4 If the upper control limit is less than 100% recovery plus the method calibration verification acceptance limit, then adjust the upper limit to 100% plus the calibration acceptance limit. For example, if CCV acceptance limits are ± 10% of the expected value, then set the upper control limit to 110%.

The basis of the new control limits must be documented. Annotation may be made directly on the printed control charts or control limits reports, and must be signed and dated.

6.0 Communicating and Implementing New Control Limits

6.1 Compile all reviewed data, control charts, and control limits reports.

6.2 Prepare a memo from QA to the affected group leader that summarizes the control limit and control chart reviews, and compares the new control limits to the old. Place a line at the bottom of the memo for the signature of the group leader. See the example in Figure 1 below.

6.3 Send the memo and compiled charts and data to the affected group leader for review. The group leader must review the data compilation, and sign the memo to signify that the selected data are representative of the current performance.

6.4 The group leader must return the signed memo and compiled control data to QA.

6.5 QA and the group leader will confer to set an implementation date for the new limits. The implementation date is the date when the control limits will be update in TALS and in any local databases used by the laboratory group (e.g., the Target database, which is used for chromatography data).

6.6 The memo and associated data and charts are scanned as an Adobe Acrobat file (i.e., pdf file) and saved to the QA public drive in the “Control Limits” subdirectory.

6.7 QA personnel are responsible for updating control limits in TALS and notifying group leaders by e-mail when limits are updated.

NOTE: For more detail on control charts see the most current revision of CW-I-T-001 on Oasis

Attachment 2. Example Control Limits Review Form

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

CONTROL CHART REVIEW

Method:		**Prep(s):**		**QC Program(s):**	
Spike List(s):					
Group:		**Group Leader:**			
Reason for review:	☐ limits update	☐ periodic routine review	☐ analytical system change		
Reviewed By:		**Date:**			

Reference TestAmerica Denver Policy QA-003P, *Quality Assurance Program*.

Purpose: Control charts and associated data are reviewed to (1) verify that the analytical system operated within statistical control; (2) verify that the data set used is truly representative of the laboratory's performance over the indicated time period, and therefore can be used to predict future performance; (3) to determine whether the existing limits should be updated based on the new data set; (4) to determine whether the newly calculated limits can be used as is or should be modified to be consistent with program requirements or continuing calibration criteria; (5) to determine whether the lab's statistical limits meet applicable program requirements.

Instructions: Use the Control Chart Module in TALS to view/print control charts for a specific time period. Examine each control chart in the attached package and document your review below. Record the results of investigations and any corrective actions taken on the appropriate control charts. Statistical control criteria are not applied to poor performing compounds since, by definition, the variability of the analytical performance of these compounds is not randomly distributed.

√	Reviewed For	Specific Measure Control Failures / Anomalies Found
	Outliers	☐ No outliers appear in any chart because only compliant data are uploaded to the LIMS. ☐ All outliers were investigated and the data and explanation or NCM number were recorded on the applicable charts. ☐ An excessive number of outliers were noted on one or more charts. The data were investigated for increased variability and an explanation is written on the applicable chart(s).
	Biases	☐ On one or more charts, the data exhibit a significant bias compared to the existing LIMS limits. The bias was investigated and an explanation is written on the applicable chart(s).
	Discontinuous Patterns	☐ On one or more control charts, a discontinuous pattern was noted. The pattern was investigated and an explanation is written on the applicable chart(s).
	Trends	☐ On one or more control charts, an upward or downward trend is noted. The trend was investigated and an explanation is written on the applicable chart(s), unless the trend was short-lived, corrected itself, and did not significantly affect data quality.
	Comparison to Method / Program Limits	☐ The newly calculated statistical limits were compared to any applicable method or program limits. Any limits that did not meet the method/program limits were investigated and an explanation written on the applicable chart(s).
	Control Limits Update	☐ For one or more control charts, the existing limits are still applicable and do not need to be changed as noted on the applicable control chart(s) or the Control Limits Summary. ☐ For one or more control charts, the control limits shall be updated to the newly calculated limits as noted on the applicable control chart(s) or the Control Limits Summary. ☐ For one or more control charts, the upper control limit is less than the CCV upper limit, therefore the limits will be updated to the newly calculated lower control limit and the upper CCV limit, as noted on the applicable control chart(s) or the Control Limits Summary. ☐ For one or more control charts, the newly calculated limits are tighter than the CCV criteria, therefore the limits will be updated to the newly calculated mean ± the CCV limits, as noted on the applicable control chart(s) or the Control Limits Summary. ☐ For one or more control charts, the newly calculated lower control limit is < 10%, therefore the lower control limit is set at 10%.

Comments:

☐ Control limits package scanned & archived by QA.	**QA Review:**		**Date:**
Entered By:	**Date:**	**Reviewed By:**	**Date:**

Revision 1 7/15/2013 G:\QA\Edit\FORMS\QA\CONTROL CHART form - TA_7-15-2013.doc

Quality Assurance Manual

TestAmerica Edison
777 New Durham Road
Edison, NJ 08817
Phone No. 732-549-3900
Fax No. 732-549-3679

www.testamericainc.com

Facility Distribution No. ELECTRONIC **Distributed To: FACILITY INTRANET**

Title Page:

Quality Assurance Manual
Approval Signatures

11/15/11

Laboratory Director – Ann Gladwell — Date

11/15/11

Quality Assurance Manager - Carl Armbruster — Date

11/15/11

Operations Manager – Mark Acierno — Date

SECTION 2. TABLE OF CONTENTS

LIST OF TABLES

LIST OF FIGURES

LIST OF APPENDICES

REFERENCED CORPORATE SOPs AND POLICIES

SOP / Policy Reference	Title
CA-Q-S-001	Solvent and Acid Lot Testing and Approval
CA-Q-S-002	Acceptable Manual Integration Practices
CA-Q-S-004	Method Compliance & Data Authenticity Audits
CA-Q-S-006	Detection Limits
CA-Q-S-008	Management Systems Review
CW-Q-S-001	Corporate Document Control and Archiving
CW-Q-S-002	Writing a Standard Operating Procedure (SOPs)
CW-L-S-002	Internal Investigation of Potential Data Discrepancies and Determination for Data Recall
CA-L-S-002	Subcontracting Procedures
CW-L-P-004	Ethics Policy
CA-L-P-002	Contract Compliance Policy
CW-F-P-002	Authorization Matrix
CW-F-P-004	Procurement and Contracts Policy
CA-C-S-001	Work Sharing Process
CA-T-P-001	Qualified Products List
CW-F-S-007	Controlled Purchases Policy
CW-F-S-018	Vendor Selection
CA-Q-M-002	Corporate Quality Management Plan
CW-E-M-001	Corporate Environmental Health & Safety Manual

REFERENCED LABORATORY SOPs

SOP Reference	Title
ED-GEN-002	Document Control
ED-GEN-003	Control of Non-Conformances and Corrective Action
ED-GEN-022	Training
ED-GEN-024	Record Storage and Retention
ED-GEN-001	Data Management and Handling
ED-GEN-021	Data Review
ED-GEN-007	Subsampling
ED-SPM-001	Sample Receipt, Login, Identification, And Storage
ED-RP-001	Reports Production
ED-GEN-011	Calibration and Use of Pipettes
ED-FLD-008, -009	Groundwater Sampling and Flow Monitoring
ED-FLD-014	Wastewater Sampling
ED-FLD-001 thru -010	Field Analytical Parameters
ED-SPM-006	Acceptance and Handling of Regulated Domestic & Foreign Soils
ED-SPM-007	Disposal of Samples and Associated Laboratory Waste

SECTION 3. INTRODUCTION, SCOPE AND APPLICABILITY

3.1 Introduction and Compliance References

TestAmerica Edison'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 NELAC Institute (TNI) Standard, dated 2009, Volume 1 Modules 2 and 4, and ISO/IEC Guide 17025:2005(E). 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 3. 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 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.
- *Manual for the Certification of Laboratories Analyzing Drinking Water (EPA 815-R-05-004, January 2005) (DW labs only)*
- *Statement of Work for Inorganics & Organics 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, 21st and on-line Editions.
- Toxic Substances Control Act (TSCA).

3.2 Terms and Definitions

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 2 for the Glossary/Acronyms.

3.3 Scope / Fields of Testing

The laboratory analyzes a broad range of environmental and industrial samples every month. Sample matrices vary among drinking water, effluent water, groundwater, hazardous waste, sludge and soils. 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 processes, reviewing results, servicing clients and tracking samples through the laboratory. The technical and service requirements of all analytical requests 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 TestAmerica Edison Work Instruction No. EDS-WI-009 (Analytical Capabilities). The approach of this manual is to define the minimum level of quality assurance and quality control necessary to meet these 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 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

The template on which this manual is based is reviewed annually by Corporate Quality Management Personnel to assure that it remains in compliance with Section 3.1. This manual itself is reviewed every two years 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. ED-GEN-002, Document Control).

SECTION 4. MANAGEMENT REQUIREMENTS

4.1 Overview

TestAmerica Edison 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, and Corporate Quality). The laboratory operational and support staff work under the direction of the Laboratory Director. The organizational structure for both Corporate & TestAmerica Edison is presented in Figure 4-1.

4.2 Roles and Responsibilities

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 briefly define each role in its relationship to the Quality Assurance Program.

4.2.1 Additional Requirements for Laboratories

The responsibility for quality resides 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 TestAmerica's Edison laboratory.

4.2.2 Laboratory Director/Lead Technical Director

TestAmerica Edison's Laboratory Director is responsible for the overall quality, safety, financial, technical, human resource and service performance of the whole laboratory and reports to the

General Manager (GM). The Laboratory Director provides the resources necessary to implement and maintain an effective and comprehensive Quality Assurance and Data Integrity Program.

Specific responsibilities include, but are not limited to:

- Serves as lead technical director for all fields of testing.
- 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.
- Ensures TestAmerica's human resource policies are adhered to and maintained.
- 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.
- Monitors standards of performance in quality control and quality assurance.
- Monitors the validity of analyses performed and data generated in the lab to assure reliable data.
- Reviews and approves all SOPs prior to their implementation and ensures all approved SOPs are implemented and adhered to.
- Interfaces with Project Management and Customer Service to forecast receipts, provide quality analytical data to clients and meet on-time delivery dates.
- Ensures that the facility has appropriate Information Technology resources and that they are 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.
- Ensures client specific reporting and quality control requirements are met.
- Captains the management team, consisting of the QA Manager, the Operations Manager, the Project Management Director, the Client Services Manager, the Service Center Manager, the Environmental, Health and Safety Manager and the Support Services Manager as direct reports.

4.2.3 Quality Assurance (QA) Manager

The QA Manager has responsibility and authority to ensure the continuous implementation of the quality system.

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 (e.g., 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:

- Serves as the focal point for QA/QC in the laboratory.
- Having functions independent from laboratory operations for which he/she has quality assurance oversight.
- Maintaining and updating the QAM.
- Monitoring and evaluating laboratory certifications; scheduling proficiency testing samples.
- Monitoring and communicating regulatory changes that may affect the laboratory to management.
- Training and advising the laboratory staff on quality assurance/quality control procedures that are pertinent to their daily activities.
- Have documented training and/or experience in QA/QC procedures and the laboratory's Quality System.
- 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 shall be investigated following procedures outlined in Section 12 and if deemed necessary may be temporarily suspended during the investigation.
- Objectively monitor standards of performance in quality control and quality assurance without outside (e.g., managerial) influence.
- Coordinating of document control of SOPs, MDLs, control limits, and miscellaneous forms and information.
- 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.
- Captains the QA team to enable communication and to distribute duties and responsibilities.

- Ensuring Communication & 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.

4.2.4 Quality Assurance (QA) Specialist

The Quality Assurance (QA) Specialist is responsible for performing data audits, special audits, assisting with external and systems audits, overseeing the maintenance of QC records, certifications, Standard Operating Procedures (SOPs), training records, DOCs, arranging and managing PT samples. Additional responsibilities may include assisting with systematic problems within the laboratory, assisting in reviewing and/or writing of Quality Assurance Project Plans, and technical and QC specifications in contracts and other functions in support of the QA Manager's responsibilities as assigned.

- Assist QA Manager in conducting QA training courses, including ethics training.
- Performs data audits.
- Assist in performing special audits as deemed necessary by data audits, client inquiries, etc.
- Assisting in, conducting and responding to external audits conducted by clients and regulatory agencies.
- Assisting in reviewing and/or writing of Quality Assurance Project Plans, and technical and QC specifications in contracts.
- Maintaining all necessary laboratory certifications.
- Arranging and managing PT samples.
- Reviewing laboratory SOPs. Writing SOPs as needed.
- Maintaining historical indices of all technical records including SOPs, QC records, laboratory data, etc.
- Ensuring maintenance of records archives.
- Assisting in and monitoring laboratory's method compliance.
- Ensuring maintenance of DOCs for all analysts.
- Ensuring maintenance of training records for all employees.

- Assisting in identification of systematic problems within laboratories.
- Recommends resolutions for ongoing or recurring nonconformance.
- Providing statistical feedback to Departments on error rates, and assisting in identifying systematic improvements to minimize errors.
- Assists in tracking of customer complaints, providing statistical feedback to the laboratory, and assisting in identifying improvements.
- Overseeing and reviewing MDL studies.
- Ensuring control charts are generated; oversees and approves setting of control limits.
- Assists in monitoring new regulations and communicating them to the laboratory.

4.2.5 LAN Analyst

The LAN Analyst reports directly to the Regional Desktop Support Supervisor. Responsibilities include:

- Works with Corporate IT to solve information systems problems and to standardize laboratory IT equipment and processes.
- Monitors and supports office automation so that LAN is operational for internal and external communications.
- Troubleshoots problems throughout laboratory relating to computers, software, telephones and other electronic equipment.
- Responsible for new user setup on network, LIMS, telephone and voice mail.
- Installs or upgrades computers and other equipment.
- Maintains tape backups for multiple computer servers including LIMS.
- 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.
- Maintains awareness of any environmental conditions of the facility housing the LIMS that may compromise LIMS raw data and informs management.

4.2.6 Operations Manager

The Operations Manager manages and directs the analytical and reports production sections of the laboratory. He/She reports directly to the Laboratory Director. Specific responsibilities include:

- Maintains awareness of any environmental conditions of the facility housing the LIMS that may compromise LIMS raw data and informs management.
- 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 Laboratory Director and QA Manager and in compliance with regulatory requirements.
- Works with the Department (Technical) Managers to ensure that scheduled instrument maintenance is completed.
- 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.

4.2.7 Environmental, Health and Safety Manager

The Environmental, Health and Safety Manager reports directly to the Laboratory Director. The duties of this position consist of:

- Supervises the Environmental, Health and Safety/Facilities Team.
- 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.
- 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.

4.2.8 EH&S/Facilities Coordinator

The EH&S/Facilities Coordinator reports directly to the Environmental, Health and Safety Manager. The duties of this position consist of:

- Monitors laboratory for unsafe conditions or acts to keep lab in compliance with the Chemical Hygiene Plan, EH&S Procedures, and company policies.
- Ensures the proper personal protective equipment is available and personnel are properly trained in its use.
- Assists the Environmental, Health and Safety Manager in the investigation of accidents, incidents, and near misses and identifies and eliminates root cause.
- Conducts monthly facility inspections for compliance with health, safety and environmental regulations and procedures. Completes and forwards monthly inspection report to safety committee and laboratory management for corrective actions.
- Conducts safety equipment checks to ensure proper working order and sufficient inventory.
- Plans and tracks completion of monthly general awareness training sessions and compliance training, including new employee EH&S orientation.
- Coordinates emergency response team to provide prompt medical attention and stabilize emergency situation. After emergency is over, assists in determining appropriate clean up procedures.
- Conducts the monthly EH&S committee meeting.
- Participates in monthly EH&S conference call.
- Reviews and maintains MSDS's for laboratory materials.
- Coordinates the management and disposal of laboratory wastes.
- Assists in the preparation and maintenance of the laboratory Integrated Contingency Plan.
- Monitors air quality in facility, including monitoring fumehoods for proper operation and ventilation.
- Maintains overall building facilities and equipment as well as administers prevention maintenance measures.
- Contacts outside contractors as necessary to repair/maintain items outside the realm of reasonable maintenance.
- Performs miscellaneous errands, buying parts for labs, janitorial supplies.
- Oversees storage facilities, files and outside storage.

4.2.9 Technical Managers (Department Managers)

The Technical Managers (Department Managers) report directly to the Operations Manager. They are accountable for all analyses and analysts under their experienced supervision. The scope of responsibility ranges from the new-hire process and existing technology through the ongoing training and development programs for existing analysts and new instrumentation. Specific responsibilities include, but are not limited to:

- Exercises day-to-day supervision of laboratory operations for the appropriate field of accreditation and reporting of results. 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. He/she develops standard costing of SOPs to include supplies, labor, overhead, and capacity (design vs. demonstrated versus first-run yield) utilization.
- Reviewing and approving, with input from the QA Manager, proposals from marketing, in accordance with an established procedure for the review of requests and contracts. This procedure addresses the adequate definition of methods to be used for analysis and any limitations, the laboratory's capability and resources, the client's expectations. Differences are resolved before the contract is signed and work begins. A system documenting any significant changes is maintained, as well as pertinent discussions with the client regarding their requirements or the results of the analyses during the performance of the contract. All work subcontracted by the laboratory must be approved by the client. Any deviations from the contract must be disclosed to the client. Once the work has begun, any amendments to the contract must be discussed with the client and so documented.
- Monitoring the validity of the analyses performed and data generated in the laboratory. This activity begins with reviewing and supporting all new business contracts, insuring data quality, analyzing internal and external 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.
- 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.
- Enhancing efficiency and improving quality through technical advances and improved LIMS utilization. Capital forecasting and instrument life cycle planning for second generation methods and instruments as well as asset inventory management.
- Coordinating sample management from "cradle to grave," insuring that no time is lost in locating samples.
- Ensures that 100% of data review undergoes two documented levels of review. Likewise ensures that all non-conformance issues are properly documented.
- Scheduling all QA/QC-related requirements for compliance, e.g., MDLs, etc..

- Captains Department personnel to communicate quality, technical, personnel, and instrumental issues for a consistent team approach.
- Responsible for the timely and accurate completion of performance evaluation samples and MDLs, for the Department.
- Ensure all logbooks are maintained, current, and properly labeled or archived.
- Report all non-conformance conditions to the QA Manager, Operations Manager, and/or Laboratory Director.
- Ensure that preventive maintenance is performed on instrumentation as detailed in the QA Manual or SOPs. He is responsible for developing and implementing a system for preventive maintenance, troubleshooting, and repairing or arranging for repair of instruments.
- Maintain adequate and valid inventory of reagents, standards, spare parts, and other relevant resources required to perform daily analysis.
- Achieve optimum turnaround time on analyses and compliance with holding times.
- Provide written responses to external and internal audit issues and coordinates audit responses with the QA Manager.

4.2.10 Laboratory Analysts and Technicians

Laboratory analysts and technicians are responsible for conducting analysis and performing all tasks assigned to them by their Department manager 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 worklists, benchsheets, lab notebooks and/or the Non-Conformance Database by means of Non-Conformance Memos (NCMs).
- Report all non-conformance situations, instrument problems, matrix problems and QC failures, which might affect the reliability of the data, to their Department (Technical) Manager, the Laboratory Director, 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.
- Suggest method improvements to the Department (Technical) Manager, the Laboratory Director, and the QA Manager. These improvements, if approved, will be incorporated within the constraints of the consensus reference methods.
- Work cohesively as a team in their 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 environmental, health and safety protocols and attend safety meetings as required.

- Attend and participate in all staff meetings.

4.2.11 Sample Control Manager

The Sample Control Manager reports to the Laboratory Director. The responsibilities are outlined below:

- Direct the logging of incoming samples into the LIMS.
- Ensure the verification of data entry from login.
- Manages the preparation and shipment of bottle kits to clients.
- Oversees the responsibilities of all Sample Control Technicians.
- Supervises the storage and disposal of all samples.

4.2.12 Client Services Manager

The Customer Service Manager reports to the Laboratory Director and serves as the primary interface between the laboratory and the Sales and Marketing staff. Responsibilities include:

- Laboratory's primary client representative.
- Ensures client complaints are handled professionally, and resolved in a timely manner.
- Compiles and interprets receipts forecast to show near term business trends.
- Manages a minimal list of projects/programs for key client accounts. (Note: sufficient time is needed to manage the PM group and the CSM must not be overwhelmed with project management.)
- Prepares proposals for new business opportunities.
- Compiles and interprets Bid Activity Report.
- Compiles and interprets receipts forecast to show near term business trends.
- Prepares proposals for new business opportunities.
- Provides general sales support to Account Executives for business development activities started in the field.
- Develops and maintains business materials and organized information resource files that include project descriptions, resumes, original proposals, boilerplates, and company qualifications materials.

4.2.13 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:

- 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 for ensuring that clients receive the proper sampling supplies, as appropriate.
- 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.
- Accountable to clients for communicating sample progress in daily status meeting 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).

4.2.14 Project Manager

The Project Managers report directly to the Director of Project Management and serve as liaisons between the laboratory and its clients. The Project Manager's responsibilities include:

- Ensure client specifications are met by communicating project and quality assurance requirements to the laboratory.
- Notify laboratory personnel of incoming projects and sample delivery schedules.
- Monitor the status of all projects in-house to ensure timely delivery of reports.
- Inform clients of project-related problems, resolving service issues and coordinating technical issues with the laboratory staff.
- 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.
- Coordinate client requests for sample containers and other services.
- Schedule sample pick-ups from client offices or project sites and notifying the laboratory staff of incoming samples.
- Coordinate subcontract work.

- Respond to client inquiries concerning sample status.
- Performs final completeness review of data packages prior to release to client.

4.2.15 Project Management Assistant

The Project Management Assistant coordinates and monitors scheduling, timely completion and maintenance of project documentation files and completion of project set up and final report review, invoicing, and EDD's. Assists the Project Manager in servicing the client's needs. Specific responsibilities include:

- Reviews login confirmation reports for accuracy and corrects as needed.
- Generates diskettes for electronic data deliverables (EDD's) for electronic delivery to clients.
- Enters data that was subcontracted to other laboratories.
- Monitors report due dates for timely delivery.
- Assists Project Manager in changing compound lists, TAT, deliverables and other client specific requirements in the LIMs project and/or job database.
- Invoices completed data packages and generates credit or debit invoices to ensure proper payment.

4.2.16 Service Center Manager

The Service Center Manager (SCM) manages the service center and acts as a liaison between the laboratory and the local client base. The SCM is in charge of maintaining the Service Center facility, managing service center couriers, samplers and other personnel, and working with sales to develop, maintain and grow the client base in the area.

- Local area primary client representative for service center location.
- May head project start up meetings to ensure project objectives are successfully met and hands off project detail to assigned Project Manager(s).
- Works with the Quality Assurance Manager and Account Executives (AE) to evaluate and establish project requirements for the service center area.
- Ensures client complaints are handled professionally, and resolved in a timely manner.
- Is in charge of scheduling service center couriers and samplers, preparing bottle orders for delivery, scheduling sample pick ups and shipping samples to the designated laboratory for analysis.
- May manage a minimal list of projects/programs for key client accounts.
- Maintains the facilities at the service center and is responsible for all EH&S policies of TestAmerica at the service center.
- Responsible for all company vehicles that operate out of the service center.
- Provides general sales support to AEs for business development activities started in the field.
- Prepares proposals for new business opportunities.

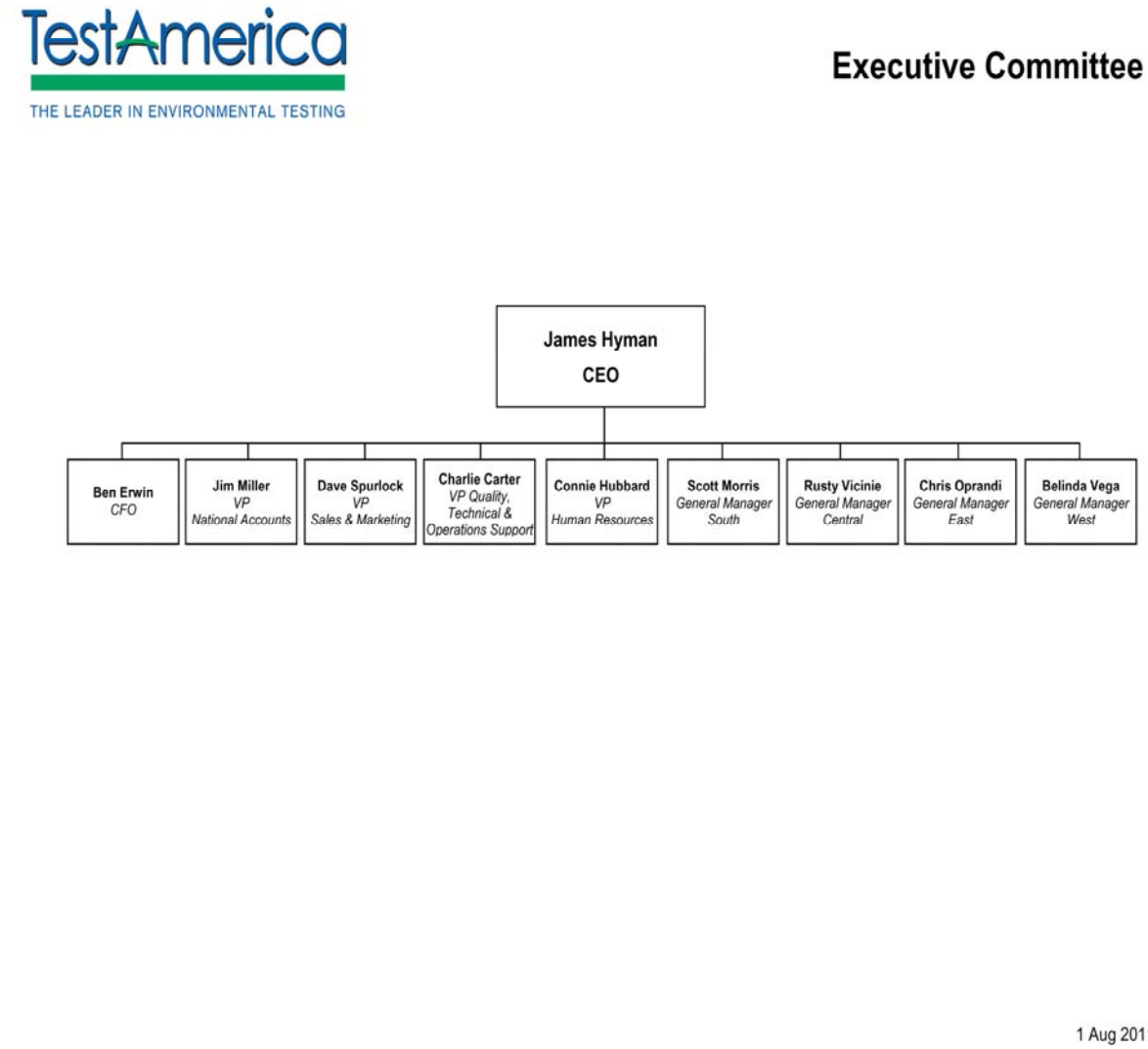
- Orders supplies (bottles, coolers, etc.) for the service center

4.3 Deputies

The following table defines who assumes the responsibilities of key personnel in their absence:

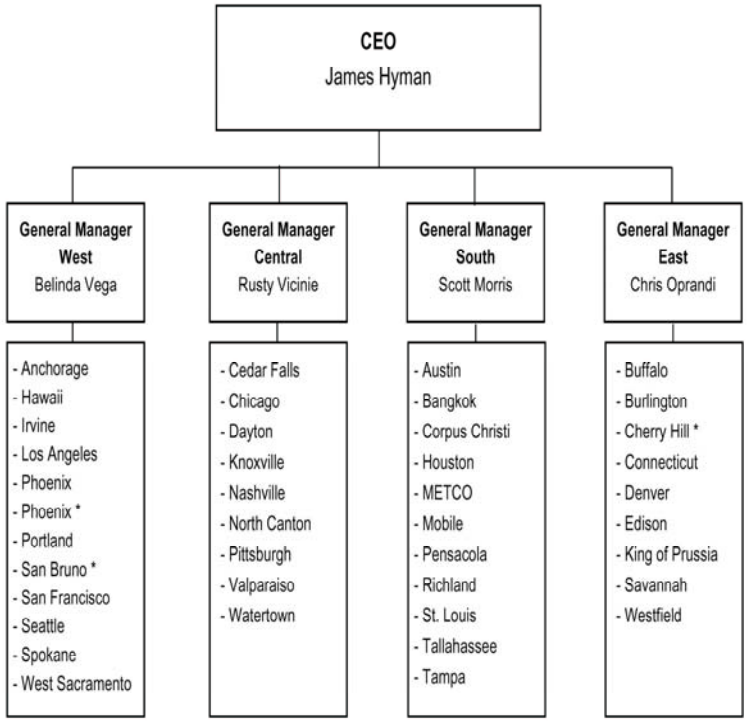
Key Personnel	Deputy
Ann Gladwell Laboratory Director	In the event of absence the Laboratory Director's responsibilities are shared by the Laboratory Operations Manager, the Quality Assurance Manager and the Client Services Manager, as appropriate
Carl Armbruster Quality Assurance Manager	Emmylou Digiacomo Quality Assurance Specialist Ann Gladwell Laboratory Director
Department (Technical) Managers	Mark Acierno Laboratory Operations Manager
David Lissy Client Services Manager	Ann Gladwell Laboratory Director
Kenwyn Williams Director of Project Management	Ann Gladwell Laboratory Director
Kene' Kasperek EH&S Manager	Edward Roche EH&S Coordinator
Kenwyn Williams Sample Control Manager	Mark McClain Sample Control Supervisor
Aidan Scott Kate Harrelson Service Center Managers	Field Services Supervisor

Figure 4-1. Corporate and Laboratory Organization Charts



TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

Operations

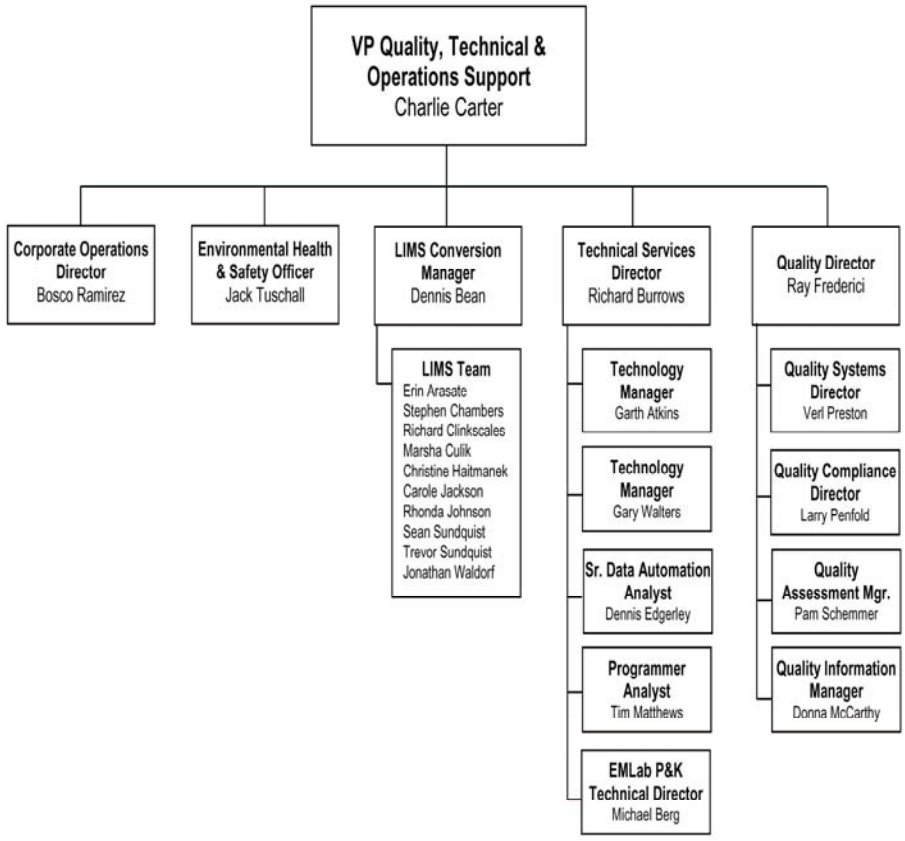


* Note: EMLab P&K microlabs report to these facilities.

1 Aug 2011

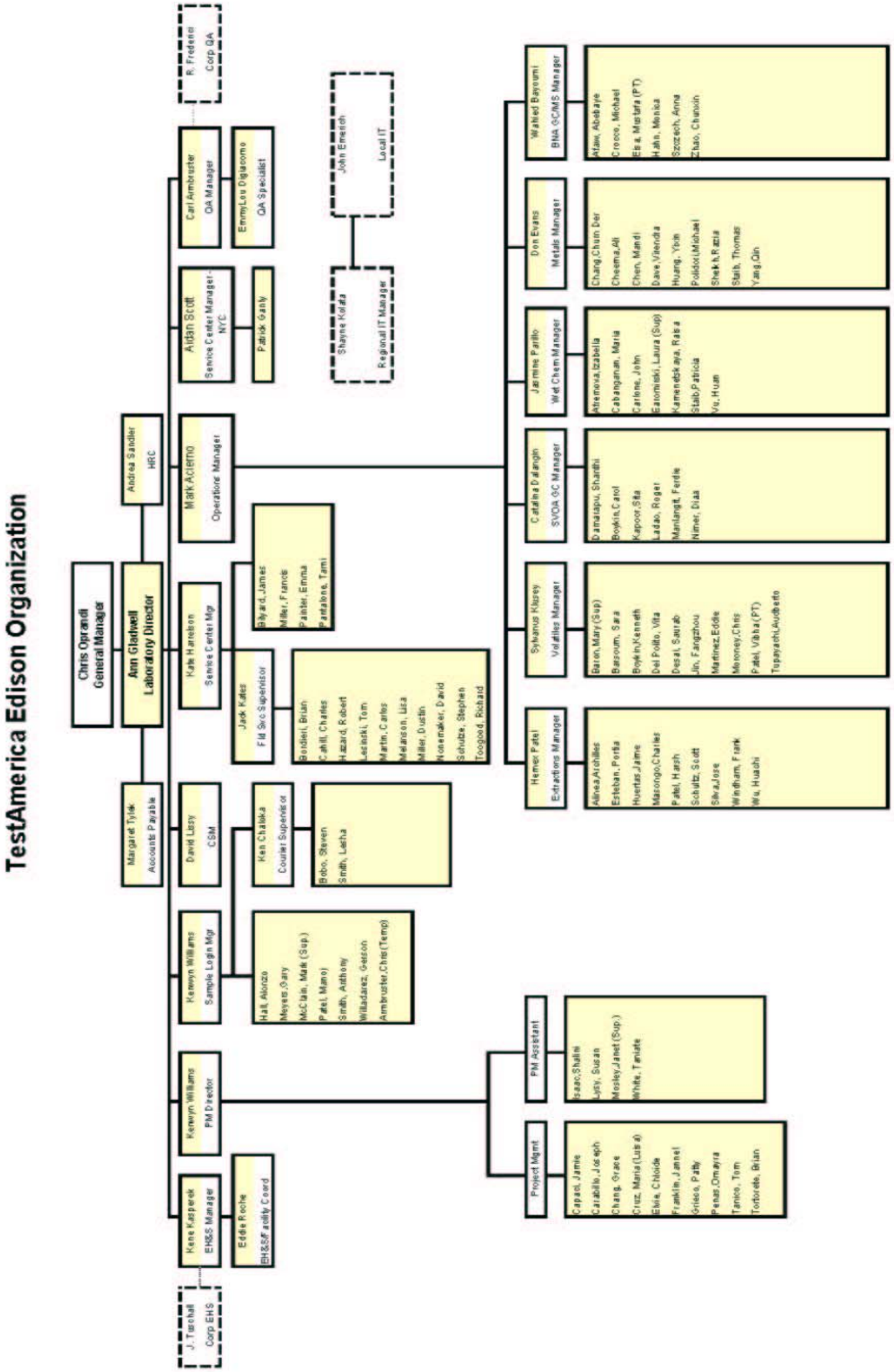
TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

Quality, Technical & Operations Support



Note:
QA Managers and EH&S Managers have a direct reporting relationship to both operations leadership and corporate functional leadership.

4 Aug 2011


TestAmerica Edison Organization
Chris Oprandi
General Manager
Ann Gladwell
Laboratory Director
Andrea Sandler
HRC
Mark Acierno
Operations Manager
R. Frederer
Corp QA
Carl Armbruster
QA Manager
EmmyLou Digiacomo
QA Specialist
Aidan Scott
Service Center Manager-NYC
Patrick Gately
John Emerich
Local IT
Shayne Kolata
Regional IT Manager
Kathi Hamelton
Service Center Mgr
Margaret Tylek
Accounts Payable
David Lassy
CSM
Kenwyn Williams
Sample Login Mgr
Hall, Alonzo
Meyers,Gary
McClain, Maki (Sup.)
Patel, Mansi
Smith, Anthony
Willadarez, Geisson
Ambruster,Chris(Temp)
Ken Chaleka
Courier Supervisor
Bobo, Steven
Smith, Lasha
Jack Kates
Fld Svc Supervisor
Goldieri, Brian
Cahill, Charles
Hazard, Robert
Lesinski, Tom
Martin, Carlos
Melanson, Lisa
Miller, Dustin
Rosenmeier, David
Schultz, Stephen
Toogood, Richard
Bilyard,James
Miller, Francis
Painter, Emma
Pantalena, Tami
Kenwyn Williams
PM Director
PM Assistant
Project Mgmt
Kane Kasperek
EH&S Manager
Eddie Roche
EH&S Facility Coord
J. Turchall
Corp EHS
Hemen Patel
Extractions Manager
Sylvanus Klarey
Volatiles Manager
Catalina Dalangin
SVOA GC Manager
Jasmine Parillo
Wet Chem Manager
Don Evans
Metals Manager
Walied Bayomi
BNA GC/MS Manager
Alfonso,Archilles
Esteban, Portia
Huertas,Jaime
Masongo,Charles
Patel, Harsh
Schultz, Scott
Silva,Jose
Windham, Frank
Wu, Huashi
Baron,Mary (Sup)
Bassoum, Sara
Boykin,Kenneth
Del Potto, Vita
Desai, Sairab
Jin, Fangzhou
Martinez,Eddie
Mooney,Chris
Patel, Vibha(PT)
Tupyashi,Audibeto
Damaraju, Shanthi
Bopkha,Carol
Kapoor,Sita
Ladao, Roger
Manlangit, Ferdie
Nimer, Dlaa
Ateenova,Izabella
Cabanganan, Maria
Carlene, John
Kazmierski, Laura (Sup)
Kamenetskaya, Rasa
Stalb,Patricia
Vu, Huan
Chang,Chun Der
Cheema,Ali
Chen, Mandi
Dave,Virendra
Huang, Yibin
Polidori,Michael
Sheikh,Razia
Stark, Thomas
Yang,Qin
Afaw, Ababaye
Crocce, Michael
Elia, Marlaria (PT)
Hahn, Monica
Szczech, Anna
Zhao, Chanxin
Capasi, Jamie
Caraballo,Joseph
Chang, Grace
Cruz, Maria (Luisa)
Elvia, Chloda
Franklin, Jamel
Griego, Patty
Perez,Omayra
Tanico, Tom
Torborde, Brian
Isaac,Shalini
Lutty, Susan
Mosley,Janet (Sup.)
White, Tamatte

SECTION 5. QUALITY SYSTEM

5.1 Quality Policy Statement

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.
- ❖ To comply with the ISO/IEC 17025:2005(E) International Standard, the 2009 TNI Standard 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

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. CW-L-P-004) and Employee Ethics Statements.
- Ethics and Compliance Officers (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. CW-L-S-002)
- Procedures and guidance for recalling data if necessary (Corporate SOP No. CW-L-S-002).
- Effective external and internal monitoring system that includes procedures for internal audits (Section 15).
- Produce results, which are accurate and include QA/QC information that meets client pre-defined Data Quality Objectives (DQOs).
- 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 Documentation

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
- Laboratory QA/QC Policy Memorandums

5.3.1 Order of Precedence

In the event of a conflict or discrepancy between policies, the order of precedence is as follows:

- 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

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.

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.

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 quantified (Reporting Limit).

5.5 Criteria for Quality Indicators

The laboratory maintains Quality Control Limits within the Method Limit Group tables in TALS (the laboratory's LIMS) that contains 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 are managed by the laboratory's QA Department. 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 development of control limits is contained in Section 24.

5.6 Statistical Quality Control

Statistically-derived precision and accuracy limits are required by selected methods (such as SW-846) and certain regulatory 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 Technical Manager 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 Method Limit Group tables in TALS (LIMS). 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 24. All calculations and limits are documented and dated when approved and effective. On occasion, a client requests contract-specified limits for a specific project.

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

The QA Manager generates QC charts using the TALS Control Chart program. In addition to their use in generating lab specific spike recovery limits and in the evaluation of MDL studies, these charts are used to determine if adjustments need to be made or for corrective actions to methods. All such findings are documented and kept on file in the QA Department.

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 16). These metrics are used to drive continuous improvement in the laboratory's Quality System.

SECTION 6. DOCUMENT CONTROL

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
- Laboratory Standard Operating Procedures (SOP)
- Laboratory Policies
- Work Instructions and Forms
- Corporate Policies and Procedures distributed outside the intranet

Corporate Quality 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. The laboratory's internal document control procedure is defined in SOP No. ED-GEN-002 (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 (CARs). Raw analytical data consists of bound logbooks, instrument printouts, any other notes, magnetic media, electronic data and final reports.

6.2 Document Approval and Issue

The pertinent elements of a document control system for each document include a unique document title and number, pagination, the total number of pages of the item or an 'end of document' page, the effective date, revision number and the laboratory's name. The QA 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 (Technical) Manager submits an electronic draft to the QA Department for suggestions and approval before use. Upon approval, QA personnel add the identifying version information to the document and retains that document as the official document on file. That document is then 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 every two years and revised as appropriate. Changes to documents occur when a procedural change warrants.

6.3 Procedures for Document Control Policy

For changes to the QA Manual, refer to SOP No. ED-GEN-002 (Document Control) Uncontrolled copies must not be used within the laboratory. Previous revisions and back-up data are stored by the QA Department. Electronic copies are stored on the Public server in the QA folder for the applicable revision.

For changes to SOPs, refer to SOP No. CW-Q-S-002, Writing a Standard Operating Procedure SOP. The SOP identified above also defines the process of changes to SOPs.

Forms, worksheets, work instructions and information are organized by department in the QA office. There is a table of contents. Electronic versions are kept on a hard drive in the QA Department; hard copies are kept in QA files. The procedure for the care of these documents is in SOP ED-GEN-002 (Document Control).

6.4 Obsolete Documents

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 according to SOP No. ED-GEN-002 (Document Control).

SECTION 7. SERVICE TO THE CLIENT

7.1 Overview

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 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

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 Sales Directors, 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 Managers / Directors
- Laboratory and/or Corporate Information Technology Managers/Directors
- Account Executives
- 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 Sales Director, Legal Contracts Director, Account Executive or Proposal *Coordinator* 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 applicable Project Manager maintains local copies of signed contracts.

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. These records are maintained in the project file by the Project Manager and/or Key Account Executive.

The contract will be distributed to and maintained by the appropriate sales/marketing personnel and the Account Executive. A copy of the contract and formal quote will be filed with the laboratory PM and the Laboratory Director.

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

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. 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 available 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 (Technical) Manager. After the modification is implemented into the laboratory process, documentation of the modification is made in 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

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 15 and 25).

The laboratory's standard procedures for reporting data are described in Section 25. Special services are also available and provided upon request. These services include:

- Reasonable access for our clients or their representatives to the relevant 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

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 non-conformances in either sample receipt or sample analysis. Project management will maintain ongoing client communication throughout the entire client project.

Technical Managers are available to discuss any technical questions or concerns that the client may have.

7.6 Reporting

The laboratory works with our clients to produce any special communication reports required by the contract.

7.7 Client Surveys

The laboratory assesses both positive and negative client feedback. The results are used to improve overall laboratory quality and client service. TestAmerica's Sales and Marketing teams periodically develops lab and client specific surveys to assess client satisfaction.

SECTION 8. SUBCONTRACTING OF TESTS

8.1 Overview

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 (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 TNI/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-TNI accredited work where required.

Project Managers (PMs), Customer Service Managers (CSM), or Account Executives (AE) (or others as defined by the lab) 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 (e.g, USDA) or contracts (e.g, certain USACE projects) may require notification prior to placing such work**.**

8.2 Qualifying and Monitoring Subcontracators

Whenever a PM, Account Executive (AE) or Customer Service Manager 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 is available on the TestAmerica intranet site. Supporting documentation is maintained by corporate offices and by the TestAmerica laboratory originally requesting approval of the subcontract lab. Verify necessary accreditation, where applicable, (e.g., on the subcontractors TNI, A2LA accreditation or State Certification).
- Firms identified in accordance with the company's Small Business Subcontracting program as small, women-owned, veteran-owned and/or minority-owned businesses;
- TNI 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. (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 and notify the finance group for JD Edwards.

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 are posted using the Vendor Performance Report.
- Information shall 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 Laboratory 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 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 (Figure 8-1) 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 TestAmerica Chain of Custody (COC). A copy of the original COC sent by the client must also be included with all samples workshared within TestAmerica. Client CoCs are only forwarded to external subcontractors when samples are shipped directly from the project site to the subcontractor lab. Under routine circumstances, client CoCs are not provided to external subcontractors.

Through communication with the subcontracted laboratory, the PM monitors the status of the subcontracted analyses, facilitates successful execution of the work, and ensures the timeliness and completeness of the analytical report.

Non-TNI accredited work must be identified in the subcontractor's report as appropriate. If TNI 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; however, this decision & justification must be documented in the project files, and the 'Purchase Order Terms And Conditions For Subcontracted Laboratory Services' must be sent with the samples and Chain-of-Custody. In the event this provision is utilized, the laboratory (e.g., PM) will be required to verify and document the applicable accreditations of the subcontractor. All other quality and accreditation requirements will still be

applicable, but the subcontractor need not have signed a subcontract with TestAmerica at this time. The comprehensive approval process must then be initiated within 30 calendar days of subcontracting.

Figure 8-1.

Example - Subcontracted Sample Form

Date/Time: _____

Subcontracted Laboratory Information:

- Subcontractor's Name: _____
- Subcontractor Point of Contact: _____
- Subcontractor's Address: _____
- Subcontractor's Phone: _____
- Analyte/Method: _____
- Certified for State of Origin: _____
- TNI Certified: Yes_____No_____
- USDA Permit (__Domestic __ Foreign) Yes_____No_____
- A2LA (or ISO 17025) Certified: Yes_____No_____
- CLP-like Required: Yes_____No_____
  (Full doc required)
- Requested Sample Due Date: _____
  (Must be put on COC)

Project Manager: _____

Laboratory Sample # Range: _____
(Only of Subcontracted Samples)

Laboratory Project Number (Billing Control #): _____

All subcontracted samples are to be sent via bonded carrier and Priority Overnight. Please attach tracking number below and maintain these records in the project files.

PM Signature_____**Date**_____

SECTION 9. PURCHASING SERVICES AND SUPPLIES

9.1 Overview

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 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 (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

Purchasing guidelines for equipment and reagents must meet 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 may check the item out of the on-site consignment system that contains items approved for laboratory use.

If an item is not available from the on-site consignment, the analyst must provide the master item number (from the master item list that has been approved by the Technical Director), 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 Technical Director prior to placing the order. The Department (Technical) Manager or the Laboratory Operations Manager places the order.

9.3.2 Receiving

It is the responsibility of the Facilities Coordinator to receive the shipment. It is the responsibility of the analyst who ordered the materials to document the date materials where received. 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 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

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, analytical reagent grade will 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 and solvents unless noted otherwise by the manufacturer or by the reference source method. Chemicals/solvents should not be used past the manufacturer's or SOPs expiration date unless 'verified' (refer to item 3 listed below).

- An expiration date cannot be extended if the dry chemical/solvent is discolored or appears otherwise physically degraded, the dry chemical/solvent must be discarded.
- Expiration dates can be extended if the dry chemical/solvent 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/solvent is used for the preparation of standards, the expiration dates can be extended 6 months if the dry chemical/solvent is compared to an unexpired independent source in performing the method and the performance of the dry chemical/solvent is found to be satisfactory. The comparison must show that the dry chemical/solvent meets CCV limits. The comparison studies are maintained in the analytical Department.

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. To prevent a tank from going to

dryness or introducing potential impurities, the pressure should be closely watched as it decreases to approximately 15% of the original reading, at which point it should be replaced. For example, a standard sized laboratory gas cylinder containing 3,000 psig of gas should be replaced when it drops to approximately 500 psig.. 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- µmho/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 greater than the specified limit, the Facility Manager and appropriate Technical Managers 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 bottleware used for sampling must be certified clean and the certificates must be maintained. If uncertified sampling bottleware is 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.

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 Manager/Laboratory Operations Manager 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.

Upon receipt of a new or used piece of equipment, an unique identification name is assigned and provided to the QA Department for inclusion on the laboratory master equipment list. IT must also be notified so that they can 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 19). For software, its operation must be deemed reliable and evidence of instrument verification must be retained by the IT Department or QA Department. Software certificates supplied by the vendors are filed with the LIMS Administrator. The manufacturer's operation manual is retained at the bench.

9.5 Services

Service to analytical instruments (except analytical balances) is performed on an as needed basis. Routine preventative maintenance is discussed in Section 20. The need for service is determined by analysts and/or Technical Managers. The service providers that perform the services are approved by the Technical Manager and/or the Laboratory Operations Manager.

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.

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.

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 and/or the Technology Director are consulted with vendor and product selection that have an impact on quality.

SECTION 10. COMPLAINTS

10.1 Overview

The laboratory considers an effective client complaint handling processes 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 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 12 (Corrective Actions) and is documented following the procedures in TestAmerica Edison SOP No. ED-GEN-003 (Control of Non-Conformances and Corrective Action).

10.2 External Complaints

An employee that receives a complaint initiates the complaint resolution process by first documenting the complaint according to TestAmerica Edison SOP No. ED-GEN-003 (Control of Non-Conformances and Corrective Action.

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 likelihood 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.

10.3 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 12. In addition, Corporate Management, Sales and Marketing and IT may initiate a complaint by contacting the laboratory or through the corrective action system described in Section 12.

10.4 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 16).

SECTION 11. CONTROL OF NON-CONFORMING WORK

11.1 Overview

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 12).

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 Department (Technical) Manager for resolution. The manager may elect to discuss it with the Lab Director and/or 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 12. This information can then be supplied to the client in the form of a footnote or a 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 19. The client may request that the compound be reported based only on the calibration. Such a request would need to be approved by the Laboratory Director and QA Manager, documented and 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 TNI (or the analytical method) requirements and the reason. Data being reported to a non-TNI state would need to note the change made to how the method is normally run.

11.2 Responsibilities and Authorities

TestAmerica's Corporate SOP entitled *Internal Investigation of Potential Data Discrepancies and Determination for Data Recall* (SOP No. CW-L-S-002, 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, the Lab Operations Manager, a Department (Technical) Manager, or a member of the QA team may 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. This information may also be documented in logbooks and/or data review checklists 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, Laboratory Operations Manager, the QA Manager, and the Department (Technical) Managers 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, 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.

11.3 Evaluation of Significance and Actions Taken

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 & Recall Procedure (SOP No. CW-L-S-002) 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 in lieu of the data recall determination form contained in TestAmerica's Corporate SOP No. CW-L-S-002.

11.4 Prevention of NonConforming Work

If it is determined that the nonconforming work could recur, further corrective actions must be made following the laboratory's corrective action system. 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.

11.5 Method Suspension / Restriction (Stop Work Procedures)

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 11.2, Paragraph 5.

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 12 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 (e.g., 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, Laboratory Operations Manager, QA Manager, Department Technical Manager) can devise a start-up plan to cover all steps from client notification through compliance and release of reports. Project Management, and the Directors 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.

SECTION 12. CORRECTIVE ACTION

12.1 Overview

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 Data Inquiry, Client Complaint and Corrective Action Report Form (CAR) (TestAmerica Edison Work Instruction No. EDS-WI-012) (refer to Figure 12-1).

12.2 General

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.

12.2.1 Non-Conformance Report (NCR) – The CAR form is used to document the following types of corrective actions:

- Deviations from an established procedure or SOP
- QC outside of limits (non-matrix related)
- Isolated reporting / calculation errors
- Client complaints
- Discrepancies in materials / goods received vs. manufacturer packing slips.

12.2.2 Corrective Action Report (CAR) – The CAR form is also used to document the following types of corrective actions:

- Questionable trends that are found in the review of NCRs.
- Issues found while reviewing NCRs 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
- Client complaints
- Data recall investigations
- Identified poor process or method performance trends
- Excessive revised reports

This will provide background documentation to enable root cause analysis and preventive action.

12.3 Closed Loop Corrective Action Process

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.

12.3.1 Cause Analysis

- Upon discovery of a non-conformance event, the event must be defined and documented. An CAR must be initiated, someone is assigned to investigate the issue and the event is investigated for cause. Table 12-1 provides 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 Department (Technical) Manager, Laboratory Director, Laboratory Operations Manager, or QA Manager (or QA designee) is consulted.

12.3.2 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 CAR is used for this documentation.

12.3.3 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; for example, 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.

12.3.4 Monitoring of the Corrective Actions

- The Department (Technical) 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 (Technical) Managers are accountable to the Laboratory Director to ensure final acceptable resolution is achieved and documented appropriately.
- Each CAR is entered into an Excel spreadsheet 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 CARs for trends. Highlights are included in the QA monthly report (refer to Section 16). 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.

12.3.5 Follow-up Audits

- 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.
- 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 15.1.4, Special Audits.)

12.4 Technical Corrective Actions

In addition to providing acceptance criteria and specific protocols for technical corrective actions in the method SOPs, 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 11). The documentation of these procedures is through the use of an CAR.

Table 12-1 includes examples of general technical corrective actions. For specific criteria and corrective actions, refer to the analytical methods or specific method SOPs. The laboratory may also maintain Work Instructions on these items that are available upon request.

Table 12-1 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 SOPs, Work Instructions, QAM Sections 19 and 20. All corrective actions are reviewed monthly, at a minimum, 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 and appropriate corrective action (e.g., reanalysis) is taken and documented.

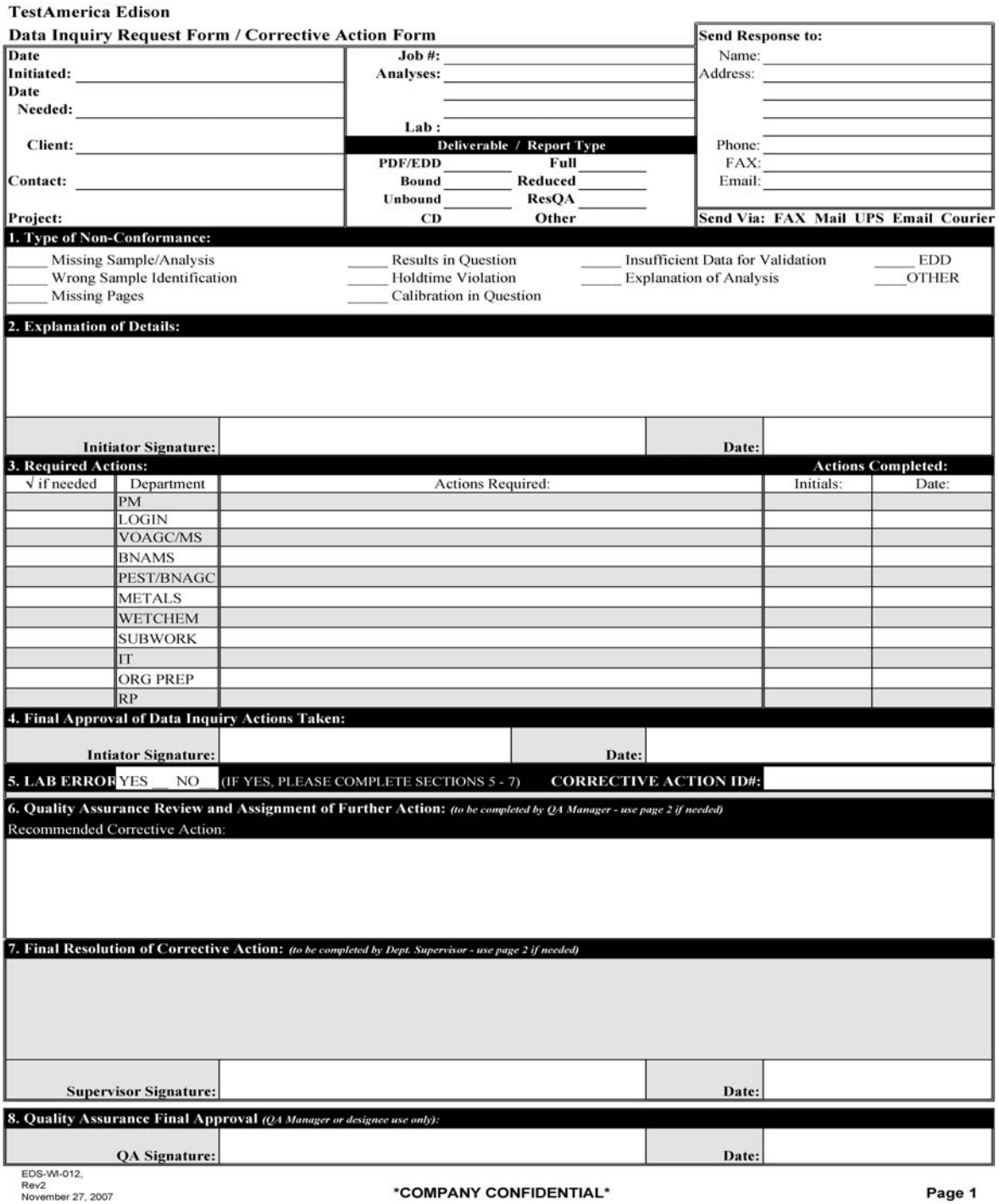
12.5 Basic Corrections

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 12-1.
Example - Corrective Action Report



TestAmerica Edison

Data Inquiry Request Form / Corrective Action Form

Date Initiated:	**Job #:**	**Send Response to:** Name:
Date Needed:	**Analyses:**	Address:
Client:	**Lab :**	
Contact:	**Deliverable / Report Type**	Phone:
Project:	**PDF/EDD** ____ **Full** ____	FAX:
	Bound ____ **Reduced** ____	Email:
	Unbound ____ **ResQA** ____	
	CD ____ **Other** ____	**Send Via: FAX Mail UPS Email Courier**

1. Type of Non-Conformance:

_____ Missing Sample/Analysis	_____ Results in Question	_____ Insufficient Data for Validation	_____ EDD
_____ Wrong Sample Identification	_____ Holdtime Violation	_____ Explanation of Analysis	____OTHER
_____ Missing Pages	_____ Calibration in Question		

2. Explanation of Details:

Initiator Signature:		**Date:**	

3. Required Actions: **Actions Completed:**

√ if needed	Department	Actions Required:	Initials:	Date:
	PM			
	LOGIN			
	VOAGC/MS			
	BNAMS			
	PEST/BNAGC			
	METALS			
	WETCHEM			
	SUBWORK			
	IT			
	ORG PREP			
	RP			

4. Final Approval of Data Inquiry Actions Taken:

Intiator Signature:		**Date:**	

5. LAB ERROR YES ___ NO ___ (IF YES, PLEASE COMPLETE SECTIONS 5 - 7) **CORRECTIVE ACTION ID#:**

6. Quality Assurance Review and Assignment of Further Action: *(to be completed by QA Manager - use page 2 if needed)*

Recommended Corrective Action:

7. Final Resolution of Corrective Action: *(to be completed by Dept. Supervisor - use page 2 if needed)*

Supervisor Signature:		**Date:**	

8. Quality Assurance Final Approval *(QA Manager or designee use only):*

QA Signature:		**Date:**	

EDS-WI-012,
Rev2
November 27, 2007

COMPANY CONFIDENTIAL

Page 1

Table 12-1. Example – General Corrective Action Procedures

QC Activity *(Individual Responsible for Initiation/Assessment)*	Acceptance Criteria	Recommended Corrective Action
Initial Instrument Blank *(Analyst)*	- Instrument response < MDL.	- Prepare another blank. - If same response, determine cause of contamination: reagents, environment, instrument equipment failure, etc..
Initial Calibration Standards *(Analyst, Department Technical Manager)*	- Correlation coefficient > 0.99 or standard concentration value. - % Recovery within acceptance range. - See details in Method SOP.	- Reanalyze standards. - If still unacceptable, remake standards and recalibrate instrument.
Independent Calibration Verification (Second Source) *(Analyst, Department Technical Manager))*	- % Recovery within control limits.	- Remake and reanalyze standard. - If still unacceptable, then remake calibration standards or use new primary standards and recalibrate instrument.
Continuing Calibration Standards *(Analyst, Data Reviewer)*	% Recovery within control limits.	- Reanalyze standard. - If still unacceptable, then recalibrate and rerun affected samples.
Matrix Spike / Matrix Spike Duplicate (MS/MSD) *(Analyst, Data Reviewer)*	- % Recovery within limits documented in TALS and/or Work Instructions	- If the acceptance criteria for duplicates or matrix spikes are not met because of matrix interferences, the acceptance of the analytical batch is determined by the validity of the LCS. - If the LCS is within acceptable limits the batch is acceptable. - The results of the duplicates, matrix spikes and the LCS are reported with the data set. - For matrix spike or duplicate results outside criteria the data for that sample shall be reported with qualifiers.

QC Activity *(Individual Responsible for Initiation/Assessment)*	Acceptance Criteria	Recommended Corrective Action
Laboratory Control Sample (LCS) *(Analyst, Data Reviewer)*	- % Recovery within limits specified in TALS and/or Work Instructions	- Batch must be re-prepared and re-analyzed. This includes any allowable marginal exceedance. When not using marginal exceedances, the following exceptions apply: 1) when the acceptance criteria for the positive control are exceeded high (i.e., high bias) and there are associated samples that are non-detects, then those non-detects may be reported with data qualifying codes; 2) when the acceptance criteria for the positive control are exceeded low (i.e., low bias), those sample results may be reported if they exceed a maximum regulatory limit/decision level with data qualifying codes. **Note:** If there is insufficient sample or the holding time cannot be met, contact client and report with flags.
Surrogates *(Analyst, Data Reviewer)*	- % Recovery within limits of method or within three standard deviations of the historical mean.	- Individual sample must be repeated. Place comment in LIMS. - Surrogate results outside criteria shall be reported with qualifiers.
Method Blank (MB) *(Analyst, Data Reviewer)*	< Reporting Limit [1]	- Reanalyze blank. - If still positive, determine source of contamination. If necessary, reprocess (i.e. digest or extract) entire sample batch. Report blank results. - Qualify the result(s) if the concentration of a targeted analyte in the MB is at or above the reporting limit AND is > 1/10 of the amount measured in the sample.
Proficiency Testing (PT) Samples *(QA Manager, Department Technical Manager)*	- 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, Department Technical Manager)*	- Defined in Quality System documentation such as SOPs, QAM, etc..	- Non-conformances must be investigated through CAR system and necessary corrections must be made.

QC Activity *(Individual Responsible for Initiation/Assessment)*	Acceptance Criteria	Recommended Corrective Action
Reporting / Calculation Errors (Depends on issue – possible individuals include: Analysts, Data Reviewers, Project Managers, Department Technical Manager, QA Manager, Corporate QA, Corporate Management)	- SOP CW-L-S-002, Internal Investigation of Potential Data Discrepancies and Determination for Data Recall.	- Corrective action is determined by type of error. Follow the procedures in SOP CW-L-S-002 or the Corrective Action SOP (ED-GEN-003).
Client Complaints *(Project Managers, Lab Director Operations Manager, Sales and Marketing)*	-	- 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 16 for an example) (QA Manager, Lab Director, Operations Manager, Department Technical Managers)	- QAM, SOPs.	- Corrective action is determined by the type of issue. For example, CARs for the month are reviewed and possible trends are investigated.
Health and Safety Violation (Safety Officer, Lab Director, Operations Manager, Department Technical Manager)	- Environmental Health and Safety (EHS) Manual.	- Non-conformance is investigated and corrected through CAR system.

Note:
1. Except as noted below for certain compounds, the method blank should be below the detection limit Concentrations up to five times the reporting limit will be allowed for the ubiquitous laboratory and reagent contaminants: methylene chloride, toluene, acetone, 2-butanone and phthalates **provided** they appear in similar levels in the reagent blank and samples. This allowance presumes that the detection limit is significantly below any regulatory limit to which the data are to be compared and that blank subtraction will not occur. For benzene and ethylene dibromide (EDB) and other analytes for which regulatory limits are extremely close to the detection limit, the method blank must be below the method detection limit

SECTION 13. PREVENTIVE ACTION / IMPROVEMENT

13.1 Overview

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 and continuous process of improvement activities 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 client satisfaction can be improved through continuous improvements to laboratory systems.

Opportunities for improvement may be discovered during management reviews, the monthly QA Metrics Report, evaluation of internal or external audits, results & evaluation of proficiency testing (PT) performance, data analysis & review processing operations, client complaints, staff observation, etc..

The monthly Management Systems Metrics Report shows performance indicators in all areas of the laboratory and 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 in evaluating the management and quality system performance on an ongoing basis and provide a tool for identifying areas for improvement.

The laboratory's corrective action process 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.

13.1.1 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.

13.1.2 Any Preventive Actions undertaken or attempted shall be taken into account during the annual Management Systems Review (Section 16). A highly detailed report is not required; however, a summary of successes and failures within the preventive action program is sufficient to provide management with a measurement for evaluation.

13.2 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 Biennial 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.

SECTION 14. CONTROL OF RECORDS

The laboratory maintains a records management 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.

14.1 Overview

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 14-1. Quality records are maintained by the QA Department in a database, 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 by Laboratory Operations under the direction of the Laboratory Operations Manager.

Table 14-1. Record Index[1]

	Record Types [1]:	Retention Time:
Technical Records	- Raw Data - Logbooks[2] - Standards - Certificates - Analytical Records - MDLs/IDLs/DOCs - 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*
QA Records	- Internal & External Audits/Responses - Certifications - Corrective/Preventive Actions - Management Reviews - 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 and Amendments - Correspondence - QAPP - SAP - Telephone Logbooks - Lab Reports	5 Years from analytical report issue*
Administrative Records	Finance and Accounting	10 years
	EH&S Manual, Permits,	7 years
	Disposal Records	Indefinitely
	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 14-2.

14.1.1 All records are stored and retained in such a way that they are secure and readily retrievable at the laboratory facility or an offsite location that provides a suitable environment to prevent damage or deterioration and to prevent loss**.** 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. 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 14-2 have lengthier retention requirements and are subject to the requirements in Section 14.1.3.

14.1.2 Programs with Longer Retention Requirements

Some regulatory programs have longer record retention requirements than the standard record retention time. These are detailed in Table 14-2 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 14-2. Example: Special Record Retention Requirements

Program	[1]Retention Requirement
Drinking Water – All States	5 years (project records) 10 years - Radiochemistry (project records)
Drinking Water Lead and Copper Rule	12 years (project records)
NY Potable Water NYCRR Part 55-2	10 years

[1]Note: Extended retention requirements must be noted with the archive documents or addressed in facility-specific records retention procedures.

14.1.3 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 19.14.1 for more information. For additional details please refer to refer to TestAmerica Edison SOP No. ED-GEN-024 (Record Storage and Retention).

14.1.4 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 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 chain of custody is stored in the laboratory's hard copy project file (in addition to the scanned copy included in the analytical report PDF). The chain of custody would indicate the name of the sampler. If any sampling notes are provided with a work order, they are kept in the project file as well. For additional details please refer to refer to TestAmerica Edison SOP No. ED-GEN-024 (Record Storage and Retention).

- 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. Reference TestAmerica Edison SOP No. ED-GEN-024 (Record Storage and Retention).

- 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 long 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 12 and 19. 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.

- Also refer to Section 19.14.1 'Computer and Electronic Data Related Requirements'.

14.2 Technical and Analytical Records

14.2.1 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. 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 sampling, performance of each analysis and reviewing results.

14.2.2 Observations, data and calculations are recorded real-time and are identifiable to the specific task.

14.2.3 Changes to hardcopy records shall follow the procedures outlined in Section 12 and 19. 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:

- laboratory sample ID code**;**
- Date of analysis; 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 or on a benchsheet.
- 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**.**

14.3 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

14.3.1 Sample Handling Records

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.

14.4 Administrative Records

The laboratory also maintains the administrative records in either electronic or hard copy form. Refer to Table 14-1.

14.5 Records Management, Storage and Disposal

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 upon request.

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.

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**.**

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 notebooks are issued on a per analysis basis, and are numbered sequentially. All data are recorded sequentially within a series of sequential notebooks. Bench sheets are filed sequentially. Standards are primarily maintained in the LIMS (this electronic record may be augmented by a logbook record. Records are considered archived when noted as such in the records management system (a.k.a., document control.)

14.5.1 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.

14.5.2 Records Disposal

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 14-1 and 14-2).

Electronic copies of records must be destroyed by erasure or physically damaging off-line storage media so no records can be read.

If a third party records management company is hired to dispose of records, a "Certificate of Destruction" is required.

SECTION 15. AUDITS

15.1 Internal Audits

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 Auditing, SOP No. CA-Q-S-004. The types and frequency of routine internal audits are described in Table 15-1. Special or ad hoc assessments may be conducted as needed under the direction of the QA staff.

Table 15-1. Types of Internal Audits and Frequency

Description	Performed by	Frequency
Quality Systems Audits	QA Department , QA approved designee, or Corporate QA	All areas of the laboratory annually
Method Audits	Joint responsibility: a) QA Manager or designee b) Technical Manager or Designee (Refer to CA-Q-S-004)	Methods Audits Frequency: 50% of methods annually
Special	QA Department or Designee	Surveillance or spot checks performed as needed, e.g., to confirm corrective actions from other audits.
Performance Testing	Analysts with QA oversight	Two successful per year for each TNI field of testing or as dictated by regulatory requirements

15.1.1 Annual Quality Systems Audit

An annual quality systems audit is required to ensure compliance to analytical methods and SOPs, TestAmerica's Data Integrity and Ethics Policies, TNI 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 for effectiveness & sustainability. The audit is divided into sections for each operating or support area of the lab, and each section is comprehensive for a given area. The area audits may be performed on a rotating schedule throughout the year to ensure adequate coverage of all areas. This schedule may change as situations in the laboratory warrant.

15.1.2 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, electronic audit miner programs (e.g., MintMiner and Chrom AuditMiner) are used to identify unusual manipulations of the data deserving closer scrutiny. QA technical audits will include all methods within a two-year period.

15.1.3 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 Department Manager (i.e., Technical Manager) or qualified designee at least every two years. It is also recommended that 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.

15.1.4 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.

15.1.5 Performance Testing

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, Non-potable Water, Soil and Hazardous Waste.

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.

15.2 External Audits

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. When requested, 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.

15.2.1 Confidential Business Information (CBI) Considerations

During on-site audits, 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 2009 TNI standards.

15.3 Audit Findings

Audit findings are documented using the corrective action process and database. 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 (i.e., Technical) Manager 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.

SECTION 16. MANAGEMENT REVIEWS

16.1 Quality Assurance Report

A comprehensive QA Report shall be prepared each month by the laboratory’s QA Department and forwarded to the Laboratory Director, 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.

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.

16.2 Annual Management Review

The senior lab management team (Laboratory Director, QA 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 goals, objectives and action items that feed into the laboratory planning system. 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 (Corporate SOP No. CA-Q-S-008 & Work Instruction No. CA-Q-WI-020) uses information generated during the preceding year to assess the “big picture” by ensuring that routine actions taken and reviewed on a monthly basis are not components of larger systematic concerns. The monthly review 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 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 management. 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.

16.3 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. CW-L-S-002) . 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 CEO, VP of Quality, Technical & Operations, General Managers and Quality Directors receive a monthly report from the Corporate Quality Director 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.

SECTION 17. PERSONNEL

17.1 Overview

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 organization chart in Figure 4-1.

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.

17.2 Education and Experience Requirements for Technical Personnel

The laboratory makes every effort to hire analytical staffs that possess 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. 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, Redox, etc.), or Titrimetric and Gravimetric Analyses	H.S. Diploma	On the job training (OJT)
GFAA, CVAA, FLAA, Single component or short list Chromatography (e.g., Fuels, 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 chromatography (e.g., Pesticides, PCB, Herbicides, 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

Specialty	Education	Experience
Department Managers (i.e,Technical Managers) - **General**	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
Department Managers (i.e,Technical Managers)– **Wet Chem** only (no advanced instrumentation)	Associates degree in an applied science or engineering or 2 years of college with 16 semester hours in chemistry	And 2 years relevant experience

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 (i.e., Technical) 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.

17.3 Training

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	All
Ethics – Comprehensive Refresher	Annually	All
Initial Demonstration of Capability (DOC)	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 19.

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 19).
- 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.

Further details of the laboratory's training program are described in the Laboratory Training SOP (TestAmerica Edison SOP No. ED-GEN-022).

17.4 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 (Policy No. CW-L-P-004) and an Ethics Statement. All initial and annual training is documented by signature on the signed Ethics Statement 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
- 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 18. ACCOMMODATIONS AND ENVIRONMENTAL CONDITIONS

18.1 Overview

The laboratory is a 42,000 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 preparation, volatile organic sample analysis, non-volatile organic sample analysis, inorganic sample analysis, and administrative functions.

18.2 Environment

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 and temperature levels in the laboratory (when appropriate).

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.

Environmental conditions of the facility housing the computer network and LIMS are regulated to protect against raw data loss.

18.3 Work Areas

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.

18.4 Floor Plan

A floor plan can be found in Appendix 1.

18.5 Building Security

Building keys are distributed to 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 19. TEST METHODS AND METHOD VALIDATION

19.1 Overview

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.

19.2 Standard Operating Procedures (SOPS)

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.

- All SOPs contain a revision number, effective date, and appropriate approval signatures. Controlled copies are available to all staff.
- Procedures for writing an SOP are incorporated by reference to TestAmerica's Corporate SOP entitled 'Writing a Standard Operating Procedure', No. CW-Q-S-002.
- 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.

19.3 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.

19.4 Selection of Methods

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), 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.

19.4.1 Sources of Methods

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.

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:

- *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*
- *Statement of Work for Inorganics & Organics 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/ 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.*
- *National Status and Trends Program, National Oceanographic and Atmospheric Administration, Volume I-IV, 1985-1994.*
- *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.

19.4.2 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. 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.

A demonstration of capability (reference TestAmerica Edison Training SOP No. ED-GEN-022) is performed whenever there is a change in instrument type (e.g., new instrumentation), method or personnel (e.g., analyst hasn't performed the test within the last 12 months).

The initial demonstration of capability must be thoroughly documented and approved by the Department Manager (i.e., Technical Manager) and QA Manager prior to independently analyzing client samples. All associated documentation must be retained in accordance with the laboratories archiving procedures.

The laboratory must have an approved SOP, demonstrate satisfactory performance, and conduct an MDL 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 quantitation limit (QL), must be at or above the lowest non-zero standard in the calibration curve and must be reliably determined. Project RLs are client specified reporting levels which may be higher than the QL. Results reported below the QL must be qualified as estimated values. Also see Section 19.6.1.3, Relationship of Limit of Detection (LOD) to Quantitation Limit (QL).
- 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: *Reporting Limit based on the low standard of the calibration curve.*

19.4.3 Initial Demonstration of Capability (IDOC) Procedures

19.4.3.1 The spiking standard used must be prepared independently from those used in instrument calibration.

19.4.3.2 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.

19.4.3.3 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 days).

19.4.3.4 Using all of the results, calculate the mean recovery in the appropriate reporting units and the standard deviations for each parameter of interest.

19.4.3.5 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.

19.4.3.6 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.

19.4.3.7 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 19.4.3.3 above.
- Beginning with 19.4.3.3 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 19.4.3.1 above.

Note: Results of successive LCS analyses can be used to fulfill the DOC requirement.

A certification statement (refer to Figure 19-1 as an example) shall be used to document the completion of each initial demonstration of capability. A copy of the certification 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.

19.5 Laboratory Developed Methods and Non-Standard Methods

Any new method developed by the laboratory must be fully defined in an SOP 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.

19.6 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.
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.

19.6.1 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.

19.6.1.1 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.

19.6.1.2 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.

19.6.1.3 Relationship of Limit of Detection (LOD) to the Quantitation Limit (QL)

An important characteristic of expression of sensitivity is the difference in the LOD and the QL. The LOD is the minimum level at which the presence of an analyte can be reliably concluded. The QL is the minimum concentration of analyte that can be quantitatively determined with acceptable 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 or LOD) and below the QL. 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 QL, 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.

19.6.1.4 Determination of Interferences

A determination that the method is free from interferences in a blank matrix is performed.

19.6.1.5 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 highest acceptable calibration concentration. The lower quantitation limit or QL cannot be lower than the lowest non-zero calibration level, and can be constrained by required levels of bias and precision.

19.6.1.6 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.

19.6.1.7 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.

19.6.1.8 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.

19.7 Method Detection Limits (MDL) / Limits of Detection (LOD)

Method detection limits (MDL) 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. MDL is also sometimes referred to as Limit of Detection (LOD). The MDL 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 MDL is determined for each analyte 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. Generally, 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. 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. [To allow for some flexibility, this low level standard may be analyzed every batch or every week or some other frequency rather than doing the study all at once. In addition, a larger number of data points may be used if the appropriate t-value multiplier is used]

Refer to the Corporate SOP No. CA-Q-S-006 for details on the laboratory's MDL process.

19.8 Instrument Detection Limits (IDL)

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.

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.

If IDL is > than the MDL, it may be used as the reported MDL.

19.9 Verification of Detection and Reporting Limits

Once an MDL is established, it must be verified, on each instrument, by analyzing a quality control sample (prepared as a sample) at no more than 3 times the calculated MDL for single analyte analyses (e.g. most wet chemistry methods, Atomic Absorption, etc.) and no more than 4 times the calculated MDL for multiple analyte methods (e.g. GC, GCMS, ICP, etc.). The analytes must be qualitatively identified. This verification 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 does not verify, then the lab will not report to the MDL, or redevelop their MDL or use the level where qualitative identification is established. MDLs must be verified at least annually.

When the laboratory establishes a quantitation limit, it must be initially verified by the analysis of a low level standard or QC sample at 1-2 times the reporting limit and annually thereafter. The annual requirement is waved for methods that have an annually verified MDL. The laboratory will comply with any regulatory requirements.

19.10 Retention Time Windows

Most organic analyses and some inorganic analyses use chromatography techniques for qualitative and quantitative determinations. For every chromatography analysis or as specific in the reference method, 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. 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.

19.11 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.

19.12 Estimation of Uncertainty of Measurement

19.12.1 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.

19.12.2 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.

19.12.3 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 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.

19.12.4 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.

19.12.5 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.

19.13 Sample Reanalysis Guidelines

Because there is a certain level of uncertainty with any analytical measurement, a sample re-preparation (where appropriate) and subsequent analysis (hereafter referred to as '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. Note: Client specific Contractual Terms & Conditions for reanalysis protocols may supersede 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 $\pm$ 1 reporting limit for samples $\leq$ 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 and reanalyze the sample a third time for confirmation if sufficient sample is available.

- 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 Laboratory Director if unsure.

19.14 Control of Data

The laboratory has policies and procedures in place to ensure the authenticity, integrity, and accuracy of the analytical data generated by the laboratory.

19.14.1 Computer and Electronic Data Related Requirements

The three basic objectives of our computer security procedures and policies are shown below. More detail is outlined in the TestAmerica Corporate IT SOPs and in TestAmerica Edison SOPs No. ED-GEN-001 (Data Management and Handling Procedures) and ED-GEN-002 (Document Control). 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.

19.14.1.1 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. Cells containing calculations must be lock-protected and controlled.
- Instrument hardware and software adjustments are safeguarded through maintenance logs, audit trails and controlled access.

19.14.1.2 Ensure Information Availability: Protection against loss of information or service is ensured 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.

19.14.1.3 Maintain Confidentiality: Ensure data confidentiality through physical access controls such as password protection or website access approval when electronically transmitting data.

19.14.2 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.

For manual data entry, e.g., Wet Chemistry, the data is reduced by the analyst and then verified by the Department (Technical) Manager 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 No. 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.

19.14.2.1 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. 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.

19.14.2.2 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**%.** Units are defined in each lab SOP.

19.14.2.3 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.

19.14.2.4 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. LIMS has a defined significant figure criterion for each analyte.

19.14.2.5 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 reviewing the quantitation report, and removing unrequested or poor spectrally-matched compounds. The analyst prints a copy of what has been entered to check for errors. This printout and the instrument's printout of calibrations, concentrations, retention times, chromatograms, and mass spectra, if applicable, are retained with

the data file. The data file is stored in a monthly folder on the instrument computer; periodically, this file is transferred to the server and, eventually, to a tape file.

19.14.3 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 12.
- 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 Managers/QA Manager at the facility. The QA Manager controls all worksheets following the procedures in Section 6.

19.14.4 Review / Verification Procedures

Review procedures are out lined in several SOPs (including but not limited to, TestAmerica Edison SOP Nos. ED-GEN-021: Data Review, ED-SPM-001:Login, and ED-RP-001:Reports Production) 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 general review concepts are discussed below, more specific information can be found in the SOPs.

19.14.4.1 The data review process at the laboratory starts at the Sample Control level. Sample Control personnel review chain-of-custody forms and input the sample information and required analyses into a computer LIMS. The Sample Control Supervisor reviews the transaction of the chain-of-custody forms and the inputted information. The Project Managers perform final review of the chain-of-custody forms and inputted information.

19.14.4.2 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 LIMS and add data qualifiers if applicable. To ensure data compliance, a different analyst or Department (Technical) Manager/Supervisor performs a second level of review. Second level review is accomplished by checking reported results against raw data and evaluating the results for accuracy. During the second level review, blank runs, QA/QC check results, initial and continuing calibration results, laboratory control samples, sample data, qualifiers and spike information are evaluated. Where calibration is not required on a daily basis, secondary review of the initial calibration results may be conducted at the time of calibration. Approximately 15% of all sample data from manual methods and from automated methods, all GC/MS spectra and all manual integrations are reviewed. Manual integrations are also electronically reviewed utilizing auditing software to help ensure compliance to ethics and manual integration policies. Issues that deem further review include the following:

- 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

19.14.4.3 Unacceptable analytical results may require reanalysis of the samples. Any problems are brought to the attention of the Laboratory Director, Project Manager, Quality Assurance Manager, Technical Manager, or Supervisor for further investigation. Corrective action is initiated whenever necessary.

19.14.4.4 The results are then entered or directly transferred into the computer database and a hard copy (or .pdf) is printed for the client.

19.14.4.5 As a final review prior to the release of the report, the Project Manager reviews the results for appropriateness and completeness. This review and approval ensures that client requirements have been met and that the final report has been properly completed. The process includes, but is not limited to, verifying that chemical relationships are evaluated, COC is followed, cover letters/ narratives are present, flags are appropriate, and project specific requirements are met.

19.14.4.6 Any project that requires a data package is subject to a tertiary data review for transcription errors and acceptable quality control requirements. The Project Manager then signs the final report. The accounting personnel also check the report for any clerical or invoicing errors. When complete, the report is sent out to the client.

19.14.5 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).

19.14.5.1 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.

19.14.5.2 Analysts shall not increase or decrease peak areas 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.

19.14.5.3 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.

19.14.5.4 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 19-1. Example - Demonstration of Capability Documentation

DEMONSTRATION OF CAPABILITIY (DOC)

Laboratory Name:_____
Laboratory Address:_____
Method:_____ Matrix:_____
Date:_____ Analyst(s):_____
Source of Analyte(s):_____

Analytical Results

Analyst	Conc. (Units)	Rep 1	Rep 2	Rep 3	Rep 4	Avg. % Recovery	% RSD

% RSD = Percent relative standard deviation = standard deviation divided by average % Recovery

Raw data reference: _____

Certification Statement:

We, the undersigned, certify that:
1. The cited test method has met Demonstration of Capability requirements.
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 method demonstration of capability are true, accurate, complete, and self-explanatory.
5. All raw data necessary to reconstruct and validate these analyses have been retained at the facility, and the associated information is well organized and available for review.
6.

_____ _____
Analyst Signature Date

_____ _____
Technical Director Signature Date

_____ _____
Quality Assurance Coordinator Signature Date

SECTION 20. EQUIPMENT and CALIBRATIONS

20.1 Overview

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. A list of laboratory instrumentation is presented in Table 20-1. The most current list of laboratory instrumentation can be found in TestAmerica Edison Work Instruction No. ED-WI-002 (Equipment Inventory).

Equipment is only operated by authorized and trained personnel. Manufacturers instructions for equipment use are readily accessible to all appropriate laboratory personnel.

20.2 Preventive Maintenance

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.

Routine preventive maintenance procedures and frequency, such as 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.

Table 20-2 lists examples of scheduled routine maintenance. It is the responsibility of each Technical Manager to ensure that instrument maintenance logs are kept for all equipment in his/her Department. Preventative maintenance procedures may also be 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.)

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.

- 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.
- 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.

- 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.

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.

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.

If an instrument is sent out for service or transferred to another facility, it must be recalibrated and verified (including new initial MDL study) prior to return to lab operations.

20.3 Support Equipment

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.

20.3.1 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.

20.3.2 pH, Conductivity, and Turbidity Meters

The pH meters used in the laboratory are accurate to ± 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.

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.

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.

20.3.3 Thermometers

All thermometers are calibrated on an annual basis with a NIST-traceable thermometer. IR thermometers, digital probes and thermocouples are calibrated quarterly.

The mercury 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 1 degree (0.5 degree or less increments are required for drinking water microbiological laboratories), and have ranges applicable to 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 No. ED-GEN-014 (Thermometer Calibration).

20.3.4 Refrigerators/Freezer Units, Waterbaths, Ovens and Incubators

The temperatures of all refrigerator units and freezers used for sample and standard storage are monitored each working day.

Ovens, waterbaths 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 > 0ºC and ≤ 6 ºC.

Specific temperature settings/ranges for other refrigerators, ovens waterbaths, and incubators can be found in method specific SOPs.

All of this information is documented in Daily Temperature Logbooks and method-specific logbooks.

20.3.5 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.

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. Refer to TestAmerica Edison SOP No. ED-GEN-011 (Calibration and Use of Lab Pipettes).

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.

20.3.6 Autoclaves

The laboratory utilizes autoclaves in the sample preparation step for certain mercury analysis procedures. These autoclaves have direct reading temperature and pressure gauges. These gauges are checked for accuracy on an annual basis.

20.3.7 Field Sampling Devices (Isco Auto Samplers)

Each Auto Sampler (ISCO) is assigned a unique identification number in order to keep track of the calibration. This number is also recorded on the sampling documentation.

The Auto Sampler is calibrated as needed based on manufacturers recommendations.

20.4 Instrument Calibrations

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 12).

Note: Instruments are calibrated initially and as needed after that and at least annually.

20.4.1 Calibration Standards

Calibration standards are prepared using the procedures indicated in the Reagents and Standards section of the determinative method SOP. If a reference method does not specify the number of calibration standards, a minimum of 3 calibration points (exception being ICP and ICP/MS methods) will be used.

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.

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 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 at least the same number of significant figures used to report the data) must be reported as having less certainty, e.g., defined qualifiers or flags (additional information may be included in the case narrative). The exception to these rules is ICP methods or other methods where the referenced method does not specify two or more standards.

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, such as air analysis where no other source or lot is available, a standard made by a different analyst at a different time or a different preparation would be considered a second source. This verification occurs immediately after the calibration curve has been analyzed, and before the analysis of any samples.

20.4.1.1 Calibration Verification

The calibration relationship established during the initial calibration must be verified initially and at least daily as specified in the laboratory method SOPs in accordance with the referenced analytical methods and in the 2009 TNI Standard. 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 verification 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 2009 TNI Std. EL-V1M4 Sec. 1.7.2.

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).

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.

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.

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).

If the results of a CCV are outside the established acceptance criteria and analysis of a second consecutive (and immediate) CCV fails to produce results within acceptance criteria, corrective action shall be performed. Once corrective actions have been completed & documented, the laboratory shall demonstrate acceptable instrument / method performance by analyzing two consecutive CCVs, or a new initial instrument calibration shall be performed.

Sample analyses and reporting of data may not occur or continue until the analytical system is calibrated or calibration verified. However, data associated with an unacceptable calibration verification may be fully useable under the following special conditions:

a). when the acceptance criteria for the CCV are exceeded high (i.e., high bias) and the associated samples within the batch are non-detects, then those non-detects may be reported with a footnote or case narrative explaining the high bias. Otherwise the samples affected by the unacceptable CCV shall be re-analyzed after a new calibration curve has been established, evaluated and accepted; or

b). when the acceptance criteria for the CCV 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 CCV shall be re-analyzed after a new calibration curve has been established, evaluated and accepted.

Samples reported by the 2 conditions identified above will be appropriately flagged.

20.4.1.2 Verification of Linear and Non-Linear Calibrations

Calibration verification for 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. (These calculations are available in the laboratory method SOPs. 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.

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 acepted.

- 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.

20.5 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.

20.6 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.

Table 20-1. Example: Instrumentation List

Example: Edison Laboratory Instrumentation List						
Instrument Type	**Manufacturer**	**Model**	**Purchase Date**	**Install Date**	**Autosampler**	**Method Performed**
METALS ICP	Thermo Jarrell Ash (2) S/N 356490	61E Trace	1998	Feb98	Yes	6010B, 200.7, CLP
	Thermo Jarrell Ash (3) S/N 493890	61E Trace	2000	Sep00	Yes	6010B, 200.7, CLP
	Thermo Jarrell Ash (4) S/N: ICP-20073407	ICAP 6500 Duo View	2007	Feb 09	Yes	6010B, 200.7, CLP
ICP-MS ICPMS1 Heat Exchanger Autosampler	Agilent Technologies 7500ce S/N JP51201560 PolyScience Agilent TechnologiesG1879B S/N G57335 Cetac S/N 120536A520	 G3272A 3370 ASX520	2006	May06	Yes	6020, 200.8
ICP-MS ICPMS2 Heat Exchanger Autosampler	 Agilent Technologies 7500ce S/N JP82802644 Agilent Technologies G1879B S/N 108500855 Cetac ASX-500 S/N US0808108A520	 G3272B 3370 G3286A	2010	June 2010	Yes	6020, 200.8
Mercury Analyzer	Leeman Labs (3) S/N HA-3010	Hydra AA	2003	Jan04	Yes	7471A, 7470, 245.1 CLP
	Leeman Labs (4) S/N HA-4008	Hydra AA	2004	Jun04	Yes	7471A, 7470, 245.1 CLP
Hotblock 1	Environmental Express Limited S/N 2772CEC1378	SC154	2003	2003	No	3050B, CLP
Hotblock 2	Environmental Express Limited S/N 2391CEC1273	SC154	2004	2004	No	3050B, CLP
Autoclave (Out of Service)	Steril-Matic S/N 95-2678	MEA 109-85-E	1996	1996	No	7471A
Hot Plate 1 (Out of Service)	Fischer Scientific S/N 1000132		Jan04	Jan04	No	200.7, 3010A, 3020A, CLP
Hot Plate 2 (Out of Service)	Fischer Scientific S/N 1000153		Oct04	Oct04	No	200.7, 3010A, 3020A, CLP
Hot Plate 3 (Out of Service)	Fischer Scientific S/N 1000168		Jul03	Jul03	No	200.7, 3010A, 3020A, CLP
Hot Plate 4 (Out of Service)	Fischer Scientific S/N 1000169		May05	May05	No	200.7, 3010A, 3020A, CLP
Hot Plate 5 (Out of Service)	Fischer Scientific S/N 1000170		Apr05	Apr05	No	200.7, 3010A, 3020A, CLP
Hot Plate 6 (Out of Service)	Fischer Scientific S/N 1000203		Dec04	Dec04	No	200.7, 3010A, 3020A, CLP
Hot Plate 7 (Out of Service)	Fischer Scientific S/N 1000210		Apr05	Apr05	No	200.7, 3010A, 3020A, CLP

Example: Edison Laboratory Instrumentation List

Instrument Type	Manufacturer	Model	Purchase Date	Install Date	Autosampler	Method Performed
Hot Plate 8 (Out of Service)	Fischer Scientific S/N 1000220		Jun05	Jun05	No	200.7, 3010A, 3020A, CLP
Hotblock 3	Environmental Express Limited S/N 4298CEC2048	SC150	2004	2004	No	200.7, 3010A, 200.8, CLP
Hotblock 4	Environmental Express Limited S/N 4507CEC2115	SC150	2006	2006	No	200.7, 3010A, 200.8, CLP
Hotblock 5	Environmental Express Limited S/N 4667CEC2183	SC150	2006	2006	No	200.7, 3010A, 200.8, CLP
Hotblock 6	Environmental Express Limited S/N 4667CEC2183	SC150	2006	2006	No	200.7, 3010A, 200.8, CLP
Hotblock 7	Environmental Express Limited S/N 2772CDC1378	SC150	2006	2006	No	200.7, 3010A, 200.8, CLP
Balance # 35	Acculab 18255989		2005	2005	No	3050B, CLP
Balance # 33	Ohaus F0461200521139		2001	2001	No	7471A
Autoclave	Steril-Matic S/N 201188	STME	2002	2002	No	7471A
GC/MS Semivolatiles (BNAMS1/GC) GC MS Tower Tray Controller	Hewlett-Packard S/N 3223A43511 S/N 3118A02442 S/N 3013A21967 S/N 3249A30680 S/N 3249A30674	 5971 7673	1986	1986	Yes	**Out of Service**
(BNAMS2/GC) GC MS Tower Tray Controller	Hewlett-Packard S/N 2618A07933 S/N 3234A04110 S/N 2704A08901 S/N 2718A08680 S/N 2607A02892	 5971 7673A	1986	1986	Yes	8270C, 625, CLP
(BNAMS3/GC) GC MS Tower Tray Controller	Hewlett-Packard S/N 3140A38366 S/N 3188A02926 S/N 3266A31274 S/N 3021A21499 S/N 3138A27180	 5971 7673	1986	1986	Yes	8270C, 625, CLP
(BNAMS4/GC) GC MS Tower Tray Controller	Hewlett-Packard S/N 3108A34490 S/N 3114A02077 S/N 2546A02861 S/N 2942A20598 S/N 2803A11211	 5971A 7673A	1986	1986	Yes	8270C, 625, CLP

Example: Edison Laboratory Instrumentation List

Instrument Type	Manufacturer	Model	Purchase Date	Install Date	Autosampler	Method Performed
(BNAMS5/GC)	Agilent Technologies		2007	2007	Yes	8270C, 625, CLP
GC	S/N CN10726100					
MS	S/N US35120328	5975C				
Tower	S/N CN72441261	7890A				
Tray	S/N CN40427800					
Controller	S/N CN40427800					
(BNAMS6/GC)	Hewlett-Packard		1990	1990	Yes	8270C, 625, CLP
GC	S/N 3336A54722					
MS	S/N 3234A04274	5971				
Tower	S/N 2843A13155	7673				
Tray	S/N 2933A11253					
Controller	S/N 3018A21811					
(BNAMS7/GC)	Hewlett-Packard		1990	1990	Yes	**Out of Service**
GC	S/N 3235A45833					
MS	S/N 3307A00368	5972				
Tower	S/N 2546602130	7673A				
Tray	S/N 2633A02968					
Controller	S/N 2511A01985					
(BNAMS8/GC)	Hewlett-Packard		1990	1990	Yes	**Out of Service**
GC	S/N 336A56444					
MS	S/N 3435A01857	5972				
Tower	S/N C11144007149	A0C-20i				
Tray	S/N C11154103496					
Controller	S/N 626059SA					
(BNAMS9/GC)	Agilent Technologies		2004	2004	Yes	8270C, 625, CLP
GC	S/N CN10349071					
MS	S/N US35120328	5973				
Tower	S/N CN35134357	7683				
Tray	S/N CN40427800					
Controller	S/N CN40427800					
(BNAMS10/GC)	Agilent Technologies		2004	2004	Yes	8270C, 625, CLP
GC	S/N CN10403063					
MS	S/N US35120373	5973				
Tower	S/N CN40334758	7683				
Tray	S/N CN40327770					
Controller	S/N CN40327770					
(BNAMS11/GC)	Agilent Technologies		2007	2007	Yes	8270C, 625, CLP
GC	S/N CN10727109					
MS	S/N US71236621	5975C				
Tower	S/N CN35134357	7890A				
Tray	S/N CN72441255					
Controller						

Example: Edison Laboratory Instrumentation List

Instrument Type	Manufacturer	Model	Purchase Date	Install Date	Autosampler	Method Performed
BNAGC2	Hewlett-Packard		1986	1986	Yes	**Out of Service**
GC	S/N 3336A55994	5890 II				
Tower 1	S/N 3004A20530	7673				
Tower 2	S/N 3613A21129					
Tray	S/N 3021A21938					
Controller	S/N 3244A30371					
BNAGC8	Hewlett-Packard		1986	1986	Yes	Screen
GC	S/N 3121A35833	5890				
Tower 1	S/N 2704805765	7673A				
Tray	S/N 3131A25914					
Controller	S/N 2921A03449					
Manifold			10/29/04	11/1/04	No	
Gases	Western Enterprise 28452	Innovator HBAC2-5-4				
GC/MS Volatiles					Yes	8260, 624, CLP, 524.2
	Agilent	5975	Feb06	Jul06		
VOAMS1	S/N US60532504					
	Agilent	6890N	Feb06	Jul06		
GC	S/N CN10606023					
	OI	4551A	Feb06	Jul06		
Autosampler	S/N D60345B194					
	OI	4660	Feb06	Jul06		
Concentrator	S/N D608466853					
	OI	SAM	Feb06	Jul06		
Spiker	S/N E610475713					
VOAMS2	Hewlett-Packard	5975C	2008	2008	Yes	8260, 624, CLP,
	S/N US80838709					
GC	Hewlett-Packard	7890A	2008	2008		
	S/N CN10813013					
Autosampler	EST	Archon 51	2008	2008		
	S/N 15264					
Concentrator	EST	Encon Evolution	2008	2008		
	S/N 104041408					
VOAMS3	Agilent	5973inert	Feb04	Aug04	Yes	8260B, 624, CLP, 524.2
	S/N US35120382					
GC	Agilent	6890N	Feb04	Aug04		
	S/N CN10406105					
Autosampler	EST	Centurion	Jun04	Aug04		
	S/N CENT140051304					
Concentrator A	EST	Encon	May04	Aug04		
	S/N 367060704					
Concentrator B	EST	Encon	May04	Aug04		
	S/N 368060704					

Example: Edison Laboratory Instrumentation List

Instrument Type	Manufacturer	Model	Purchase Date	Install Date	Autosampler	Method Performed
VOAMS4	Hewlett-Packard S/N US80838712	5975C	2008	2008	Yes	8260, 624, CLP,
GC	Hewlett-Packard S/N CN10813014	7890A	2008	2008		
Autosampler 1	OI S/N 15266	4552	2008	2008		
Concentrator	OI S/N D809466076	2008	2008	2008		
VOAMS5	Hewlett-Packard S/N 3234A04198	5971	1996	1996	Yes	8260B, 624, CLP, 524.2
GC	Hewlett-Packard S/N 3033A33368	5890 II	1996	1996		
Autosampler	Archon S/N 11957-696A	5100A	1996	1996		
Concentrator	OI S/N D310219	4560	1996	1996		
VOAMS6	Agilent VOAMS6 S/N US35120322	5973inert	Feb04	Apr04	Yes	624, 524.2, CLP
GC	Agilent S/N CN10406076	6890N	Feb04	Apr04		
Autosampler	OI S/N D54645B461	4551A	Nov05	Dec05		
Concentrator	OI S/N D548466579	4660	Nov05	Dec05		
Spiker	OI S/N C425475656	SAM	Jun04	Jul04		
VOAMS7	Agilent S/N US43110514	5973inert	Oct 04	Nov 04	Yes	624, 524.2,8260 CLP
GC	Agilent S/N CN10437064	6890N	Oct 04	May 06		
Autosampler	Teledyne Tekmar S/N US08121007	Solatek	Tekmar swap	May 08		
Concentrator	Teledyne Tekmar S/N US08007007	Stratum	Tekmar swap	May 08		
VOAMS8	Hewlett-Packard S/N 3118A02630	5971	1998	1998	Yes	8260B, 624, CLP, 524.2
GC	Hewlett-Packard S/N 3126A36935	5890 II	1998	1998		
Autosampler	EST Archon S/N 12206	5100A	1998	1998		
Concentrator	OI S/N I418460464	4560	1998	1998		

Example: Edison Laboratory Instrumentation List

Instrument Type	Manufacturer	Model	Purchase Date	Install Date	Autosampler	Method Performed
VOAMS9	Hewlett-Packard	5971	1998	1998	Yes	8260B, 624, CLP, 524.2
	S/N 3118A03332					
GC	Hewlett-Packard	5890 II	1998	1998		
	S/N 3203A40292					
Autosampler	EST Archon	5100A	1998	1998		
	S/N 12207					
Concentrator	OI	4560	1998	1998		
	S/N C302089					
VOAMS10	Hewlett-Packard	5972	1997 (Whippany acquisition)	July /2000 (In Edison)	Yes	8260, 624, CLP, 524.2
	S/N 3307A00392					
GC	Hewlett-Packard	5890	Unknown	1997 (In Whippany)		
	S/N 2728414257					
Autosampler	Teledyne Tekmar	Aquatek 70	Mar06			
	S/N 94312017			May 2008		
Concentrator	Tekmar	3000	1997			
	S/N 94087010					
VOAMS11	Agilent	5973N	Jun03	Jul03	Yes	8260B, 624, CLP, 524.2
	S/N US30965664					
GC	Agilent	6890N	Jun03	Jul03		
	S/N CN10324011					
Autosampler	EST Archon	5100A	Jun03	Jul03		
	S/N 13970					
Concentrator	EST	Encon	Jun03	Jul03		
	S/N 279061703					

Example: Edison Laboratory Instrumentation List

Instrument Type	Manufacturer	Model	Purchase Date	Install Date	Autosampler	Method Performed
VOAMS12	Agilent S/N US43110519	5973inert	Oct04	Nov04	Yes	8260, 624, CLP, 524.2
GC	Agilent S/N CN10439051	6890N	Oct04	Jun05		
Autosampler	EST S/N 14448	Archon 5100A	May05	Jun05		
Concentrator	EST S/N 430051605	Encon	May05	Jun05		
Turbo Pump Upgrade	Agilent S/N 56115832	Performance	Jun05	Jun05		
VOAMS13	Agilent S/N US43110517	5973inert	Oct04	Nov04	Yes	8260, 624, CLP, 524.2
GC	Agilent S/N CN10439052	6890N	Oct04	Jun05		
Autosampler	EST S/N 14449	Archon 5100A	May05	Jun05		
Concentrator	EST S/N 431051605	Encon	May05	Jun05		
Turbo Pump Upgrade	Agilent S/N 56069171	Performance	Jun05	Jun05		
Balance #22	Mettler 2115517886	PB1501	1997	1997	No	8260, 8015 GRO
Balance #50	Ohaus 1125573353	Explorer Pro	2006	2006	No	8260, 8015 GRO
Balance # 103	Denver Instruments 126008		2009	2009	No	8260, 8015. GRO
Oven Drying	Fisher Isotemp Oven 502N0045	13-246-516G	2/15/2005	3/3/2005	N0	
Oven Drying	Baxter 199012	DX-1	2000	2000	No	

Example: Edison Laboratory Instrumentation List

Instrument Type	Manufacturer	Model	Purchase Date	Install Date	Autosampler	Method Performed
GC Volatiles					Yes	8015B (GRO)
	Agilent	6890N	Mar06	May06		
GC1	S/N US10610006					
	OI	4552	Feb06	May06		
Autosampler	S/N 14608					
	OI	4660	Feb06	May06		
Concentrator	S/N D607466340					
	OI	4551A	Feb06	May06		
Autosampler	S/N D60745B342					
	OI	4660	Feb06	May06		
Concentrator	S/N D607466341					
	OI	SAM	Feb06	May06		
Spiker	S/N E610475713					
GC2	Hewlett-Packard	5890II	1993	1993	Yes	Screening/3810
	S/N 2921A23492					
Autosampler 1	Tekmar	7050	Jun04	Jul04		
	S/N US04156005					
Headspace 1	Tekmar	7000	Jun04	Jul04		
	S/N US04156003					
Autosampler 2	Tekmar	7050	Jun04	Jul04		
	S/N US04148014					
Headspace 2	Tekmar	7000	Jun04	Jul04		
	S/N US04163001					
GC3	Hewlett-Packard	5890II	1996	1996	Yes	8015B (GRO)
	S/N 3310A49242					
PID	OI	4430	1996	1996		
	S/N 91-I107					
Autosampler	Dynatech Archon	5100	1996	1996		
	S/N 11780-795					
Concnetrator	OI	4560	1996	1996		
	S/N J437460274					
SCREEN1/2 GC	Hewlett-Packard	5890 II	1989	1989	Yes	Screening
	S/N 2950A29246					
Autosampler 1	Tekmar	7050	1989	1989		
	S/N 91025014					
Headspace 1	Tekmar	7000	1989	1989		
	S/N 91163066					
Autosampler 2	Tekmar	7050	1989	1989		
	S/N 91168012					
Headspace 2	Tekmar	7000	1989	1989		
	S/N 90255003					

Example: Edison Laboratory Instrumentation List

Instrument Type	Manufacturer	Model	Purchase Date	Install Date	Autosampler	Method Performed
SCREEN3/4 GC	Hewlett-Packard S/N 2908A21857	5890	1998	1998	Yes	Screening/3810
Autosampler 1	Tekmar S/N 91346013	7050	1998	1998		
Headspace 1	Tekmar S/N 91339015	7000	1998	1998		
Autosampler 2	Tekmar S/N 90256011	7050	1998	1998		
Headspace 2	Tekmar S/N 91025010	7000	1998	1998		
H-Nu PID	H-nu Systems S/N 801023	PI101	1989	1989	No	Headspace Screening
Hood Ductless Fume	Air Science P41007	PurAir15	Oct04	Nov04	No	
GC Semivolatiles BNAGC1 GC Network Injector Module Tray	Agilent Technologies S/N US10248079 S/N CN24428026 S/N CN24322270	 6890N G2613A G2614A	2003	2005	Yes	NJDEP-OQA-QAM-025
BNAGC4 GC Network Injector Module 1 Injector Module 2 Tray	Agilent Technologies S/N US10610005 S/N CN43820808 S/N CN43820804 S/N CN43830663	 6890N G2913A G2914A G2614A	Feb06	Apr06	Yes	8015B DRO/Fingerprints QAM-025
BNAGC5 GC Tower Tray Controller	Hewlett-Packard S/N 2728A14513 S/N 2704A0854 S/N 2920A10887 S/N 01866	 5890 7673	1997	1997	Yes	8015B Alcohols
BNAGC6 GC Tower 1 Tower 2 Tray Controller	Hewlett-Packard S/N 3203A40054 S/N 3120A28315 S/N 3202A27987 S/N 3228A29094 S/N 3138A27180	 5890 II 7673	1997	1997	Yes	8015B Amines
BNAGC7 GC Tower 1 Tray Controller	Hewlett-Packard S/N 2443A03923 S/N 2546A02013 S/N 2718A05293 S/N 2929A15891	 5890 7673A	1999	1999	Yes	8015B Glycols

Example: Edison Laboratory Instrumentation List

Instrument Type	Manufacturer	Model	Purchase Date	Install Date	Autosampler	Method Performed
Pest/PCB						
	Hewlett-Packard		1992	1992	Yes	8081, CLP
GC1	S/N 2612A07669	5890A				
GC Mainframe	S/N CN22321930	G1513A				
Injector Module	S/N CN00005085	G1512A				
Controller	S/N US72101578	18596C				
Tray						
GC3	Hewlett-Packard		1992	1992	Yes	Herbicides
Series II GC	S/N 3223A42873	5890A				
Injector Module	S/N 3228A31372	18593B				
Controller	S/N 3049A23890	18594B				
Tray	S/N 3202A27453	18596B				
GC4	Hewlett-Packard		1997	1997	Yes	8081
Series II Plus GC	S/N 336A54563	5890A				
Injector Module	S/N 3013A22344	18593B				
Controller	S/N 3227A29129	18594B				
Tray	S/N 3624A42191	18596B				
GC5	Agilent Technologies		2002	2002	Yes	8081
GC Network	S/N US10226033	6890N				
Injector Module	S/N CN22025340	G2613A				
Tray	S/N CN21420543	G2614A				
GC6	Hewlett-Packard		1998	1998	Yes	608
GC Mainframe	S/N 2950A26642	5890A				
Injector Module	S/N CN13420438	G1513A				
Controller	S/N CN00004777	G1512A				
Tray	S/N US20407961	18596C				
GC7	Hewlett-Packard		1998	1998	Yes	8082
GC Mainframe	S/N 3029A29927	5890A				
Injector Module	S/N C11144007141	18593A				
Controller	S/N 626059	18594A				
Tray	S/N C11154103504	18596A				
GC8	Agilent Technologies		2000	2000	Yes	8082
GC Plus	S/N US00004463	6890				
Injector Module	S/N CN15221154	G1513A				
Controller	S/N 3631A05939	G1512A				
Tray	S/N 3050A23572	18596C				
GC9	Agilent Technologies		2001	2001	Yes	8082
GC Plus	S/N US00043694	6890				
Injector Module	S/N CN13420437	G1513A				
Controller	S/N CN00004150	G1512A				
Tray	S/N US13807350	18596C				

Example: Edison Laboratory Instrumentation List

Instrument Type	Manufacturer	Model	Purchase Date	Install Date	Autosampler	Method Performed
GC11 GC Plus Injector Module Controller Tray	Agilent Technologies S/N US00008746 S/N US64600228 S/N US72202100 S/N US22408138	 6890 G2513A G2512A 18596C	2003	2003	Yes	CLP
Wet Chemistry						
Spectrophotometer	HACH S/N 1205122	DR2800	2007	2007	No	365.2, 7196A, 353.2, 410.4
Spectrophotometer	HACH S/N 1204684	DR2800	2007	2007	No	365.2, 7196A, 353.2, 410.4
Spectrophotometer	HACH S/N 11204422	DR2800	2007	2007	No	7196A, USGS
Turbidimeter	HF Scientific S/N 200604033	Micro 100	2006	2006	No	180.1, SM 2130B
Ion Selective Meter	Orion S/N 006825	720A	1994	1994	No	350.1+ .2, 340.2, 150.1
Ion Selective Meter	Orion S/N 092904	720A+	2007	2007	No	350.1+ .2, 340.2, 150.1
pH Meter	Orion S/N 010005	320	2002	2002	No	Cr6+
pH Meter	Orion S/N 009986	320	2002	2002	No	350.1/4500 NH3 H
pH Meter	Orion 320 S/N 016995	320	2002	2002	No	TCLP (1311)
pH meter	Orion 320 S/N 017414	320		2009	No	4500-H B
Oven	VWR S/N 0402001	1320	2001	2001	No	2540C
Oven	VWR	1300U	2001	2001	No	2540C
Oven	VWR	1305U	2001	2001	No	2540B
Oven	Fisher	230G	1997	1997	No	2540B, 2540D
Oven (Muffle Furnace)	Fisher S/N 901N002	550-14	2002	2002	No	160.4
Oven drying	VWR	1320	2001	2001	No	
Balance #27	A&D 12315883	HR-200	2005	2005	No	Gen. chem.
Balance #29	A&D 12315872	HR-200	2005	2005	No	160.1, 160.2
Balance #26	Sartorius 3503054	1712MP8	2003	2003	No	Gen. chem.
Balance #51	Ohaus 7125010794	Scout Pro	2006	2006	No	1311 (TCLP), 3060A
Balance #100	Mettler 122423439		2006	2006	No	Lloyd Kahn (TOC)
Balance # 101	Denver Instrument 126009		2009	4/16/2009	No	Gen. chem.
Water Bath	Precision S/N 9302-112	50	1995	1995	No	7196A
Water Bath	Precision S/N 9305-024	50	1995	1995	No	7196A
Water System (Log-in)	Millipore S/N 07348-C		1990	1990	No	

Example: Edison Laboratory Instrumentation List

Instrument Type	Manufacturer	Model	Purchase Date	Install Date	Autosampler	Method Performed
Water System (Extr. room)	Barnstead S/N 1191020210415	D11911	1995	1995	No	
FTIR	Perkin Elmer S/N 139038	1600	1991	1991	No	418.1
Printer	Epson S/N 61P107612	FX-870	2003	2003	No	418.1
Fixed IR	Buck Scientific S/N 1026	404	2004	2004	No	418.1
COD reactor	HACH S/N 980300017418	45600	2007	2007	No	410.4, 5220D
COD reactor	HACH S/N 900402268	45600	2007	2007	No	410.4, 5220D
COD reactor	HACH S/N 1202323	DRB 200	2007	2007	No	410.4, 5220D
COD reactor	HACH S/N 1209887	DRB 200	2007	2007	No	410.4, 5220D
Auto-analyzer	Lachat S/N A83000	QUICKEM 8000	1997	1997	Yes	335.3, 420.2, 353.2, 351.2, 350.1+ .2
Auto-analyzer	Lachat S/N 8300-1658	8000 Series	2000	2000	Yes	335.3, 350.1+ .2
TOC Soil Analyzer (2)	Thermo Electron Corp. S/N 20034945	Flash EA 1112 Series	2004	2004	Yes	Lloyd Kahn's method
Printer	Epson S/N 41NE28676	LQ570	1997	1997	No	415.1
TOC Analyzer	Shimadzu S/N H51104335164	TOC-VCSH	2006	2006	Yes	Lloyd Kahn's method, 415.1, 9060, 5310B
Autosampler	Shimadzu S/N H52104301656SA	ASI-V	2006	2006	Yes	415.1, 5310B, 9060
Solid Sample Module	Shimadzu S/N H52504300040NK	SSM-500A	2006	2006	Yes	Lloyd Kahn's method
BOD Meter	YSI S/N 97S0534AE	5000	1998	1998	No	405.1
Incubator	GCA Precision Scientific		1998	1998	No	405.1
Hot Plate	Fischer Scientific S/N 103N0071		2001	2001	No	365.2
Hot Plate	Corning S/N 370301092774	PC-400	2007	2007	No	1311
Hot Plate	Fischer Scientific S/N 390502148495	PC-420	2007	2007	No	Lloyd Khan Method
Hot Plate	Fischer Scientific S/N 220897070707	PC-620	2007	2007	No	351.2
Conductivity Meter	Fischer Scientific S/N AB 81209007	Accumetab30	2002	2002	No	120.1, 9050A
Vortex mixer	Thermolyne S/N 632000855604	M63215	2002	2002	No	351.2
Dishwasher	Miele Professional S/N 208479	G7783CD	2003	2003	No	Glassware
Easy-Dist Distillation	Westco S/N 1095		2003	2003	No	350.1+ .2, 420.2, 9066
Easy-Dist Distillation	Westco S/N J097		2003	2003	No	335.3, 9012A & B
Easy-Dist Distillation	Westco S/N 1063		2007	2007	No	350.1+.2, 420.2, 353.3, 9012A&B
Easy-Dist Distillation	Westco S/N 1110		2007	2007	No	353.3, 420.2, 9066
Discreet Analyzer (1)	Konelab S/N S2019177	20	2003	2003	Yes	Automated Wet Chem

Example: Edison Laboratory Instrumentation List

Instrument Type	Manufacturer	Model	Purchase Date	Install Date	Autosampler	Method Performed
Discreet Analyzer (2)	Konelab S/N 2519236	20	2003	2003	Yes	Automated Wet Chem
Dell Computer	Dell S/N 246175		2003	2003	No	Automated Wet Chem (Konelab)
BOD Aerator	Thomas Scientific S/N 1187	DOA-P104d-AA	1998	1998	No	405.1
BOD Plus Assay Liquid Handler DO meter YSI 52	Mantech Assoc., Inc. S/N 27OC3XB215 S/N O3C0812 AM	 221 & 222 52CE	2003	2003	Yes	405.1
PC-Titration Plus Autotitrator Interface Titra-Rinse 1 Titra-Rinse 2 Buret Module 1 Buret Module 2 Titration Module	Mantech Assoc., Inc S/N MS-0H4-373 S/N MS-0G4-198 S/N MS-0G4-200 S/N MS-0H4-627 S/N MS-0H4-625 S/N MS-0B5-657	 PC-1000-102/4 PC-1000-408 PC-1000-408 PC-1104-00 PC-1104-00 PC-1300-475	2004	2004	Yes	310.1, 2320B – Alkalinity 2320B – Carbonate, Bicarbonate 4500 CO2D – Carbon Dioxide 130.2, 2340C – Hardness
Ion Chromatograph Pump #1 Pump #2 Conduct. Detector Injector & Oven 2-Ch Interface Liq. Handling #1 Liq. Handling #2 Dil. Autosampler	Metrohm Peak, Inc. S/N 04187 S/N 04197 S/N 03181 S/N 04147 S/N 04184 S/N 04154 S/N 04118 S/N 03198	 818 818 819 820 830 833 833 838	May05	May05	Yes	7199
Ion chromatograph Pump #1 Pump #2 UV-VIS Detector IC Interface Separation Center Sample Processor	Metrohm Peak, Inc. SS4818011006190 SS1818011003192 SS1153001010101 SS1830002003180 SS1820023003168 SS1838001009171	 818 818 1010 (Bischoff) 830 820 838	Feb 2010	Feb 2010	Yes	7199
Filter pump	Emerson S/N SA55-NXGTB 4142		1997		No	Sample Filtering
Filter pump	Emerson S/N G8ECX	SA55JXgtd-4144	2002	2002	No	Sample Filtering
Redox meter	VWR S/N 001149	8005	1997	1997	No	SM2580
Rotator 1	AP & R Machine & Tool S/N 222307		2003	2003	No	600/8000/CLP
Rotator 2	AP & R Machine & Tool S/N 222306		2003	2003	No	600/8000/CLP
Rotator 3	AP & R Machine & Tool S/N 222305		2003	2003	No	600/8000/CLP
Rotator 4	AP & R Machine & Tool S/N 222304		2003	2003	No	600/8000/CLP
Rotator 5	AP & R Machine & Tool S/N 222303		2003	2003	No	600/8000/CLP

Example: Edison Laboratory Instrumentation List

Instrument Type	Manufacturer	Model	Purchase Date	Install Date	Autosampler	Method Performed
Rotator 6	AP & R Machine & Tool S/N 222302		2003	2003	No	600/8000/CLP
TCLP Extraction1 Apparatus/Timer included	Assoc. Design and Mfg. Co. S/N 1352	3740-12 BRE	1997	1997	No	1311 TCLP, ZHE
TCLP Extraction2 Apparatus/Timer included	Assoc. Design and Mfg. Co. S/N 1053	3740-12 BRE	1997	1997	No	1311 TCLP, ZHE
TCLP Extraction3 Apparatus/Timer included	Assoc. Design and Mfg. Co. S/N 1249	3740-12 BRE	1997	1997	No	1311 TCLP, ZHE
TCLP Extraction4 Apparatus/Timer included	Environmental Express Limited S/N 3384-12-473	LE 1002	May05	May05	No	1311 TCLP, ZHE
TCLP Extraction5 Apparatus/Timer included	Environmental Express Limited S/N 3384-12-472	LE 1002	May05	May05	No	1311 TCLP, ZHE
TCLP Extraction6 Apparatus/Timer included	Assoc. Design and Mfg. Co. S/N 2125	3740-12 BREII	Jul06	Sep06	No	1311 TCLP, ZHE
TCLP Extraction7 Apparatus/Timer included	Assoc. Design and Mfg. Co. S/N 2126	3740-12 BREII	Jul06	Sep06	No	1311 TCLP, ZHE
SAMPLE LOGIN						
Balance #13	Satorius S/N 50709085	LC421	1995	1995	No	composite
Balance #104	Denver Instruments S/N 126006		2009	2009	No	% Solids
Isotemp Oven 1	Fisher S/N 410B01117	637G	Mar05	Mar05	No	%Solids
Isotemp Oven 2	Fisher S/N 505N0063	637G	Jun05	Jun05	No	%Solids
ORGANIC EXTRACTIONS						
N-EVAP #1	Organomation S/N 51004	8125	2004	2004	No	600/8000/CLP
N-EVAP #2	Organomation S/N 10253	N-EVAP 112	1990	1990	No	600/8000/CLP
Water Bath #1	Fisher Scientific S/N 605021280	15-491	2005	2005	No	600/8000/CLP
Water Bath #2	Fisher Scientific S/N (204272)	15-491	2007	2007	No	600/8000/CLP
Sonicator #0 (Controller)	Tekmar SN 19606F (Asset # 36339)	TM600-2				
Sonicator #1 (Controller)	Sonic & Material, Inc. S/N 38701H (Asset #36362)	VCX 500	2006	2006	No	8000/CLP
Sonicator #2 (Controller)	Sonic & Material, Inc. S/N 38710H (Asset #36361)	VCX 500	2006	2006	No	8000/CLP

Example: Edison Laboratory Instrumentation List

Instrument Type	Manufacturer	Model	Purchase Date	Install Date	Autosampler	Method Performed
Sonicator Horn #3	Tekmar S/N 29281	CV17	1990	1990	No	8000/CLP
Sonicator Horn #4	Tekmar S/N illegible	CV17	1990	1990	No	8000/CLP
Sonicator #5 (Controller)	Sonic & Material, Inc. S/N 41748 M+ (Asset # 36363)	VCX 500	2004	2004	No	8000/CLP
Sonicator #6 (Controller)	Sonic & Material, Inc. S/N 41755 M+(Asset # 36364)	VCX 500	2004	2004	No	8000/CLP
Sonicator Horn # 7	Sonic & Material, Inc. S/N 3353027	CV33				
Sonicator Horn # 8	Sonic & Material, Inc. S/N 3353028	CV33				
Sonicator Horn # 9	Sonic & Material, Inc. S/N 3342405	CV33				
Sonicator Horn # 10	Sonic & Material, Inc. S/N 3342408	CV33				
Muffle Furnace #1	Thermolyne S/N 40800875	F6010	1990	1990	No	600/8000/CLP
Muffle Furnace #2	Thermolyne S/N (warn out)	F6028C	1990	1990	No	600/8000/CLP
Large Muffle Furnace	Wilt Industries S/N 041213	210	2001	2001	No	600/8000/CLP
Dishwasher #1	Miele Professional S/N 53075564	G7783CD	2003	2003	No	608/8000/CLP
Dishwasher #2	Miele Professional S/N 53075571	G7783CD	2003	2003	No	608/8000/CLP
Vacuum Pump #1	Emerson electric MLD S/N UNL231171	5KH36KN90HX	1990	1990	No	600/8000/CLP
Vortex	Scientific Industries S/N 2-318564	6560	1995	1995	No	600/8000/CLP
Electric Mixer	Barnstead/Thermolyne S/N 125404091646		1995	1995	No	600/8000/CLP
Mini Hotplate/Stir	VWR Scientific S/N 33918-604	220	1995	1995	No	600/8000/CLP
Centrifuge #1	Sigma S/N 78646	2-5	2001	2001	No	600/8000/CLP
Centrifuge #2	Sigma S/N 78647	2-5	2001	2001	No	600/8000/CLP
Centrifuge #3 (Out of Service)	Sigma S/N 80226	2-5	2001	2001	No	600/8000/CLP
Balance # 60	Ohaus S/N 7125471186	Scout Pro	2007	2007	No	600/8000/CLP
Balance #28	A&D S/N 12315879	HR-200	2005	2005	No	600/8000/CLP

Example: Edison Laboratory Instrumentation List

Instrument Type	Manufacturer	Model	Purchase Date	Install Date	Autosampler	Method Performed
Balance #30	A&D S/N 12315880	HR-200	2005	2005	No	600/8000/CLP
Soxtherm 1 Controller Chiller	OI Analytical S/N 4012358 S/N 4012351 S/N 10200022	Type 07-5101	2002	2002	No	8000
Soxtherm 2 Controller Chiller	OI Analytical S/N 4010018 S/N 4010088 S/N 10200022	Type 07-5101	2002	2002	No	8000
Soxtherm 3 Controller Chiller	OI Analytical S/N 4012359 S/N 4002805 S/N 10365037	Type 07-5101	2002	2002	No	8000
Soxtherm 4 Controller Chiller	OI Analytical S/N 429023 S/N 4022012 S/N 101365037	Type 07-5101	2002	2002	No	8000
Soxtherm 5 Controller Chiller	Gerhardt S/N 4073032 S/N 4051753 S/N 107344070 (Thermo)	SOX 416	2007	2007	No	8000
Soxtherm 6 Controller Chiller	Gerhardt S/N 4073033 S/N 4051753 S/N 107344070 (Thermo)	SOX 416	2007	2007	No	8000
Soxtherm 7 Controller Chiller	Gerhardt S/N 4073030 S/N 4051753 S/N 107344069 (Thermo)	SOX 416	2007	2007	No	8000
Soxtherm 8 Controller Chiller	Gerhardt S/N 4073031 S/N 4051753 S/N 107344069 (Thermo)	SOX 416	2007	2007	No	8000
Soxtherm 9 Controller Chiller	OI Analytical S/N 4012357 S/N 4012354 S/N 101361126	Type 07-5101	2003	2003	No	8000

Example: Edison Laboratory Instrumentation List

Instrument Type	Manufacturer	Model	Purchase Date	Install Date	Autosampler	Method Performed
Soxtherm10 Controller Chiller	OI Analytical S/N 4010016 S/N 4012353 S/N 101361126	Type 07-5101	2003	2003	No	8000
Soxtherm11 Controller Chiller	OI Analytical S/N 4012356 S/N 480017 S/N 102002024	Type 07-5101	2005	2005	No	8000
Soxtherm12 Controller Chiller	OI Analytical S/N 4033530 S/N 401812 S/N 102002024	Type 07-5101	2005	2005	No	8000
Soxtherm13 Controller Chiller	Gerhardt S/N 4031667 S/N 4051747 S/N 101361121	SOX416 1177PD	2006	2006	No	8000
Soxtherm 14	Gerhardt S/N 4031666 S/N 4051747 S/N 101361121	SOX416	2006	2006	No	8000
Soxtherm 15	Gerhardt S/N 4051583 S/N 4051747 S/N 10650017 (VWR)	SOX416	2006	2006	No	8000
Soxtherm 16	Gerhardt S/N 4051582 S/N 4051747 S/N 10650017 (VWR)	SOX416	2006	2006	No	8000
Wrist Action Shaker 1	Burrell S/N	75	2003	2003	No	8151
Wrist Action Shaker 2	Labline S/N 12910443	3589	2003	2003	No	8151
Field Services pH/Temp meter	Thermo Orion 15035	250A+	2000	2000	No	pH, Temperature
Conductivity meter	HACH 21000005660	Sension 5	2002	2002	No	Conductivity
DO meter	HACH 0200001321	Sension 6	2002	2002	No	Dissolved Oxygen
DO meter	HACH 001200002352	Sension 6	2000	2000	No	Dissolved Oxygen
Turbidity meter	La Motte 0119-0997	2020	1998	1998	No	Turbidity
Turbidity meter	La Motte 3897-5102	2020	2002	2002	No	Turbidity

Example: Edison Laboratory Instrumentation List

Instrument Type	Manufacturer	Model	Purchase Date	Install Date	Autosampler	Method Performed
Turbidity meter	LaMotte 3649-3802	2020	2002	2002	No	Turbidity
pH/ORP meter	Cole Parmer 643409	05669-20			No	pH, Oxidation reduction
pH/ORP meter	HACH 31100003358	Sension 1	2005	2005	No	pH, Oxidation reduction
Cond./Salinity/ TDS meter	HACH 30500006215	Sension 5			No	Conductivity, Salinity, TDS
pH/ ORP meter	HACH 050400020239	Sension 1	2005	2005	No	pH, Oxidation reduction
pH/ ORP meter	HACH 050400022762	Sension 1	2005	2005	No	pH, Oxidation reduction
Cond./Salinity/ TDS meter	HACH 050300013668	Sension 5	2005	2005	No	Conductivity, Salinity, TDS
Cond./Salinity/ TDS meter	YSI 93L12159	33			No	Conductivity, Salinity, TDS
Turbidity meter	LaMotte ME 10036	2020e	2005	2005	No	Turbidity
Turbidity meter	LaMotte ME 10117	2020e	2005	2005	No	Turbidity
Cond./Salinity/ TDS meter	HACH 050506C50148	Sension 5	2005	2005	No	Conductivity, Salinity, TDS
DO meter	HACH 050500C60212	Sension 6	2005	2005	No	Dissolved oxygen
DO meter	HACH 050500C60066	Sension 6	2005	2005	No	Dissolved oxygen
pH/ ORP meter	HACH 050600C10445	Sension 1	2005	2005	No	pH, Oxidation reduction
pH/ ORP meter	HACH 4030004162	Sension 1	2005	2005	No	pH, Oxidation reduction
DO meter	Hach 040800001267		2006	2006	No	Dissolved Oxygen
Conductivity meter	Hach 050100002708		2006	2006	No	Conductivity
DO meter	Hach 040700001191		2006	2006	No	Dissolved Oxygen
pH/ mV meter	Hach 040200003831		2006	2006	No	pH, mV
Conductivity meter	Hach 050100002707		2006	2006	No	Conductivity
DO meter	Hach 030500007618		2006	2006	No	Dissolved Oxygen
pH/ mV	Hach 041200004666		2006	2006	No	pH, mV
Turbidity meter	LaMotte 4969-1604		2006	2006	No	Turbidity
Turbidity meter	LaMotte 4943-1604		2006	2006	No	Turbidity
Turbidity meter	LaMotte 1909-2900		2006	2006	No	Turbidity
pH/mV meter	Hach 041200002902		2006	2006	No	pH, mV
pH/mV meter E-019	Hach 41200002933	Sension 1	2006	2006	No	pH, mV
Conductivity meter E-027	Hach 050500C50193	Sension 5	2006	2006	No	Conductivity
pH meter E-028	Hach 040800010007	Sension 1	2006	2006	No	pH meter

Example: Edison Laboratory Instrumentation List

Instrument Type	Manufacturer	Model	Purchase Date	Install Date	Autosampler	Method Performed
pH/mV meter M-039	Hach 0804C410063	Sension 1				pH/ORP
pH/mV meter M-034	Hach 06070C710134	Sension 1	Oct06	Oct06	No	pH/ORP
Conductivity meter M-028	Hach 050500C50288	Sension 5	Aug05	Aug05	No	Conductivity
DO meter M-032	Hach 05070C360249	Sension 6	Nov06	Nov06	No	DO
pH/mV meter M-036	Hach 07080C710259	Sension 1	Oct07	Oct07	No	pH/ORP
pH/mV meter M-030	Hach 050600C10468	Sension 1	Aug05	Aug05	No	pH/ORP
pH/mV meter M-037	Hach 08020c110145	Sension 1	Mar08	Mar08	No	pH/ORP
DO meter E-030	Hach 07120C260018	Sension 6	2008	2008	No	DO
pH E-031	Thermo Orion 018168	Model 230			No	pH
pH/ORP E-029	Hach 07070C610178	Sension1	2008	2008	No	pH/ORP
DO E-032	YSI 01F0708AA	55/25 FT			No	DO
pH E-033	Thermo Orion 017788	Model 230A			No	pH
pH E-034	Thermo Orion 017630	Model 230A			No	pH
Chlorine meter CL-007	Hach 040200011290	Pocket Colorimeter II	2006	2006	No	330.5, SM 18th 4500 Cl G
Chlorine meter CL-002	Hach 020100174404	Pocket Colorimeter	2006	2006	No	330.5, SM 18th 4500 Cl G
Chlorine meter CL-003	Hach 040200011345	Pocket Colorimeter II	2006	2006	No	330.5, SM 18th 4500 Cl G
Chlorine meter CL-004	Hach 961200102549	Pocket Colorimeter	2006	2006	No	330.5, SM 18th 4500 Cl G
Chlorine meter CL-006	Hach 030400034505	Pocket Colorimeter	2005			
Chlorine meter CL-005	Hach 020100174252	Pocket Colorimeter	2006			
Chlorine meter CL-008	Hach 4796-4900	Colorimeter 1200				
Colorimeter M-040	Hach 041050031426	48450-60 DR/850			No	
Water level meter	Solonist S/N 37993		Jan05	Feb05	No	
Water level meter	Solonist S/N 37995		Jan05	Feb05	No	
Water level meter	Solonist S/N 42807		Jan06	Jan06	No	
Water level meter	Fisher				No	
PID meter	RAE Systems S/N 110-010953	PGM-7600	May05	May05	No	
PID meter	RAE Systems S/N 110-010984	Mini RAE 2000	May05	May05	No	
PID meter	RAE Systems S/N 110-01094	Mini Rae 2000	May05	May05	No	
PID meter	RAE Systems S/N 103958	Plus Classic	Jan05	Jan05	No	

Example: Edison Laboratory Instrumentation List

Instrument Type	Manufacturer	Model	Purchase Date	Install Date	Autosampler	Method Performed
PID meter	PE Photovac S/N DQGD302	2020			No	
Comp sampler	ISCO S/N 205C01376	603704001-3700	May05	May05	Yes	
Comp sampler	ISCO S/N 205C01380	603704001-3700	May05	May05	Yes	
Comp sampler	ISCO S/N 204G00984	3700			Yes	
Comp sampler	ISCO S/N 05248-001	2700			Yes	
Comp sampler	ISCO	2700			Yes	
Comp sampler	ISCO	2700			Yes	
Comp sampler	ISCO	2700			Yes	
Submersible pump	Grundfos S/N 05141-8349	MP1 / 1A106003	May05	May05	No	
Submersible pump	Grundfos S/N 05141-8361	MP1 / 1A106003	May05	May05	No	
Submersible pump	Grundfos S/N 0621-0014	A1A106003P1	Jul06	Jul06	No	
Submersible pump	Grundfos S/N 06029591				No	
Submersible pump	Grundfos S/N 98490294				No	
Submersible pump	Grundfos				No	
Submersible pump	Grundfos				No	
Submersible pump	Grundfos				No	
Submersible pump	Proactive S/N 1371	SS Monsoon	July06	Jul06	No	
Pump control box	Grundfos S/N H0412210120	91126028	May05	May05	No	
Pump control box	Grundfos S/N H0412210120	91126028	May05	May05	No	
Pump control box	Grundfos S/N P1940304254		May05	May05	No	
Pump control box	Grundfos S/N 203831		May05	May05	No	
Pump control box	Grundfos S/N H0303130012		May05	May05	No	
Pump control box	Grundfos S/N 9517		May05	May05	No	
Pump control box	Grundfos		May05	May05	No	
Pump control box	ProActive	Low-flow with power booster	Jul06	Jul06	No	
Trash pump	North Star S/N E06	10633	2007	2007	No	
Generator	Honda S/N EB-3000C	EZGP-1145763	May05	May05	No	
Generator	Honda S/N EB-3000C	EZGP-1151238	Jun05	Jun05	No	
Generator	Honda S/N EZGL1002930	EB-3000C	2005	2005	No	
Generator	Honda				No	

Example: Edison Laboratory Instrumentation List

Instrument Type	Manufacturer	Model	Purchase Date	Install Date	Autosampler	Method Performed
Generator	Honda				No	
Control Pack	QED S/N MP15-1300	MP-15	May05	May05	No	
Control Pack	QED S/N MP15-1297	MP-15	May05	May05	No	
Control Pack	QED S/N MP15-1298	MP-15	May05	May05	No	
Control Pack	QED S/N MP15-1299	MP-15	May05	May05	No	
Control Pack	QED	MP-15	May05	May05	No	
Control Pack	QED	MP-15	May05	May05	No	
Control Pack	QED	MP-15	May05	May05	No	
Control Pack	QED	MP-15	May05	May05	No	
Control Pack	QED	MP-15	May05	May05	No	
Bladder Pump	QED S/N 10993	MP-SPK-4P	May05	May05	No	
Bladder Pump	QED S/N 10997	MP-SPK-4P	May05	May05	No	
Bladder Pump	QED S/N 10995	MP-SPK-4P	May05	May05	No	
Bladder Pump	QED S/N 10996	MP-SPK-4P	May05	May05	No	
Bladder Pump	QED S/N 11191	MP-SPK-4P	May05	May05	No	
Bladder Pump	QED S/N 11192	MP-SPK-4P	May05	May05	No	
Bladder Pump	QED 11512	MP-SPK-4P	May05	May05	No	
Bladder Pump	QED 10948	MP-SPK-4P	May05	May05	No	
Bladder Pump	QED 10949	MP-SPK-4P	May05	May05	No	
Bladder Pump	QED	MP-SPK-4P	May05	May05	No	
Bladder Pump	QED	MP-SPK-4P			No	
Bladder Pump	QED	MP-SPK-4P			No	
Peristaltic Pump	Solonist S/N 002562	410			No	
Peristaltic Pump	Solonist S/N 002071	410			No	
Peristaltic Pump	Solonist S/N 001979	410			No	
Peristaltic Pump	Solonist S/N 002642	410			No	
Peristaltic Pump	ISCO	Accuwell 150 portable pump			No	
Peristaltic Pump	ISCO	Accuwell 150 portable pump			No	
Peristaltic Pump	ISCO	Accuwell 150 portable pump			No	
Peristaltic Pump	ISCO	Accuwell 150 portable pump			No	

Example: Edison Laboratory Instrumentation List

Instrument Type	Manufacturer	Model	Purchase Date	Install Date	Autosampler	Method Performed
Peristaltic Pump	ISCO	Accuwell 150 portable pump			No	
Peristaltic Pump	ISCO	Accuwell 150 portable pump			No	
Centrifugal Pump	Teel S/N 3021	2P110B			No	
Centrifugal Pump	Teel S/N 0036	2P110B			No	
Centrifugal Pump	Teel S/N 0034	2P110B			No	
Centrifugal Pump	Teel S/N 1962	2P110B			No	
Centrifugal Pump	Teel	2P110B			No	
Compressor	Coleman / Honda S/N D02812339	CT5090412	Jun05	Jun05	No	
Compressor	Honda/Campbell Hausfeld S/N VT697203AJ				No	
Multi-probe meter YSI-1	YSI S/N 06F1362AC	556 MPS	Jul06	Jul06	No	
GPS	Ashtech 10564	110454-01			No	
Oil/Water Interface probe	Testwell					
Oil/Water Interface probe	Testwell					
Oil/Water interface Probe	Solonist 122-008699-1	122	Sept07	Sept07	No	
Oil/Water interface probe	Solonist S/N 122 007364-1		Aug06	Aug06	No	

Table 21-2. Example: Schedule of Routine Maintenance

Instrument	Procedure	Frequency
Leeman Mercury Analyzer	Check tubing for wear Fill rinse tank with 10% HCl Change dryer tube Fill reductant bottle with 10% Stannous Chloride	Daily Daily As needed Daily
ICP	Check pump tubing Check liquid argon supply Check fluid level in waste container Check filters Clean or replace filters Check torch Check sample spray chamber for debris Clean and align nebulizer Check entrance slit for debris Change printer ribbon Replace pump tubing	Daily Daily Daily Weekly As required Daily Monthly Monthly Monthly As required As required
ICP MS	Change pump tubing Clean torch Check / clean nebulizer Clean cones Check air filters Check multiplier voltages & do cross calibration Replace sample uptake tubing Check rotary pump oil Check oil mist filters Check chiller water level	Weekly or As required Weekly or As required Weekly or As required Weekly or As required Weekly or As required Weekly or As required Weekly or As required Weekly or As required Monthly Monthly
UV-Vis Spectrophotometer	Clean ambient flow cell Precision check/alignment of flow cell Wavelength verification check	As required As required Semi-annually
Auto Analyzers	Clean sampler Check all tubing Clean inside of colorimeter Clean pump well and pump rollers Clean wash fluid receptacle Oil rollers/chains/side rails Clean optics and cells	Daily Daily Daily Quarterly Weekly Weekly Quarterly
Gas Chromatograph/Mass Spectrometer (GC/MS)	Ion gauge tube degassing Pump oil-level check Pump oil changing Analyzer bake-out Analyzer cleaning Resolution adjustment COMPUTER SYSTEM AND PRINTER: Air filter cleaning Change data system air filter Printer head carriage lubrication Paper sprocket cleaning Drive belt lubrication	As required Monthly Annually As required As required As required As required As required As required As required As required

Table 21-2. Example: Schedule of Routine Maintenance

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
	Glass wool replacement	As required
	Check system for gas leaks with SNOOP	W/cylinder change as required
	Check for loose/frayed wires and insulation	Monthly
	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
Photoionization Detector (PID)	Change O-rings	As required
	Clean lamp window	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 fuses in power supply	As required
	Filter all samples	Daily
	Change autosampler rotor/stator	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	0.01M KCl calibration	Daily
	Conductivity cell cleaning	As required
Turbidimeter	Check light bulb	Daily, when used
Deionized/Distilled Water	Daily conductivity check	Daily
	Check deionizer light	Daily
	Monitor for VOA’s	Daily
	System cleaning	As required
	Replace cartridge & large mixed bed resins	As required
Drying Ovens	Temperature monitoring	Daily
	Temperature adjustments	As required
Refrigerators/ Freezers	Temperature monitoring	Daily
	Temperature adjustment	As required
	Defrosting/cleaning	As required
Vacuum Pumps/ Air Compressor	Drained	Weekly
	Belts checked	Monthly
	Lubricated	Semi-annually
pH/Specific Ion Meter	Calibration/check slope	Daily
	Clean electrode	As required

Table 21-2. Example: Schedule of Routine Maintenance

Instrument	Procedure	Frequency
BOD Incubator	Temperature monitoring Coil and incubator cleaning	Daily Monthly
Centrifuge	Check brushes and bearings	Every 6 months or as needed
Water baths	Temperature monitoring Water replaced	Daily Monthly or as needed

SECTION 21. MEASUREMENT TRACEABILITY

21.1 Overview

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 20.3). With the exception of Class A Glassware and glass microliter syringes, quarterly accuracy checks are performed for all mechanical volumetric devices. Wherever possible, subsidiary or peripheral equipment is checked against standard equipment or standards that are traceable to national or international standards. Class A Glassware and glass microliter syringes should be routinely inspected for chips, acid etching or deformity (e.g., bent needle). If the Class A glassware or syringe is suspect, the accuracy of the glassware will be assessed prior to use.

21.2 NIST-Traceable Weights and Thermometers

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.

21.3 Reference Standards / Materials

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. The receipt of all reference standards must be documented. Reference standards are labeled with a unique Standard Identification Number and expiration date. All documentation received with the reference standard is retained as a QC record and references the Standard Identification Number.

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, such as air analysis 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 the Corporate Environmental Health & Safety Manual or laboratory SOPs. For safety requirements, please refer to method SOPs and the laboratory Environmental Health and Safety Manual.

Standards and reference materials shall not be used after their expiration dates unless their reliability is verified by the laboratory and their use is approved by the Quality Assurance Manager. The laboratory must have documented contingency procedures for re-verifying expired standards.

21.4 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 applicable analytical Departments. 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 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.

21.4.1 All standards, reagents, and reference materials must be labeled in an unambiguous manner. Standards are logged into the laboratory's LIMS system, and are assigned a unique identification number. The following information is typically recorded in the electronic database within the LIMS.

- Standard ID
- 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
- Comment box (text field)

Records are maintained (either electronically or hard-copy) 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.

21.4.2 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 (***Specify from LIMS or logbook***)
- Special Health/Safety warnings if applicable

Records must also be maintained of the date of receipt for commercially purchased items or date of preparation for laboratory prepared items. Special Health/Safety warnings must also be available to the analyst. This information is maintained by the facility Environmental Health and Safety Coordinator.

21.4.3 In addition, the following information may be helpful:

- 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)
- Recommended Storage Conditions
- Concentration (if applicable)
- Initials of analyst preparing standard or opening container

All containers of prepared reagents must include an 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.

SECTION 22. SAMPLING

22.1 Overview

The laboratory provides the following sampling and field services. Sampling procedures are described in the following SOPs as applicable:

- Groundwater Sampling (TestAmerica Edison SOP #s ED-FLD-008 and ED-FLD-009)
- Wastewater Sampling (TestAmerica Edison SOP # ED-FLD-014)
- Potable Sampling
- Waste Sampling
- Soil and Sediment Sampling
- Flow Monitoring (TestAmerica Edison SOP #s ED-FLD-008 and ED-FLD-009)
- Field Parameter Analysis (TestAmerica Edison SOPs ED-FLD-001 thru ED-FLD-007, ED-FLD-010)
- Cleaning and Decontamination of Field Equipment (see individual SOPs listed above and TestAmerica Edison SOP# ED-GEN-013)

22.2 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.

22.2.1 Preservatives

Upon request, preservatives are provided to the client in pre-cleaned sampling containers. In some cases containers may be purchased pre-preserved from the container supplier. Whether prepared by the laboratory or bought pre-preserved, 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

22.3 Definition of Holding Time

The date and time of sampling documented on the 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 programs that determine holding time compliance based on the date and specific time of analysis compared to the time of sampling regardless of how long the holding time is.

22.4 Sampling Containers, Preservation Requirements, Holding Times

The preservation and holding time criteria specified in the laboratory SOPs are derived from the source documents for the methods. If method required holding times 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.

22.5 Sample Aliquots / Subsampling

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 subsample or aliquot of the sample provided for analysis.

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.

Guidelines on taking sample aliquots & subsampling are located SOP No. ED-GEN-007 (Subsampling).

SECTION 23. HANDLING OF SAMPLES

Sample management procedures at the laboratory ensure that sample integrity and custody are maintained and documented from sampling/receipt through disposal.

23.1 Chain of Custody (COC)

The COC form is the written documented history of any sample and is 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 23-1.

23.1.1 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 23-1). 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
- The container description
- The total number of each type of container
- 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.

When the sampling personnel deliver the samples directly to TestAmerica personnel, 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 personnel. 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. When sampling personnel deliver the samples through a common carrier (Fed-Ex, UPS), the CoC relinquished date/time is completed by the field personnel and samples are released to the carrier. Samples are only considered to be received by lab when personnel at the fixed laboratory facility 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 receipt from the courier is stored in log-in by date; it lists all receipts each date.

23.1.2 Legal / Evidentiary Chain-of-Custody

The laboratory may, upon special request, adhere to legal/evidentiary chain of custody requirements. If TestAmerica agrees to such procedures the samples are identified for legal/evidentiary purposes on the COC, login will complete the custody seal retain the shipping record with the COC, and initiate an internal COC for laboratory use by analysts and a sample disposal record.

23.2 Sample Receipt

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.

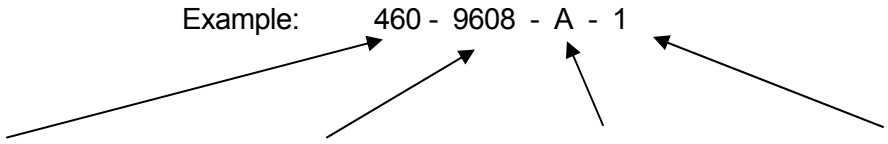
23.2.1 Laboratory Receipt

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 via the Sample Receipt application within TALS (the laboratory LIMS) and brought to the immediate attention of the appropriate Project Manager who will, in turn, contact 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.

23.2.1.1 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 Edison Laboratory (Location 460). 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: 460 - 9608 - A - 1 - **A** **Secondary Container Occurrence**

Example: 460-9608-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.

23.3 Sample Acceptance Policy

The laboratory has a written sample acceptance policy (Figure 23-2) 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.

23.3.1 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.

23.3.2 Any deviations from these checks 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 are not met, the laboratory shall either:

- Retain all correspondence and/or records of communications with the client regarding the disposition of rejected samples, or

- Fully document any decision to proceed with sample analysis that does not meet sample acceptance criteria.

Once sample acceptance is verified, the samples are logged into the LIMS according SOP No. ED-SPM-001.

23.4 Sample Storage

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, freezers or protected locations suitable for the sample matrix. Sample containers designated for metals only analysis are stored un-refrigerated. 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 every two weeks.

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 30 days after delivery of the final report to the client, which meets or exceeds most sample holding times. After 30 days the samples are disposed of or, upon client request moved to an sample archive area where they are stored for an additional time period agreed upon with the client or dictated by the applicable analytical program (ex. USEPA CLP).

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.

23.5 Hazardous Samples and Foreign Soils

To minimize exposure to personnel and to avoid potential accidents, hazardous and foreign soil samples are stored in an isolated area designated for hazardous waste only.

Procedures for the handling and storage of hazardous samples is addressed in the TestAmerica Corporate Safety Manual (TestAmercia Document No. CW-E-M-001) and in TestAmerica Edison SOP No. ED-SPM-001 (Sample Receipt, Login, Identification, And Storage).

Procedures for the acceptance and handling of USDA regulated domestic and foreign soils are detailed in TestAmerica SOP No. ED-SPM-006 (Procedure for Acceptance and Handling of Regulated Domestic and Foreign Soil).

23.6 Sample Shipping

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 is enclosed for those samples requiring water/solid volatile organic analyses. The chain-of-custody form is signed by the sample control technician 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.

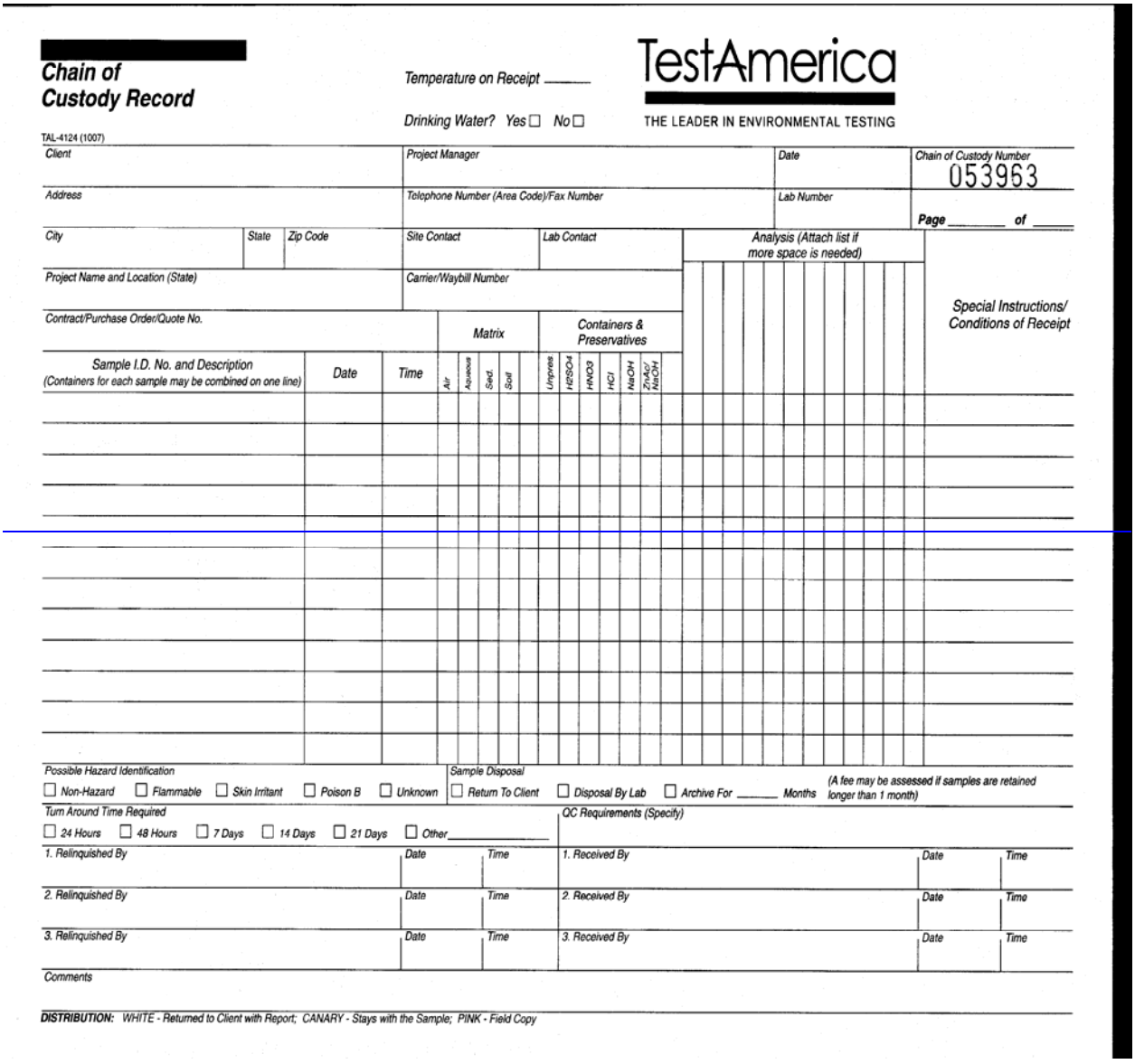
Note: If a client does not request trip blank analysis on the COC or other paperwork, the laboratory will not analyze the trip blanks that were supplied. However, in the interest of good client service, the laboratory will advise the client at the time of sample receipt that it was noted that they did 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.

23.7 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, TestAmerica Edison SOP No. ED-SPM-007 (Disposal of Samples and Associated Laboratory Waste). All procedures in the laboratory Environmental, Health and Safety Manual are followed during disposal. Samples are normally maintained in the laboratory no longer than 2 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, 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).

Figure 24-1. Chain of Custody (COC)



Chain of Custody Record

Temperature on Receipt _____

Drinking Water? Yes ☐ No ☐

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

TAL-4124 (1007)

Client	Project Manager	Date	Chain of Custody Number 053963
Address	Telephone Number (Area Code)/Fax Number	Lab Number	Page _____ of _____
City / State / Zip Code	Site Contact	Lab Contact	Analysis (Attach list if more space is needed)
Project Name and Location (State)	Carrier/Waybill Number		Special Instructions/ Conditions of Receipt
Contract/Purchase Order/Quote No.	Matrix	Containers & Preservatives	

Sample I.D. No. and Description (Containers for each sample may be combined on one line)	Date	Time	Air	Aqueous	Sed.	Soil	Unpres	H_2SO_4	HNO_3	HCl	NaOH	ZnAc/ NaOH

Possible Hazard Identification
☐ Non-Hazard ☐ Flammable ☐ Skin Irritant ☐ Poison B ☐ Unknown

Sample Disposal
☐ Return To Client ☐ Disposal By Lab ☐ Archive For _____ Months
(A fee may be assessed if samples are retained longer than 1 month)

Turn Around Time Required
☐ 24 Hours ☐ 48 Hours ☐ 7 Days ☐ 14 Days ☐ 21 Days ☐ Other_____

QC Requirements (Specify)

1. Relinquished By	Date	Time	1. Received By	Date	Time
2. Relinquished By	Date	Time	2. Received By	Date	Time
3. Relinquished By	Date	Time	3. Received By	Date	Time

Comments

DISTRIBUTION: WHITE - Returned to Client with Report; CANARY - Stays with the Sample; PINK - Field Copy

Figure 23-2. 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 noted 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.

1) Samples must arrive with labels intact with a Chain of Custody filled out completely. The following information must be recorded.
   - Client name, address, phone number and fax number (if available)
   - Project name and/or number
   - The sample identification
   - Date, time and location of sampling
   - The collectors name
   - The matrix description
   - The container description
   - The total number of each type of container
   - 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 date and time of receipt must be recorded between the last person to relinquish the samples and the person who receives the samples in the lab, and they must be exactly the same.
   - Information must be legible

2) Samples must be properly labeled.
   - Use durable labels (labels provided by TestAmerica are preferred)
   - Include a unique identification number
   - Include sampling date and time & sampler ID
   - Include preservative used.
   - Use indelible ink
   - Information must be legible

3) Proper sample containers with adequate volume for the analysis and necessary QC are required for each analysis requested.

4) Samples must be preserved according to the requirements of the requested analytical method.

5) Most analytical methods require chilling samples to 4° C (other than water samples for metals analysis). For these methods, the criteria are met if the samples are chilled to below 6° C and above freezing (0°C). For methods with other temperature criteria (e.g. some bacteriological methods require ≤ 10 °C), the samples must arrive within ± 2° C of the required temperature or within the method specified range. **Note:** Samples that are hand delivered to the laboratory immediately after collection may not have had time to cool sufficiently. In this case the samples will be considered acceptable as long as there is evidence that the chilling process has begun (arrival on ice).

5i.) Samples that are delivered to the laboratory on the same day they are collected may not meet the requirements of Section 5. In these cases, the samples shall be considered acceptable if the samples were received on ice.

5ii.) If sample analysis is begun within fifteen (15) minutes of collection, thermal preservation is not required.

5iii.) Thermal preservation is not required in the field if the laboratory receives and refrigerates the sample within fifteen (15) minutes of collection.

- Chemical preservation (pH) will be verified prior to analysis and documented, either in sample control or at the analyst's level. The project manager will be notified immediately if there is a discrepancy. If analyses will still be performed, all affected results will be flagged to indicate improper preservation.

- **For Volatile Organic analyses in drinking water (Methods 502.2 or 524.2**). Residual chlorine must be neutralized prior to preservation. If there is prior knowledge that the samples are not chlorinated, state it on the COC and use the VOA vials pre-preserved with HCl. The following are other options for a sampler and laboratory where the presence of chlorine is not known:

  - 1. Test for residual chlorine in the field prior to sampling.
    - If no chlorine is present, the samples are to be preserved using HCl as usual.
    - If chlorine is present, add either ascorbic acid or sodium thiosulfate prior to adding HCl.

  - 2. Use VOA vials pre-preserved with sodium thiosulfate or ascorbic acid and add HCl after filling the VOA vial with the sample.

- **FOR WATER SAMPLES TESTED FOR CYANIDE (by Standard Methods or EPA 335)**
  - In the Field: Samples are to be tested for Sulfide using lead acetate paper prior to the addition of Sodium Hydroxide (NaOH). If sulfide is present, the sample must be treated with Cadmium Chloride and filtered prior to the addition of NaOH.

    - If the sulfide test and treatment is not performed in the field, the lab will test the samples for sulfide using lead acetate paper at the time of receipt and if sulfide is present in the sample, the client will be notified and given the option of retaking the sample and treating in the field per the method requirements or the laboratory can analyze the samples as delivered and qualify the results in the final report.

  - It is the responsibility of the client to notify the laboratory if thiosulfate, sulfite, or thiocyanate are known or suspected to be present in the sample. This notification may be on the chain of custody. The samples may need to be subcontracted to a laboratory that performs a UV digestion. If the lab does not perform the UV digestion on samples that contain these compounds, the results must be qualified in the final report.

- The laboratory must test the sample for oxidizing agents (e.g. Chlorine) prior to analysis and treat according to the methods prior to distillation. (ascorbic acid or sodium arsenite are the preferred choice).

6) Sample Holding Times
   - TestAmerica will make every effort to analyze samples within the regulatory holding time. Samples must be received in the laboratory with enough time to perform the sample analysis. Except for short holding time samples (< 48hr HT) sample must be received with at least 48 hrs (2 working days) remaining on the holding time for us to ensure analysis.

   - Analyses that are designated as "field" analyses (Odor, pH, Dissolved Oxygen, Disinfectant Residual; a.k.a. Residual Chlorine, and Redox Potential) should be analyzed ASAP by the field sampler prior to delivering to the lab (within 15 minutes). However, if the analyses are to be performed in the laboratory, TestAmerica will make every effort to analyze the samples within 24 hours from receipt of the samples in the testing laboratory. Samples for "field" analyses received after 4:00 pm on Friday or on the weekend will be analyzed no later than the next business day after receipt (i.e., Monday, unless Monday is a holiday). Samples will remain refrigerated and sealed until the time of analysis. The actual times of all "field" sample analyses are noted in the final report. Samples analyzed in the laboratory will be qualified on the final report with an 'H' to indicate holding time exceedance.

7) All samples submitted for Volatile Organic analyses must have a Trip Blank submitted at the same time. TestAmerica will supply a blank with the bottle order.

8) The project manager will be notified if any sample is received in damaged condition. TestAmerica will request that a sample be resubmitted for analysis.

9) Recommendations for packing samples for shipment.

   - Pack samples in Ice rather than "Blue" ice packs.

   - Soil samples should be placed in plastic zip-lock bags. The containers often have dirt around the top, do not seal very well and are prone to intrusion from the water which results from melted ice.

   - Water samples are best package when wrapped with bubble-wrap or paper (newspaper, or paper towels) and then placed in plastic zip-lock bags.

   - Fill cooler void spaces with bubble wrap.

SECTION 24. ASSURING THE QUALITY OF TEST RESULTS

24.1 Overview

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 20, 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.

24.2 Controls

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.

24.3 Negative Controls

Table 24-1. Example – Negative Controls

Control Type	Details
Method Blank (MB)	are used to assess preparation and analysis for possible contamination during the preparation and processing steps.
	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.
	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. The method blank goes through all of the steps of the process (including as necessary: filtration, clean-ups, etc.).
	Reanalyze or qualify associated sample results when the concentration of a targeted analyte in the blank is at or above the reporting limit as established by the method or by regulation, AND is greater than 1/10 of the amount measured in the sample.
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.
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.

Table 24-1. Example – Negative Controls

Control Type	Details
Trip Blank [1]	are required to be submitted by the client with each shipment of samples requiring aqueous and solid volatiles analyses (or as specified in the client's project plan). 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.
Field Blanks [1]	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)
Equipment Blanks [1]	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. (TNI)
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

[1] When known, these field QC samples 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.

24.4 Positive Controls

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.

24.4.1 Method Performance Control - Laboratory Control Sample (LCS)

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.

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.

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.).

The specific frequency of use for LCS during the analytical sequence is defined in the specific standard operating procedure for each analysis. It is generally 1 for each batch of samples; not to exceed 20 environmental samples.

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.

- For methods that have 1-10 target analytes, spike all components.
- For methods that include 11-20 target analytes, spike at least 10 or 80%, whichever is greater.
- For methods with more than 20 target analytes, spike at least 16 components.
- Exception: Due to analyte incompatibility in pesticides, Toxaphene and Chlordane are only spiked at client request based on specific project needs.
- 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.

24.5 Sample Matrix Controls

Table 24-3. Sample Matrix Control

Control Type	Details	
Matrix Spikes (MS)	Use	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;

Table 24-3. Sample Matrix Control

Control Type	Details	
	Typical Frequency [1]	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.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. Refer to the method SOP for complete details
	Description	essentially a sample fortified with a known amount of the test analyte(s).
Surrogate	Use	Measures method performance to sample matrix (organics only).
	Typical Frequency [1]	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. 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.
	Description	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.
Duplicates[2]	Use	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.
	Typical Frequency [1]	Duplicate samples are usually analyzed with methods that do not require matrix spike analysis.
	Description	Performed by analyzing two aliquots of the same field sample independently or an additional LCS.
Internal Standards	Use	Are spiked into all environmental and quality control samples (including the initial calibration standards) to monitor the qualitative aspect of organic and some inorganic analytical measurements.
	Typical Frequency [1]	All organic and ICP methods as required by the analytical method.
	Description	Used to correct for matrix effects and to help troubleshoot variability in analytical response and are assessed after data acquisition. Possible sources of poor internal standard response are sample matrix, poor analytical technique or instrument performance.

[1] See the specific analytical SOP for type and frequency of sample matrix control samples.
[2] 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.

24.6 Acceptance Criteria (Control Limits)

As mandated by the test method and regulation, each individual analyte in the LCS, MS, or Surrogate Spike is evaluated against the control limits 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 control limits. When this occurs, the regulatory or project limits will supersede the laboratory's in-house limits.

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.

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. Control limits are

established per method (as opposed to per instrument) regardless of the number of instruments utilized.

Laboratory generated % Recovery acceptance (control) limits are generally established by taking ± 3 Standard Deviations (99% confidence level) from the average recovery of a minimum of 20-30 data points (more points are preferred).

- 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).

- 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 laboratory control limits are not consistent with DQOs, then alternatives must be considered, such as method improvements or use of an alternate analytical method.

- 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.

- The maximum acceptable recovery limit will be 150%.

- The maximum acceptable RPD limit will be 35% for waters and 40% for soils. The minimum RPD limit is 10%.

- If either the high or low end of the control limit changes by ≤ 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.

24.6.1 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.

24.6.1.1 The QA Department generates and reviews Quality Control Limit Summaries using the TALS Control Chart module. These tables summarize the updated, proposed precision and accuracy acceptability limits for each applicable analysis performed at TestAmerica Edison Once the QA Department is satisfied that the proposed limits are satisfactory the tables are forwarded to the applicable Department (Technical) Manager for final review. Once the proposed limits have been reviewed they entered into the appropriate TALS Method Limit Group database and approved for use (effectively replacing the existing limits in the database). The Quality Assurance Department maintains an archive of all limits used within the laboratory.

24.6.2 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 12) is also initiated if an LCS exceeds the acceptance limits. Sample results may be qualified and reported without reanalysis if:

- The analyte results are below the reporting limit and the LCS is above the upper control limit.
- If the analytical results are above the relevant regulatory limit and the LCS is below the lower control limit.

24.6.3 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 the lab's method SOPs and in Section 12.

24.6.4 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.

24.7 Additional Procedures to Assure Quality Control

The laboratory has written and approved method SOPs to assure the accuracy of the test method including calibration (see Section 20), use of certified reference materials (see Section 21) and use of PT samples (see Section 15).

A discussion regarding MDLs, Limit of Detection (LOD) and Limit of Quantitation (LOQ) can be found in Section 19.

- Use of formulae to reduce data is discussed in the method SOPs and in Section 20.
- Selection of appropriate reagents and standards is included in Section 9 and 21.
- A discussion on selectivity of the test is included in Section 5.
- Constant and consistent test conditions are discussed in Section 18.
- The laboratories sample acceptance policy is included in Section 23.

SECTION 25. REPORTING RESULTS

25.1 Overview

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 conflict between client requests and laboratory ethics or regulatory requirements, the laboratory's ethical and legal requirements are paramount, and the laboratory will work with the client during project set up to develop an acceptable solution. Refer to Section 7.

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 19.

25.2 Test Reports

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 is printed on laboratory letterhead, reviewed, and signed by the appropriate project manager. At a minimum, the standard laboratory report shall contain the following information:

25.2.1 A report title (e.g. Analytical Report For Samples) with a "sample results" column header.

25.2.2 Each report cover page printed on company letterhead, which includes the laboratory name, address and telephone number.

25.2.3 A unique identification of the report (e.g. work order number) 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.

25.2.4 A copy of the chain of custody (COC).

25.2.5 The name and address of client and a project name/number, if applicable.

25.2.6 Client project manager or other contact

25.2.7 Description and unambiguous identification of the tested sample(s) including the client identification code.

25.2.8 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.

25.2.9 Date reported or date of revision, if applicable.

25.2.10 Method of analysis including method code (EPA, Standard Methods, etc).

25.2.11 Reporting limit.

25.2.12 Method detection limits (if requested)

25.2.13 Definition of Data qualifiers and reporting acronyms (e.g. ND).

25.2.14 Sample results.

25.2.15 QC data consisting of method blank, surrogate, LCS, and MS/MSD recoveries and control limits.

25.2.16 Condition of samples at receipt including temperature. This may be accomplished in a narrative or by attaching sample login sheets

25.2.17 A statement expressing the validity of the results, that the source methodology was followed and all results were reviewed for error.

25.2.18 A statement to the effect that the results relate only to the items tested and the sample as received by the laboratory.

25.2.19 A statement that the report shall not be reproduced except in full, without prior express written approval by the laboratory coordinator.

25.2.20 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.

25.2.21 When TNI accreditation is required, the lab shall certify that the test results meet all requirements of TNI or provide reasons and/or justification if they do not.

25.2.22 The laboratory includes a cover letter.

25.2.23 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.

25.2.24 When soil samples are analyzed, a specific identification as to whether soils are reported on a “wet weight” or “dry weight” basis.

25.2.25 Appropriate laboratory certification number for the state of origin of the sample, if applicable.

25.2.26 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., partial report). A complete report must be sent once all of the work has been completed.

25.2.27 Any non-TestAmerica subcontracted analysis results are provided as a separate report on the official letterhead of the subcontractor. All TestAmerica subcontracting is clearly identified on the report as to which laboratory performed a specific analysis.

25.2.28 Non-accredited tests shall be clearly identified in the case narrative when claims of accreditation to the TNI standard are made.

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.

.

25.3 Reporting Level or Report Type

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 25.2 above.
- Level II (also called 'Results/QA) is a Level I report plus summary information, including results for the method blank reported to the laboratory MDL, percent recovery for laboratory control samples and matrix spike samples, and the RPD values for all MSD and sample duplicate analyses.
- NJDEP Reduced Deliverables Format which contains, at minimum, the elements listed in the current NJDEP Technical Requirements for Site Remediation, N.J.A.C. 7:26E.
- NJDEP Full Deliverables Format (Non-USEPA CLP Methods) which contains, at minimum, the elements listed in the current NJDEP Technical Requirements for Site Remediation, N.J.A.C. 7:26E.
- NJDEP Full Deliverables Format (USEPA CLP Methods) which contains, at minimum, the elements listed in the current NJDEP Technical Requirements for Site Remediation, N.J.A.C. 7:26E.
- NYSDEC ASP 'A' and 'B' Deliverables Format which contain, at minimum, the elements listed in the current *New York State Department of Environmental Conservation Analytical Services Protocol*.

In addition to the various levels of QC packaging, the laboratory also provides reports in diskette deliverable form. Initial reports may be provided to clients by facsimile. All faxed reports are followed by hardcopy. Procedures used to ensure client confidentiality are outlined in Section 25.6.

25.3.1 Electronic Data Deliverables (EDDs)

EDDs are routinely offered as part of TestAmerica's services. TestAmerica Edison offers a variety of EDD formats including NJ Hazsite Deliverables, Excel, Dbase, GISKEY, and Text Files.

EDD specifications are submitted to the IT Department by the PM for review and undergo the contract review process. 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. The validation of the code is retained by the IT staff coding the EDD.

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.

25.4 Supplemental Information for Test

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.

Numeric results with values outside of the calibration range, either high or low are qualified as 'estimated'.

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 TNI sample acceptance requirements such as improper container, holding time, or temperature.

Where applicable, a statement on the estimated uncertainty of measurements; information on uncertainty is needed when a client's instructions so require.

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 QA Department. 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.

25.5 Environmental Testing Obtained From Subcontractors

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).

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 TestAmerica are reported to the client on the subcontract laboratory's original report stationary and the report includes any accompanying documentation.

25.6 Client Confidentiality

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.

25.6.1 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:

This material is intended only for the use of the individual(s) or entity to whom it is addressed, and may contain information that is privileged and confidential. If you are not the intended recipient, or the employee or agent responsible for delivering this material to the intended recipient, you are hereby notified that any dissemination, distribution or copying of this communication is strictly prohibited. If you have received this communication in error, please notify us immediately by telephone at the 1-800-765-0980 (or for e-mails: please notify us immediately by e-mail or by phone (1-800-765-0980) and delete this material from any computer).

25.7 Format of Reports

The format of reports is designed to accommodate each type of environmental test carried out and to minimize the possibility of misunderstanding or misuse.

25.8 Amendments to Test Reports

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 12).

The revised report is retained on the Archive data server, as is the original report. The revised report is stored in the Archive data server under the sample number followed by "Rev (n)" where 'n' is the revision number. The revised report will have the words "Revision (n)" on the report cover page beneath the report date. Additionally, a section entitled "Revised Report" will appear on the Case Narrative page. A brief explanation of the reasons for the re-issue will be included in this section.

25.9 Policies on Client Requests for Amendments

25.9.1 Policy on Data Omissions or Reporting Limit Increases

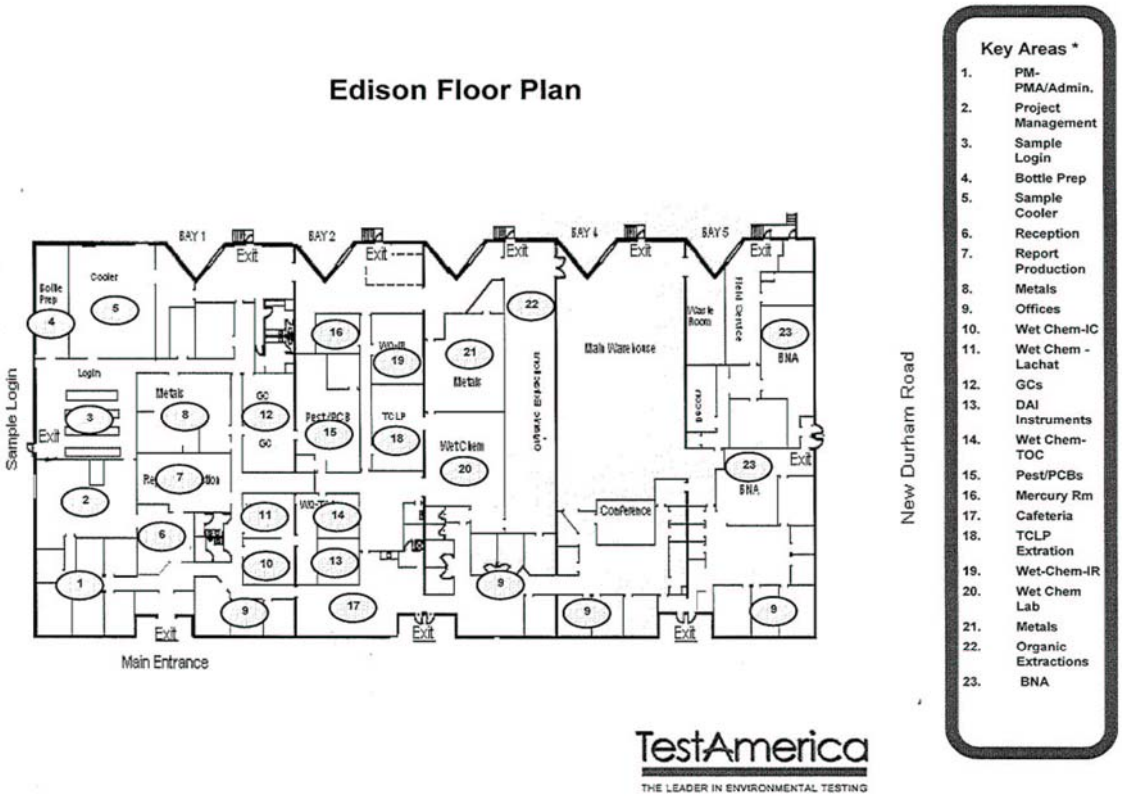
Fundamentally, our policy is simply to not omit previously reported results (including data qualifiers) or to not raise reporting limits and report sample results as ND. This policy has few exceptions. Exceptions are:

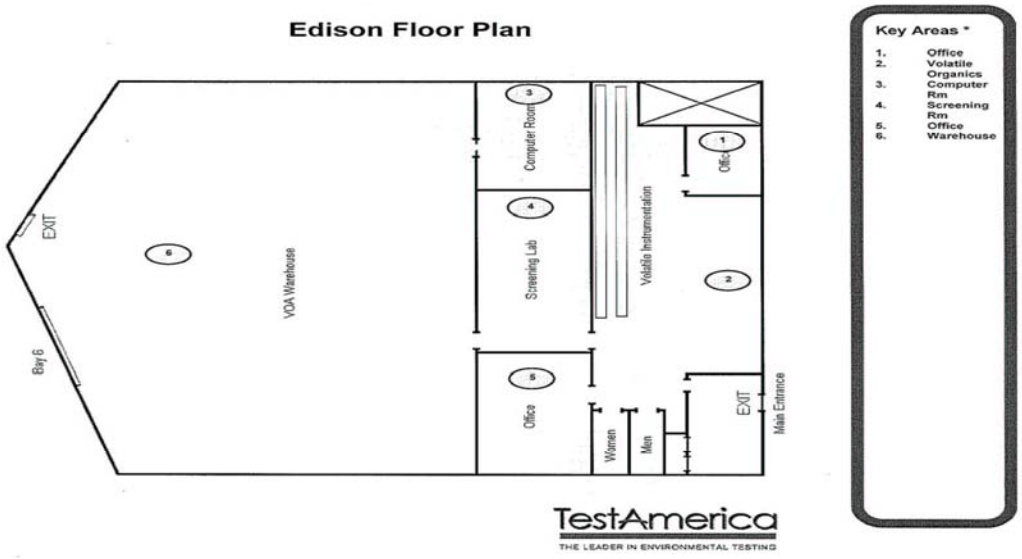
- Laboratory error.
- Sample identification is indeterminate (confusion between COC and sample labels).
- An incorrect analysis (not analyte) was requested (e.g., COC lists 8315 but client wanted 8310). A written request for the change is required.
- Incorrect limits reported based on regulatory requirements.
- 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.

25.9.2 Multiple Reports

TestAmerica does not issue multiple reports for the same work order 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. Laboratory Floor Plan





Appendix 2. Glossary/Acronyms (EL-V1M2 Sec. 3.1)

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.

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.

Analytical Uncertainty: A subset of Measurement Uncertainty that includes all laboratory activities performed as part of the analysis. (TNI)

Assessment: The evaluation process used to measure or establish the performance, effectiveness, and conformance of an organization and/or its systems to defined criteria (to the standards and requirements of laboratory accreditation). (TNI)

Audit: A systematic and independent examination of facilities, equipment, personnel, training, procedures, record-keeping, data validation, data management, and reporting aspects of a system to determine whether QA/QC and technical activities are being conducted as planned and whether these activities will effectively achieve quality objectives. (TNI)

Batch: Environmental samples that 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 (1) to twenty (20) environmental samples of the same quality systems 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 twenty-four (24) hours. An **analytical batch** is composed of prepared environmental samples (extracts, digestates or concentrates) which are analyzed together as a group. An analytical batch can include prepared samples originating from various quality system matrices and can exceed twenty (20) samples. (TNI)

Bias: The systematic or persistent distortion of a measurement process, which causes errors in one direction (i.e., the expected sample measurement is different from the sample's true value). (TNI)

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)

Calibration: A set of operations that establish, under specified conditions, the relationship between values of quantities indicated by a measuring instrument or measuring system, or values represented by a material measure or a reference material, and the corresponding values realized by standards. (TNI)

1) In calibration of support equipment the values realized by standards are established through the use of reference standards that are traceable to the International System of Units (SI).

2) In calibration according to methods, the values realized by standards are typically established through the use of Reference Materials that are either purchased by the laboratory with a certificate of analysis or purity, or prepared by the laboratory using support equipment that has been calibrated or verified to meet specifications.

Calibration Curve: The mathematical relationship between the known values, such as concentrations, of a series of calibration standards and their instrument response. (TNI)

Calibration Standard: A substance or reference material used to calibrate an instrument (QAMS)

Certified Reference Material (CRM): A reference material accompanied by a certificate, having a value, measurement uncertainty, and stated metrological traceability chain to a national metrology institute. (TNI).

Chain of Custody (COC) Form: Record that documents the possession of the samples from the time of collection to receipt in the laboratory. This record generally includes: the number and types of containers; the mode of collection; the collector; time of collection; preservation; and requested analyses. (TNI)

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.

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. TNI 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. (TNI)

Conformance: An affirmative indication or judgment 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).

Data Reduction: The process of transforming the number of data items by arithmetic or statistical calculations, standard curves, and concentration factors, and collation into a more useable form. (TNI)

Deficiency: An unauthorized deviation from acceptable procedures or practices, or a defect in an item. (ASQC)

Demonstration of Capability: A procedure to establish the ability of the analyst to generate analytical results of acceptable accuracy and precision. (TNI)

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)

Equipment Blank: Sample of analyte-free media which has been used to rinse common sampling equipment to check effectiveness of decontamination procedures.

External Standard Calibration: Calibrations for methods that do not utilize internal standards to compensate for changes in instrument conditions.

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 of Accreditation: Those matrix, technology/method, and analyte combinations for which the accreditation body offers accreditation.

Holding Times: The maximum time~~s~~ that samples may be held prior to analyses and still be considered valid or not compromised. (40 CFR Part 136)

Internal Standard: A known amount of standard added to a test portion of a sample as a reference for evaluating and controlling the precision and bias of the applied analytical test method. (TNI)

Internal Standard Calibration: Calibrations for methods that utilize internal standards to compensate for changes in instrument conditions.

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.

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 of the procedure unless otherwise noted in a reference method. 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.

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(s) of Detection (LOD) [a.k.a., Method Detection Limit (MDL)]: A laboratory's estimate of the minimum amount of an analyte in a given matrix that an analytical process can reliably detect in their facility. (TNI)

LOD Verification [a.k.a., MDL Verification]: A processed QC sample in the matrix of interest, spiked with the analyte at no more than 3X the LOD for single analyte tests and 4X the LOD for multiple analyte tests and processed through the entire analytical procedure.

Limit(s) of Quantitation (LOQ) [a.k.a., Reporting Limit]: The minimum levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be reported with a specified degree of confidence. (TNI)

Quality System (QS) 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 or Saline/Estuarine —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 & Emissions: 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. (TNI)

Matrix Spike (spiked sample or fortified sample): A sample prepared, taken through all sample preparation and analytical steps of the procedure unless otherwise noted in a referenced method, by adding a known amount of target analyte to a specified amount of sample for which an independent test result 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 Spike Duplicate (spiked sample or fortified sample duplicate): A replicate matrix spike prepared and analyzed to obtain a measure of the precision of the recovery for each analyte.

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.

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)

Negative Control: Measures taken to ensure that a test, its components, or the environment do not cause undesired effects, or produce incorrect test results.

Non-conformance: An indication, judgment, or state of not having met the requirements of the relevant specifications, contract, or regulation.

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.

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.

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. (TNI)

Preservation: Any conditions under which a sample must be kept in order to maintain chemical and/or biological integrity prior to analysis. (TNI)

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. (TNI)

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. (TNI)

Proficiency Test Sample (PT): A sample, the composition of which is unknown to the laboratory and is provided to test whether the laboratory can produce analytical results within specified acceptance criteria. (TNI)

Quality Assurance: An integrated system of management activities involving planning, implementation, assessment, reporting and quality improvement to ensure that a process, item, or service is of the type of quality needed and expected by the client. (TNI)

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 that measures the attributes and performance of a process, item, or service against defined standards to verify that they meet the stated requirements established by the customer; operational techniques and activities that are used to fulfill requirements for quality; also the system of activities and checks used to ensure that measurement systems are maintained within prescribed limits, providing protection against "out of control" conditions and ensuring that the results are of acceptable quality. (TNI)

Quality Control Sample: A sample used to assess the performance of all or a portion of the measurement system. One of any number of samples, such as Certified Reference Materials, a quality system matrix fortified by spiking, or actual samples fortified by spiking, intended to demonstrate that a measurement system or activity is in control. (TNI)

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. (TNI)

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 activities. (TNI)

Raw Data: The documentation generated during sampling and analysis. This documentation includes, but is not limited to, field notes, electronic data, magnetic tapes, untabulated sample results, QC sample results, print outs of chromatograms, instrument outputs, and handwritten records. (TNI)

Record Retention: The systematic collection, indexing and storing of documented information under secure conditions.

Reference Material: Material or substance one or more properties of which are sufficiently homogeneous and well established to be used for the calibration of an apparatus, the assessment of a measurement method, or for assigning values to materials. (TNI)

Reference Standard: Standard used for the calibration of working measurement standards in a given organization or a given location. (TNI)

Sampling: Activity related to obtaining a representative sample of the object of conformity assessment, according to a procedure.

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: The ability to analyze, distinguish, and determine a specific analyte or parameter from another component that may be a potential interferent or that may behave similarly to the target analyte or parameter within the measurement system. (TNI)

Sensitivity: The capability of a method or instrument to discriminate between measurement responses representing different levels (e.g., concentrations) of a variable of interest. (TNI)

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.

Standard: The document describing the elements of laboratory accreditation that has been developed and established within the consensus principles of standard setting and meets the approval requirements of standard adoption organizations procedures and policies. (TNI)

Standard Operating Procedures (SOPs): A written document which details the method for an operation, analysis, or action, with thoroughly prescribed techniques and steps. SOPs are officially approved as the methods for performing certain routine or repetitive tasks. (TNI)

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)

Technical Manager: A member of the staff of an environmental laboratory who exercises actual day-to-day supervision of laboratory operations for the appropriate fields of accreditation and reporting of results

Technology: A specific arrangement of analytical instruments, detection systems, and/or preparation techniques.

Traceability: The ability to trace the history, application, or location of an entity by means of recorded identifications. In a calibration sense, traceability relates measuring equipment to national or international standards, primary standards, basic physical constants or properties, or reference materials. In a data collection sense, it relates calculations and data generated throughout the project back to the requirements for the quality of the project. (TNI)

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
DOC – Demonstration of Capability
DQO – Data Quality Objectives
DUP - 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
ICP/MS – ICP/Mass Spectrometry
ICV – Initial Calibration Verification
IDL – Instrument Detection Limit
IH – Industrial Hygiene
IS – Internal Standard
LCS – Laboratory Control Sample
LCSD – Laboratory Control Sample Duplicate
LIMS – Laboratory Information Management System
LOD – Limit of Detection
LOQ – Limit of Quantitation
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
NELAP - National Environmental Laboratory Accreditation Program
PT – Performance Testing
TNI – The NELAC Institute
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 – Standard Deviation
SOP – Standard Operating Procedure
TAT – Turn-Around-Time
VOA – Volatiles
VOC – Volatile Organic Compound

Appendix 3. Laboratory Certifications, Accreditations, Validations

TestAmerica Edison maintains accreditations, certifications, and approvals 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:

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

TestAmerica Certifications

Laboratory	Program	Authority	Identification	Expiration Date
TestAmerica Edison	Delaware DNREC	Delaware	N/A	12/31/2011
TestAmerica Edison	NELAC	New Jersey	12028	06/30/2012
TestAmerica Edison	NELAC	New York	11452	04/01/2012
TestAmerica Edison	NELAC	Pennsylvania	68-00522	02/29/2012
TestAmerica Edison	State Program	Connecticut	PH-0200	09/30/2012
TestAmerica Edison	State Program	Rhode Island	LAO00132	12/30/2011
TestAmerica Edison	USDA	USDA	NJCA-003-08	03/11/2014

Page 1 of 1 9/17/11

For more information, or to contact a local TestAmerica representative nearest you, please visit our website at www.testamericainc.com

The certificates and parameter lists (which may differ) are available, upon request, from a laboratory representative. 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.

Title: TSS, Analysis of Total Suspended Solids in Water and Wastewater Samples By Standard Method 2540D

Once printed, this is considered an uncontrolled document

Approvals (Signature/Date):

12/04/2012	12/04/2012
Mark Acierno Date	Kene' Kasperek Date
Operations Manager	Health & Safety Manager /Coordinator
12/04/2012	12/04/2012
Carl Armbruster Date	Ann Gladwell Date
Quality Assurance Manager	Laboratory Director

Facility Distribution No. Electronic **Distributed To:** Facility Intranet

1.0 Scope and Application

1.1 Analytes, Matrix(s), and Reporting Limits

This SOP is applicable to SM 2540 D for the determination of total suspended solids in drinking, surface and saline waters, domestic and industrial waste. The laboratory's reporting limit is 10 mg/L.

On occasion clients may request modifications to this SOP. These modifications are handled following the procedures outlined in Sections 7 (Review of Work Request) and 19 (Test Methods and Method Validation) of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

2.0 Summary of Method

A well-mixed sample is filtered through a glass fiber filter. Residue, non-filterable, is defined as those solids that are retained by a glass fiber filter and dried to a constant weight at 103-105^{O}C. The amount of residue in mg/L is calculated by taking the difference of the initial weight of the Double Weigh filters and the weight of the filters with dried residue.

3.0 Definitions

For a complete list of definitions refer to Appendix 2 in the most current revision of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

4.0 Interferences

4.1. Filtration apparatus, filter material, pre-washing, post-washing, and drying temperature are specified because these variables have been shown to affect results.

4.2. Samples high in filterable residue may be subject to a positive interference. Care must be taken when washing the filterable residue from the filter. Diluting the sample will also minimize this interference.

4.3. Excessive residue on the filter can form a water-entrapping crust, limit the sample volume to that yielding no more than 200 mg residue.

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001) 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 coats and closed-toe, nonabsorbent shoes are a minimum.

5.1. Specific Safety Concerns or Requirements

Be very careful when putting in and getting out samples from the oven. It is very hot, wear heat resistant gloves and use tongs.

5.2. Primary Materials Used

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.

6.0 Equipment and Supplies

6.1. Instrumentation

None

6.2. Supplies

- Double Weigh Filters – pre-weighed and verified, 47mm glass Fiber Filter – Environmental Express, Cat # F93447MM-X
- Desiccators
- Drying Oven Capable of holding a temperature of 103-105^{O}C
- Analytical Balance – capable of accurate weighing to 0.1mg.
- 100 ml Graduated Cylinder
- 1000 ml Filtering Flask
- Glass Filter Holder
- Vacuum Filtration Apparatus
- De-ionized Water 18 megohm

7. Reagents and Standards

7.1. Reagents

None

7.2. Standards

- Laboratory control sample (LCS) – purchased from ERA, for stability and storage information refer to manufacturer's instructions.

8. Sample Collection, Preservation, Shipment 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	Sample Container	Min. Sample Size	Preservation	Holding Time	Reference
Waters	P,FP,G[1]	100 mL	Cool ≤ 6 °C	7 Days	40 CFR Part 136.3

[1] 'P' is polyethylene, 'FP' is fluoropolymer, 'G' is glass.

9. Quality Control

9.1. Sample QC - The following quality control samples are prepared with each batch of samples.

Quality Controls	Frequency	Control Limit
Method Blank (MB)	Daily	< Rpt. Limit
Laboratory Control Sample (LCS)	Daily	85-115%
Matrix Duplicate (MD)[1]	1 in 20 or fewer samples	RPD< 5%

[1] The sample selection for Matrix Duplicate is randomly selected, unless specifically requested by a client.

9.1.1. Method Blank: A blank must be analyzed each time samples are analyzed. Use deionized water for the blank and the results must be below the reporting limit. Method Blank results serve as a quality check for the Double Weigh filters.

9.1.2. Laboratory Control Sample: A Laboratory Control Sample is obtained from an outside vendor and is used to measure method performance on the matrix being analyzed. LCS is run daily. LCS results serve as a quality check for the Double Weigh filters.

9.1.3. Matrix Duplicate: A duplicate is analyzed by using a second aliquot of sample. The relative percent difference (RPD) for wastewater and drinking water samples must be within 5%.

9.2. Instrument QC

None

10. Procedure

10.1. Sample Preparation

10.1.1. Log sample IDs in TALS>Analyst Desktop II.

10.1.2. Record the corresponding filter ID and weights in TALS>worksheet tab.

10.1.3. Arrange samples according to the sample order listed in TALS worksheet. This will ensure that the assigned filter disc is used for the correct sample.

10.2. Calibration

10.2.1. Analytical balances are checked daily to verify calibrations, (see TestAmerica Edison SOP No. ED-GEN-010, *Calibration of Analytical Balances*, current revision).

10.3. Sample Analysis

10.3.1. Assemble the filtering apparatus with the pre-weighed filter disc wrinkled side up. Apply vacuum and wet the filter with a small volume of deionized water to seat it against the support.

10.3.2. Shake liquid sample vigorously and rinse a 100 ml graduated cylinder with a sufficient volume of sample. Check to ensure that the right sample corresponds to the correct filter disc. Fill a graduated cylinder with 100 ml of sample or a quantity of sample that will filter and quantitatively transfer to the funnel. Record volume of sample used for analysis in TALS on the worksheet tab. Turn vacuum on. **Note:** Pour sample immediately after shaking to prevent suspended solids from settling to the bottom.

10.3.3. After the sample has completely filtered, rinse the graduated cylinder, filter disc, non-filterable residue, and funnel wall with three small portions of deionized water allowing complete drainage between washings. Continue vacuum until filtration is complete.

10.3.4. Stop vacuum and carefully remove the filter from the filter support into the designated aluminum weigh dish. Dry filter in an oven at 103-105^{O}C for one hour. **Note:** Check and record oven temperature before placing samples in the oven. Mark the temperature on the oven chart posted on the oven door and record analyst's initial.

10.3.5. Record the time the samples are placed in and out of the oven and the temperature of the oven in TALS batch information page.

10.3.6. Remove filter from oven and place in a desiccator. After filter has cooled (usually 20 minutes), weigh to 0.1 mg and record in TALS worksheet tab.

10.3.7. Place samples back in the oven for another hour and repeat procedure in Sec 10.3.5 and 10.3.6.

10.3.8. Check if a constant weight is obtained or if weight change is less than 4% of the previous weighing or 0.5 mg whichever is less.

10.3.9. Repeat drying cycle if necessary.

11.0. Calculations / Data Reduction

11.1. Accuracy:

$$\text{LCS \% Recovery} = \frac{\text{observed concentration}}{\text{known concentration}} \times 100$$

11.2. Precision (RPD):

$$\text{Matrix Duplicate (MD)} = \frac{|\text{orig. sample value - dup. sample value}|}{[(\text{orig. sample value + dup. sample value})/2]} \times 100$$

11.3. Concentration = TSS (mg/L) = $\frac{W_2 - W_1 * 1000}{V}$

Where:
W_2 = Weight of filter and residue in mg
W_1 = Weight of filter in mg
V = Sample volume in ml

11.4. Data Reduction

11.4.1. Record special notes and observations in the "worksheet" tab (i.e. sample appearance and notes on why samples were rejected).

11.4.2. Analyst must fill out the Wet Chem Data Review checklist (WI# EDS-WI-008) during the first level review. After the batch is second level reviewed, the checklist is filed in wetchem department.

12.0. Method Performance

12.1. Method Detection Limit Study (MDL)

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 the laboratory's MDL procedure in Section 19 (Test Methods and Method Validation) of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM). 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 unless method requirements require a greater frequency.

12.2. Demonstration of Capabilities

For DOC procedure refer to Section 19 in the most current revision of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

12.3. Training Requirements

Refer to TestAmerica SOP No. ED-GEN-022, Training, for the laboratory's training program.

13.0. 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 Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

14.0. Waste Management

14.1. 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 TestAmerica SOP No. ED-SPM-008 (Laboratory Waste Disposal Procedures).

14.2. There are no special waste streams produced by method.

15.0. References / Cross-References

15.1. Standard Methods for the Examination of Water and Wastewater , 18th Edition, American Public Health Association, Baltimore Maryland, 1992, SM 2540 D.

15.2. TestAmerica Edison Document No. ED-QA-LQM, *Laboratory Quality Manual*, most current revision.

15.3. TestAmerica Edison SOP ED-GEN-022, *Training*, most current revision.

15.4. TestAmerica Edison SOP No. ED-GEN-010, *Calibration of Analytical Balances*, current revision.

15.5. TestAmerica Edison SOP No. ED-SPM-008, *Laboratory Waste Disposal Procedures*, current revision.

15.6. TestAmerica Corporate Environmental Health and Safety Manual, Document No. CW-E-M-001, most current revision.

15.7. TestAmerica Edison Work Instruction # EDS-WI-008, Wetchem Data ReviewChecklist, most current revision.

16.0. Method Modifications:

None

17.0. Attachments

None

18.0. Revision History

- Revision 7, dated 04 December 2012
  - Sec 1, 3 and 12: Revised LQM section references to reflect the most current LQM revision.

- Revision 6, dated 02 November 2010
  - Sec. 4.3: Section added.
  - Sec. 9.1: Revised LCS limits from “manufacturer’s limits” to “85-115%.”
  - Sec. 10.0: Changed data recording from analytical logbook to TALS since all data is now recorded in TALS.
  - Sec. 11.4: Added data reduction section.
  - Sec 15: Added applicable references.

- Revision 5, dated 08 October 2008
  - Revised SOP format in accordance with TestAmerica Corporate Quality SOP No. CW-QS-002 (Rev 0) Writing a Standard Operating Procedure (SOP).
  - Sec. 1.1: Deleted method EPA 160.2 to comply with the Method Update Rule.
  - Sec. 8 & 9: Reformat both sections into Table format.
  - Section 9.1.3: Revised RPD limit to 5% for all matrices as per Method 2540D.
  - Sec. 10: Clarified Sample preparation and analysis procedure.
  - Section 10.3.7: Added criteria for constant weight, ‘*less than 4% of the previous weighing*.’
  - Section 15: Deleted EPA reference Method 160.2. Added applicable references.

- Revision 4, 01 August 2006
  - Section 4.1: Replaced ‘Pro-Weigh’ filters with ‘Double Weigh’ filters.

Title: Analysis of Alkalinity in Water, Wastewater and Soil by Manual Titration or Auto-Titrator, Standard Method 2320 B-11

Once printed, this is considered an uncontrolled document

Approvals (Signature/Date):

Jasmine Parillo 06/28/13 Jasmine Parillo Date Department Manager	Kene' Kasperek 06/27/13 Kene' Kasperek Date Health & Safety Manager /Coordinator
Carl Armbruster 06/27/13 Carl Armbruster Date Quality Assurance Manager	Ann Gladwell 06/27/13 Ann Gladwell Date Laboratory Director

1.0 Scope and Application

1.1 Analytes, Matrix(s), and Reporting Limits

Standard Method 2320 B-11 is applicable to the determination of alkalinity in drinking, surface waters, saline waters, domestic and industrial wastes. The laboratory's reporting limit for aqueous samples is 5.0 mg/L total alkalinity as CaCO3.

Method 2320 B-11 modified is applicable to the determination of alkalinity in soil. The laboratory's reporting limit is 20 mg/kg total alkalinity as CaCO3.

The following forms of alkalinity may be determined using this method: total alkalinity, hydroxide alkalinity, carbonate alkalinity and bicarbonate alkalinity.

On occasion clients may request modifications to this SOP. These modifications are handled following the procedures outlined in Sections 7 (Review of Work Request) and 19 (Test Methods and Method Validation) of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

2.0 Summary of Method

The level of alkalinity contained in a sample is determined by titrating with acid to a pH of 8.3 (if applicable) and then to 4.5. The sample aliquot should be selected such that the titration volume used does not exceed 50ml.

3.0 Definitions

For a complete list of definitions refer to Appendix 2 in the most current revision of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

4.0 Interferences

Dissolved gas can affect alkalinity; prevent undo agitation or exposure to atmosphere as much as possible.

Soaps, oily matter, suspended solids or precipitates may coat the electrode. Allow electrode to come to equilibrium between additions of titrant and clean electrode occasionally. Do not filter, dilute or alter sample.

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001) 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 coats and closed-toe, nonabsorbent shoes are a minimum.

5.1. Specific Safety Concerns or Requirements

There are no specialized safety concerns associated with this method.

5.2. 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 (2)	Hazards	Exposure Limit (1)	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 – Exposure limit refers to the OSHA regulatory exposure limit.			
2- Always add acid to water to prevent violent reactions.			

6.0 Equipment and Supplies

6.1. Instrumentation

- MAN-TECH PC-Titration Plus
- pH meter Thermo Orion, Model 320
- combination electrode: PerpHecT Ross Electrode

6.2. Supplies

- Burette, 10 ml and 25 ml. (Class A)
- Beakers, 150 ml
- Magnetic stirrer and Teflon-coated magnets
- Eppendorf Pipettes, varying volumes
- Volumetric flasks, assorted (Class A)
- Graduated cylinder (Class A)

7. Reagents and Standards

7.1. Reagents

7.1.1. 0.05 N Sodium carbonate solution: Place 0.625 grams Na_2CO_3 (dried 4 hours at 250^{o}C) in a 250 ml volumetric flask and dilute to mark with deionized water. Solution is good for one week; refrigerate until ready to use.

7.1.2. Ross pH electrode filling solution, 3M KCL: Cat. No. 810007; for storage and stability information, see manufacturer's instructions. Note: Do not use any filling solution that contains silver, as silver will damage the electrode.

7.1.3. pH buffer solution of 4, 7, and 10: Purchased commercially; store at room temperature, for stability information refer to manufacturer's instructions.

7.2. Standards

Standardize titrants every three months by the following method and document standardization in the standardization logbook.

7.2.1. **0.10 N H_2SO_4**: Dilute 3.0ml conc. H_2SO_4 with de-ionized water to 1000ml. This reagent is stable for 6 months and should be stored at room temperature. Standardize against 0.05 N Na_2CO_3 solution (Sec. 7.1.1).

7.2.1.1. Place 40.0 ml freshly prepared 0.05 N Na_2CO_3 solution into a 500 ml erlenmeyer flask. Add about 60 ml of deionized water.

7.2.1.2. Titrate with 0.1 N H_2SO_4 until pH = 5.0.

7.2.1.3. Cover flask with a watchglass and boil for 3 minutes.

7.2.1.4. Cool and continue titrating to pH 4.5.

7.2.2. **0.02 N H_2SO_4:** Purchased commercially. For stability and storage information, refer to manufacturer's instructions. Standardize against 0.05N Na_2CO_3 solution (Sec. 7.1.1):

7.2.2.1. Same as above except use only 15.0 ml Na_2CO_3 solution and bring up to 100 ml with deionized water.

7.2.2.2. Titrant Concentration (N)= $\frac{2.50 \times A}{53.00 \times B}$

Where: A= vol. Na_2CO_3 solution (ml)
B= vol. titrant (ml)

8. Sample Collection, Preservation, Shipment 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	Sample Container	Min. Sample Size	Preservation	Holding Time[2]	Reference
Waters	P,FP,G[1]	50 mLs	Cool, ≤ 6°C	14 Days	40 CFR Part 136.3
Soils	P,FP,G[1]	50 grams	Cool, ≤ 6°C	14 Days	Not applicable

[1] P' is polyethylene, 'FP' is fluoropolymer, 'G' is glass. Fill container completely, cap tightly and limit headspace. Do not filter, concentrate or alter samples in any way. Avoid sample agitation and prolonged exposure to the air.
[2] Holding time is 14 days but it is strongly urged to analyze as soon as possible.

9. Quality Control

9.1. Sample QC

Open a quality control batch every two weeks or every 20 samples, whichever is first. The following quality control samples are prepared with each batch of samples.

Quality Controls	Frequency	Control Limit
Method Blank (MB)	Daily, 1 in 20 or fewer samples	< Reporting Limit
Laboratory Control Sample (LCS)[1]	Daily, 1 in 20 or fewer samples	Vendor specified QC limits
Matrix Duplicate (MD)[1]	1 in 20 or fewer samples	Statistical Limits [2]

[1] The sample selection for MD is randomly selected, unless specifically requested by a client.
[2] Statistical control limits are updated annually and are updated into LIMS.

9.1.1. Method Blank: A blank must be analyzed each time samples are analyzed. Use deionized water for the blank and the results must be below the reporting limit. If not all samples associated with the method blank must be re-analyzed following an acceptable Method Blank.

9.1.2. Laboratory Control Sample (LCS): LCS is obtained from an outside vendor and is used to measure method performance on the matrix being analyzed. LCS is run daily; results must be within vendor specified QC limits. If LCS fails, all samples must be re-analyzed following an acceptable LCS.

9.1.3. Matrix Duplicate: A duplicate is analyzed by using a second aliquot of sample. The relative percent difference (RPD) must be within laboratory generated control limits.

9.2. Instrument QC

None

10. Procedure

10.1. Calibration

10.1.1. Check the flow rate ml/min of the sampler daily or prior to sample analysis as follows:

- Place sample needle (input) in a vial filled with water.
- Set sample pump to 'Forward.'
- Place the sample needle (output) in a graduated cylinder and measure the flow rate in ml/min.
- Repeat the above procedure three times and take the average measurements.
- Set sample back to 'Auto.'
- In 'PC titrate' window, click on 'Interface,' then 'Hardware set-up.'
- Click on 'Digital/Amplifier' tab.
- Click on 'Extended Digital I/O' tab.
- Highlight the row for 'Sample pump'
- Enter the measured average flow rate in 'Flow rate ml/min' box and click 'ok.

10.1.2. Enter the final concentration (N) of the titrant H_2SO_4 in the PC-titrator.

10.1.2.1. From the Main menu, highlight 'Set up.'

10.1.2.2. Click on "Titration Method,' and 'Load.' Select 'Alkalinity,' then click OK.

10.1.2.3. This will bring up H_2SO_4 as the titrant. Type in the obtained concentration (N) value (Sec 7.2.2.2).

10.1.2.4. Click 'SAVE.' A blank box will appear on the screen, type in 'H_2SO_4 standardized on mm/dd/yy,' then click 'Done.'

10.1.3. Calibrate the instrument (PC titrator) daily.

10.1.3.1. Fill vials 1, 2, and 3 with 25 ml of buffer 4, 7, and 10. **Note:** After the last buffer, place a vial filled with diluted buffer 4, this does not need to be typed into the sequence. The diluted buffer 4 will fill the electrode cell after the analysis has been completed and protect the electrodes from drying out.

10.1.3.2. Click twice on the PC-Titrate V3 icon to open the program.

10.1.3.3. Click on the Calibration pH 4-7-10 icon from the list of icons on the bottom on the screen.'

10.1.3.4. Click 'Start' (DO NOT click 'ok'). Instrument will start calibration by measuring the pH of each buffer (4, 7 and 10).

10.1.3.5. After the last buffer (pH 10) is measured, click on 'Calibration Result' tab. If calibration validity is 'True' click 'Ok.' If the calibration validity is 'False', check for crystals around the electrode, check to make sure that the probe has enough filling solution, and recalibrate the instrument.

10.1.3.6. Print Calibration Report:

10.1.3.6.1. Go to 'Titrator.' Select 'Examine Calibrations.'

10.1.3.6.2. From the 'Port' drop down menu, click on '1.'

10.1.3.6.3. From the 'Calibration ID' drop down menu, click 'pH-cal- 4-7-10.'

10.1.3.6.4. Click 'Print this Calibration,' and choose the printer destination. Click 'Done' and 'Ok.'

10.1.4. Calibration for manual titration: see TestAmerica SOP No. ED-WET-060 (Analysis of pH for Waters and Drinking water Measured Electrochemically) for information on calibrating the pH meter.

10.2. Sample Analysis

10.2.1. Manual titration (Water samples):

10.2.1.1. Rinse all glassware well with deionized water.

10.2.1.2. Transfer 100 ml or aliquot of a well-mixed sample to specimen cup with as little agitation as possible.

10.2.1.3. Record initial pH of sample in analytical logbook.

10.2.1.4. Titrate to a pH of 8.3 if carbonate and bicarbonate alkalinity are required, and proceed to next step.

10.2.1.5. Titrate with standardized acid to pH of 4.5 and record volume of acid. Sample may be swirled to mix in acid, but avoid excessive agitation. If alkalinity is <20ppm use 0.02N H_2SO_4.

10.2.1.6. If alkalinity is less than 20 ppm, continue to titrate to 4.20 and note added titrant.

10.2.2. Manual titration (Soil samples):

10.2.2.1. Homogenize sample as per procedure in TestAmerica Edison SOP No. ED-GEN-007 (Subsampling).

10.2.2.2. Rinse all glassware well with deionized water.

10.2.2.3. Weigh 50.0g of sample into a 140ml specimen cup and add 50 ml of deionized water and mix. Avoid excessive agitation.

10.2.2.4. Stir the sample for 5 minutes and let it stand for approximately 30 minutes.

10.2.2.5. Decant as much liquid for titration.

10.2.2.6. Record the initial pH. If total alkalinity is required, proceed to the next step. For all other forms of alkalinity proceed to Sec. 10.2.4.

10.2.2.7. Titrate with standardized acid to pH of 4.5 and record volume of acid. Sample may be swirled to mix in acid, but avoid excessive agitation. If alkalinity is <20ppm use 0.02N H_2SO_4.

10.2.2.8. If alkalinity is less than 20 ppm, continue to titrate to 4.20 and note added titrant

.

10.2.3. Instrument: MANTECH PC titrator:

10.2.3.1. From the main menu, highlight 'Titrator,' then click 'Run Titration.' This will bring the timetable screen.

10.2.3.2. Determine the number of samples, including MB and LCS, that will be run and add the required number of spaces/rows needed on the timetable by clicking on 'ADD X ROWS'. This will expand the rows in the timetable.

Note: If more rows were added than desired, click on the sample row (this will highlight the sample), then click on 'DELETE THE HIGHLIGHTED SAMPLE.'

10.2.3.3. Double click the first row under the heading SCHEDULE, A pop-up menu will appear, select 'Alkalinity 25 ml.' Do the same for each sample starting on the second row or use the Copy command.

10.2.3.4. Click on the row below the heading ORDER NUMBER then click on AUTO-GENERATE ORDER NUMBER. The order number (i.e. 20060331-1) will appear under the 'Order Number' column. Do the same for each sample starting on second row or use the Copy command.

10.2.3.5. Under the heading SAMPLE NAME and starting at row 1 (numbers can be seen on the left hand side of the timetable), type in RINSE in row 1, MB in row 2, and type in LCS in row 3.

Note: Use deionized water for the RINSE sample to flush the system.

10.2.3.6. Continue on to row 3 and enter the Job number and Sample ID in the format 'Job#-XXXXXX.'

10.2.3.7. For QC samples, use the suffix 'DU' on the sample number (i.e. 460-19450-B-2 DU).

10.2.3.8. A new MB and LCS should be analyzed after 20 samples.

10.2.3.9. Enter the VIAL information: Under the heading VIAL, type in the vial number in the sample tray that corresponds to the sample identified in the timetable. Note: Always use vial #1 for sample number 1, this will ensure that vial numbers will match the row numbers.

10.2.3.10. Once the timetable information is complete and accurate, save this information by clicking on SAVE AS. A pop up menu will appear, type in the file ID in the format 'ALKmmddyy' (next to 'ENTER NEW TEXT').

10.2.3.11. Click on 'Create Using Current Sample ID's' then click OK. Pop up menu will close.

10.2.3.12. Fill the vials with sample aliquot using the timetable as a reference. **Note**: After the last sample aliquot, place a vial filled with the diluted buffer 4. This vial does not need to be typed into the sequence but, it will fill the electrode cell with the diluted buffer 4 to protect the electrodes until the instrument is used again.

10.2.3.13. Click 'Start' to begin the run.

10.2.3.14. Once the run is finished, the report will automatically print.

10.2.4. Procedure for All forms of Alkalinity:

10.2.4.1. Follow procedures for Total Alkalinity. If pH of initial sample is greater than 8.3, titrate initially to a pH of 8.3 and record volume of titrant in logbook. This will be equal to P (or the phenolphthalein alkalinity) in the calculations. Then continue titrating to a pH of 4.5 and record in logbook.

11.0. Calculations / Data Reduction

11.1. Accuracy:

$$\text{LCS \% Recovery} = \frac{\text{observed concentration}}{\text{known concentration}} \times 100$$

11.2. Precision (RPD):

$$\text{Matrix Duplicate (MD)} = \frac{|\text{orig. sample value - dup. sample value}|}{[(\text{orig. sample value + dup. sample value})/2]} \times 100$$

11.3. Total Alkalinity:

$$\text{Alkalinity (mg/l } CaCO_3) = \frac{A \times N \times 50000}{B}$$

A = volume of acid, ml.
N = concentration of acid, N.
B = volume of sample, ml

11.4. $$\text{Alkalinity (mg/Kg } CaCO_3) = \frac{A \times N \times 50000}{B}$$

A = volume of acid, ml.
N = concentration of acid, N.
B = weight of sample in grams

11.5. Total Alkalinity low level, (< 20 mg/l)

$$\text{Alkalinity} = \frac{(2A - B) \times N \times 50000}{C}$$

A = vol. acid to pH = 4.5, ml.
B = vol. acid from pH = 4.5 to 4.2, ml.
C = vol. of sample, ml

11.6. Calculations for All Forms of Alkalinity:

11.6.1. Carbonate alkalinity is present when phenolphthalein alkalinity is not zero but less than total. Hydroxide alkalinity is present if phenolphthalein alkalinity is more than half the total. Bicarbonate alkalinity is present if phenolphthalein alkalinity is zero or less than half the total.

11.6.2. Calculate different forms of alkalinity using relationships in chart below:

Result of Titration	Hydroxide as CaCO3	Carbonate as CaCO3	Bicarbonate as CaCO3
P = 0	0	0	T
P < 0.5T	0	2P	T - 2P
P = 0.5T	0	2P	0
P > 0.5T	2P - T	2(T - P)	0
P = T	T	0	0

Where P = phenolphthalein alk. and T = total alk

11.7. PC-Titrator Calculation:

Total Alkalinity calculation at pH 4.5 and p-alk at pH 8.3 is automatically performed by the instrument. If alkalinity is < 20 ppm, it will apply the volume used at pH 4.2 in the calculation. Final results are reported in mg/L.

11.8. Exporting Data from PC titrate to TALS:

11.8.1. From the main menu, click "reporting" then click "prepare and/or print shazam reports."

11.8.2. Go to "File," then "open report." In "reports" folder, select "water analysis historical data report.srw." Click "open."

11.8.3. Select "filter on the left side of the grid and the "rundate" column and/or time "runtime" column. Click on each box to open filter screens and select appropriate date/time.

11.8.4. Click "preview report" tab at the top to display the chosen data.

11.8.5. Click "file" then "export." Click on "…" box next to the file name box. Go to the drop down menu by "look in" at the top and choose "c:" drive and choose "export data" folder. Type in the file name in the box at the bottom, then click "open."

11.8.6. Use the drop down menu under "file type" to select "FixedFieldASCII File (*.txt)." Click "ok."

11.8.7. Go to "shortcut to mantechFT.exe" icon on the desktop. Click "…" button next to the raw data box. Choose the appropriate file and click "open." On the left side, choose the appropriate analyte from "available analytes" box (i.e. FCO2 for carbon dioxide and for Alkalinity select all of the following: talk, bcarb, carb, hydrx).

11.8.8. Click "transfer file." The data will now be in TALS.

11.9. Data Reduction

11.9.1. All reagent information is recorded on the "batch information" page. Use the "worksheet" tab if additional pages are necessary.

11.9.2. Record special notes and observations on the "worksheet" tab (i.e. sample appearance and notes on why samples were rejected or diluted).

11.9.3. All raw data is attached as a pdf file. The raw data includes the instrument report and calibration curve.

11.9.4. The analyst must fill out the Wet Chem Data Review checklist (WI# EDS-WI-008) during the first level review. The batch is second level reviewed and the checklist is filed in the Wet Chemistry department.

12.0. Method Performance

12.1. Method Detection Limit Study (MDL)

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 the laboratory's MDL procedure in Section 19 (Test Methods and Method Validation) of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM). 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 unless method requirements require a greater frequency. MDLs are typically generated via the TestAmerica LIMS (TALS) control chart module.

12.2. Demonstration of Capabilities

For Demonstration of Capabilities (DOC) procedure refer to Section 19 in the most current revision of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM). DOCs are typically generated via the TestAmerica LIMS (TALS) control chart module.

12.3. Training Requirements

Refer to TestAmerica Edison SOP No. ED-GEN-022, Training, for the laboratory's training program.

13.0. 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 Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

14.0. Waste Management

14.1. 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 TestAmerica SOP No. ED-SPM-008 (Laboratory Waste Disposal Procedures).

14.2. The following waste streams are produced when this method is carried out:

- Acidic waste generated by the analysis is collected in a waste container which is periodically dumped down the sink with water.

15.0. References / Cross-References

15.1. Standard Methods for the Examination of Water and Wastewater, 22th Edition, American Public Health Association, American Washington, DC, 2012, SM 2320 B (Editorial Revisions, 2011).

15.2. TestAmerica Edison Document No. ED-QA-LQM, *Laboratory Quality Manual*, most current revision.

15.3. TestAmerica Edison SOP ED-GEN-022, *Training*, most current revision.

15.4. TestAmerica Edison SOP No. ED-SPM-008, *Laboratory Waste Disposal Procedures*, current revision.

15.5. TestAmerica Edison SOP No. ED-GEN-007, *Subsampling*, current revision.

15.6. TestAmerica Corporate Environmental Health and Safety Manual, Document No. CW-E-M-001, most current revision.

15.7. TestAmerica Edison Work Instruction # EDS-WI-008, Wet Chemistry Data Review Checklist, most current revision.

15.8. TestAmerica Edison SOP ED-WET-060, Analysis of pH for Waters and Drinking water Measured Electrochemically, most current revision.

16.0. Method Modifications:

Item	Method No.	Modification
Sec. 10.2.2.3 – 10.2.2.5	SM 2320B	Added procedure for the analysis of alkalinity in soil, following the procedure in SW846 Method 9040C (Analysis of soil and waste pH) where a 1:1 weight to volume (g/ml) is prepared, mixed, decanted and analyzed.

17.0. Attachments

None

18.0. Revision History

- Revision 9, dated 28 June 2013
  - Throughout document: Updated Standard Methods reference to currently implemented and accredited methods, SM 2320 B -11.
  - Throughout document: updated references to Lab Quality Manual section numbers as necessary.
  - Minor formatting edits throughout document
  - Sec. 9.1: Changed acceptance limits for LCS from 85-115% to vendor specified QC limits.
  - Sections 12.1 and 12.2: included statements referencing use of TALS control chart in the generation of MDLs and DOCs.

- Revision 8, dated 14 June 2011

  - Sec. 7.1.2: Added Ross pH electrode filling solution to list of reagents.
  - Sec. 7.1.3: Added pH buffers 4, 7 and 10 to list of reagents.
  - Sec. 9.1.1: Revised to include the corrective action taken if MB is greater than RL.
  - Sec. 9.1.2: Revised the corrective action taken if LCS is outside the acceptable range.
  - Sec. 10.1.1: Revised to include the checking of the sampler's flow rate as part of the daily instrument calibration.
  - Sec 10.1.4: Section added.
  - Sec 15: Added SOP No. ED-WET-060 in the list of references.

- Revision 7, dated 17 November 2010

  - Sec 3: Updated the LQM reference for the list of definitions.
  - Sec. 9.1 Table & 9.1.2: Revised LCS control limits to 85-115%.
  - Sec. 11.8: Added information on exporting data to TALS.
  - Sec. 11.9: Added data reduction section in accordance with TALS
  - Sec 15: Added applicable references.

- Revision 6, dated 10 November 2008

  - Revised SOP format in accordance with TestAmerica Corporate Quality SOP No. CW-QS-002 (Rev 0) Writing a Standard Operating Procedure (SOP).
  - Combined SOP ED-WET-040 (Analysis of Alkalinity in Soil) with this SOP (ED-WET-039). Retired SOP ED-WET-040 at the effective date of this SOP.
  - Sec. 1.1: Deleted method EPA 310.1 to comply with the Method Update Rule.
  - Added information for soil matrix where applicable (i.e. Sec. 1.1, Sec. 8, etc.).
  - Sec. 6: Deleted Erlenmeyer flasks & hotplate; added beakers & graduated cylinder
  - Section 7.2: Added shelf life and storage requirements.
  - Section 8 & 9: Reformat both sections into Table format.
  - Sec 9.1.2: Added the procedure for adjusting the flow rate of the sampler when LCS fails.
  - Sec. 10.2.2: Added the soil procedure as written in SOP No. ED-WET-040.
  - Sec 10.2.3: Changed the order of instrument operation to reflect actual laboratory procedure
  - Sec 10.2.3.5: Revised to include RINSE sample in the analysis log.
  - Sec 10.2.3.12: Expanded to include the use of Buffer 4 to fill the electrode cell.
  - Section 15: Deleted EPA reference 310.1; added applicable references
  - Sec. 16: Added method modification to include reason and reference for the modification.
  - Revised SOP Title to include Soil matrix

- Revision 5, dated 02 November 2007
  - Section 6 Standards. Revised the frequency for the standardization of titrants (0.10

N H2SO4 and 0.02N H2SO4 from two weeks to three months.

Title: Analysis of Total Hardness: EDTA Titration in Water by Standard Method 2340 C

Once printed, this is considered an uncontrolled document

Approvals (Signature/Date):

Jasmine Parillo — 03/06/12	Kene' Kasperek — 03/07/12
Jasmine Parillo — Date	Kene' Kasperek — Date
Department Manager	Health & Safety Manager /Coordinator
Carl Armbruster — 03/06/12	Ann Gladwell — 03/06/12
Carl Armbruster — Date	Ann Gladwell — Date
Quality Assurance Manager	Laboratory Director

1.0 Scope and Application

1.1. This SOP is applicable to the titrimetric analysis of hardness by Standard Method 2340 C. This method is applicable to drinking, surface, and saline waters, domestic and industrial wastes.

1.2. The method is suitable for all concentration ranges of hardness; however, in order to avoid large titration volumes, use a sample aliquot containing not more than 25 mg $CaCO_3$. The laboratory's reporting limit is 5.0 mg $CaCO_3$ /L.

1.3. On occasion clients may request modifications to this SOP. These modifications are handled following the procedures outlined in Sections 7 (Review of Work Request) and 19 (Test Methods and Method Validation) of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

2.0 Summary of Method

An aliquot of sample is titrated using Sodium EDTA at a pH of 10.0±0.1. Sodium EDTA when added to a solution with Calcium and Magnesium ion at pH of 10.0±0.1, and a small amount of dye such as Eriochrome Black T, the ions are complexed and the solution turns from wine red to blue marking the endpoint.

3.0 Definitions

For a complete list of definitions refer to Appendix 2 in the most current revision of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

4.0 Interferences

Some metal ions interfere with the titration by causing an indistinct end point. At a higher concentration of heavy metals, an alternate method should be chosen.

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001) 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 coats and closed-toe, nonabsorbent shoes are a minimum.

5.1. Specific Safety Concerns or Requirements

None

5.2. 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.
Ammonium Hydroxide	Corrosive Poison	50 ppm-TWA	Vapors and mists cause irritation to the respiratory tract. Causes irritation and burns to the skin and eyes.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

6.1. Instrumentation

- N/A

6.2. Supplies

- Burette, 10 ml and 25 ml (Class A)
- Cups
- Magnetic stirrer, Teflon coated magnets
- Eppendorf Pipettes, varying volumes
- various Volumetric Flasks (Class A): 100 mLs; 200 mLs; 500 mLs; 1000 mLs
- Erlenmeyer flask – for standardization
- pH paper, pH 10-12 (accuracy to 0.20)
- pH meter

7. Reagents and Standards

7.1. Reagents

7.1.1. Eriochrome Black T Indicator: Mix together 0.5 g Eriochrome Black T and 100 g NaCl. Prepare every 6 months; store at room temperature.

7.1.2. Sodium Chloride: Crystal AR; ACS grade. For stability and storage information, refer to manufacturer's instructions.

7.1.3. Ammonium Hydroxide Solution (3N): Transfer 210 ml of concentrated Ammonium Hydroxide to a 1000-ml volumetric flask. Dilute to volume with reagent water. Store at room temperature and discard after 6 months.

7.1.4. Hydrochloric Acid, ACS Grade (1:1): Transfer 250 ml of reagent water to a 1-L beaker. Slowly and with constant stirring add 250 ml of Hydrochloric Acid. If the solution becomes very hot or starts to boil or sputter, stop the addition immediately. Allow the solution to cool thoroughly before continuing. Transfer solution to a glass bottle. Store at room temperature and discard after 6 months.

7.1.5. Methyl Red Indicator, ACS Grade: Transfer 0.10 g of Methyl Red to a 100-ml volumetric flask. Dilute to volume with reagent water. Store in refrigerator and discard after 6 months.

7.1.6. Buffer Solution: Purchased pre-made; Fisher/certified APHA standard. Follow manufacturer's instructions for storage and stability. If laboratory prepared: dissolve 1.179 g of EDTA and 0.780 g of Magnesium Sulfate Heptahydrate in 50 ml of reagent water. Add this solution to 16.9 g of ammonium chloride and 143 ml of ammonium hydroxide in a 250-ml volumetric flask. Dilute to volume with reagent water. Store in a polyethylene bottle; stopper tightly to prevent loss of ammonia (NH3) or pick up of carbon dioxide (CO2). Discard after one month or when 2 ml added to a sample fails to maintain a pH of 10.0 +0.1 at the titration end point.

7.2. Standards

7.2.1. Standard Calcium Carbonate Solution (0.02N): Weigh 1.000 g of Calcium Carbonate into a 500-ml conical flask. Place a funnel in the erlenmeyer flask neck and add, a little at a time, 1:1 Hydrochloric Acid until all the Calcium Carbonate is dissolved. Add 200 ml of reagent water and boil for a few minutes to expel carbon dioxide. Cool, add a few drops of Methyl Red Indicator, and adjust to the intermediate orange color by adding 3N Ammonium Hydroxide Solution or 1:1 Hydrochloric Acid, as required. Transfer quantitatively to a 1000-ml volumetric flask and dilute to volume with reagent water. Store in a polyethylene container at room temperature and discard after 6 months.

7.2.2. EDTA Titrant, 0.02N: purchased commercially-Aqua Solutions/ACS grade. Follow manufacturer's instructions for storage and stability. Standardize EDTA every three months. If laboratory prepared: transfer 3.723 g of disodium ethylene diamine tetra-acetate dihydrate to a 1000-ml volumetric flask. Dilute to volume with reagent water. Store in a polyethylene container in a refrigerator and discard after 6 months. Check with standard calcium solution (Sec 7.2.1) by titration every two weeks because of gradual deterioration.

7.2.2.1. EDTA Standardization Procedure

Place 10.0 ml of standard calcium solution in a plastic cup containing about 50 ml of distilled water. Add 1 ml of buffer solution. Add a small scoop of Eriochrome Black T Indicator. Titrate slowly with stirring until the last reddish tinge disappears. Add the last few drops at 5 second intervals, until the end point blue color. Total titration time should be 5 minutes or less from the time of buffer addition.

$$\text{Normality of EDTA titrant} = \frac{Nc \times Vc}{Ve}$$

Where:

Nc = Normality of the Standard Calcium Carbonate Solution (0.02N)
Vc = volume of Standard Calcium Carbonate Solution used (10 ml)
Ve = volume of EDTA titrant used, ml

8. Sample Collection, Preservation, Shipment 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	Sample Container	Min. Sample Size	Preservation	Holding Time	Reference
Waters	Polyethylene, glass	50 mLs	HNO_3, pH < 2; Cool 4 ± 2°C	180 Days	40 CFR Part 136.3

9. Quality Control

9.1. Sample QC

9.1.1. Method Blank: A blank must be analyzed each time samples are analyzed. Deionized water is used for the blank and results must be below the reporting limit.

9.1.2. Laboratory Control Sample (LCS): An LCS must be analyzed each time samples are analyzed. The LCS for hardness is a whole volume quality control sample purchased from Environmental Resources Associates (ERA). Results must be within 85-115% of the certified value.

9.1.3. Matrix Duplicate: A duplicate is analyzed by using a second aliquot of sample. The relative percent difference (RPD) must be within laboratory generated control limits.

9.2. Instrument QC

N/A

10. Procedure

10.1. Sample Preparation

10.1.1. Allow all solutions and samples to come to room temperature before analysis.

10.2. Sample Analysis

10.2.1. Medium Level Procedure:

10.2.1.1. Prepare a reagent blank by transferring 50 ml of reagent water to a 125-ml Erlenmeyer flask.

10.2.1.2. Transfer 25 ml of sample to a 125-ml Erlenmeyer flask and dilute sample to 50 ml with de-ionized water.

10.2.1.3. Just prior to titration, add 1 to 2 ml of buffer solution (Sec 7.1.6). Usually 1 ml is sufficient to give a pH of 10.0 to 10.1. Verify that the sample pH is 10.0 ± 0.10 using pH papers. If necessary check sample pH with a pH meter.

10.2.1.4. Add a small scoop of dry Indicator (Sec 7.1.1).

10.2.1.5. Titrate the reagent blank and samples, completing each titration within 5 minutes of adding the buffer solution and Indicator Solution.

10.2.1.6. Record volume of sample used and titrant EDTA used in ml in TALS.

10.2.1.7. Repeat the titration for any samples requiring more than 15 ml of titrant using a smaller sample volume. Add sufficient reagent water to make a total volume of 50 ml before adding buffer solution and Indicator.

10.2.2. Low Level Procedure:

10.2.2.1. Prepare a reagent blank by transferring 100 ml of reagent water to a 250-ml Erlenmeyer flask.

10.2.2.2. Transfer 100 ml or 50 ml of sample to a 250-ml Erlenmeyer flask.

10.2.2.3. Just prior to titration, use proportionate amount of buffer and indicator: add 4 ml of Buffer Solution for 100 ml sample or 1 to 2 ml of buffer solution for a 50 ml sample. Usually 1 ml is sufficient to give a pH of 10.0 to 10.1. Verify that the sample pH is 10.0 ± 0.10 using pH papers. If necessary, check sample pH with a pH meter.

10.2.2.4. Add a scoop of dried powder indicator (Sec 7.1.1).

10.2.2.5. Titrate the reagent blank and samples to a true blue color endpoint, completing each titration within 5 minutes of adding the Buffer.

10.2.2.6. Record volume of sample used and titrant EDTA used in ml in TALS.

10.2.2.7. Repeat the titration for any samples requiring more than 5 ml of titrant using the medium level procedure.

11.0. Calculations / Data Reduction

11.1. Precision (RPD):

$$\text{Matrix Duplicate (MD)} = \frac{|\text{orig. sample value - dup. sample value}|}{[(\text{orig. sample value + dup. sample value})/2]} \times 100$$

11.2. Concentration =

$$\text{Hardness (EDTA) as mg } CaCO_3/L = \frac{Ne \times Ve \times 50000}{Vs}$$

Where:

Ne = Normality of EDTA titrant
Ve = Volume of EDTA titrant used, ml
Vs = Volume of sample, ml

11.3. Data reduction :

11.3.1. All reagent information is recorded on the “View Batch Information” page. Use the ‘Worksheet tab’ if additional pages are necessary.

11.3.2. Record special notes and observations in the “worksheet” tab (i.e. sample appearance and notes on why samples were rejected or diluted).

11.3.3. If any data is recorded in the logbook, it must be scanned and attached to the TALS batch as a pdf file.

11.3.4. Analyst must fill out the Wet Chem Data Review checklist (WI# EDS-WI-008) during the first level review. The batch is second level reviewed and the checklist is filed in wetchem department.

12.0. Method Performance

12.1. Method Detection Limit Study (MDL)

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 the laboratory's MDL procedure in Section 19 (Test Methods and Method Validation) of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM). 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 unless method requirements require a greater frequency.

12.2. Demonstration of Capabilities

For DOC procedure refer to Section 19 in the most current revision of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

12.3. Training Requirements

Refer to TestAmerica SOP No. ED-GEN-022, Training, for the laboratory's training program.

13.0. 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 Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

14.0. Waste Management

14.1. 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 TestAmerica SOP No. ED-SPM-008 (Laboratory Waste Disposal Procedures).

14.2. The following waste streams are produced when this method is carried out:

There are no special waste streams associated with this method.

15.0. References / Cross-References

15.1. Standard Methods for the Examination of Water and Wastewater , 18th Edition, American Public Health Association, Baltimore Maryland, 1992, SM 2340 C.

15.2. TestAmerica Edison Document No. ED-QA-LQM, *Laboratory Quality Manual*, most current revision.

15.3. TestAmerica Edison SOP ED-GEN-022, *Training*, most current revision.

15.4. TestAmerica Edison SOP No. ED-SPM-008, *Laboratory Waste Disposal Procedures*, current revision.

15.5. TestAmerica Edison Work Instruction # EDS-WI-008, Wetchem Data Review Checklist, most current revision.

15.6. TestAmerica Corporate Environmental Health and Safety Manual, Document No. CW-E-M-001, most current revision.

16.0. Method Modifications:

Item	Method No.	Modification
10.2.2.2 & 10.2.2.3	Standard Methods 2340C	A 50 ml sample volume can be used for low level hardness as long as the ratio of the sample volume and the buffer volume and indicator are maintained.

17.0. Attachments

N/A

18.0. Revision History

- Revision 5, dated 07 March 2012
  - Sec 1 & 12: Revised LQM reference to reflect the most current LQM revision.
  - Sec 6.2: Updated list of supplies.
  - Sec 10.2.1.3 & 10.2.2.3: Added procedure for checking sample pH after the addition of buffer. Subsequent sections adjusted accordingly.
  - Sec 15: Added applicable references.

- Revision 4, dated 04 March 2010

  - Revised SOP format in accordance with TestAmerica Corporate Quality SOP No. CW-QS-002 (Rev 0) Writing a Standard Operating Procedure (SOP).
  - Sec 1.1: Deleted reference to method 130.2 in accordance with the MUR; added reference SM 2340C.
  - Sec 2: Expanded method summary.
  - Sec 7: Added reagent grade, storage and stability information.
  - Sec 7.1: Deleted 1N Ammonium Hydroxide in the list of reagents. Also deleted text in the procedure: '*neutralize with 1N Ammonium hydroxide*,' (previously in sections 10.2.1.2 and 10.2.2.1). The use of this reagent was only referenced in Method 130.2.

- Sec 10.2.2.2 & 10.2.2.3: Revised to include 50 ml sample volume as an option and a note was added to use proportionate amount of indicator and buffer to the sample.
- Sec 11: Revised data reduction procedures in accordance with new TALS.
- Sec 15: deleted EPA method 130.2 on the list of reference; added applicable references.
- Sec 16: Added Method modification to include the use of 50 ml sample volume.
- Added revision history.

Title: Analysis of pH for Waters and Drinking water Measured Electrochemically by Method EPA 150.1, SM 4500 H $^{+}$B and SW-846 9040B/9040C

Once printed, this is considered an uncontrolled document

Approvals (Signature/Date):

Jasmine Parillo 08/02/12	Kene' Kasperek 08/03/12
Jasmine Parillo Date	Kene' Kasperek Date
Department Manager	Health & Safety Manager /Coordinator
Carl Armbruster 08/02/12	Ann Gladwell 08/02/12
Carl Armbruster Date	Ann Gladwell Date
Quality Assurance Manager	Laboratory Director

1.0 Scope and Application

1.1 Analytes, Matrix(s), and Reporting Limits

1.1.1 This SOP is applicable to the determination of pH in aqueous wastes and multiphase wastes where the aqueous phase constitutes at least 20% of the total volume using Method SW846 9040B/9040C and Standard Methods 4500 H^+ B. For the determination of pH in drinking water samples, use Method EPA 150.1.

1.1.2 On occasion clients may request modifications to this SOP. These modifications are handled following the procedures outlined in Sections 7 (Review of Work Request) and 19 (Test Methods and Method Validation) of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

2.0 Summary of Method

The pH of a water sample is determined electrometrically using a combination electrode.

3.0 Definitions

For a complete list of definitions refer to Appendix 2 in the most current revision of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

4.0 Interferences

Coatings of oily materials or particulates can impair electrode response. Any coatings may be removed by wiping and rinsing with distilled water. Additional cleaning may be necessary as stated in the manufacturer's manuals.

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001) 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 coats and closed-toe, nonabsorbent shoes are a minimum.

5.1. Specific Safety Concerns or Requirements

None

5.2. Primary Materials Used

There are no materials with a health rating of 3 or 4 used in this method. **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.

6.0 Equipment and Supplies

6.1 Instrumentation

- pH meter (capable of calibrating to pH buffers 4-10 range): Thermo Scientific, Orion Star LogR
- combination electrode: PerpHecT Ross Electrode

6.2 Supplies

- Temperature compensating probe or combination electrode capable of temperature compensation
- Specimen cups
- Teflon coated stir bars
- magnetic stirrers

7.0 Reagents and Standards

7.1. Reagents

7.1.1. Ross pH electrode filling solution, 3M KCl: Cat No. 810007; for storage and stability information, see manufacturer's instructions. Note: Do not use any filling solution that contains silver, as silver will damage the electrode.

7.1.2. pH cleaning solution: for storage and stability information, see manufacturer's instructions.

7.1.3. Reagent Water -18 megohm Reagent grade Type II water.

7.2. Standards

7.2.1. pH buffer solution of 2, 4, 7, 10, and 12.0: Purchased commercially; store at room temperature, for stability information refer to manufacturer's instructions.

8.0 Sample Collection, Preservation, Shipment 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	Sample Container	Min. Sample Size	Preservation	Holding Time	Reference
Waters	Glass or plastic	100 mLs	Cool 4 ± 2°C	Analyze immediately	40 CFR Part 136.3

9.0 Quality Control

9.1. Sample QC

9.1.1. Method Blank: A blank must be analyzed each time samples are analyzed. Use deionized water for the blank.

9.1.2. Laboratory Control Sample (LCSSRM): The LCS must be performed daily or for each batch of 20 samples, whichever is more frequent. The LCS is currently being purchased from an outside vendor, ERA. The results must be within vendor specified QC limits. If LCS result is not within the acceptable range, all samples associated with the LCS must be re-analyzed following an acceptable LCS.

9.1.3. Sample Duplicate*:* A duplicate is analyzed by using a second aliquot of sample. One duplicate sample must be analyzed for each batch of 20 samples. The relative percent difference (RPD) for wastewater and drinking water samples must be within laboratory generated control limits.

9.2. Instrument QC

9.2.1. Continuing Calibration Verification (CCV): A buffer check is analyzed after the initial calibration of the pH meter and after every tenth sample or every 3 hours, as applicable. If the pH buffer reading differs by more than 0.05 units, the pH meter must be recalibrated and all samples analyzed after the last good CCV must be reanalyzed.

10.0 Procedure

10.1. Sample Preparation

10.1.1. Allow samples to come to room temperature before measuring. Samples must be within 2 degrees of buffer solution temperature. Do not alter samples in any way by filtering or diluting.

10.2. Calibration

10.2.1. Calibrating the pH meter:

10.2.1.1. In the measurement mode, press “line select” button until the arrow icon points to the top line. Press the “arrow up” button until the **pH** icon is shown then press the “calibrate” button to begin calibration.

10.2.1.2. Rinse the electrode, and ATC probe if being used, with deionized water and place into the buffer 4.0.

10.2.1.3. Wait for the **pH** icon to stop flashing. When the **pH** icon stops flashing, the meter will display the temperature-corrected pH value for the buffer.

10.2.1.4. Press the “calibrate” button to proceed to the next calibration point.

10.2.1.5. Rinse the electrode, and ATC probe if being used, with deionized water and place into the buffer 10.0. Wait for the **pH** icon to stop flashing.

10.2.1.6. Press the “measure” button to save and end the calibration.

10.2.1.7. The meter will display the slope. Slope must be within 92-102%. Record the calibration data (i.e. 4.00, 10.00) and the slope in the appropriate pH meter calibration logbook.

10.2.1.8. Read back buffers 4.00, 7.00, and 10.00 in ‘analyzed mode’ to verify the calibration and record results in the calibration logbook.

10.2.1.9. The buffer solution must be +/- 0.05 standard units of the actual value.

10.3. Sample Analysis

10.3.1. Fill the specimen cup partially full with sample and add well-rinsed magnetic stir bar.

10.3.2. Place cup on stirrer and carefully lower probe into sample solution.

10.3.3. Gently stir the sample at a constant rate and monitor pH reading until stable. Do not entrain air in sample if possible.

10.3.4. Repeat above with fresh aliquot of sample and continue until the difference between sample readings is less than 0.1 pH units.

10.3.5. If the sample pH is greater than 10.0, a pH buffer of 12.0 is checked. **Note**: When measuring the buffer 12.0, ensure that it is measured at the manufacturer’s specified temperature of 25ºC.

10.3.5.1. If the pH is over 12.0, adjust the results by adding the correction value from the nomograph to the observed pH reading. See attachment 1 for the nomograph.

10.3.6. If sample pH is less than 4.0, a pH buffer of 2.0 is checked.

10.3.7. Thoroughly rinse and gently wipe the electrode between measurements. Rinse probe with acetone if grease or oil coats the glass electrode.

10.3.8. Record pH, temperature and the time of analysis in the pH logbook and in TALS.

11.0. Calculations / Data Reduction

11.1. Accuracy:

$$\text{ICV / CCV, LCS \% Recovery} = \frac{\text{observed concentration}}{\text{known concentration}} \times 100$$

11.2. Precision (RPD):

$$\text{Matrix Duplicate (MD)} = \frac{|\text{orig. sample value - dup. sample value}|}{[(\text{orig. sample value + dup. sample value})/2]} \times 100$$

11.3. Data Reduction

11.3.1. All data is recorded in the logbook and attached to the batch as a pdf file.

11.3.2. Record special notes and observations in the “worksheet” tab (i.e. sample appearance).

11.3.3. Record reagent information in the prep batch information (this can be viewed in the “batch information” page).

11.3.4. Analyst must fill out the Wet Chem Data Review checklist (WI# EDS-WI-008) during the first level review. After the batch is second level reviewed, the checklist is filed in wetchem department.

12.0. Method Performance

12.1. Method Detection Limit Study (MDL)

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 the laboratory’s MDL procedure in Section 19 (Test Methods and Method Validation) of TestAmerica Edison’s Quality Assurance Manual (ED-QA-LQM). 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 unless method requirements require a greater frequency.

12.2. Demonstration of Capabilities

For DOC procedure refer to Section 19 in the most current revision of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

12.3. Training Requirements

Refer to TestAmerica SOP No. ED-GEN-022, Training, for the laboratory's training program.

13.0. 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 Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

14.0. Waste Management

14.1. 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 TestAmerica SOP No. ED-SPM-008 (Laboratory Waste Disposal Procedures).

14.2. There are no specific waste streams generated with this method.

15.0. References / Cross-References

15.1. Methods for Chemical Analysis of Water and Wastewaters, EMSL-Cincinnati, EPA/600/4-79-020, March 1983 and 1979; Test Method 150.1.

15.2. Standard Methods for the Examination of Water and Wastewater , 18th Edition, American Public Health Association, Baltimore Maryland, 1992, SM 4500-H^+ B.

15.3. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, U.S. Environmental Protection Agency, Office of Solid Waste and Emergency Response. U.S. Government Printing Office: Washington, DC, SW846 Method 9040B, Revision 2, January 1995.

15.4. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, U.S. Environmental Protection Agency, Office of Solid Waste and Emergency Response. U.S. Government Printing Office: Washington, DC, SW846 Method 9040C, Revision 3, November 2004.

15.5. TestAmerica Edison Document No. ED-QA-LQM, *Laboratory Quality Manual*, most current revision.

15.6. TestAmerica Edison SOP ED-GEN-022, *Training*, most current revision.

15.7. TestAmerica Edison SOP No. ED-SPM-008, *Laboratory Waste Disposal Procedures*, current revision.

15.8. Orion PerpHecT ROSS Electrode User Guide-Thermo Electron Corporation.

15.9. TestAmerica Edison Work Instruction # EDS-WI-008, Wetchem Data Review Checklist, most current revision.

15.10. TestAmerica Corporate Environmental Health and Safety Manual, Document No. CW-E-M-001, most current revision.

16.0. Method Modifications:

N/A

17.0. Attachments

Attachment 1: Nomograph for pH values greater than 12.0

18.0. Revision History

- Revision 8, dated 03 August 2012
  - Sec 6.1: Replaced pH meter model # to Orion Star LogR - capable of calibrating to pH buffers 4-10 range.
  - Sec 10.2.1.1 and 10.2.1.6: Revised calibration procedures to reflect the new pH meter; subsequent sections adjusted accordingly.
- Revision 7, dated 19 June 2012
  - Revised throughout to include references to the most recent revision of the method, SW846 9040C, Revision 3, November 2004..
  - Updated references to Lab Quality Manual section numbers.
  - Revised Section 9.1.2 to read “The results must be within vendor specified QC limits.”

- Revision 6, dated 20 June 2011
  - Sec. 9.1.1: Removed text from MB section “the results must be less than the reporting limit.”
  - Sec. 9.1.2: Added the corrective action taken if LCS result is outside of the acceptable range.
  - Sec. 10.2.1.5: Added slope criteria.

- Revision 4, dated 09 October 2009
  - Revised SOP format in accordance with TestAmerica Corporate Quality SOP No. CW-QS-002 (Rev 0) Writing a Standard Operating Procedure (SOP).
  - Sec 1.1.1: Clarified which method is applicable to aqueous wastes (9040B & 4500 H B) and drinking water samples (150.1).
  - Sec 6.1.1: Replaced probe filling solution Saturated Ag/Ag Cl filling solution with 3M KCl.
  - Section 6.2.1: Changed the 12.45 pH buffer solution to 12.00 pH buffer
  - Section 7: Added storage and stability information.
  - Section 10.3.5: Added note that the buffer should be measured at the manufacturer's specified temperature of 25 deg C.
  - Section 10.3.5.1: Added nomograph information
  - Sec 11.3: Revised new data reduction procedures in accordance with new TALS
  - Sec. 15: Added applicable reference.
  - Sec 17: Added attachment, nomograph

- Revision 4b, dated 29 June 2009
  - Section 9.1: Added detailed procedure for calibrating the pH meter
  - Added text: *After calibrating the pH meter and prior to sample analysis, verify the calibration by reading back the 4.00, 7.00, and 10.00 buffers. Record readings in the analytical logbook.* Delete text: *After initial calibration, verify by recording values for all buffers used.*

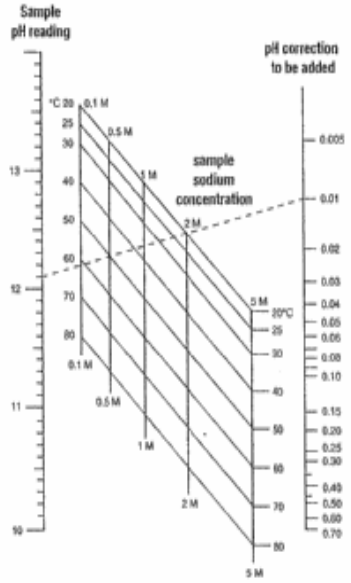
- Revision 3, dated March 2007
  - Section 1.1 and 15.2 Inserted 'B' in the Standard method reference (SM 4500 H^+) to indicate the complete method number (SM 4500 H^+ B).

Attachment 1

Nomograph for pH values > 12.0

Interferences

Sodium ion is the principal interference of the pH electrode, causing increasing error at higher pH (lower hydrogen ion activities) and at higher temperatures. Because the pH membrane is composed of special low sodium error glass, error due to sodium is negligible when measuring at pH values less than 12. When measuring at pH values greater than 12, add the correction value from the nomograph in **Figure 5** to observed pH reading.



Example:

pH reading	12.10
Sodium concentration	0.5 M
Temperature	50 °C
Correction	0.01
Corrected pH reading	12.11

18

***** Taken from Thermo Orion pH electrode Instruction manual

Title: TOC, Analysis of Total Organic Carbon in Aqueous Samples By Method No. EPA 5310B

Once printed, this is considered an uncontrolled document

Approvals (Signature/Date):

10/05/12	10/06/12
Mark Acierno Date	Kene' Kasperek Date
Operations Manager	Health & Safety Manager /Coordinator
10/05/12	10/05/12
Carl Armbruster Date	Ann Gladwell Date
Quality Assurance Manager	Laboratory Director

1.0 Scope and Application

1.1. This SOP describes the procedure to measure the organic carbon in drinking, surface and saline waters, domestic and industrial wastes using Standard Methods, 18th Edition,Test Method 5310B.

1.2. The carbonaceous analyzer measures all of the carbon in a sample. The manner of preliminary treatment of the sample and instrument settings defines the types of carbon being measured. Forms of carbon that are measured by this method are:

- Soluble, nonvolatile organic carbon such as sugars
- Soluble, volatile organic carbons such as mercaptans
- Insoluble, partially volatile carbons such as oils
- Insoluble particulate carbonaceous matter such as cellulose filters
- Soluble or insoluble carbonaceous matter adsorbed or insoluble organic suspended matter such as oily matter adsorbed on silt particles.

1.3. This procedure is applicable to homogeneous samples that can be injected by means of a microliter syringe.

1.4. The method detection limit is 1.0 mg/l total organic carbon. Samples with total organic carbon concentrations higher than 100 mg/l must be diluted first with deionized water.

1.5. On occasion clients may request modifications to this SOP. These modifications are handled following the procedures outlined in Sections 7 (Review of Work Request) and 19 (Test Methods and Method Validation) of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

2.0 Summary of Method

2.1. Organic carbon in a sample is converted to carbon dioxide by catalytic combustion. The CO_2 formed can be measured directly by a non-dispersive infrared gas analyzer. The NDIR outputs a detection signal that generates a peak whose area is proportional to the TOC concentration of the sample. Dissolved organic carbon is filtered through a 0.45um filter and preserved prior to sample analysis.

2.2. Inorganic Carbon is decomposed to become CO2 when a sample is introduced into an IC reactor vessel where a carrier gas is flowing. The IC concentration is determined similar to TOC run.

3.0 Definitions

For a complete list of definitions refer to Appendix 2 in the most current revision of

TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

4.0 Interferences

4.1. Carbonate and bicarbonate interference. Remove by acidification and purging sample with nitrogen.

4.2. Acidification and purging can result in the loss of volatile organic substances.

4.3. Homogeneity of samples can cause a problem if the sample cannot be readily injected into the instrument.

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001) 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 coats and closed-toe, nonabsorbent shoes are a minimum.

5.1. Specific Safety Concerns or Requirements

The auto sampler has a probe that is sharp; use caution not to stick yourself.

The furnace is very hot and can cause severe burns if touched.

5.2. 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
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.

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.			

6.0 Equipment and Supplies

6.1. Instrumentation

- Shimadzu TOC-V-CSH/CPN
- Shimadzu TOC-L
- Autosampler: ASI-V, SAI-L
- Analytical Balance
- Printer

6.2. Supplies

- Scintillation Vials
- Air, Ultra Grade 50 psig
- Eppendorf Pipettes, varying volumes
- Whatman syringe filters & 0.45 um syringe filters

7.0. Reagents and Standards

7.1. Reagents

7.1.1. Sulfuric Acid, conc.: Reagent grade.

7.2. Standards

7.2.1. Potassium Hydrogen Phthalate, KHP, stock (2000 mg/l TOC): Partially fill a 100-ml amber flask with deionized water and dissolve 0.426 g of potassium hydrogen phthalate (reagent grade). Bring to volume with deionized water. Store at 4 ° C. Prepare every six months.

7.2.2. Prepare a fresh 100 mg/l KHP standard solution by diluting 5 ml of 2000 mg/L stock solution (section 7.2.1) to a final volume of 100 ml with deionized water. Also prepare a 10 mg/l and a 1.0 mg/l KHP standard solution by diluting 10 ml and 1.0 ml of the 100 mg/l solution to a final volume of 100 ml with deionized water, respectively. Acidify all standard solutions and a 0-ppm standard with conc. H_2SO_4 to a pH of 2. Store at 4°C. Prepare every three months.

7.2.3. Potassium Hydrogen Phthalate, KHP, secondary stock (2000 mg/l TOC): prepared similar to section 7.2.1 but taken from a different manufacturer or lot number. Store at 4°C. Prepare every six months.

7.2.4. Inorganic Carbon stock solution (2000 mg/L): Dissolve 0.70 g of Sodium bicarbonate, $NaHCO_3$ and 0.882 g of Sodium Carbonate, Na_2CO_3 (heated for 1 hour at 285°C and cooled) in deionized water in 100 ml volumetric flask. Bring the volume to mark with DI water. Do not acidify, prepare every six months. Standard solution is used for TIC determination.
Prepare running standard solutions at 0, 1.0 ppm, 10.0 ppm and 100 ppm concentrations.

8.0. Sample Collection, Preservation, Shipment 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	Sample Container	Min. Sample Size	Preservation[1]	Holding Time	Reference
Waters	Polyethylene, glass	50 mLs	H_2SO_4 or HCl to pH < 2; Cool 4 ±2°C	28 Days	40 CFR Part 136.3

[1] Samples for TIC determination should be taken from the unpreserved sample.

Note: Samples for dissolved organic carbon must be filtered in the field before acid is added to the sample. If the sample is to be filtered in the lab, no preservative is added to the sample until the sample is filtered.

9.0. Quality Control

9.1. **Sample QC** - The following quality control samples are prepared with each batch of samples. Samples can be put into a batch for up to 2 weeks after the initial date; that is the date the first sample was analyzed. Up to 20 samples can be put in a batch. This does not include MS, MSD, Blank, and Laboratory Control Sample.

9.1.1. Method/ Preparation Blank: For every QC batch, prepare a Method Blank that consists of deionized water which is pH adjusted to ≤ 2.0 and is carried through the entire analytical procedure. This will identify any system/ process contamination. The results of this analysis must fall below the reporting limit.

9.1.2. Filtering Blank: If Dissolved Organic Carbon (DOC) is to be determined, analyze a blank sample which has been filtered through a 0.45 um-pore-diameter filter.

9.1.3. Matrix Spike and Matrix Spike Duplicate: Two portions of the same sample (matrix spike and matrix spike duplicate) are spiked with 50ppm (1.25 ml of 2000 ppm stock (section 7.2.1) is spiked into 50 ml volumetric flask and brought up to the final volume with the sample aliquot). The recovery for MS/MSD and RPD must be within laboratory generated control limits. See Section 11.0 to calculate recoveries.

9.1.4. Laboratory Control Sample (LCSRM): The laboratory control sample is run daily and is obtained from an outside vendor. It is used to measure method performance on the matrix being analyzed. The recovery must be within the manufacturer specified limits.

9.2. **Instrument QC**

9.2.1. Initial Calibration Verification (ICV), 50 mg/L: The curve must be verified by analyzing an Initial Calibration Verification solution immediately after the calibration curve. The ICV is approximately at mid-range standard (50 mg/L) and is prepared by adding 2.5 ml of the KHP, secondary source, (Sec. 7.2.3) into a 100 ml volumetric flask and brought to the mark with deionized water. The value obtained must not differ from the true value by more than 10%. If it does, the problem must be corrected, the instrument recalibrated and the ICV reanalyzed.

9.2.2. Continuing Calibration Verification (CCV), 50 mg/L: The validity of the calibration curve must be verified periodically during the analysis. A Continuing Calibration Verification is prepared the same way as ICV (see section 9.2.1). The solution must be analyzed following every ten samples; "samples" include matrix spike, matrix spike duplicate, laboratory blank, Laboratory Control Sample (LCS) and environmental samples. The value obtained for the CCV must not differ from the true value by more than 10%. If it does, the problem must be corrected and the previous ten samples reanalyzed following the last good calibration verification.

9.2.3. Initial and Continuing Calibration Blank (ICB/CCB): Following each calibration verification (CCV), a calibration blank must be analyzed. Use deionized water which is pH adjusted to ≤ 2.0. The results of this analysis must fall below the reporting limit.

10.0. Procedure

10.1. Equipment preparation:

10.1.1. Instrument:TOC-V-CSH/CPN and ASI-V

10.1.2.1. Turn on power of TOC-VCSH and ASI-V. Turn Air, Ultra Zero pressure up to 50 psig.

10.1.2.2. Allow instrument to stabilize for about 30 minutes until screen shows “Ready” (combustion temperature at 680°C).

10.1.2.3. On the computer desktop click on “TOC-Control V” icon. This will open the ‘Control V Sample Table’ window, click File then New.

10.1.2.4. Select “Sample Run.” This will bring up a system selection screen. For TOC determination, choose system “TOC-V CSH,” click “ok” and “save.” For TIC, select system “TOC-V W/ ASI.”

10.2. Calibration

10.2.1. Instrument: TOC-V-CSH/CPN and ASI-V

10.2.1.1. Prepare the standards as described in section 7.2.2 and begin setting up the instrument.

10.2.1.2. Select ‘Calibration Curve’ to run new calibration.

10.2.1.3. Click ‘Next’ twice and select ‘NPOC’ in the analysis box.

10.2.1.4. Uncheck ‘Zero shift’ box to disable, and check ‘Multiple injection’ box.

10.2.1.5. Enter file name in the format: HVYYMMDD (i.e. HV080403).

10.2.1.6. Click ‘Next’ and set the following conditions:

- Number of injections: 4
- Sparge time: 10 minutes

10.2.1.7. Enter the calibration concentration points in mg/L (0, 1.0, 10.0, and 100).

10.2.1.8. Click 'Next' twice then click 'Finish.'

10.2.1.9. From the drop down menu, click on 'File' then 'New.'

10.2.1.10. Select 'Sample Run' and click 'ok' twice.

10.2.1.11. Click on 'Save' to save the calibration curve.

10.2.1.12. Click on the #1 position (row 1). From the drop down menu, select 'Insert,' and 'Calibration curve.'

10.2.1.13. Double click on the calibration curve file just created.

10.2.1.14. Enter the position of each of the vials (i.e. 1-4), click 'ok.'

10.2.1.15. From the taskbar, click the 'Connect' (lightning) icon, then select 'Use Settings on PC.'

10.2.1.16. From the taskbar, click 'Start,' (traffic light icon), then click 'Standby' and 'ok.'

10.2.1.17. Uncheck 'External acid addition' box to disable, and then click 'Start.'

10.2.1.18. After completion of measurement, check calibration and linearity of range, correlation coefficient should be $\geq$ 0.997.

10.2.2. Calibration standards are prepared and analyzed every 3 months or when QC requirements fail, whichever is first.

10.3. Sample Analysis:

10.3.1. Instrument: TOC-V-CSH/CPN and ASI-V

10.3.1.1. Check that sample pH is less than 2 or acidify with H_2SO_4. Pour well-mixed samples into scintillation vials and position them in the autosampler. Be sure to fill all the vials between initial sample and final sample in the turntable without leaving any empty positions.

10.3.1.2. Follow Section 10.1.2.1-10.1.2.4 (Equipment preparation). Select 'Sample' and '2008 Data;' folder. The 2008 Data folder stores the data for the particular year.

10.3.1.3. The system will create a filename for the analysis (i.e. TOC-2008_04_03_14_41_49_0.t32). Click 'Save' to save the analysis on this file.

10.3.1.4. When the excel Table opens up, click 'Connect' icon and select 'Settings on PC.'

10.3.1.5. From the drop-down menu, select 'Insert' and 'Auto Generate.'

10.3.1.6. From the message box, click 'Method.' Select the appropriate method (i.e. 5310B).

10.3.1.7. Click 'Next,' then enter the No. of Samples and Start vial position.

10.3.1.8. Click 'Next' twice, then click 'Finish' and 'ok.'

10.3.1.9. From the taskbar, click the 'Start' icon then click 'Standby' and 'ok.'

10.3.1.10. Uncheck the 'External Acid addition,' and click 'Start.'

10.3.1.11. Take reading of sample from two separate aliquots and record the average of two results. Dilute samples that are over the calibration range and record dilution in TALS; denote dilution with @ symbol.

10.3.1.12. For TIC DETERMINATION:

Ensure that there is sufficient amount of 25% H3PO4 into the IC reagent container.

If IC reagent does not flow into the vessel, run the program for REGENERATION OF IC SOLUTION on MAINTENANCE SCREEN. Also, check the joint of IC reagent container for any leakage.

Follow section 10.3.2 for analysis.

10.3.1.13. For DOC Determination:

- If samples are lab filtered, the unpreserved sample must be filtered through a 0.45um filter.
  Note: Whatman 0.45um syringe filter must be rinsed with deionized water prior to use.
- Collect the required volume of filtrate and immediately acidify the filtrate with H2SO4 to a pH of <2.

11.0. Calculations / Data Reduction

11.1. Accuracy:

$$\text{ICV / CCV, LCS \% Recovery} = \frac{\text{observed concentration}}{\text{known concentration}} \times 100$$

$$\text{MS \% Recovery} = \frac{\text{(spiked sample) - (unspiked sample)}}{\text{spiked concentration}} \times 100$$

11.2. Precision (RPD):

$$\text{Matrix Duplicate (MD)} = \frac{|\text{orig. sample value - dup. sample value}|}{[(\text{orig. sample value + dup. sample value})/2]} \times 100$$

11.3. Concentration = mg/ L = A x B

Where:

A = sample concentration on instrument (mg/L)
B = dilution factor

11.4. Data Reduction

11.4.1. Instrument data is imported to TALS by following these steps:

11.4.1.1. Once the run is complete, highlight all samples in the sample table.

11.4.1.2. Go to 'File' then from the drop down menu, select 'ASCII export options,' then click 'Misc' tab and uncheck 'Export Strings in Quotation Marks.' Click 'Ok.'

11.4.1.3. Go to 'File' then from the drop down menu, select 'ASCII export.' This opens up a save window, choose 'C:/TALS Export.' Then click 'Open.' Enter the file name and click 'Save.'

11.4.1.4. From the desktop, choose 'TALS export' icon. Browse for the file by clicking on the '…' button and select the file, click 'Open.' Next, use the drop down menu to choose the analyte (i.e. TOC, TIC, or DOC). Then hit 'Transfer file.'

11.4.1.5. The data has now been transferred to TALS.

11.4.2 Record special notes and observations in the 'worksheet' tab (i.e. sample appearance and notes on why samples were rejected).

11.4.3 Record reagent information in the prep batch information (this can be viewed in the 'batch information' page).

11.4.4 All raw data is attached as a pdf file. The raw data includes the instrument report and calibration curve.

11.4.5 Analyst must fill out the Wet Chem Data Review Checklist (WI# EDS-WI-008) during the first level review. After the second level review, the checklist is filed in the wetchem department.

12.0. Method Performance

12.1. Method Detection Limit Study (MDL)

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 the laboratory's MDL procedure in Section 19 (Test Methods and Method Validation) of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM). 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 unless method requirements require a greater frequency.

12.2. Demonstration of Capabilities

For DOC procedure refer to Section 19 in the most current revision of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

12.3. Training Requirements

Refer to TestAmerica SOP No. ED-GEN-022, Training, for the laboratory's training program.

13.0. 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 Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

14.0. Waste Management

14.1. 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 TestAmerica SOP No. ED-SPM-008 (Laboratory Waste Disposal Procedures).

14.2. The following waste streams are produced when this method is carried out:

Waste TOC Acid: This material contains sulfuric acid is collected in 5 gallon poly containers at satellite accumulation and will be submitted for elementary neutralization using 50 % sodium hydroxide (Siedler Chemical SC-1824-03) and sodium bicarbonate (Siedler Chemical SC-0219-25) to a pH of 6 – 9 in the primary tank. Once pH has been established the primary tank is transferred through filter housing to a secondary tank. The pH is rechecked. If the pH is within specifications, the secondary tank is released to the municipal sewer system

15.0. References / Cross-References

15.1. Standard Methods for the Examination of Water and Wastewater , 18th Edition, American Public Health Association, Baltimore Maryland, 1992, SM 5310B.

15.2. Shimadzu TOC-5000/ASI-5000 Systems Manual.

15.3. TestAmerica Edison Document No. ED-QA-LQM, *Laboratory Quality Manual*, most current revision.

15.4. TestAmerica Edison SOP ED-GEN-022, *Training*, most current revision.

15.5. TestAmerica Edison SOP No. ED-SPM-008, *Laboratory Waste Disposal Procedures*, current revision.

15.6. TestAmerica Corporate Environmental Health and Safety Manual, Document No. CW-E-M-001, most current revision.

15.7. TestAmerica Edison Work Instruction # EDS-WI-008, Wetchem Data Review Checklist, most current revision.

16.0. Method Modifications:

None

17.0. Attachments

None

18.0. Revision History

- Revision 8, dated 08 October 2012
  - Added Sec 1.5; updated LQM reference in Sec 12 to reflect the most current LQM revision.
  - Deleted all references to TOC-5000 and ASI-5000 (i.e. equipment list, calibration procedure); sections adjusted where applicable.
  - Sec 6.1: Added TOC instrument Shimadzu TOC-L and autosampler SAI-L to list of equipment.
  - Sec 10.2.2 (previously sec 10.2.3): Revised preparation frequency for the calibration standards from 6 months to 3 months.
  - Sec 10.3.1.13 (previously Sec. 10.3.2.13): Added note that syringe filters must be rinsed with DI prior to use.

- Revision 7, dated 08/03/2010
  - Section 3: Updated the LQM reference for the list of definitions.
  - Sec. 9.2.3: Revised to include ICB; added acidified deionized water.
  - Added dissolved organic procedures in Sections 2.1, 6.2, 8.0 and 10.3.2.13.
  - Sec 11.4: Revised data reduction procedures in accordance with new TALS.
  - Sections 13 and 14: Sections updated to reflect the most current TestAmerica Corporate Quality SOP No. CW-QS-002 (Writing a Standard Operating Procedure (SOP)).
  - Sec. 15: Added applicable references.

- Revision 6, dated 04/07/2008
  - Updated to format as per TestAmerica SOP format.
  - Modified sample sparge time from 2 minutes to 10 minutes to comply with method 5310B, (Section 10.1.1.4).
  - Deleted instrument condition for soil; not applicable.
  - Added secondary stock solution, (section 7.2.3).
  - Added Filtering blank in sample QC when analyzing DOC (section 9.1.2).
  - Added procedure for the Calibration and Sample analysis for instrument TOC-V-CSH/CPN and ASI-V, (sections 10.2.2 and 10.3.2).

- Revision 5, dated 11/12/07
  - Section 1.1. Deleted reference to Method 415.1; USEPA Method Update Rule has removed the method from use.
  - Section 4. Apparatus and Materials. Added new TOC instrument and autosampler:
    4.3.ShimadzuTOC-V-CSH/CPN
    4.4. Autosampler ASI-V
  - Section 9. Procedure.
    Section 9.2. Added equipment preparation procedure for the Shimadzu TOC-V-CSH/CPN. Subsequent sections adjusted accordingly.
    Section 9.5. Added sample analysis procedure for the Shimadzu TOC-V-CSH/CPN. Subsequent sections adjusted accordingly
  - Section 15.1 References. Deleted EPA 600/4-79-020, Test Method 415.1 reference as a result of the Method Update Rule.

Title: TOC, The Determination of Total Organic Carbon in Solid Samples by Lloyd Kahn Method

Once printed, this is considered an uncontrolled document

Approvals (Signature/Date):

Jasmine Parillo	8/2/12 Date	Kene' Kasperek	8/2/12 Date
Department Manager		Health & Safety Manager /Coordinator	
Carl Armbruster	8/1/12 Date	Ann Gladwell	8/1/12 Date

1.0 Scope and Application

1.1 Analytes, Matrix(s), and Reporting Limits

This SOP is applicable to the determination of organic carbon in soil samples and ocean sediments using the Lloyd Kahn method. The minimum reporting value is 100 mg/kg.

On occasion clients may request modifications to this SOP. These modifications are handled following the procedures outlined in Sections 7 (Review of Work Request) and 19 (Test Methods and Method Validation) of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

2.0 Summary of Method

2.1. A sample is acidified and heated to allow inorganic carbon to effervesce as carbon dioxide.

2.2. The sample is then heated in oxygen to 900°C to allow complete combustion of carbon.

2.3. The compounds are swept by the carrier gas to a copper catalyst reduction reactor where excess oxygen is consumed.

2.4. The water is then trapped in a magnesium perchlorate filter.

2.5. Following the removal of water, the compounds are separated on a chromatographic column and a thermal conductivity detector detects the carbon dioxide.

3.0 Definitions

For a complete list of definitions refer to Appendix 20 in the most current revision of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

4.0 Interferences

4.1. Volatile organics may be lost in the removal of carbonates and bicarbonates resulting in a low bias.

4.2. Maintain the samples at 4°C and analyze within the holding time to minimize bacterial decomposition and volatilization of organic compounds.

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001) 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 coats and closed-toe, nonabsorbent shoes are a minimum.

5.1. Specific Safety Concerns or Requirements

The furnace is very hot and can cause severe burn if touched

5.2. 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
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.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

6.1. Instrumentation

- Thermo Electron FlashEA 1112 NC Soil Analyzer
- Printer
- Analytical Balance

6.2. Supplies

- Tin disks, 30 mm
- Forceps/ Spatula
- Dropper
- Hot plate set at 75°C
- Sealing Device
- Mortar and pestle

7.0 Reagents and Standards

7.1. Reagents

7.1.1. Phosphoric Acid solution, 1:1 by volume.

7.1.2. Oxygen: Ultra high purity

7.1.3. Helium: Ultra high purity

7.2. Standards

7.2.1. Aspartic acid, 36.09% Carbon, High range TOC, primary: CAS# 56-84-8; CE Elantech Inc. Refer to manufacturer's instructions for stability and storage information.

7.2.2. Aspartic acid, 36.09% Carbon, High range TOC, secondary: CAS# 56-84-8; CE Elantech Inc. Secondary source must have a Lot number different from the primary standard. Refer to manufacturer's instructions for stability and storage information.

7.2.3. Soil reference material 5G NC, 2.29%C, Low range TOC, primary: Product# 33840025; Thermo Scientific. Material is non- hygroscopic, store at 20-25°C. For stability information, refer to manufacturer's instructions.

7.2.4. Soil reference material 5G NC, 2.29%C, Low range TOC, secondary: Product# 33840025; Thermo Scientific. Secondary source must have a Lot number different from the primary standard. Material is non-hygroscopic, store at 20-25°C. For stability information, refer to manufacturer's instructions.

8.0 Sample Collection, Preservation, Shipment 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	Sample Container	Min. Sample Size	Preservation	Holding Time	Reference
Soils/ Sediments	Glass	3 grams	Cool 4 ± 2°C	14 Days	Lloyd Kahn Method

9.0 Quality Control

9.1 Sample QC - The following quality control samples are prepared with each batch of samples

9.1.1. Method Blank: A method blank must be analyzed each time samples are analyzed at a frequency of 1 per 20 samples. An empty sample tin disk is used as the method blank. The results must be below the reporting limit. If not, all samples associated with the MB must be re-analyzed.

9.1.2. Laboratory Control Sample: The LCS is obtained from an outside vendor and is used to measure method performance on the matrix being analyzed. The LCS is run daily or every 20 samples whichever is more frequent. Weigh approx. 100 mg of the LCS standard and analyze in the same manner as the sample. Select the appropriate standard for the appropriate range of standard. If the low range calibration is used, take the aliquot from the low range LCS and enter the LCS in TALS as "LLCS;" results will be compared against the QC limit type 'LLCSREC' in TALS. If the high range calibration is used, take the aliquot from the high range LCS and enter the LCS in TALS as "LCS;" results are compared to the QC limit type 'LCSREC' in TALS. The results must be within the vendor's certified limits. If not, all samples associated with the LCS must be re-analyzed.

9.1.3. Matrix Quadruplicate: One quadruplicate sample analysis is required per batch. Percent relative standard deviation (%RSD) of quadruplicate analysis must be below the control limits established by the laboratory. If the CCB is greater than the RL, all samples following the last good calibration blank must be re-analyzed.

9.2. Instrument QC

9.2.1. Initial Calibration Verification (ICV): The curve must be verified by analyzing an Initial Calibration Verification solution at the midpoint of the calibration range. Select the appropriate standard for the appropriate range of solid sample (Sec 7.2.2 for high range TOC or 7.2.4 for the low range TOC) and weigh 4-5 mg of the standard. The value obtained must not differ from the true value by more than 10%. If it does, the problem must be corrected, the instrument recalibrated, and the ICV reanalyzed.

9.2.2. Continuing Calibration Verification (CCV): To verify the validity of the calibration curve, a Continuing Calibration Verification (CCV) must be analyzed following every ten sample analyses. "Samples" include method blanks, LCS, matrix duplicates and environmental samples. Prepare the sample the same way as the ICV. The CCV source must be different from the primary standard. The value obtained for the CCV must be within 10% of the true value, otherwise the problem must be corrected and the previous ten samples reanalyzed following the last good calibration verification.

9.2.3. Initial and Continuing Calibration Blank (ICB/CCB): Following each calibration verification, a calibration blank must be analyzed. The results of this analysis must fall below the reporting limit. Use the empty sample tin disk for the Initial Calibration Blank and Continuing Calibration Blank.

10. Procedure

10.1. Equipment Preparation

10.1.1. Turn on oxygen gas pressure up to 300 kPa and helium pressure to 300 kPa.

10.1.2. Turn on power of FlashEA 1112 analyzer.

10.1.3. Double click on 'EA' icon button; then on 'NC-Soils' button and press 'OK.'

10.1.4. Open 'Edit Elemental Analyzer' icon, turn on the furnaces, press 'Send' then 'OK'.

10.1.5. Open 'View Elemental Status'. Once the instrument is at temperature, and the green ready light is on, auto adjust the detector to 1000 microvolts.

10.2. Calibration

10.2.1. Prepare and analyze calibration standards every 3 months or when QC requirements fail, whichever is first.

10.2.2. Press the 'Edit Sample Table' icon.

10.2.3. Press the 'Fill Sample Table' icon. Select a unique name for the 'File Name'. Select the 'Number of Samples' you want filled in and press 'OK'. The Eager 300 program will index the 'File Name' and 'Sample Name' according to the number of samples selected.

10.2.4. Bypass : Prepare a 'Bypass run' by weighing approximately 15 mg of standard into a tin disk. Seal standard and place in the autosampler.

10.2.5. Blank: Fold and seal an empty tin disk and place in the autosampler.

10.2.6. Calibration standards:

10.2.6.1. Click on 'Balance'. Press 'Receive weight from balance'. Weigh 2.00 mg of the standard (Sec 7.2.1 for High range TOC or 7.2.3 for low-range TOC). Wait for a stable reading, and then press the third leftmost 'Operating key 'of the balance. The reading of the sample weight is transmitted to the 'Sample table.' Seal and place the standard in the autosampler.

10.2.6.2. Repeat procedure # 10.2.6.1 for the 5.00 mg, 10.00 mg, 15.00 mg, 20.00 mg, & 25.00 mg standards.

10.2.7. Insert a blank line on the sample table after the calibration standards to stop the sequence.

10.2.8. Move 'Act' to the 'Bypass' sample row.

10.2.9. Press 'OK' to exit the sample table. Go to 'File' and save the method.

10.2.10. Click 'Run' under the drop down menu and select 'Start Sequence of Samples.' Click 'OK' and analysis will start.

10.2.11. To view the sample, click 'View' and select 'Sample Being Acquired.'

10.2.12. After the calibration is completely run, click on the 'Summary Results' icon to view data. Check calibration and linearity of range, correlation coefficient should be equal to or greater than 0.995.

10.3. Sample Analysis

10.3.1. Add 25-150 mg of the homogenized soil sample to a 20 mL glass septa vial. Add 1:1 Phosphoric Acid solution drop wise until the effervescence is no longer observed.

10.3.2. Place the vial on a 75 °C hot plate for 10-15 minutes or until the sample is completely dry.

10.3.3. Remove sample from the hot plate then place sample in dessicant to cool.

10.3.4. Using a glass stir rod, crush the sample in the vial until the sample is of a fine grain consistency. The sample is now ready for analysis. Note: % solid will be determined on a separate sample aliquot.

10.3.5. Open program 'Eager 300 EA 1112. Open file 'NC soil' then open the sample table.

10.3.6. Scan sample IDs or manually type them in the sample table. Enter CCV and CCB every 10 sample. Insert a blank line at the end of the sequence to stop the instrument from continuing on with the run.

10.3.7. Under the filename column, enter filename: YYMMDDsample order (i.e. 100624001…100624005).

10.3.8. Prepare each sample by packing the soil into each tin. Add at least 25 mg of soil and up to 150 mg of soil to the weigh disk. Place sample on the balance and press 'enter.' The sample weight will be uploaded into the sample table, column: weight.

10.3.9. Load sample into the autosampler and click 'Save Method.'

10.3.10. Under the Drop down menu, click 'Run' and select 'Run Sequence' or click '⇨,' then click 'start now.'

10.3.11. When all samples have been measured, go to the 'Summarize Results'. All sample measurement results will be displayed, then print.

10.3.12. Set instrument to Standby.

11.0. Calculations / Data Reduction

11.1. Accuracy:

$$\text{ICV / CCV, LCS \% Recovery} = \frac{\text{observed concentration}}{\text{known concentration}} \times 100$$

$$\text{MS \% Recovery} = \frac{\text{(spiked sample) - (unspiked sample)}}{\text{spiked concentration}} \times 100$$

11.2. Precision (% RSD):

$$\%RSD = \frac{100 \times s}{A}$$

Where: s = standard deviation (quadruplicate measurements)
A = mean average

11.3. TOC (mg/kg) = % Carbon X 10,000 / (% solids x 100)

NOTE: All dry weight corrections are made in LIMS at the time the final report is prepared.

11.4. Data reduction:

11.4.1. Instrument data is imported to TALS by following these steps:

11.4.1.1. Once the run is complete, highlight all samples in the sample table.

11.4.1.2. Go to 'File' then from the drop down menu, select 'ASCII export options,' then click 'Misc' tab and uncheck 'Export Strings in Quotation Marks.' Click 'Ok.'

11.4.1.3. Go to 'File' then from the drop down menu, select 'ASCII export.' This opens up a save window, choose 'C:/TALS Export.' Then click 'Open.' Enter the file name and click 'Save.'

11.4.1.4. From the desktop, choose 'TALS export' icon. Browse for the file by clicking on the '…' button and select the file, click 'Open.' Next, use the drop down menu to choose the analyte (i.e. TOC, TIC, or DOC). Then hit 'Transfer file.'

11.4.1.5. The data has now been transferred to TALS.

11.4.2 Record special notes and observations in the 'worksheet' tab (i.e. sample appearance and notes on why samples were rejected).

11.4.3 Record reagent information in the prep batch information (this can be viewed in the 'batch information' page).

11.4.4 All raw data is attached as a pdf file. The raw data includes the instrument report and calibration curve.

11.4.5 Analyst must fill out the Wet Chem Data Review Checklist (WI# EDS-WI-008) during the first level review. After the second level review, the checklist is filed in the wetchem department.

12.0 Method Performance

12.1. Method Detection Limit Study (MDL)

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 the laboratory's MDL procedure in Section 19 (Test Methods and Method Validation) of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM). 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 unless method requirements require a greater frequency.

12.2. Demonstration of Capabilities

For DOC procedure refer to Section 19 in the most current revision of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

12.3. Training Requirements

Refer to TestAmerica SOP No. ED-GEN-022, Training, for the laboratory's training program.

13.0 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 Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

14.0 Waste Management

14.1. 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 TestAmerica SOP No. ED-SPM-008 (Laboratory Waste Disposal Procedures).

14.2. There are no specific waste streams generated by this method.

15.0 References / Cross-References

15.1. "Determination of Total Organic Carbon in Sediment", Lloyd Kahn, Quality Assurance specialist, USEPA Region II, Edison, NJ, July 27, 1988.

15.2. FlashEA 1112 Elemental Analyzer Operating Manual, Thermo Electron Corporation, September 2003 Edition.

15.3. TestAmerica Edison Work Instruction # EDS-WI-008, Wetchem Data Review Checklist, most current revision.

11.3. TestAmerica Edison Document No. ED-QA-LQM, *Laboratory Quality Manual*, most current revision.

11.4. TestAmerica Edison SOP No. ED-GEN-022, *Training*, most current revision.

15.4. TestAmerica Edison SOP No. ED-SPM-008, *Laboratory Waste Disposal Procedures*, current revision.

15.5. TestAmerica Corporate Environmental Health and Safety Manual, Document No. CW-E-M-001, most current revision.

16. Method Modifications:

None

17. Attachments

None

18. Revision History

- Revision 5, dated 02 August 2012
  - Sec 1.1, 3 and 12: Revised LQM references to reflect the most current LQM revision.
  - Sec. 9.1.1: Added corrective action procedure when MB is greater than the RL.
  - Sec.9.1.2: Clarified procedures for low-range LCS and high-range LCS. Also, included the corrective action procedure when LCS is outside the acceptance limits.
  - Sec 9.2.3: Added corrective procedure when ICB/CCB is greater than RL.
  - Sec 10.2.6.1: Clarified sources for the cal standards (low and high range).

- Revision 4, dated 30 July 2010

  - Sec 7.1.1 & 10.3.1: Replaced 10% phosphoric acid solution with 1:1 phosphoric acid solution.
  - Sec 8: Added sediments as matrix.
  - Sec 10.2.1: Revised frequency of calibration.
  - Sec 10.3.4: Added notes on % solid determination.

- Revision 3, dated 29 June 2010

  - Revised SOP format in accordance with TestAmerica Corporate Quality SOP No. CW-QS-002 (Rev 0) Writing a Standard Operating Procedure (SOP).
  - Sec 6.2 & 10.3.2: Replaced oven with hotplate.
  - Sec 7: Revised to include all standards utilized for this SOP, including stability and storage information.
  - Sec 9: Updated Sample and Instrument QC to include spiking procedures.
  - Sec 10.3: Updated section to reflect actual lab procedure.
  - Sec 11.2: Added % RSD calculation for the quadruplicate measurements.
  - Sec 15: Added applicable references.
  - Added this Revision history.

- Revision 2, dated 11 July 2007

  - Section 9.2.4. Sample Analysis:Detailed procedure for sample preparation such as the amount of sample to weigh, drying time, stirring and crushing of sample is inserted in now identified as section 9.2.4.1 thru 9.2.4.4, and section 9.2.4.7. Subsequent section numbers adjusted.
  - Section 9.2.4.10. (previously identified as section 9.2.4.5.). Revised to include the formula used for calculating the final results.

SAP Worksheet #15 -- Reference Limits and Evaluation Table
(UFP-QAPP Manual Section 2.8.1)

Matrix: SOIL
Analytical Group: LCMS PFCs

Analyte	CAS Number	Project Action Limit (ug/Kg)	Project Action Limit Reference[1]	Project Quantitation Limit Goal (ug/Kg)	Laboratory-specific[2]	
					QLs (ug/Kg)	MDLs (ug/Kg)
Perfluorobutane Sulfonate (PFBS)	29420-43-3				0.8	0.14
Perfluorobutyric acid (PFBA)	375-22-4				0.8	0.12
Perfluorodecane Sulfonate (PFDS)	67906-42-7				0.8	0.30
Perfluorodecanoic acid (PFDA)	335-76-2				0.8	0.27
Perfluorododecanoic acid (PFDoA)	307-55-1				2.0	0.57
Perfluoroheptanoic acid (PFHpS)	375-85-9				0.8	0.12
Perfluorohexane Sulfonate (PFHxS)	108427-53-8				0.8	0.12
Perfluorohexanoic acid (PFHxA)	307-24-4				0.8	0.15
Perfluorononanoic acid (PFNA)	375-95-1				0.8	0.22
Perfluorooctane Sulfonamide (FOSA)	754-91-6				0.8	0.098
Perfluorooctanoic acid (PFOA)	335-67-1				0.8	0.23
Perfluorooctanoic Sulfonate (PFOS)	1763-23-1				0.8	0.14
Perfluoropentanoic acid (PFPA)	2706-90-3				0.8	0.24
Perfluorotetradecanoic acid (PFTeA)	376-06-7				2.0	1.0
Perfluorotridecanoic acid (PFTriA)	72629-94-8				0.8	0.32
Perfluoroundecanoic acid (PFUnA)	2058-94-8				0.8	0.32

[1]List the type and source of the PAL used for each matrix specific analyte (e.g. Background, HH-MCL, HH-region III RBC, eco-WQC, eco-Region III BTAG, etc.)

[2]Laboratory-specific MDLs and QLs are limits that an individual laboratory can achieve when performing a specific analytical method. MDLs may be subject to update.

SAP Worksheet #15 -- Reference Limits and Evaluation Table
(UFP-QAPP Manual Section 2.8.1)

Matrix: Water
Analytical Group: LCMS PFCs

Analyte	CAS Number	Project Action Limit (ug/L)	Project Action Limit Reference[1]	Project Quantitation Limit Goal (ug/L)	Laboratory-specific[2]	
					QLs (ug/L)	MDLs (ug/L)
Perfluorobutane Sulfonate (PFBS)	29420-43-3				0.02	0.00824
Perfluorobutyric acid (PFBA)	375-22-4				0.02	0.00980
Perfluorodecane Sulfonate (PFDS)	67906-42-7				0.02	0.00915
Perfluorodecanoic acid (PFDA)	335-76-2				0.02	0.00782
Perfluorododecanoic acid (PFDoA)	307-55-1				0.03	0.0149
Perfluoroheptanoic acid (PFHpS)	375-85-9				0.03	0.0132
Perfluorohexane Sulfonate (PFHxS)	108427-53-8				0.03	0.00697
Perfluorohexanoic acid (PFHxA)	307-24-4				0.02	0.00291
Perfluorononanoic acid (PFNA)	375-95-1				0.04	0.0174
Perfluorooctane Sulfonamide (FOSA)	754-91-6				0.02	0.00571
Perfluorooctanoic acid (PFOA)	335-67-1				0.02	0.00979
Perfluorooctanoic Sulfonate (PFOS)	1763-23-1				0.03	0.0133
Perfluoropentanoic acid (PFPA)	2706-90-3				0.03	0.0109

Perfluorotetradecanoic acid (PFTeA)	376-06-7				0.03	0.0147
Perfluorotridecanoic acid (PFTriA)	72629-94-8				0.04	0.0177
Perfluoroundecanoic acid (PFUnA)	2058-94-8				0.02	0.00689

[1]List the type and source of the PAL used for each matrix specific analyte (e.g. Background, HH-MCL, HH-region III RBC, eco-WQC, eco-Region III BTAG, etc.)

[2]Laboratory-specific MDLs and QLs are limits that an individual laboratory can achieve when performing a specific analytical method. MDLs may be subject to update.

SAP Worksheet #24 -- Analytical Instrument Calibration Table
(UFP-QAPP Manual Section 3.2.2)

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria[1]	Corrective Action (CA)	Person Responsible for CA	SOP Reference[2]
PFCs	ICAL. Minimum of five points initial calibration for all target analytes	Initial calibration prior to sample analysis. Perform instrument re-calibration once per year minimum.	Coefficient of calibration: r ≥0.990 linear. r^2≥0.990 2nd order. RSD <15%	Instrument and standards are checked. Correct problem. Repeat initial calibration	Lab Manager/ Analyst	DV-LC-0012
	ICV. Initial calibration verification from a 2nd source.	Immediately following minimum five-point initial calibration	All analytes within 70-130% recovery	Correct problem then repeat initial calibration	Lab Manager/ Analyst	DV-LC-0012
	Detection Limit Calibration Verification (DLCK)	0.2 ug/L standard analyzed	70 - 130%	Correct problem then repeat initial calibration	Lab Manager/ Analyst	DV-LC-0012
	CCV. Continuing calibration verification	50 µg/L and 100 µg/L alternately before sample analysis and after every 10 samples	70 - 130%	Recalibrate the instrument; reanalyze any samples associated with failed CCV. If CCV > 130% and samples ND, the samples can be reported flagged without reanalysis.	Lab Manager/ Analyst	DV-LC-0012
	IS. Internal Standard recovery.	All samples and standards	50 - 150% Historical limits – see individual analytes for specific limits.	Re-extract if no matrix effect is obvious. Dilute if matrix effect is obvious.	Lab Manager/ Analyst	DV-LC-0012
	Mass calibration with PEG or other appropriate material bracketing mass calibration range	Annually or after major maintenance or if large amounts of drift are observed in the tune.	+/- 0.5 amu for all compounds	Re-calibrate	Lab Manager/ Analyst	DV-LC-0012
	Tune (while infusing or evaluation of analyte response in low standard). Check mass of spectral ion intensities.	Prior to ICAL	+/- 0.5 amu for all compounds	Perform mass calibration	Lab Manager/ Analyst	DV-LC-0012

1- This is a summary of the acceptance criteria; refer to the method SOP for specific or more information.
2- SOPs are reviewed/revised on an annual schedule. The current version will be followed at the time of sample receipt.

SAP Worksheet #28 -- Laboratory QC Samples Table

(UFP-QAPP Manual Section 3.4)

Matrix	Water / Soil
Analytical Group	LCMS
Analytical Method / SOP Reference[2]	PFCs DV-LC-0012

QC Sample	Frequency/ Number	Method/SOP QC Acceptance Limits[1]	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blank	1/Batch (20 samples)	No Target Compounds>1/2RL; no common lab contaminants >RL.	If sufficient sample is available, reanalyze samples. Qualify data as needed. Report results if sample results >10x blank result or sample results ND.	Analyst / Section Supervisor	Accuracy/Bias-Contamination	No Target Compounds>1/2RL; no common lab contaminants >RL.
Laboratory Control Sample	1/Batch (20 samples)	Refer to the DV-LC-0012 Attachment for LCS control limits.	If sufficient sample is available, reanalyze samples. Qualify data as needed.	Analyst / Section Supervisor	Accuracy/Bias	Laboratory % Recovery Control Limits
Matrix Spike/Matrix Spike Duplicate	1/Batch (20 samples)	Refer to the DV-LC-0012 Attachment for LCS control limits.	Determine root cause; flag MS/MSD data; discuss in narrative.	Analyst / Section Supervisor	Accuracy/Bias/ Precision	Laboratory % Recovery / RPD Control Limits
Surrogates	Every sample and standards	Refer to the DV-LC-0012 Attachment for Surrogate control limits.	Check calculations and instrument performance; recalculate, reanalyze.	Analyst / Section Supervisor	Accuracy/Bias	Laboratory % Recovery Control Limits

1- This is a summary of the acceptance criteria; refer to the method SOP for specific or more information.

2- SOPs are reviewed/revised on an annual schedule. The current version will be followed at the time of sample receipt.

APPENDIX B

FIELD SAMPLING PLAN

APPENDIX B
FIELD SAMPLING PLAN

Perfluorinated Compounds Work Plan

Prepared for
Solvay Specialty Polymers USA, LLC
10 Leonard Lane
West Deptford, NJ 08086

Prepared by

200 Harry S. Truman Pkwy.
Suite 330
Annapolis, MD 21401

November 15, 2013

CONTENTS

LIST OF FIGURES

LIST OF TABLES

ACRONYMS AND ABBREVIATIONS

COC	chain-of-custody
DGPS	differential global positioning system
FSP	field sampling plan
GPS	global positioning system
HSP	health and safety plan
Integral	Integral Consulting Inc.
MUA	Municipal Utility Authority
NJDEP	New Jersey Department of Environmental Protection
NOAA	National Oceanic and Atmospheric Administration
PFC	perfluorinated compound
QA/QC	quality assurance/quality control
QAPP	quality assurance project plan
Site	West Deptford, New Jersey, Plant
Solvay	Solvay Specialty Polymers USA, LLC
SOP	standard operating procedure

1 INTRODUCTION

This document presents the field sampling plan (FSP) that has been prepared on behalf of Solvay Specialty Polymers USA, LLC (Solvay) to study perfluorinated compounds (PFCs) at the West Deptford, New Jersey, Plant site (the Site). Additional information on the Site history and a summary of existing data are provided in the work plan.

The 234-acre Site is located at 10 Leonard Lane in West Deptford Township, Gloucester County, New Jersey, on the Delaware River across from the Philadelphia Airport. Active plant operations occur on approximately 34 acres of the property, with the remainder either in a natural state or used for agriculture. The far northern area of the site contains dredge spoils placed there by the U.S. Army Corps of Engineers in the 1960s (ERM 2013). The western edge of the property borders Little Mantua Creek, with undeveloped property located to the east, and a rail line to the south.

Prior to 1970 the property was used for agriculture. Fluorocarbon manufacturing began when Pennwalt constructed a facility in 1970. Manufacturing ceased in 1977 and a new facility was constructed from 1983 to 1985; production of vinylidene fluoride monomers and polymers using PFCs in the manufacturing process started in 1985 (ERM 2013). The facility was purchased by Elf Atochem in 1989 and operated until it was sold to Ausimont USA, Inc. in 1990. The Solvay Group acquired the holding of the parent company of Ausimont in May 2002, and changed the name to Solvay Solexis, Inc. on January 1, 2003. The company and facility name were then changed to Solvay Specialty Polymers USA, LLC on October 31, 2012 (ERM 2013).

The primary objective of the work plan is to determine if PFCs are present in Site groundwater and/or surface water and sediments in the Delaware River.

To execute this investigation, Integral Consulting Inc. (Integral) will conduct the fieldwork and data analysis outlined in this FSP and in the quality assurance project plan (QAPP). The names and quality assurance responsibilities of key project personnel for Integral who will be involved in sampling and analysis activities are provided in Figure A-1 of the QAPP.

1.1 OVERVIEW

The sampling design as provided in the work plan incorporates a number of different components and sampling media. The sampling design can be summarized as follows:

- **Groundwater**—Groundwater samples will be collected at 31 existing monitoring wells and tested for the analytes presented in Table B-1. These wells were chosen to provide a representative set of wells that capture the various aquifer units and subdivisions with a lateral spatial extent sufficient to determine the presence and extent of PFCs in

groundwater beneath the site. The wells that will be sampled during this field sampling event were selected from upgradient, within the axes, at the lateral bounds of the property, and along a transect of the downgradient property boundary.

- **Surface Water**—Surface water samples will be collected from 26 stations in the Delaware River. The sampling stations were placed at six locations that were previously sampled by the Delaware River Basin Commission in 2007-2009 (MacGillivray 2012), at three additional locations to provide greater spatial coverage in the Delaware River, two locations at the confluence of adjacent creeks with the Delaware River, and one location offshore of a Publically Owned Treatment Works outfall adjacent to the Site.
- **Sediment**—Surface sediment will be collected from 15 stations in the Delaware River and subsurface sediment will be collected from an additional 11 stations. Surface water and sediment stations will be co-located.

1.2 DOCUMENT ORGANIZATION

This FSP describes the field methods that will be used to collect samples to meet the objectives of the work plan. The background, rationale, data quality objectives, and overall study design are described in detail in the QAPP. Section 2 of this FSP describes the field procedures and sample packaging and shipping requirements that will be followed by the technical team during the field study. Section 3 summarizes field documentation and chain-of-custody (COC) procedures. Field data reporting and field custody procedures are discussed in Section 4.

The following documents are provided as attachments to this FSP:

- Site Health and Safety Plan (HSP). This document describes the specific requirements and procedures that will be implemented to minimize the safety risk to personnel who carry out the field study program for groundwater, surface water, and sediment collection (Attachment B1).
- Standard Operating Procedures (SOPs). The SOPs describe the procedures that will be used to collect groundwater, surface water, and surface and subsurface sediments (Attachment B2).
- Field Forms. This attachment contains examples of various forms that will be used during field sampling, including a corrective action record, a field change request form, and a COC form (Attachment B3).

2 SAMPLING PROCEDURES

The following sections describe the detailed procedures and methods that will be used during the proposed field sampling events, including sampling procedures, recordkeeping, sample handling, storage, and field quality control procedures. Sample collection and processing will be conducted in accordance with the SOPs provided in Attachment B2. Depending on field conditions, procedures specified in the referenced SOPs may be modified if necessary. All field activities will be conducted in accordance with the HSP that is provided as Attachment B1.

2.1 SCHEDULE

The perfluorinated compounds work plan and associated QAPP and FSP will be submitted to the New Jersey Department of Environmental Protection (NJDEP) for review on November 15, 2013. Pending approval of the work plan by NJDEP, Integral, on behalf of Solvay, will commence onsite groundwater sampling and surface water and sediment sampling in the Delaware River. The onsite groundwater sampling event and the surface water and sediment sampling event are each expected to take 2 weeks. Laboratory analytical time will be completed in 10 days. An additional 5 weeks will be required after laboratory analysis for data validation (Figure B-1).

Sampling of Municipal Utility Authority (MUA) wells has already begun under the FSP addenda, starting with West Deptford. Sampling at six additional identified MUAs will occur after personnel are contacted to schedule sampling and obtain information on well construction, status of operation, procedures for handling purge water of inactive wells, and site-specific health and safety considerations.

2.2 FIELD EQUIPMENT AND SUPPLIES

Field equipment and supplies include sampling equipment, decontamination supplies, sample containers, coolers, shipping containers, log books and forms, personal protection equipment, and personal gear. Protective wear (e.g., gloves) is required to minimize the possibility of cross-contamination between sampling locations. Additional information on protective wear required for this project is provided in Attachment B1.

Sample jars, preservatives, distilled/deionized water, coolers, and packaging material for the samples will be supplied by the analytical laboratory. Pursuant to the NJDEP sampling guidance (NJDEP 2005), sample containers will not be held for more than 4 days between sample container shipment from the testing laboratory and sample shipment back to the laboratory for analysis. Details on the numbers and type of sample containers are provided in the QAPP and in Table B-2 of this FSP. The field lead and field personnel in charge of sample

handling in the field will use a sample matrix table (Tables B-3, B-4, and B-5) as a quality control check to ensure that all samples have been collected at a given station. These tables include the total number and type of sample jars required for each analysis at each sampling station.

Commercially available, pre-cleaned jars will be used for the samples, and the testing laboratories will maintain a record of certification from the suppliers. The bottle shipment documentation will include batch numbers. With this documentation, jars can be traced to the supplier, and bottle-wash analysis results can be reviewed. The bottle-wash certificate documentation will be archived in Integral's project file.

Sample containers will be clearly labeled at the time of sampling. Labels will include the task name, sample number, sampler's initials, analyses to be performed, and sample date and time. Sample numbering and identification procedures are described in detail in Sections 3.3 and 3.4.

2.3 STATION POSITIONING

Latitude and longitude coordinates will be obtained at each station where samples are collected on the Delaware River. A differential global positioning system (DGPS) will be used to document the sample collection locations. The standard projection method to be used during field activities is New Jersey state plane feet (NAD83); coordinates will be collected in the field using WGS84. The positioning objective is to accurately determine and record the positions of all sampling locations to within ±2 m. Proposed sediment sampling location coordinates are provided in Table B-6.

The DGPS unit consists of a global positioning system (GPS) receiver and a differential receiver located at a horizontal control point. At the control point, the GPS-derived position is compared with the known horizontal location, offsets or biases are calculated, and the correction factors are telemetered to the GPS receiver. Positioning accuracies on the order of ±1 to 3 m can be achieved by avoiding the few minutes per day when the satellites are not providing the appropriate quality of signal (SOP AP-06). The GPS unit provides the operator with a listing of the time intervals during the day when accuracies are decreased. Avoidance of these time intervals permits the operator to maintain better positioning accuracy (SOP AP-06).

2.4 SAMPLING VESSEL

Surface water and sediment samples will be collected from a sampling vessel. The sampling boat or barge will have enough space to process the sediment cores onboard and accommodate a minimum of five people—three sampling team members, the vessel's operator, and one NJDEP oversight individual (if required)—and the following gear: hydraulic (or similar) winch and cable capable of deploying a vibracorer and van Veen grab sampler (or similar equipment) for sediment collection, sample collection and compositing equipment, sample coolers, and

multiple sampling equipment boxes containing sample jars and other ancillary equipment. The vessels used for sampling will have navigational lights, anchors, and basic sonar (e.g., fathometer). The vessel operator will be thoroughly familiar with the area of the river to be navigated.

2.5 WEATHER

During sampling activities weather using the following web site:

Weather conditions and forecasts: National Oceanic and Atmospheric Administration (NOAA) site for the Philadelphia area http://graphical.weather.gov/sectors/phi.php#tabs.

2.6 SPECIAL SAMPLING CONCERNS

Because PFCs are also found in numerous everyday items, the following special precautions will be taken during all sampling activities:

- No use of Teflon®-containing materials (e.g., Teflon® tubing, bailers, tape, sample jar lid liners, plumbing paste)
- No Tyvek® clothing will be worn onsite
- Clothes treated with stain- or rain-resistant coatings will be avoided or go through several washings prior to use onsite
- No Post-It® notes will be brought onsite
- No fast food wrappers, disposable cups or microwave popcorn will be brought onsite
- After handing any of the above items, field personnel's will wash their hands thoroughly with soap and water prior to any sampling activities
- No use of chemical (blue) ice packs or foil will be allowed.

Nitrile gloves will be worn during all sample collection activities.

2.7 EQUIPMENT DECONTAMINATION

2.7.1 Groundwater

Prior to pump installation and collection of the each groundwater sample, the pump will be decontaminated and fitted with new quarter-inch polyethylene tubing, eliminating the need for field decontamination of the tubing.

Pump decontamination procedures are as follows:

1. Disassemble pump and dispose of used bladder
2. Tap water rinse
3. Vigorously scrub all parts with brush and laboratory-grade standard detergent (e.g., Alconox® or Liquinox®)and tap water
4. Generous rinse with tap water or store-purchased distilled /deionized water
5. Isopropyl alcohol rinse and air dry followed by rinse with laboratory-grade distilled/deionized water
6. Air dry
7. Install new bladder in the pump
8. Reassemble pump

Water level measuring tapes and the flow-through cell will be decontaminated following above procedures in steps 2 through 6.

2.7.2 Surface Water

Before surface water sampling begins at a location, the water collection bottle (e.g., Niskin or Go-Flo bottle, or similar) will be rinsed with river water, scrubbed with a standard detergent (e.g., Alconox® or Liquinox®), rinsed with river water to remove all detergents, and set aside to drain. The water collection bottle will be rinsed with site water three times immediately prior to collecting the sample. Non-dedicated sampling equipment will be decontaminated following procedures in SOP SW-01 (Attachment B2), except that no solvent or acid rinses will be used.

If the water level at a given station is not deep enough to use a water collection bottle (i.e., nearshore samples), then a peristaltic pump with disposable tubing will be used. Dedicated or disposable quarter-inch polyethylene tubing will be used to collect the samples. This will eliminate the need for field decontamination of the tubing.

2.7.3 Sediment

Before surface sediment sampling begins at a location, the grab sampler will be scrubbed with a standard detergent (e.g., Alconox® or Liquinox®), rinsed with water (river, tap, or deionized water), air-dried, and rinsed with river water. Equipment used for compositing the sediment samples (i.e., stainless-steel pot, bowls, and spoons) will follow the same basic decontamination sequence, except that the final rinse will be with laboratory-grade distilled/deionized water. After cleaning, the decontaminated stainless-steel spoon will be placed inside the decontaminated stainless-steel pot and will be covered with the stainless-steel lid for the pot to protect it from possible contamination.

Prior to subsurface sampling, all core liners will be washed in sequence with a standard detergent (e.g., Alconox® or Liquinox®), rinsed with laboratory grade distilled/deionized water, and then air-dried. During storage and transport, decontaminated core liners will be capped at both ends to prevent contamination.

All non-dedicated sampling equipment that comes into contact with the sediment samples (e.g., core catchers, grab samplers, core liners, stainless-steel bowls, pots, and spoons) will be decontaminated prior to use and between samples. Non-dedicated sampling equipment will be decontaminated following procedures in SOP SD-01 (Attachment B2), except that no solvent rinse will be used.

2.8 GROUNDWATER SAMPLING

As discussed previously in Section 1.1, groundwater samples will be collected from selected Site wells for onsite sampling at the Solvay facility (Figure B-2). Samples will be collected directly into bottles provided by the certified contract laboratory from the sample port on the flow-through cell.

Low-flow purging and sampling methods will be employed to collect groundwater samples from Site wells identified in Table B-7. This method was selected based on prior use at the Site to collect volatile organic compound samples, reduced production of purge water, and limited disruption to groundwater conditions during sampling. Field procedures are to follow NJDEP and U.S. Environmental Protection Agency guidance (NJDEP 2005; USEPA 2010). Unlike prior sampling events, no Teflon® disposable bladders or Teflon®-lined tubing will be used to minimize risk of PFC contamination from the sampling equipment. Easy to disassemble and clean low-flow bladder pumps with disposable polyethylene bladders will be employed for sample collection (e.g., Solinst 407 Integra). Dedicated or disposable quarter-inch polyethylene tubing will be used to avoid the need for decontamination of tubing.

Pump intakes will be placed at the midpoint of the well screens and pumps will be secured with non-Teflon® coated cord.

2.8.1 Stabilization Parameters

As per NJDEP guidance, stabilization parameters will be monitored during purging to determine if well stability has been achieved prior to sampling. Purging will continue until respective measurements fall within the ranges stated below for three consecutive measurements or 4 hours have elapsed, at which time a sample will be collected and the attempts to reach stabilization documented. Measurements will be collected every 5–6 minutes.

Drawdown	<0.3ft
pH	± 0.1 unit

Specific Conductance	± 3%
Temperature	± 3%
Dissolved Oxygen	± 10%
Turbidity	± 10% for values > 1 NTU
ORP/Eh	± 10 millivolts

Care will be taken to minimize the length of tubing between the top of the well casing and the flow-through chamber. A low volume transparent flow-through cell will be used to allow visual observation of water conditions during purging. The flow-through cell will be connected such that water enters the bottom of the cell and exits the top.

Prior to connection of the flow-through cell, tubing will be flushed for up to 10 minutes while initial drawdown measurements are made. Probes used in the flow-through cell will be calibrated in the field prior to the day's sampling event by personnel certified for the collection of stabilization parameters in New Jersey.

Water level measurements will be recorded prior to pump installation. Following pump installation the water level probe will be suspended in the well at the point representing 0.3-ft drawdown. Water level readings will be recorded every 5 minutes at the time stabilization parameters are measured.

Pump installation will occur following initial water level measurements. Prior to installation, the pump will be properly decontaminated and fitted with appropriate tubing. Once the pump is prepared for installation, the pump will be lowered in a manner to minimize any disturbance to the well by slowly lowering the pump to the target sampling interval.

The purge rate will be set initially within a range of 100 mL/min to 500 mL/min. The initial few minutes of purge water will be discharge to the waste water container prior to connection to the flow-through cell. Tubing will be connected to the cell with the pump continuing to operate. If drawdown measurements indicate greater than 0.3 ft of drawdown, the pumping rate will be decreased, but not to less than 100 mL/min. Due to the highly permeable nature of the aquifer being sampled, it is not anticipated that drawdown to the well screen will occur. Following initial adjustments to the pumping rate, stabilization parameters will begin to be recorded.

When water quality parameters have stabilized, sample collection will proceed from the needle valve at the sample port.

2.9 SURFACE WATER SAMPING

Surface water samples will be collected outside the main channel, near the shoreline, and will avoid areas impacted by dredging activity in the river. Surface water samples will be collected at mid-depth relative to river bottom at each station. Water depth will be measured with a lead

line from the boat deck or with a fathometer mounted on the research vessel's hull. Surface water samples will be collected by using either water collection bottles without Teflon® lining (e.g., Niskin or Go-Flo bottles, or similar) or a peristaltic pump and tubing. Both methods will provide acceptable samples and they may be used interchangeably by the field crew depending on field conditions.

The preferred sampler is a water bottle because a large volume of water can be collected quickly. However, if there is insufficient water depth at a given station to use a water bottle (i.e., nearshore samples), then a peristaltic pump with disposable tubing will be used. Sampling methods using water bottles and peristaltic pumps with tubing are described in SOPs SW-05 and SW-04, respectively.

As stated above, the sample matrix table (Table B-4) shows the total number of sample jars for each analysis needed at each sampling station. Integral's field lead and field personnel in charge of sample handling will use this table as a quality control check to ensure that all samples at a given station are collected and that the appropriate sample container is used for each sample.

2.9.1 Sample Handling

Gloved hands are required for sample collection and handling, as described above. Field staff will wear appropriate non-contaminating, disposable, powderless nitrile gloves during the entire sampling operation. The field sampling team will change gloves frequently and before each surface water sample is collected.

Gloved hands are required for all operations that involve equipment that comes into contact with the surface water sample, including the following activities:

- Handling the sample bottle
- Handling the discharge end of the water collection bottle or sample tubing.

The surface water samples will be placed in labeled, laboratory-cleaned sample containers (Table B-2). Each sample container will be clearly labeled with the task name, sample number, type of analysis to be performed, date and time, and initials of person(s) preparing the sample. Immediately after sample containers are filled, the samples will be stored on wet ice (4 ± 2°C); no chemical (blue) ice packs will be used during the sampling event. The sampling team leader is responsible for maintaining sample integrity throughout the sampling event.

2.9.2 Water Quality Measurements

In addition to surface water collection, general water quality parameters (i.e., water temperature, pH, dissolved oxygen, salinity, oxidation-reduction potential, turbidity, and

conductivity) will be measured *in situ* at all sampling locations using a multi-probe (e.g., YSI 650/6600). An extra, sacrificial 500-mL high-density polyethylene aliquot of water will be collected at each surface water station (from mid-depth), kept at ambient temperature, and the sonde will be inserted completely within the sample until a stable reading is retrieved by the meter.

The multi-probe will be calibrated at the beginning of every day according to manufacturer specifications (see user manual). The name(s) of the person(s) performing the calibration will be recorded in the field logbook that is used during the sampling event. Calibration records must be kept so the results of the water quality monitoring are traceable. Information and general instructions for field measurement of water quality parameters are provided in SOP SW-06.

2.10 SEDIMENT SAMPLING

As discussed previously in Section 1.1, surface and subsurface sediment chemistry samples will be collected from historic Delaware River stations (Figure B-3) and from the Delaware River near the Site (Figure B-4). Sediment samples will be collected outside the main channel, near the shoreline, and will avoid areas impacted by dredging activity in the river. The following sections describe the sampling equipment, sampling methods, sample handling, and shipping for sediments.

2.10.1 Surface Sediment Sampling

Surficial sediment samples (0–6 in.; 0–15 cm) will be collected at 15 stations with a stainless-steel van Veen grab sampler (or equivalent type of equipment) depending upon the conditions encountered in the field. Sediment samples will be collected in accordance with standard methods used by USEPA (1997). Methods for surface sediment sampling are provided in SOPs SD-04 and SD-13.

One surface sediment sample will be collected at each location. A minimum of three grabs will be collected at each station to create a composite sample of surface sediment. The holding time requirements for the sediment samples following field collection are specified in Table B-2. The samples will be analyzed for the target analytes listed in Table B-1. Additional sediment from each station will be archived for possible future analysis, if necessary.

Material collected with the grab sampling device will be evaluated by the Integral field lead for acceptability using the following criteria:

- The sampler is not overfilled
- Overlying water may be present
- The overlying water (if present) is not excessively turbid

- The sediment surface is relatively undisturbed
- An adequate penetration depth is attained (i.e., to enable sampling of the undisturbed surface sediment).

If a sample fails to meet any of the above criteria, it will be rejected and discarded away from the station. Removal of material from the sample will be documented in the field log book.

After a sediment sample is judged to be acceptable, any overlying water will be siphoned off and the upper 6 in. (15 cm) of sediment will be collected in accordance with guidelines (USEPA 1997). Decontaminated stainless-steel spoons will be used to collect the sediment from the grab sampler. A stainless-steel ruler will be used to ensure that the sampling criterion for adequate penetration depth has been met and that the correct amount (i.e., 6 in. [15 cm]) of sediment has been removed from the grab sampler.

Surface sediments from the grab samples will be placed into a decontaminated, stainless-steel pot and homogenized using a stainless-steel spoon or other stainless-steel mixing implement until the sediment attains a visually uniform color and texture. The sediment sample in the pot will be covered with the pot's lid until a sufficient volume of sediment (approximately 1 L per station) is collected. Sediment subsamples will then be removed for the various kinds of laboratory analyses and for archiving.

The composite surface sediment samples will be placed in labeled, laboratory-cleaned sample containers without Teflon®-lined lids (Table B-2). Each sample container will be clearly labeled with the task name, sample number, type of analysis to be performed, date and time, and initials of person(s) preparing the sample. Containers that will be frozen (i.e., archived samples) will have 0.5–1 in. (1.3–2.6 cm) of headspace above the sediment to prevent the jars from breaking during storage at the laboratory. Immediately after sample containers are filled, the samples will be stored on ice (4 ± 2°C); no chemical (blue) ice packs will be used during the sampling event.

As stated above, the sample matrix table (Table B-5) shows the total number of sample jars for each analysis needed at each sampling station. Integral's field lead and field personnel in charge of sample handling will use this table as a quality control check to ensure that all samples at a given station are collected and that the appropriate sample container is used for each sample.

2.10.2 Subsurface Sediment Sampling

During subsurface sediment sampling, river gauge height and tides will be monitored using the following web sites:

- Real-time information on wind direction, wind speed, and river elevation: U.S. Geological Service (USGS - NWIS) http://waterdata.usgs.gov/nwis/sw

- Tides: NOAA site at http://tidesandcurrents.noaa.gov/.

Subsurface sediment will be collected at 11 stations using a coring device (e.g., vibracorer with core catcher, an impact coring device, piston core, or equivalent type of equipment) depending on conditions encountered in the field. Sampling methods for subsurface sediment sampling are provided in SOPs SD-08, SD-12, and SD-13, respectively.

A minimum of one core will be collected at each station. If sample volume requirements dictate the need for additional sediment, then additional co-located core(s) (or a grab sample if additional sediment is needed for the surface interval only) will be collected and the sediment will be composited. The holding time requirements for the sediment samples following field collection are specified in Table B-2. The samples will be analyzed for the target analytes listed in Table B-1. If there is a sufficient volume of sediment within a core interval, then additional sediment from each interval will be archived for possible future analysis, if necessary.

A minimum diameter of 3 in. (7.6 cm) will be used for all cores. The following sediment intervals in the core will be sampled: 0–6 in. (0–15 cm), 6–12 in. (15–30 cm), 12–24 in. (30–61 cm), 24–36 in. (61–91 cm), and >36 in. (>91 cm) to refusal or to a maximum depth of 6 ft (2 m). Any separate sediment horizons that are observed in the core will be noted on the field form (Attachment B3), but will not alter these collection intervals. Sediment will be collected from the entire sediment interval and a discrete sample from the composited, homogenized sediment will be collected. Shorter core lengths will be accepted if native materials are encountered, based on visual inspection of the core, or if multiple attempts (i.e., two attempts) at coring a given sampling location do not provide the anticipated core length.

The core's position will be monitored by observing the angle of the winch line while the corer is being lowered in the water column. When the inlet of the corer is approximately 2 m above the sediment, the corer will stop being lowered, the boat location confirmed, and the angle of the hydrowire determined. When the angle of the hydrowire is less than 5 degrees, the corer will be lowered into the sediment at a rate of 30 cm/s or less. If the weather is windy or tidal conditions warrant it, the boat will be anchored before the core is lowered. Cable will be released through the winch until there is slack in the line. If the boat drifts significantly (e.g., because of wind or tidal conditions), slack in the line will be permitted only briefly to prevent pulling the corer out at an angle.

The corer will be retrieved at a controlled rate to minimize agitation of the core. Retrieval will be stopped as soon as the top of the corer reaches the water surface. If a core catcher is not installed at the bottom end of the core, a plug may be inserted in the bottom end of the corer to prevent the core from slipping out when the corer is raised out of the water. The corer will be brought on board the sampling vessel and immediately stabilized to prevent it from tipping or falling. Care will be taken at all times to keep the corer in a vertical position. After the corer is secured onboard the sampling vessel, the Lexan® or polyethylene (or similar) liner that contains the sample will be removed from the corer barrel and inspected.

Each core will be evaluated by Integral's field lead for acceptability using the following criteria:

- The sediment surface is relatively undisturbed
- Any overlying water is not excessively turbid

At least 80 percent core recovery relative to penetration is achieved.

If a sediment core fails to meet any of the above criteria, it will be rejected.

If less than 80 percent core recovery versus penetration is achieved, the recovered core will be retained but considered insufficient, and another attempt to recover a sediment core at the same location will be conducted. If the specified penetration depth is not achieved after two attempts, the station may be relocated slightly. If the slight relocation of the station does not improve the penetration depth, the station may be temporarily abandoned and Integral's project manager will be notified.

After the cores have been collected, both ends of the cores designated for chemical analysis will be securely capped; labeled with the station identifier, core section, and sediment orientation; and fastened in an upright position. The overlying water will be siphoned or drained off.

Processing of the core may occur either on the sampling vessel or at a specified location onshore. The core liner will be laid out horizontally on a clean work surface. The content of the core will either be extruded with a plunger onto a clean sheet of polyethylene plastic sheeting (e.g., visqueen) or the core liner will be cut lengthwise and the core split open. All cores will be placed next to a tape measure and a station identifier and photographed. Cores will be inspected for physical characteristics and described on a core profile form (see Attachment B3).

Sediment touching the sides of the core tube will be excluded from each sample. The sediment from each core section will be homogenized with a decontaminated stainless-steel mixing implement (e.g., spoon) until the sediment attains a visually uniform color and texture. The sediment sample in the decontaminated stainless-steel bowl until a sufficient volume of sediment (approximately 1 L per core interval) is collected. Sediment subsamples will then be removed for the various kinds of laboratory analyses and for archiving.

The subsurface sediment composite samples will be placed in labeled, laboratory-cleaned sample containers without Teflon®-lined lids (Table B-2). Each sample container will be clearly labeled with the task name, sample number, type of analysis to be performed, date and time, and initials of person(s) preparing the sample. Containers that will be frozen (i.e., archived samples) will have 0.5–1 in. (1.3–2.6 cm) of headspace above the sediment to prevent the jars from breaking during storage at the laboratory. Immediately after sample containers are filled, the samples will be stored on ice (4 ± 2°C); no chemical (blue) ice packs will be used during the sampling event.

As stated above, the sample matrix table (Table B-5) shows the total number of sample jars for each analysis needed at each sampling station. Integral's field lead and field personnel in charge of sample handling in the field will use this table as a quality control check to ensure that all samples at a given station are collected and that the appropriate sample container is used for each sample.

2.11 FIELD QUALITY CONTROL

Field quality control samples will be used to assess sample variability and evaluate potential sources of contamination. The types of quality control samples that will be collected for during the proposed sampling events are described in this section. Detailed information on quality assurance and quality control (QA/QC) procedures, limits, and reporting are described in detail in the QAPP. The estimated numbers of field quality control samples to be collected are listed in the sample matrix tables (Tables B-3, B-4, and B-5). If quality control problems are encountered, they will be brought to the attention of Integral's quality assurance coordinator. Corrective actions, if appropriate, will be implemented to meet the task's data quality indicators.

2.11.1 Groundwater

Field quality control samples for the groundwater investigation will include field duplicates, equipment blanks, travel blanks, field blanks, and decontamination water blanks for non-laboratory supplied water. The procedures and rationale for collecting these samples are described below.

- **Field duplicate** – Sample will be used to assess the variability in concentrations of co-located samples due to the combined effects of sample processing in the field and laboratory as well as chemical analysis. A blind field duplicate sample will be collected for at least 5 percent of groundwater samples. Samples will be assigned unique numbers and will not be identified as field duplicates to the laboratory.
- **Equipment blank** – Sample will be collected to help identify possible contamination from the sampling environment or from the sampling equipment (e.g., pump). Equipment blanks will be collected for at least 10 percent of the groundwater sampling stations.

  The equipment blank will be collected by filling a 1000-mL decontaminated, graduated cylinder with distilled/deionized water supplied by the laboratory performing the analysis. A decontaminated pump will be placed into the graduated cylinder with sample tubing and plumbing fittings attached. The required field blank samples will then be collected by operating the pump. As water is removed from the cylinder, additional distilled/deionized water will be added as needed until sufficient sample

volume has been collected to perform the required analyses. Due to the larger water volumes required, field blank water may not be supplied in the same identical containers as the sample being collected, as provided for by NJDEP guidance for low-flow purging and sampling.

- **Travel (or trip) blank** – Sample will be used to determine whether or not contaminants may have been introduced during the shipment of the groundwater samples from the field to the laboratory for PFC analyses only. Trip blanks will be prepared at the testing laboratory by pouring distilled/deionized water into two, 250 mL bottles and tightly closing the lids. Each container will be inverted and tapped lightly to ensure no air bubbles exist.

  The trip blanks will be transported unopened to and from the field in the cooler with the PFC samples. A trip blank will be labeled and placed inside the cooler that contains newly collected PFC samples and it remains in the cooler at all times. A trip blank must accompany samples at all times in the field. One trip blank will be sent with each cooler of samples shipped to the testing laboratory for PFC analysis.

- **Field (or environmental) blank** – Sample will be prepared in the field to evaluate potential background concentrations present in the air and in the distilled/deionized water used for the final decontamination rinse. Field blanks will be collected at a minimum frequency of 10 percent.

  An environmental blank in the field will be prepared by opening the laboratory-prepared sample bottle while at a sample collection site, filling the sample bottle with distilled/deionized water, and then sealing it. The environmental blank will be assigned a unique sample number, and the labeled bottle will be sent to the laboratory with the field samples.

- **Decontamination water blank** – Sample of the water used for the gross decontamination step of the groundwater sampling equipment. This blank will be collected once during the groundwater sampling event.

2.11.2 Surface Water

Field quality control samples for the surface water investigation will include the same types of samples as used for groundwater, except without the decontamination water blank. The procedures and rationale for collecting these samples are described below.

- **Field duplicate** – Sample used to assess the variability in concentrations of co-located samples due to the combined effects of sample processing in the field and laboratory as well as chemical analysis. A blind field duplicate sample will be collected at a minimum

frequency of 5 percent and will not be identified as a quality control sample to the laboratory.

- **Equipment blank** – Sample will be collected to help identify possible contamination from the sampling environment or from the sampling equipment (e.g., water collection bottle). Equipment blanks will be collected for at least 10 percent of the surface water sampling stations.

  The equipment blank will be collected by rinsing the decontaminated sample collection equipment with distilled/deionized water and collecting the rinsate.

- **Travel (or trip) blank** – Sample will be used to help identify whether or not contaminants may have been introduced during the shipment of the aqueous samples from the field to the laboratory for PFC analyses only. Trip blanks are prepared at the testing laboratory by pouring distilled/deionized water into two, 250 mL bottles and tightly closing the lids. Each container will be inverted and tapped lightly to ensure no air bubbles exist.

  Collection procedures for trip blanks are discussed in the groundwater section above.

- **Field (or environmental) blank** – Sample will be prepared in the field to evaluate potential background concentrations present in the air and in the distilled/deionized water used for the final decontamination rinse. Field blanks will be collected at a minimum frequency of 10 percent.

  Collection procedures for environmental blanks are discussed in the groundwater section above.

2.11.3 Sediment

Field quality control samples for sediment samples will include field split samples, equipment blanks, and filter blanks. The procedures and rationale for collecting these samples are described below.

- **Field duplicate** – Samples will be used to assess the variability in concentrations of co-located samples due to the combined effects of sample processing in the field and laboratory as well as chemical analysis. Blind duplicate (field split samples) will be collected at a minimum frequency of 10 percent. Samples will be assigned unique numbers and will not be identified as field splits to the laboratory. Field duplicate samples will be collected from both surface and subsurface sediment samples for chemical analysis. A minimum of one field split sample will be collected for each kind of sample collected.
- **Equipment blank** – Samples will be collected to help identify possible contamination from the sampling environment or from the sampling equipment (e.g., stainless-steel

grab sampler, coring device, spoons, bowls, and pots). Equipment blanks will be generated at approximately 10 percent of the sediment sampling stations at a minimum. Field equipment blanks will be collected from equipment used to collect both surface and subsurface sediment samples for chemical analysis. All equipment samples will be clearly noted in the field log (e.g., sample identifier, equipment type, date and time of collection, analysis, and filter lot number).

The equipment blank will be collected by rinsing the decontaminated sample collection equipment (grab sampler or corer and sample compositing equipment [stainless-steel bowl or pot, and spoon]) with distilled/deionized water and collecting the rinsate.

2.12 SAMPLE PACKAGIN AND TRANSPORT

As mentioned above, sample coolers and packing materials will be supplied by the analytical laboratories. Individual sample jars will be labeled and placed into plastic bags and sealed. Samples will then be packed in a cooler lined with a large plastic bag. Glass jars will be packed to prevent breakage and separated in the cooler by bubble wrap or other shock-absorbent material. Ice in sealed plastic bags will then be placed in the cooler to maintain a temperature of approximately 4°C (±2°C). When the cooler is full, the COC form will be placed into a zip-locked bag and taped to the inside lid of the cooler. A temperature blank will be added to each cooler. Each cooler will be sealed with two COC seals, one each on the front and side of the cooler. Labels indicating "This End Up" with an arrow and "Fragile" will be attached to each cooler.

The shipping containers will be clearly labeled (i.e., name of task, time and date container was sealed, person sealing the cooler, and company name and address) for positive identification. These packaging and shipping procedures are in accordance with U.S. Department of Transportation regulations (49 CFR 173.6 and 49 CFR 173.24). Coolers containing samples for chemical analyses will be transported to the laboratory by courier or overnight shipping service.

After the samples have been received by the laboratory, they will be stored under refrigeration (4 ± 2°C). Archive sediment samples collected from each composite sample for possible future analysis will be stored frozen at –20°C.

2.13 STUDY DERIVED WASTES

Any excess phosphate-free, detergent-bearing liquid wastes from decontamination or any groundwater, surface water, or surface sediment sample remaining after processing will be deposited in the vicinity of the collection area. Purge water from the groundwater sample collection process will be containerized in drums and temporarily stored at a secure location in the plant for treatment and disposal via the onsite treatment system. Any subsurface sediment

present at the end of the sampling event or other non-liquid investigation derived waste will be managed by the Site. This study derived waste will be containerized (e.g., 55-gallon drums) and disposed of in accordance with Federal and State requirements using a NJDEP-licensed waste disposal company.

3 FIELD DOCUMENTATION

The integrity of each sample from the time of collection to the point of data reporting must be maintained. Proper record-keeping and COC procedures will allow samples to be traced from collection to final disposition. Representative photographs will be taken of each area where samples are collected. A photograph will be taken of each surface sediment sample and each subsurface sediment interval collected for testing. Site photographs from various angles and close-up views of the overall conditions will also be collected.

3.1 FIELD LOGBOOK

All field activities and observations will be noted in a logbook. The field logbook will be a bound document and may contain individual field and sample log forms (depending on the sampling activity). Information will include personnel, date, time, station designation, sampler, types of samples collected, and general observations. Any changes that occur during sampling (e.g., personnel, responsibilities, or deviations from the FSP) and the reasons for these changes will be documented. The logbook will identify onsite visitors (if any) and the number of photographs taken at each sampling location. Integral's field lead is responsible for ensuring that the respective field logbook for each kind of media sampled and all field data forms are correct. Requirements for log book entries will include the following:

- Logbooks will be bound, with consecutively numbered pages
- Removal of any pages, even if illegible, will be prohibited
- Entries will be made legibly with black (or dark) waterproof ink
- Unbiased, accurate language will be used
- Entries will be made while activities are in progress or as soon afterward as possible (the date and time that the notation is made should be recorded, as well as the time of the observation itself)
- Each consecutive day's first entry will be made on a new, blank page
- The date and time, based on a 24-hour clock (e.g., 0900 for 9:00 a.m. and 2100 for 9:00 p.m.), will appear on each page.

In addition to the preceding requirements, the person recording the information must sign and date the last page of the field logbook for each sampling day. If more than one individual makes entries in the logbook on any given day, then each recorder must initial and date their respective entries.

Logbook corrections will be made by drawing a single line through the original entry, allowing the original entry to be read. The corrected entry will be written alongside the original. Corrections will be initialed and dated and may require a footnote for explanation.

The type of information that may be included in the field logbook and/or field data forms includes the following:

All Types of Sampling

- Task name and task number
- Start date and end date
- Weather conditions
- Name of person making entries and other field staff
- Onsite visitors, if any
- Observations made during sample collection, including any complications, and other details associated with the sampling effort

Specific information on each type of sampling activity that will be recorded in the field logbook is provided below.

Groundwater Sampling

- Calibration and QA/QC results for field instruments used
- Description of all sampling/water quality monitoring equipment used including make, model, and material types for equipment contacting groundwater (e.g., tubing, bladder, pump body, and seals, cord)
- Water conditions including approximate air temperature
- For each well sampled the following will be noted:
  - Well number, sample identifier, and sample number
  - Date and time of sample collection
  - Measuring point description
  - Static water level, date, time, and measurement technique prior to pump installation
  - Presence and thickness of any nonaqueous-phase liquid encountered
  - Pump depth
  - Pumping rate
  - Water quality parameters, time, and drawdown at 5-minute intervals

- Any observations or problems encountered.

Surface Water and Sediment Sampling

- Sampling vessel, if any
- Station number, sample identifier, and sample number for each sample to be submitted for laboratory analysis
- Date and collection time of each sample
- The specific date and time with corresponding station number associated with the sampling location coordinates derived from DGPS
- The sample number, date and time of collection, equipment type, and the lot number for the box of filter papers used for field quality control samples
- Sample description (source and appearance, such as color, oil sheens, odor, and other debris, and sediment type, presence of anthropogenic material and presence and type of biological structures)
- Sediment penetration depth (nearest 0.5 cm) based on sediment depth at the center of the excavation
- Any visible debris near any of the sampling locations
- Any surface vegetation or debris that is removed from the sampling location prior to sampling
- The locations of any surface water runoff or seeps that are located near any of the sampling stations.

In addition, a sampling location map will be updated during each sampling activity and will be maintained throughout each sampling event. All log books must be completed at the time that any observations are made. Copies of all log books and forms will be retained by the technical team.

3.2 CHAIN-OF-CUSTODY PROCEDURES

Samples are in custody if they are in the custodian's view, stored in a secure place with restricted access, or placed in a container secured with custody seals (see SOP AP-03). A COC record will be signed by each person who has custody of the samples and will accompany the samples at all times. Copies of the COC will be included in laboratory and QA/QC reports. Attachment B3 contains an example of the COC form that will be used during the proposed field events.

At a minimum, the form will include the following information:

- Site name or contract number
- Field lead's name and team members responsible for collection of the listed samples
- Collection date and time for each sample
- Sample type (i.e., sample for immediate analysis or archive)
- Requested analyses
- Sample preservation information (if any)
- Name of the carrier relinquishing the samples to the transporter, noting date and time of transfer and the designated sample custodian at the receiving facility.

Integral's field lead (or delegate) will be the designated field sample custodian for each sampling event and will be responsible for all sample tracking and COC procedures for the samples collected in the field. The field sample custodian will be responsible for final sample inventory and will maintain sample custody documentation. The field sample custodian will complete COC forms prior to removing samples from the field. Upon transferring samples to the laboratory sample custodian (if a local laboratory is selected) or shipping courier (as appropriate), the field sample custodian will sign, date, and note the time of transfer on the COC form. The original COC form will be transported with the samples to the laboratories. All samples will be shipped to the testing laboratories in coolers sealed with custody seals.

Each laboratory will designate a sample custodian who will be responsible for receiving samples and documenting their progress through the laboratory analytical process. The sample custodian for each laboratory will establish the integrity of the custody seals upon sample arrival at the laboratory. The laboratory sample custodian will also ensure that the COC and sample tracking forms are properly completed, signed, and initialed upon receipt of the samples.

When the laboratory receives the samples, the laboratory sample custodian will conduct an inventory by comparing sample labels to those on the COC document. The custodian will enter the sample number into a laboratory tracking system by task code and sample designation. The custodian will assign a unique laboratory number to each sample and will be responsible for distributing the samples to the appropriate analyst or for storing samples at the correct temperature in an appropriate secure area.

3.3 WELL AND STATION NUMBERING

All wells and stations will be assigned a unique identification code based on a designation scheme designed to suit the needs of the field personnel, data management, and data users. Groundwater stations will be labeled using the previously assigned well designation. Surface water and sediment station numbers will begin with "SSI" to indicate the Solvay site

investigation. This will be followed by a three-digit number (e.g., 001, 002, 003). Surface water and sediment station numbers will increase as the stations move upstream. An example station number for a co-located surface water and sediment would be SSI021.

Station numbers will not be recorded on sample labels or COC forms to prevent analytical laboratories from seeing the relationships between samples and stations.

3.4 SAMPLE IDENTIFIERS

Each sample from a given station will also have a unique label identifier. Sample identifiers will be established before field sampling begins and assigned to each sample as it is collected. Sample identifiers consist of codes designed to fulfill three purposes: 1) to identify related samples (i.e., field split samples) to ensure proper data analysis and interpretation; 2) to obscure the relationships between samples so that laboratory analysis will be unbiased by presumptive similarities between samples; and 3) to track individual sample containers to ensure that the laboratory receives all of the material associated with a single sample. To accomplish these purposes, each container is assigned a sample number and a tag number. These codes and their uses are described below:

- A sample identifier for each sample will be created as follows: the well or station number (e.g., SSI021), followed by the month, day, and year that the sample was collected (e.g., 022814), and this would be followed by a two-letter code for the kind of sample collected at a given location (GW = groundwater, SW= surface water, GR = sediment grab sample, CR = sediment core sample). In addition, subsurface core samples will also have a final series of numbers attached to the sample identifier that will distinguish between the different sample intervals of the core (e.g., 0–6 in. [0–15 cm], 6–12 in. [15–30 cm], 12–24 in. [30–61 cm], 24–36 in. [61–91 cm], and >36 in. [>91 cm]). An example sample identifier for a groundwater sample collected on November 20, 2013, at Well M/H-1D would be M/H-1D--112013-GW. Example sample identifiers for a co-located surface water, surface sediment sample would be SSI008-022814-SW and SSI008-022814-GR1. Example sample identifiers for a co-located surface water and sediment coring station would be SSI011-022814-SW, and SSI011-022814-CR1-0-30, SSI011-022814-CR1-30-60, and so on. If a second core were required at a given station to obtain the required sample volume, then example sample identifiers for this second core would be SSI011-022814-CR2-0-30, SSI011-022814-CR2-30-60, SSI011-022814-CR2-60-90, and so on.

  Each field split sample will have the same sample identifier, but with "-DUP" at the end of the sample identifier (e.g., SSI011-022814-SW-DUP).

- The sample number is an arbitrary number assigned sequentially to each sample collected (e.g., GW0001, GW0002, SW0001, SW0002, SD0001, SD0002). All subsamples of

a composited field sample will have the same sample number. Each field split sample will have a different sample number, and the sample numbers of related field quality control samples may not share any content. The sample number will appear on the sample containers and the COC forms.

- A unique numeric sample tag number will be attached to each sample container. The tag number is a preprinted number on the sample label. If the amount of material (i.e., everything associated with a single sample number) is too large for a single container, each container will have the same sample number and a different sample label with a unique sample tag number. A sample will also be split between containers if a different preservation technique is used for each container (i.e., because different analyses will be conducted). The tag number will appear on the COC forms. Tag numbers will be used by laboratories only to confirm that they have received all of the containers that were filled and shipped. Data will be reported by sample number.
- For equipment blanks, sequential numbers starting at 900 will be assigned instead of station numbers. For example, the first equipment blank for a surface sediment sample collected with a grab sampler will be labeled as SDEQ-901G, whereas the second equipment blank for a subsurface sediment sample collected with a coring device will be labeled as SDEQ-902C (SD = sediment, EQ = equipment blank, G = grab sampler, and C = core). Other types of field quality control blanks will be numbered in a similar fashion: TB = travel blank, FB = field blank, and DB = decontamination water blank.

4 FIELD DATA MANAGEMENT AND REPORTING PROCEDURES

During field operations, effective data management is critical to providing consistent, accurate, and defensible data and data products. Daily field records (a combination of field logbooks, field forms, if any, and COC forms) will make up the main documentation for field activities. Upon completion of sampling, field notes, data sheets (if any), and COC forms will be scanned to create an electronic record. Field data will be manually entered into the project database. One hundred percent of the transferred data will be verified based on hard copy records. Electronic quality assurance checks to identify anomalous values will also be conducted following entry.

5 REFERENCES

ERM. 2012. Interim submittal for ground water. Solvay Solexis, Inc. West Deptford, NJ Facility ISRA Case Nos. E89231, E90205, E20020018 Program Interest No. 015010.

ERM. 2013. Free/residual product and ground water interim remedial investigation submittal. Solvay Specialty Polymers USA, LLC West Deptford, NJ Facility ISRA Case Nos. E89231, E90205, Program Interest No. 015010.

MacGillivray, A.R. 2012. Contaminants of emerging concern in the tidal Delaware River, pilot monitoring study 2007–2009. Delaware River Basin Commission, West Trenton, NJ.

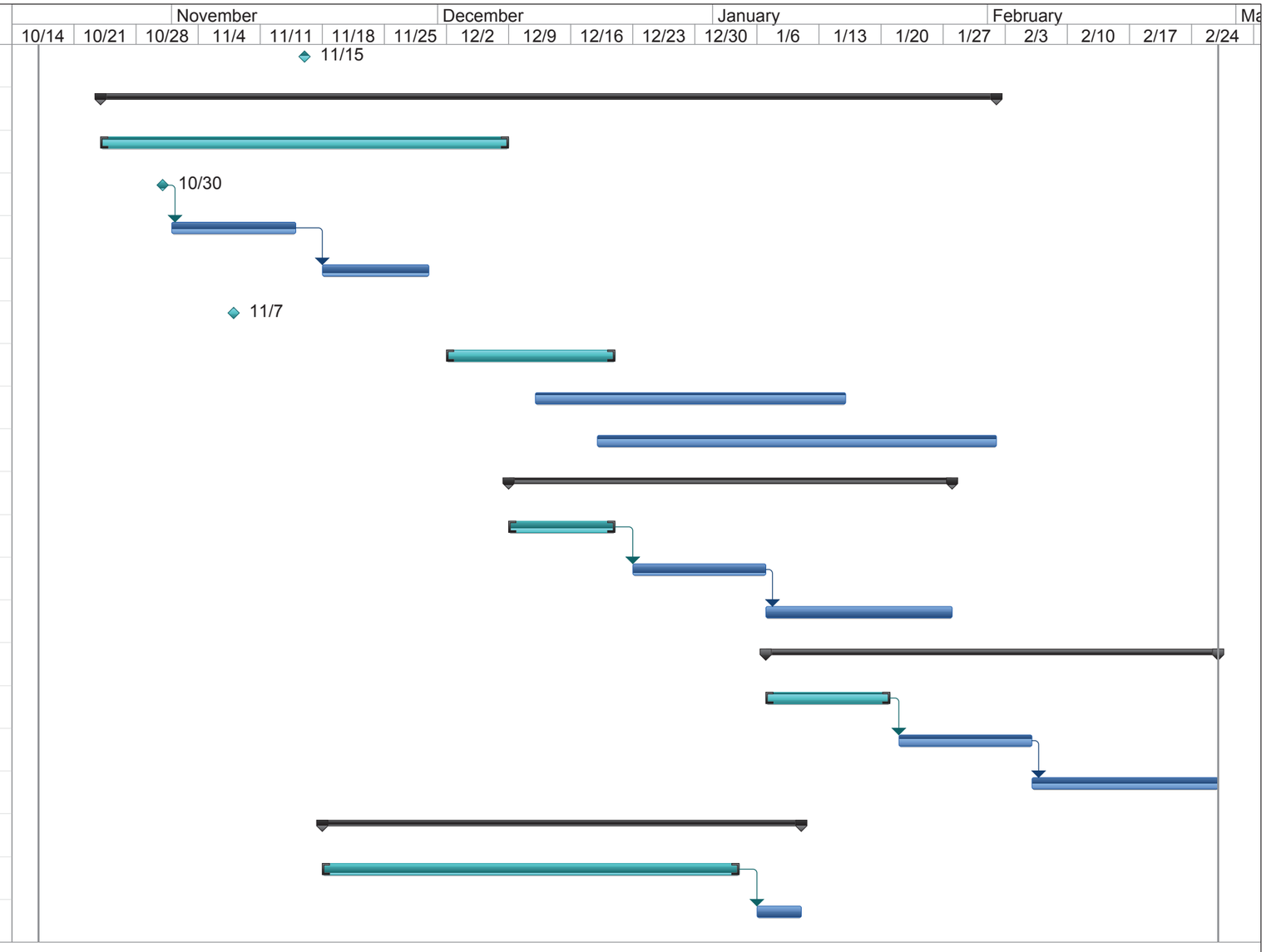
NJDEP. 2005. Field sampling procedures manual. New Jersey Department of Environmental Protection, Trenton, NJ.

USEPA. 1997. Recommended quality assurance and quality control guidelines for the collection of environmental data in Puget Sound. In: Recommended protocols for measuring selected environmental variables in Puget Sound. U.S. Environmental Protection Agency, Puget Sound Estuary Program, Seattle, WA.

USEPA. 2010. Low stress (low flow) purging and sampling procedures for the collection of groundwater samples from monitoring wells. U. S. Environmental Protection Agency, Washington, DC. Revised January 19, 2010.

FIGURES

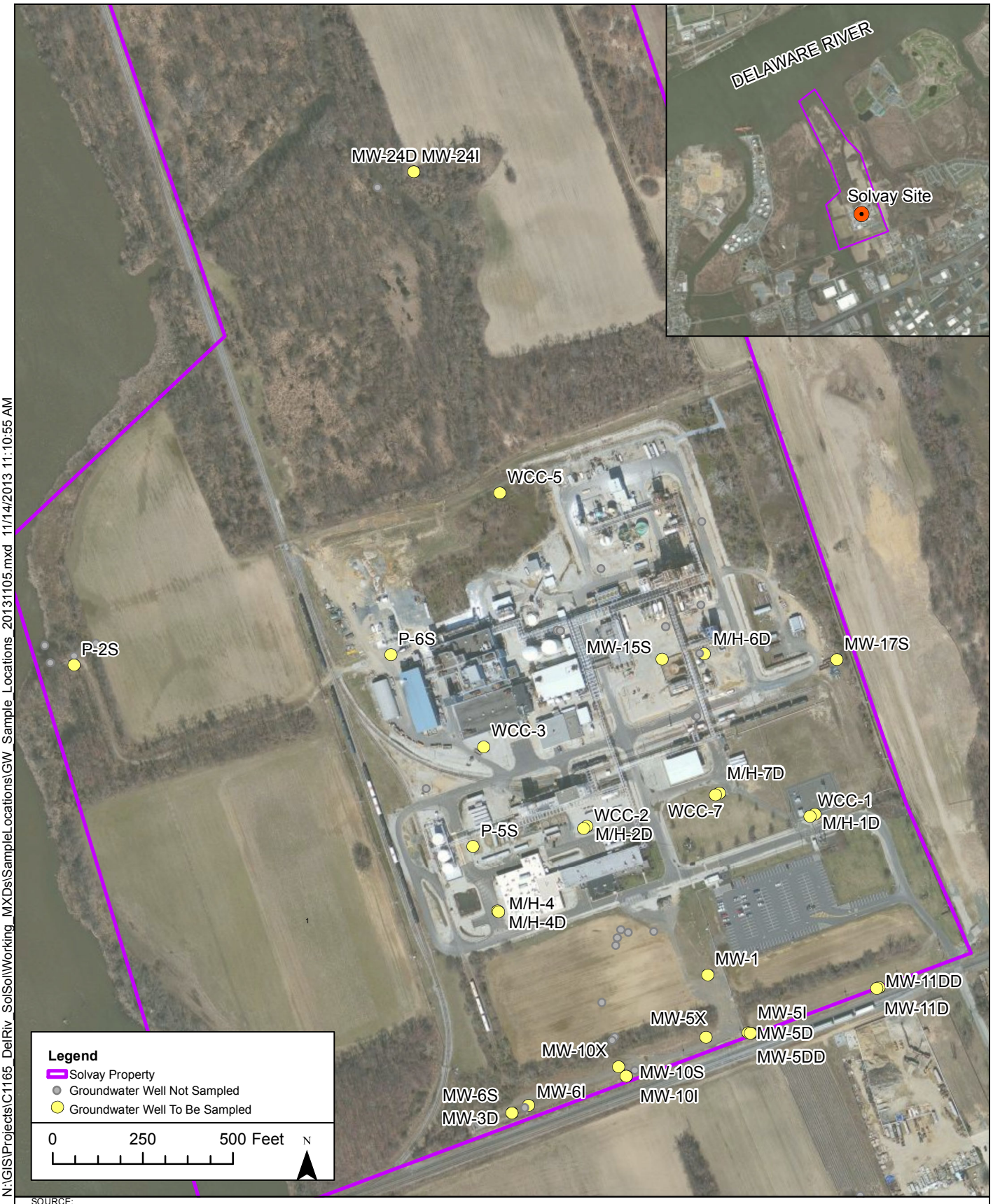
ID	Task Name	Start	Finish
1	Work Plan to NJDEP	Fri 11/15/13	Fri 11/15/13
2	**Field MUA**	**Thu 10/24/13**	**Sat 2/1/14**
3	Municipal Outreach and Scheduling	Thu 10/24/13	Mon 12/9/13
4	W. Deptford - Sampling	Wed 10/30/13	Wed 10/30/13
5	W. Deptford - Lab Analysis and Reporting	Fri 11/1/13	Thu 11/14/13
6	W. Deptford - External Data Validation	Mon 11/18/13	Fri 11/29/13
7	NJDEP Letter to MUAs	Thu 11/7/13	Thu 11/7/13
8	Six MUAs - Sampling	Mon 12/2/13	Fri 12/20/13
9	Six MUAs - Lab Analysis and Reporting	Thu 12/12/13	Wed 1/15/14
10	Six MUAs - External Data Validation	Thu 12/19/13	Sat 2/1/14
11	**Field Groundwater Onsite Wells**	**Mon 12/9/13**	**Mon 1/27/14**
12	GW - Sampling	Mon 12/9/13	Fri 12/20/13
13	GW - Lab Analysis and Reporting	Mon 12/23/13	Mon 1/6/14
14	GW - External Data Validation	Tue 1/7/14	Mon 1/27/14
15	**Field Surface Water and Sediment**	**Tue 1/7/14**	**Wed 2/26/14**
16	SW & Sediment - Sampling	Tue 1/7/14	Mon 1/20/14
17	SW & Sediment - Lab Analysis and Reporting	Wed 1/22/14	Wed 2/5/14
18	SW & Sediment - External Data Validation	Thu 2/6/14	Wed 2/26/14
19	**Air Modeling**	**Mon 11/18/13**	**Fri 1/10/14**
20	Run Modeling Scenarios	Mon 11/18/13	Fri 1/3/14
21	Reporting	Mon 1/6/14	Fri 1/10/14



MUA - Municipal Utility Authority
GW - Groundwater
SW - Surface Water

integral consulting inc.

Figure B-1.
Gantt Chart of Proposed Project Schedule for Sampling and Lab Validation

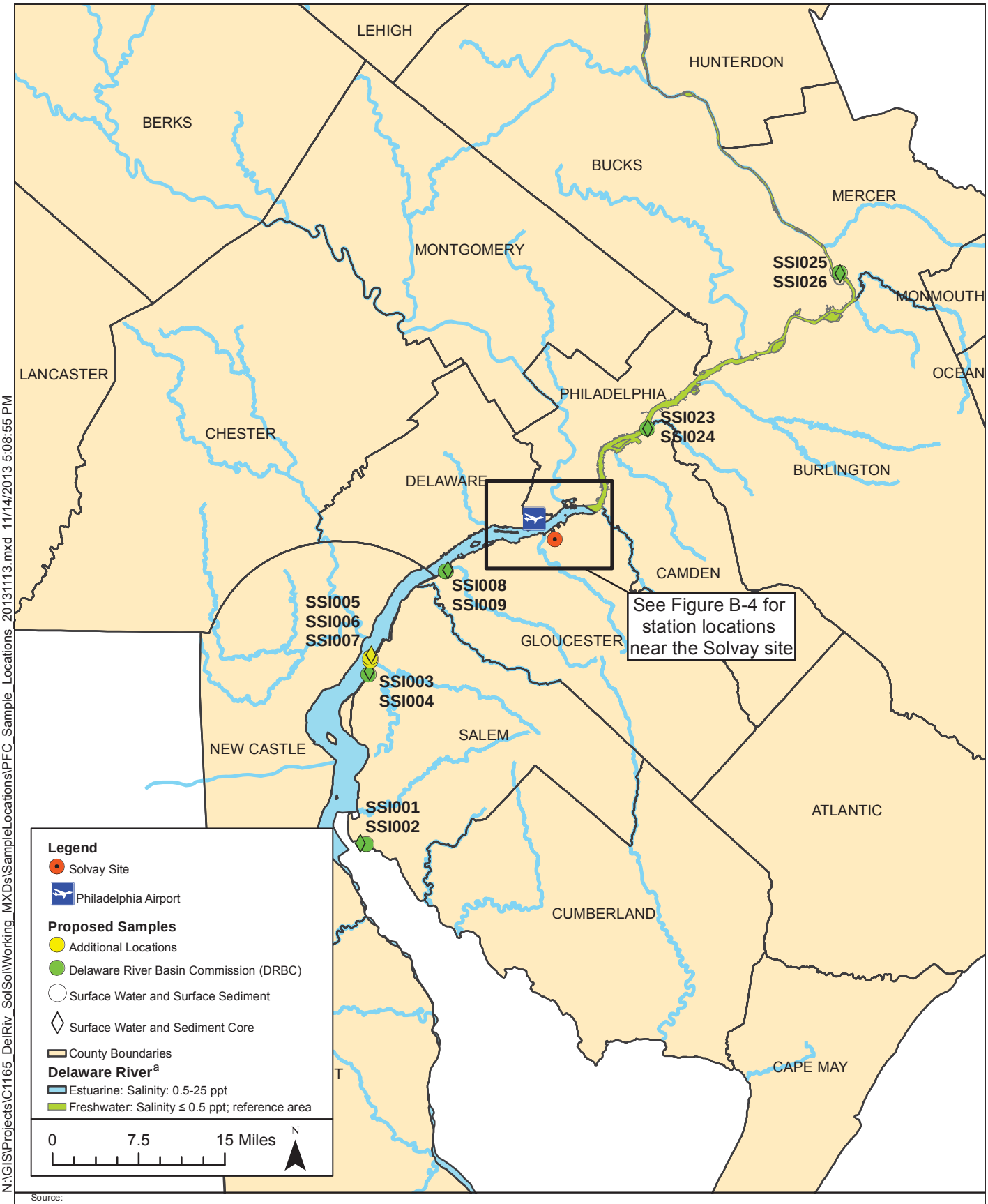


N:\GIS\Projects\C1165_DelRiv_SolSol\Working_MXDs\SampleLocations\GW_Sample_Locations_20131105.mxd 11/14/2013 11:10:55 AM

SOURCE:
Aerial Imagery: (c) 2010 Microsoft Corporation and its data suppliers.

integral consulting inc.

Figure B-2.
Proposed Groundwater Sampling Locations

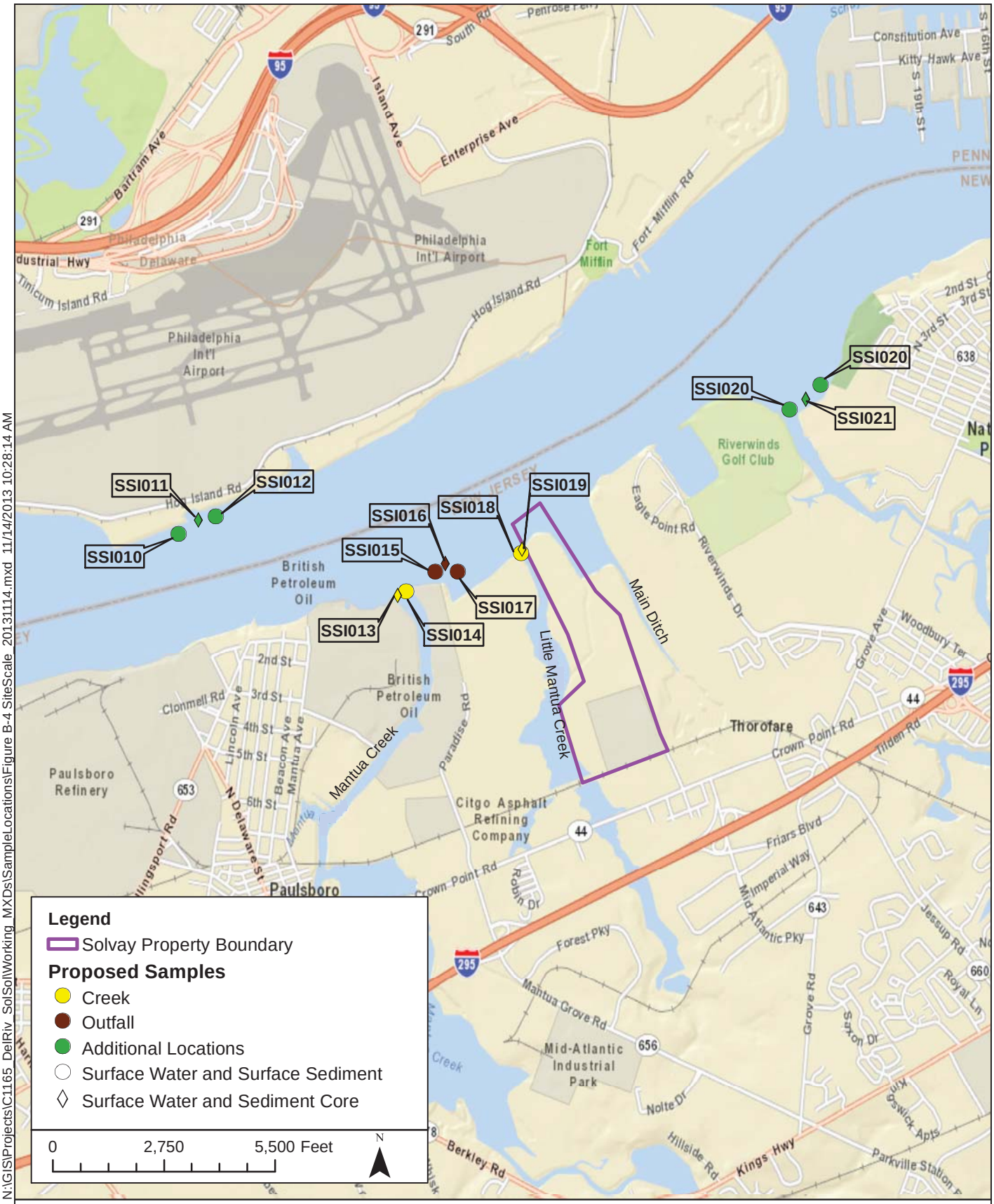


N:\GIS\Projects\C1165_DelRiv_SolSol\Working_MXDs\SampleLocations\PFC_Sample_Locations_20131113.mxd 11/14/2013 5:08:55 PM

Source:
a. NJDEP (2012)

integral consulting inc.

Figure B-3.
Proposed Co-Located Surface Water and Sediment Sampling Locations near Historic DRBC Monitoring Locations



N:\GIS\Projects\C1165_DelRiv_SolSol\Working_MXDs\SampleLocations\Figure B-4 SiteScale_20131114.mxd 11/14/2013 10:28:14 AM

integral consulting inc.

Figure B-4.
Proposed Co-Located Surface Water and Sediment Sampling Locations near the Site

TABLES

Table B-1. Chemicals of Interest

Analyte	Formula	CAS Number	Groundwater	Surface Water	Sediment
PFCs					
Perfluorooctanoic acid (PFOA;C8)	$C_7F_{15}COOH$	335-67-1	X	X	X
Perfluorononanoic acid (PFNA;C9)	$C_8F_{17}COOH$	375-95-1	X	X	X
Perfluorodecanoic acid (PFDA; C10)	$C_9F_{19}COOH$	335-76-2	X	X	X
Perfluoroundecanoic acid (PFUnDA; C11)	$C_{10}F_{21}COOH$	2058-94-8	X	X	X
Perfluorododecanoic acid (PFDoDA; C12)	$C_{11}F_{23}COOH$	307-55-1	X	X	X
Perfluorotridecanoic acid (PFTrDA; C13)	$C_{12}F_{25}COO^-$	72629-94-8	X	X	X
Perfluorooctanesulfonic acid (PFOS; C8)	$C_8F_{17}SO_3H$	1763-23-1	X	X	X
Conventional Parameters					
Total organic carbon	--	7440-44-0		X	X
Total suspended solids	--	--		X	
pH	--	12408-02-5		X	
Hardness, Total	--	--		X	
Alkalinity	--	--		X	
Grainsize	--	--			X
Field Water Quality Parameters					
Temperature			X	X	
pH			X	X	
Dissolved oxygen			X	X	
Salinity			X	X	
Conductivity			X	X	
Turbidity			X	X	
Oxidation-reduction potential (ORP)			X	X	

Notes:

CAS = Chemical Abstracts Service registry number

PFC = perfluorinated compound

X = samples will be collected and analyzed for these parameters

-- = not available

Table B-2. Sample Containers, Preservation, and Holding Time Requirements

Analyses	Container [a] Type	Container [a] Size	Preservation	Holding Time	Minimum Sample Size
Water Samples (Groundwater)					
PFCs	HDPE	2 x 250 mL	4 ± 2°C	7 days to extraction 40 days from extraction to analysis	250 mL
Water Samples (Surface Water)					
PFCs	HDPE	2 x 250 mL	4 ± 2°C	7 days to extraction 40 days from extraction to analysis	250 mL
Total organic carbon	AG	250 mL	4 ± 2°C, H_2SO_4 to pH<2	28 days	50 mL
Total suspended solids	HDPE	1,000 mL	4 ± 2°C	7 days	100 mL
pH	HDPE	250 mL	4 ± 2°C	Analyze immediately	100 mL
Hardness, total	HDPE	500 mL	4 ± 2°C, HNO_3 to pH<2	6 months	50 mL
Alkalinity	HDPE	500 mL	4 ± 2°C	14 days	50 mL
Sediments					
PFCs	Wide mouth HDPE	250 mL	4 ± 2°C	14 days to extraction 40 days from extraction to analysis	10 g
Grainsize	WMG	16 oz.	4 ± 2°C	6 months	500 g
Total organic carbon	WMG	4 oz.	4 ± 2°C	14 days	3 g

Notes:

AG = amber glass
HDPE = high density polyethylene
HNO_3 = nitric acid
H_2SO_4 = sulfuric acid
PFC = perfluorinated compound
PP = polypropylene
WMG = wide mouth glass

[a] The size and number of containers may be modified by the analytical laboratory.

Table B-3. Field Sample Collection Matrix for Groundwater

					Groundwater Chemistry		Equipment Rinsate Blank	
					PFCs (C8 to C13)		PFCs (C8 - C14)	
					2 Sample Bottles		2 Sample Bottles	
					250 mL HDPE[a]	250 mL HDPE[a]	250 mL HDPE[a]	250 mL HDPE[a]
					250 mL[f]		250 mL[f]	
Well Number	Sample ID	Zone	Sample Number	Sample Type[e]	Refrigerated	Refrigerated	Refrigerated	Refrigerated
Site								
☐ M/H-1D	M/H-1D-xxxxxx[d]-GW	Deep	GW0001	Normal	Tag #_____	Tag #_____	NA	NA
☐ M/H-2D	M/H-2D-xxxxxx[d]-GW	Deep	GW0002	Normal	Tag #_____	Tag #_____	NA	NA
☐ M/H-4	M/H-4-xxxxxx[d]-GW	Shallow	GW0003	Normal	Tag #_____	Tag #_____	NA	NA
☐ M/H-4D	M/H-4D-xxxxxx[d]-GW	Intermediate	GW0004	Normal	Tag #_____	Tag #_____	NA	NA
☐ M/H-6D	M/H-6D-xxxxxx[d]-GW	Intermediate	GW0005	Normal	Tag #_____	Tag #_____	NA	NA
☐ M/H-7D	M/H-7D-xxxxxx[d]-GW	Intermediate	GW0006	Normal	Tag #_____	Tag #_____	NA	NA
☐ MW-1	MW-1-xxxxxx[d]-GW	Shallow	GW0007	Normal	Tag #_____	Tag #_____	NA	NA
☐ MW-10I	MW-10I-xxxxxx[d]-GW	Intermediate	GW0008	Normal	Tag #_____	Tag #_____	NA	NA
☐ MW-10S	MW-10S-xxxxxx[d]-GW	Shallow	GW0009	Normal	Tag #_____	Tag #_____	NA	NA
☐ MW-10X	MW-10X-xxxxxx[d]-GW	Confined	GW0010	Normal	Tag #_____	Tag #_____	NA	NA
☐ MW-10X	MW-10X-xxxxxx[d]-GW-DUP	Confined	GW0011	Field Duplicate [b]	Tag #_____	Tag #_____	NA	NA
☐ MW-11D	MW-11D-xxxxxx[d]-GW	Intermediate	GW0012	Normal	Tag #_____	Tag #_____	NA	NA
☐ MW-11DD	MW-11DD-xxxxxx[d]-GW	Deep	GW0013	Normal	Tag #_____	Tag #_____	NA	NA
☐ MW-15S	MW-15S-xxxxxx[d]-GW	Shallow	GW0014	Normal	Tag #_____	Tag #_____	NA	NA
☐ MW-17S	MW-17S-xxxxxx[d]-GW	Shallow	GW0015	Normal	Tag #_____	Tag #_____	NA	NA
☐ MW-24D	MW-24D-xxxxxx[d]-GW	Deep	GW0016	Normal	Tag #_____	Tag #_____	NA	NA
☐ MW-24I	MW-24I-xxxxxx[d]-GW	Intermediate	GW0017	Normal	Tag #_____	Tag #_____	NA	NA
☐ MW-3D	MW-3D-xxxxxx[d]-GW	Deep	GW0018	Normal	Tag #_____	Tag #_____	NA	NA
☐ MW-5D	MW-5D-xxxxxx[d]-GW	Intermediate	GW0019	Normal	Tag #_____	Tag #_____	NA	NA
☐ MW-5DD	MW-5DD-xxxxxx[d]-GW	Deep	GW0020	Normal	Tag #_____	Tag #_____	NA	NA
☐ MW-5I	MW-5I-xxxxxx[d]-GW	Intermediate	GW0021	Normal	Tag #_____	Tag #_____	NA	NA
☐ MW-5I	MW-5I-xxxxxx[d]-GW-DUP	Intermediate	GW0022	Field Duplicate [b]	Tag #_____	Tag #_____	NA	NA
☐ EQ Blank	GWEQ-901	NA	EQ0001	Equipment rinsate blank [c]	NA	NA	Tag #_____	Tag #_____
☐ MW-5X	MW-5X-xxxxxx[d]-GW	Confined	GW0023	Normal	Tag #_____	Tag #_____	NA	NA

Table B-3. Field Sample Collection Matrix for Groundwater

					Groundwater Chemistry		Equipment Rinsate Blank	
					PFCs (C8 to C13)		PFCs (C8 - C14)	
					2 Sample Bottles		2 Sample Bottles	
					250 mL HDPE[a]	250 mL HDPE[a]	250 mL HDPE[a]	250 mL HDPE[a]
					250 mL[f]		250 mL[f]	
Well Number	Sample ID	Zone	Sample Number	Sample Type[e]	Refrigerated	Refrigerated	Refrigerated	Refrigerated
☐ MW-6I	MW-6I-xxxxxx[d]-GW	Intermediate	GW0024	Normal	Tag #_____	Tag #_____	NA	NA
☐ MW-6S	MW-6S-xxxxxx[d]-GW	Shallow	GW0025	Normal	Tag #_____	Tag #_____	NA	NA
☐ P-2S	P-5S-xxxxxx[d]-GW	Shallow	GW0026	Normal	Tag #_____	Tag #_____	NA	NA
☐ P-5S	P-5S-xxxxxx[d]-GW	Shallow	GW0027	Normal	Tag #_____	Tag #_____	NA	NA
☐ P-6S	P-6S-xxxxxx[d]-GW	Shallow	GW0028	Normal	Tag #_____	Tag #_____	NA	NA
☐ WCC-1	WCC-1-xxxxxx[d]-GW	Shallow	GW0029	Normal	Tag #_____	Tag #_____	NA	NA
☐ WCC-2	WCC-2-xxxxxx[d]-GW	Shallow	GW0030	Normal	Tag #_____	Tag #_____	NA	NA
☐ WCC-3	WCC-3-xxxxxx[d]-GW	Shallow	GW0031	Normal	Tag #_____	Tag #_____	NA	NA
☐ WCC-5	WCC-5-xxxxxx[d]-GW	Shallow	GW0032	Normal	Tag #_____	Tag #_____	NA	NA
☐ WCC-7	WCC-7-xxxxxx[d]-GW	Shallow	GW0033	Normal	Tag #_____	Tag #_____	NA	NA
☐ WCC-7	WCC-7-xxxxxx[d]-GW-DUP	Shallow	GW0034	Field Duplicate [b]	Tag #_____	Tag #_____	NA	NA
☐ EQ Blank	GWEQ-902	NA	EQ0002	Equipment rinsate blank [c]	NA	NA	Tag #_____	Tag #_____

Notes:

HDPE = high density polyethylene

NA = not applicable

PFC = perfluorinated compound

[a] The size and number of containers may be modified.

[b] Blind field duplicate samples will be collected at a minimum frequency of 1 field duplicate sample per 10 groundwater samples.

[c] An equipment rinsate blank sample will be collected at a minimum frequency of 1 per 20 groundwater samples.

[d] Where "xxxxxx" = month, day, and year that the sample was collected (e.g., November 20, 2013 would be 112013).

[e] In addition to the samples listed in this table, a field (or environmental) blank will be collected at a minimum frequency of 10 percent, and a travel or trip blank will be sent with each PFC sample to the analytical laboratory. Also, one decontamination water blank will be collected during the groundwater sampling event.

[f] Minimum sample size.

Table B-4. Field Sample Collection Matrix for Surface Water

				Surface Water Chemistry							Equipment Rinsate Blank	
				PFCs (C8 to C13)		TOC	TSS	pH	Total Hardness	Alkalinity	PFCs (C8 - C14)	
				2 Sample Bottles							2 Sample Bottles	
				250 mL HDPE[a]	250 mL HDPE[a]	250 ML Ag[a]; H_2SO_4	1 L HDPE[a]	250 mL HDPE[a]	500 mL HDPE[a]; HNO_3	500 mL HDPE[a]	250 mL HDPE[a]	250 mL HDPE[a]
				250 mL[e]		50 mL[e]	100 mL[e]	100 mL[e]	50 mL[e]	50 mL[f]	250 mL[e]	
Well Number	Sample ID	Sample Number	Sample Type[f]	Refrigerated	Refrigerated	Refrigerated	Refrigerated	Refrigerated	Refrigerated	Refrigerated	Refrigerated	Refrigerated
Site												
☐ SSI001	SSI001-xxxxxx[d]-SW	SW0001	Normal	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	NA	NA
☐ SSI002	SSI002-xxxxxx[d]-SW	SW0002	Normal	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	NA	NA
☐ SSI003	SSI003-xxxxxx[d]-SW	SW0003	Normal	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	NA	NA
☐ SSI004	SSI004-xxxxxx[d]-SW	SW0004	Normal	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	NA	NA
☐ SSI005	SSI005-xxxxxx[d]-SW	SW0005	Normal	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	NA	NA
☐ SSI006	SSI006-xxxxxx[d]-SW	SW0006	Normal	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	NA	NA
☐ SSI007	SSI007-xxxxxx[d]-SW	SW0007	Normal	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	NA	NA
☐ SSI008	SSI008-xxxxxx[d]-SW	SW0008	Normal	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	NA	NA
☐ SSI009	SSI009-xxxxxx[d]-SW	SW0009	Normal	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	NA	NA
☐ SSI010	SSI010-xxxxxx[d]-SW	SW0010	Normal	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	NA	NA
☐ SSI010	SSI010-xxxxxx[d]-SW-DUP	SW0011	Field Duplicate [b]	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	NA	NA
☐ SSI011	SSI011-xxxxxx[d]-SW	SW0012	Normal	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	NA	NA
☐ SSI012	SSI012-xxxxxx[d]-SW	SW0013	Normal	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	NA	NA
☐ SSI013	SSI013-xxxxxx[d]-SW	SW0014	Normal	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	NA	NA
☐ SSI014	SSI014-xxxxxx[d]-SW	SW0015	Normal	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	NA	NA
☐ SSI015	SSI015-xxxxxx[d]-SW	SW0016	Normal	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	NA	NA
☐ SSI016	SSI016-xxxxxx[d]-SW	SW0017	Normal	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	NA	NA
☐ SSI017	SSI017-xxxxxx[d]-SW	SW0018	Normal	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	NA	NA
☐ SSI018	SSI0018-xxxxxx[d]-SW	SW0019	Normal	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	NA	NA

Table B-4. Field Sample Collection Matrix for Surface Water

				Surface Water Chemistry							Equipment Rinsate Blank	
				PFCs (C8 to C13)		TOC	TSS	pH	Total Hardness	Alkalinity	PFCs (C8 - C14)	
				2 Sample Bottles							2 Sample Bottles	
Well Number	Sample ID	Sample Number	Sample Type[f]	250 mL HDPE[a]	250 mL HDPE[a]	250 mL AG[a]; H_2SO_4	1 L HDPE[a]	250 mL HDPE[a]	500 mL HDPE[a]; HNO_3	500 mL HDPE[a]	250 mL HDPE[a]	250 mL HDPE[a]
				250 mL[e]		50 mL[e]	100 mL[e]	100 mL[e]	50 mL[e]	50 mL[f]	250 mL[e]	
				Refrigerated	Refrigerated	Refrigerated	Refrigerated	Refrigerated	Refrigerated	Refrigerated	Refrigerated	Refrigerated
☐ SSI019	SSI019-xxxxxx[d]-SW	SW0020	Normal	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	NA	NA
☐ SSI020	SSI020-xxxxxx[d]-SW	SW0021	Normal	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	NA	NA
☐ SSI020	SSI020-xxxxxx[d]-SW-DUP	SW0022	Field Duplicate [b]	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	NA	NA
☐ EQ Blank	SWEQ-901	EQ0001	Equipment rinsate blank [c]	NA	NA	NA	NA	NA	NA	NA	Tag #_____	Tag #_____
☐ SSI021	SSI021-xxxxxx[d]-SW	SW0023	Normal	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	NA	NA
☐ SSI022	SSI022-xxxxxx[d]-SW	SW0024	Normal	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	NA	NA
☐ SSI023	SSI023-xxxxxx[d]-SW	SW0025	Normal	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	NA	NA
☐ SSI024	SSI024-xxxxxx[d]-SW	SW0026	Normal	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	NA	NA
☐ SSI025	SSI025-xxxxxx[d]-SW	SW0027	Normal	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	NA	NA
☐ SSI026	SSI026-xxxxxx[d]-SW	SW0028	Normal	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	NA	NA
☐ SSI026	SSI026-xxxxxx[d]-SW-DUP	FB0002	Field Duplicate [b]	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	NA	NA
☐ EQ Blank	SWEQ-902	EQ0002	Equipment rinsate blank [c]	NA	NA	NA	NA	NA	NA	NA	Tag #_____	Tag #_____

Notes:

AG = amber glass
HDPE = high density polyethylene
NA = not applicable
PFC = perfluorinated compound
TOC = total organic carbon
TSS = total suspended solid

[a] The size and number of containers may be modified.

[b] Blind field duplicate samples will be collected at a minimum frequency of 1 field duplicate sample per 10 surface water samples.

[c] An equipment rinsate blank sample will be collected at a minimum frequency of 1 per 20 surface water samples.

[d] Where "xxxxxx" = month, day, and year that the sample was collected (e.g., February 28, 2014 would be 022814).

[e] Minimum sample size.

[f] In addition to the samples listed in this table, a field (or environmental) blank will be collected at a minimum frequency of 10 percent, and a travel or trip blank will be sent with each PFC sample to the analytical laboratory.

Table B-5. Field Sample Collection Matrix for Surface and Subsurface Sediment

				Sediment Chemistry				Equipment Rinsate Blank	
							Archive		
				PFCs (C8 to C13)	Grain Size	TOC	TBD	PFCs (C8 - C14)	
				250 mL wide mouth HDPE	500 mL WMG	125 mL WMG	250 mL WMG [a]	250 mL HDPE[a]	250 mL HDPE[a]
				10 g[e]	500 g[e]	3 g[e]	NA	250 mL[e]	
Station Number	Sample ID	Sample Number	Sample Type	Refrigerated	Refrigerated	Refrigerated	Frozen	Refrigerated	Refrigerated
Site									
☐ SSI001	SSI001-xxxxxx[d]-GR	SD0001	Normal	Tag #_____	Tag #_____	Tag #_____	Tag #_____	NA	NA
☐ SSI002	SSI002-xxxxxx[d]-CR-0-15	SD0002	Normal	Tag #_____	Tag #_____	Tag #_____	Tag #_____	NA	NA
☐	SSI002-xxxxxx[d]-CR-15-30	SD0003	Normal	Tag #_____	Tag #_____	Tag #_____	Tag #_____	NA	NA
☐	SSI002-xxxxxx[d]-CR-30-61	SD0004	Normal	Tag #_____	Tag #_____	Tag #_____	Tag #_____	NA	NA
☐	SSI002-xxxxxx[d]-CR-61-91	SD0005	Normal	Tag #_____	Tag #_____	Tag #_____	Tag #_____	NA	NA
☐	SSI002-xxxxxx[d]-CR-91-TBD[f]	SD0006	Normal	Tag #_____	Tag #_____	Tag #_____	Tag #_____	NA	NA
☐ SSI003	SSI003-xxxxxx[d]-GR	SD0007	Normal	Tag #_____	Tag #_____	Tag #_____	Tag #_____	NA	NA
☐	SSI003-xxxxxx[d]-GR-DUP	SD0008	Field Duplicate [b]	Tag #_____	Tag #_____	Tag #_____	NA	NA	NA
☐ SSI004	SSI004-xxxxxx[d]-CR-0-15	SD0009	Normal	Tag #_____	Tag #_____	Tag #_____	Tag #_____	NA	NA
☐	SSI004-xxxxxx[d]-CR-15-30	SD0010	Normal	Tag #_____	Tag #_____	Tag #_____	Tag #_____	NA	NA
☐	SSI004-xxxxxx[d]-CR-30-61	SD0011	Normal	Tag #_____	Tag #_____	Tag #_____	Tag #_____	NA	NA
☐	SSI004-xxxxxx[d]-CR-61-91	SD0012	Normal	Tag #_____	Tag #_____	Tag #_____	Tag #_____	NA	NA
☐	SSI004-xxxxxx[d]-CR-91-TBD[f]	SD0013	Normal	Tag #_____	Tag #_____	Tag #_____	Tag #_____	NA	NA
☐ SSI005	SSI005-xxxxxx[d]-GR	SD0014	Normal	Tag #_____	Tag #_____	Tag #_____	Tag #_____	NA	NA
☐ SSI006	SSI006-xxxxxx[d]-GR	SD0015	Normal	Tag #_____	Tag #_____	Tag #_____	Tag #_____	NA	NA
☐ SSI007	SSI007-xxxxxx[d]-CR-0-15	SD0016	Normal	Tag #_____	Tag #_____	Tag #_____	Tag #_____	NA	NA
☐	SSI007-xxxxxx[d]-CR-15-30	SD0017	Normal	Tag #_____	Tag #_____	Tag #_____	Tag #_____	NA	NA
☐	SSI007-xxxxxx[d]-CR-30-61	SD0018	Normal	Tag #_____	Tag #_____	Tag #_____	Tag #_____	NA	NA

Table B-5. Field Sample Collection Matrix for Surface and Subsurface Sediment

				Sediment Chemistry				Equipment Rinsate Blank	
							Archive		
				PFCs (C8 to C13)	Grain Size	TOC	TBD	PFCs (C8 - C14)	
				250 mL wide mouth HDPE	500 mL WMG	125 mL WMG	250 mL WMG [a]	250 mL HDPE[a]	250 mL HDPE[a]
				10 g[e]	500 g[e]	3 g[e]	NA	250 mL[e]	
Station Number	Sample ID	Sample Number	Sample Type	Refrigerated	Refrigerated	Refrigerated	Frozen	Refrigerated	Refrigerated
☐	SSI007-xxxxxx[d]-CR-61-91	SD0019	Normal	Tag #_____	Tag #_____	Tag #_____	Tag #_____	NA	NA
☐	SSI007-xxxxxx[d]-CR-91-TBD[f]	SD0020	Normal	Tag #_____	Tag #_____	Tag #_____	Tag #_____	NA	NA
☐	SSI007-xxxxxx[d]-CR-91-TBD[f]-DUP	SD0021	Field Duplicate [b]	Tag #_____	Tag #_____	Tag #_____	NA	NA	NA
☐ EQ Blank	SDEQ-901C	EQ0001	Equipment rinsate blank [c]	NA	NA	NA	NA	Tag #_____	Tag #_____
☐ SSI008	SSI008-xxxxxx[d]-GR	SD0022	Normal	Tag #_____	Tag #_____	Tag #_____	Tag #_____	NA	NA
☐ SSI009	SSI009-xxxxxx[d]-CR-0-15	SD0023	Normal	Tag #_____	Tag #_____	Tag #_____	Tag #_____	NA	NA
☐	SSI009-xxxxxx[d]-CR-15-30	SD0024	Normal	Tag #_____	Tag #_____	Tag #_____	Tag #_____	NA	NA
☐	SSI009-xxxxxx[d]-CR-30-61	SD0025	Normal	Tag #_____	Tag #_____	Tag #_____	Tag #_____	NA	NA
☐	SSI009-xxxxxx[d]-CR-61-91	SD0026	Normal	Tag #_____	Tag #_____	Tag #_____	Tag #_____	NA	NA
☐	SSI009-xxxxxx[d]-CR-91-TBD[f]	SD0027	Normal	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____
☐ SSI010	SSI010-xxxxxx[d]-GR	SD0028	Normal	Tag #_____	Tag #_____	Tag #_____	Tag #_____	NA	NA
☐	SSI010-xxxxxx[d]-GR-DUP	SD0029	Field Duplicate [b]	Tag #_____	Tag #_____	Tag #_____	NA	NA	NA
☐ SSI011	SSI011-xxxxxx[d]-CR-0-15	SD0030	Normal	Tag #_____	Tag #_____	Tag #_____	Tag #_____	NA	NA
☐	SSI011-xxxxxx[d]-CR-15-30	SD0031	Normal	Tag #_____	Tag #_____	Tag #_____	Tag #_____	NA	NA
☐	SSI011-xxxxxx[d]-CR-30-61	SD0032	Normal	Tag #_____	Tag #_____	Tag #_____	Tag #_____	NA	NA
☐	SSI011-xxxxxx[d]-CR-61-91	SD0033	Normal	Tag #_____	Tag #_____	Tag #_____	Tag #_____	NA	NA
☐	SSI011-xxxxxx[d]-CR-91-TBD[f]	SD0034	Normal	Tag #_____	Tag #_____	Tag #_____	Tag #_____	NA	NA
☐ SSI0012	SSI012-xxxxxx[d]-GR	SD0035	Normal	Tag #_____	Tag #_____	Tag #_____	Tag #_____	NA	NA
☐ SSI013	SSI013-xxxxxx[d]-CR-0-15	SD0036	Normal	Tag #_____	Tag #_____	Tag #_____	Tag #_____	NA	NA

Table B-5. Field Sample Collection Matrix for Surface and Subsurface Sediment

				Sediment Chemistry				Equipment Rinsate Blank	
							Archive		
				PFCs (C8 to C13)	Grain Size	TOC	TBD	PFCs (C8 - C14)	
				250 mL wide mouth HDPE	500 mL WMG	125 mL WMG	250 mL WMG [a]	250 mL HDPE[a]	250 mL HDPE[a]
				10 g[e]	500 g[e]	3 g[e]	NA	250 mL[e]	
Station Number	Sample ID	Sample Number	Sample Type	Refrigerated	Refrigerated	Refrigerated	Frozen	Refrigerated	Refrigerated
☐	SSI013-xxxxxx[d]-CR-15-30	SD0037	Normal	Tag #_____	Tag #_____	Tag #_____	Tag #_____	NA	NA
☐	SSI013-xxxxxx[d]-CR-30-61	SD0038	Normal	Tag #_____	Tag #_____	Tag #_____	Tag #_____	NA	NA
☐	SSI013-xxxxxx[d]-CR-61-91	SD0039	Normal	Tag #_____	Tag #_____	Tag #_____	Tag #_____	NA	NA
☐	SSI013-xxxxxx[d]-CR-91-TBD[f]	SD0040	Normal	Tag #_____	Tag #_____	Tag #_____	Tag #_____	NA	NA
☐ SSI014	SSI014-xxxxxx[d]-GR	SD0041	Normal	Tag #_____	Tag #_____	Tag #_____	Tag #_____	NA	NA
☐	SSI014-xxxxxx[d]-GR-DUP	SD0042	Field Duplicate [b]	Tag #_____	Tag #_____	Tag #_____	NA	NA	NA
☐ EQ Blank	SDEQ-902G	EQ0002	Equipment rinsate blank [c]	NA	NA	NA	NA	Tag #_____	Tag #_____
☐ SSI015	SSI015-xxxxxx[d]-GR	SD0043	Normal	Tag #_____	Tag #_____	Tag #_____	Tag #_____	NA	NA
☐ SSI016	SSI016-xxxxxx[d]-CR-0-15	SD0044	Normal	Tag #_____	Tag #_____	Tag #_____	Tag #_____	NA	NA
☐	SSI016-xxxxxx[d]-CR-15-30	SD0045	Normal	Tag #_____	Tag #_____	Tag #_____	Tag #_____	NA	NA
☐	SSI016-xxxxxx[d]-CR-30-61	SD0046	Normal	Tag #_____	Tag #_____	Tag #_____	Tag #_____	NA	NA
☐	SSI016-xxxxxx[d]-CR-61-91	SD0047	Normal	Tag #_____	Tag #_____	Tag #_____	Tag #_____	NA	NA
☐	SSI016-xxxxxx[d]-CR-91-TBD[f]	SD0048	Normal	Tag #_____	Tag #_____	Tag #_____	Tag #_____	NA	NA
☐ SSI017	SSI017-xxxxxx[d]-GR	SD0049	Normal	Tag #_____	Tag #_____	Tag #_____	Tag #_____	NA	NA
☐	SSI017-xxxxxx[d]-GR-DUP	SD0050	Field Duplicate [b]	Tag #_____	Tag #_____	Tag #_____	NA	NA	NA
☐ SSI018	SSI018-xxxxxx[d]-GR	SD0051	Normal	Tag #_____	Tag #_____	Tag #_____	Tag #_____	NA	NA
☐ SSI019	SSI019-xxxxxx[d]-CR-0-15	SD0052	Normal	Tag #_____	Tag #_____	Tag #_____	Tag #_____	NA	NA
☐	SSI019-xxxxxx[d]-CR-15-30	SD0053	Normal	Tag #_____	Tag #_____	Tag #_____	Tag #_____	NA	NA
☐	SSI019-xxxxxx[d]-CR-30-61	SD0054	Normal	Tag #_____	Tag #_____	Tag #_____	Tag #_____	NA	NA

Table B-5. Field Sample Collection Matrix for Surface and Subsurface Sediment

				Sediment Chemistry				Equipment Rinsate Blank	
							Archive		
				PFCs (C8 to C13)	Grain Size	TOC	TBD	PFCs (C8 - C14)	
				250 mL wide mouth HDPE	500 mL WMG	125 mL WMG	250 mL WMG [a]	250 mL HDPE[a]	250 mL HDPE[a]
				10 g[e]	500 g[e]	3 g[e]	NA	250 mL[e]	
Station Number	Sample ID	Sample Number	Sample Type	Refrigerated	Refrigerated	Refrigerated	Frozen	Refrigerated	Refrigerated
☐	SSI019-xxxxxx[d]-CR-61-91	SD0055	Normal	Tag #_____	Tag #_____	Tag #_____	Tag #_____	NA	NA
☐	SSI019-xxxxxx[d]-CR-91-TBD[f]	SD0056	Normal	Tag #_____	Tag #_____	Tag #_____	Tag #_____	NA	NA
☐	SSI019-xxxxxx[d]-CR-91-TBD[f]-DUP	SD0057	Field Duplicate [b]	Tag #_____	Tag #_____	Tag #_____	NA	NA	NA
☐ EQ Blank	SDEQ-903C	EQ0003	Equipment rinsate blank [c]	NA	NA	NA	NA	Tag #_____	Tag #_____
☐ SSI020	SSI020-xxxxxx[d]-GR	SD0058	Normal	Tag #_____	Tag #_____	Tag #_____	Tag #_____	NA	NA
☐ SSI021	SSI021-xxxxxx[d]-CR-0-15	SD0059	Normal	Tag #_____	Tag #_____	Tag #_____	Tag #_____	NA	NA
☐	SSI021-xxxxxx[d]-CR-15-30	SD0060	Normal	Tag #_____	Tag #_____	Tag #_____	Tag #_____	NA	NA
☐	SSI021-xxxxxx[d]-CR-30-61	SD0061	Normal	Tag #_____	Tag #_____	Tag #_____	Tag #_____	NA	NA
☐	SSI021-xxxxxx[d]-CR-61-91	SD0062	Normal	Tag #_____	Tag #_____	Tag #_____	Tag #_____	NA	NA
☐	SSI021-xxxxxx[d]-CR-91-TBD[f]	SD0063	Normal	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____	Tag #_____
☐ SSI022	SSI022-xxxxxx[d]-GR	SD0064	Normal	Tag #_____	Tag #_____	Tag #_____	Tag #_____	NA	NA
☐ SSI023	SSI023-xxxxxx[d]-GR	SD0065	Normal	Tag #_____	Tag #_____	Tag #_____	Tag #_____	NA	NA
☐	SSI023-xxxxxx[d]-GR-DUP	SD0066	Field Duplicate [b]	Tag #_____	Tag #_____	Tag #_____	NA	NA	NA
☐ SSI024	SSI024-xxxxxx[d]-CR-0-15	SD0067	Normal	Tag #_____	Tag #_____	Tag #_____	Tag #_____	NA	NA
☐	SSI024-xxxxxx[d]-CR-15-30	SD0068	Normal	Tag #_____	Tag #_____	Tag #_____	Tag #_____	NA	NA
☐	SSI024-xxxxxx[d]-CR-30-61	SD0069	Normal	Tag #_____	Tag #_____	Tag #_____	Tag #_____	NA	NA
☐	SSI024-xxxxxx[d]-CR-61-91	SD0070	Normal	Tag #_____	Tag #_____	Tag #_____	Tag #_____	NA	NA
☐	SSI024-xxxxxx[d]-CR-91-TBD[f]	SD0071	Normal	Tag #_____	Tag #_____	Tag #_____	Tag #_____	NA	NA
☐ SSI025	SSI025-xxxxxx[d]-GR	SD0072	Normal	Tag #_____	Tag #_____	Tag #_____	Tag #_____	NA	NA

Table B-5. Field Sample Collection Matrix for Surface and Subsurface Sediment

				Sediment Chemistry				Equipment Rinsate Blank	
							Archive		
				PFCs (C8 to C13)	Grain Size	TOC	TBD	PFCs (C8 - C14)	
				250 mL wide mouth HDPE	500 mL WMG	125 mL WMG	250 mL WMG [a]	250 mL HDPE[a]	250 mL HDPE[a]
				10 g[e]	500 g[e]	3 g[e]	NA	250 mL[e]	
Station Number	Sample ID	Sample Number	Sample Type	Refrigerated	Refrigerated	Refrigerated	Frozen	Refrigerated	Refrigerated
☐ EQ Blank	SDEQ-904G	EQ0004	Equipment rinsate blank [c]	NA	NA	NA	NA	Tag #_____	Tag #_____
☐ SSI026	SSI026-xxxxxx[d]-CR-0-15	SD0073	Normal	Tag #_____	Tag #_____	Tag #_____	Tag #_____	NA	NA
☐	SSI026-xxxxxx[d]-CR-15-30	SD0074	Normal	Tag #_____	Tag #_____	Tag #_____	Tag #_____	NA	NA
☐	SSI026-xxxxxx[d]-CR-30-61	SD0075	Normal	Tag #_____	Tag #_____	Tag #_____	Tag #_____	NA	NA
☐	SSI026-xxxxxx[d]-CR-61-91	SD0076	Normal	Tag #_____	Tag #_____	Tag #_____	Tag #_____	NA	NA
☐	SSI026-xxxxxx[d]-CR-91-TBD[f]	SD0077	Normal	Tag #_____	Tag #_____	Tag #_____	Tag #_____	NA	NA
☐	SSI026-xxxxxx[d]-CR-91-TBD[f]-DUP	SD0078	Field Duplicate [b]	Tag #_____	Tag #_____	Tag #_____	NA	NA	NA

Notes:

NA = not applicable
PFC = perfluorinated compound
TBD = to be determined
TOC = total organic carbon
WMG = wide mouth glass

[a] The size and number of containers may be modified.
[b] Blind field duplicate samples will be collected at a minimum frequency of 1 field duplicate sample per 10 sediment samples; location may change due to field conditions.
[c] An equipment rinsate blank sample will be collected at a minimum frequency of 1 per 20 sediment samples.
[d] Where "xxxxxx" = month, day, and year that the sample was collected (e.g., February 28, 2014 would be 022814).
[e] Minimum sample size.
[f] Maximum of 183 cm or to refusal.

Table B-6. Geographic Coordinates of Proposed Sampling Locations for the Field Investigation[a]

Station Type	Well or Station Number	Coordinates		Coordinates	
		Northing[b]	Easting[b]	Latitude[c]	Longitude[c]
Groundwater Stations					
Shallow	M/H-4	368399	292485	39.8427	-75.2110
Shallow	MW-1	368224	293066	39.8422	-75.2089
Shallow	MW-10S	367943	292840	39.8414	-75.2097
Shallow	MW-15S	369101	292940	39.8446	-75.2094
Shallow	MW-17S	369100	293425	39.8446	-75.2077
Shallow	MW-6S	367840	292523	39.8411	-75.2109
Shallow	P-2S	369084	291306	39.8445	-75.2152
Shallow	P-5S	368581	292415	39.8432	-75.2113
Shallow	P-6S	369113	292187	39.8446	-75.2121
Shallow	WCC-1	368664	293351	39.8434	-75.2079
Shallow	WCC-2	368631	292722	39.8433	-75.2102
Shallow	WCC-3	368857	292444	39.8439	-75.2112
Shallow	WCC-5	369562	292490	39.8459	-75.2110
Shallow	WCC-7	368723	293088	39.8436	-75.2089
Intermediate	M/H-4D	368402	292482	39.8427	-75.2110
Intermediate	M/H-6D	369116	293056	39.8446	-75.2090
Intermediate	M/H-7D	368729	293099	39.8436	-75.2088
Intermediate	MW-10I	367943	292840	39.8414	-75.2097
Intermediate	MW-11D	368186	293536	39.8415	-75.2098
Intermediate	MW-24I	370455	292250	39.8483	-75.2119
Intermediate	MW-5D	368061	293188	39.8418	-75.2085
Intermediate	MW-5I	368061	293185	39.8418	-75.2085
Intermediate	MW-6I	367840	292522	39.8411	-75.2109
Deep	M/H-1D	368670	293364	39.8434	-75.2079
Deep	M/H-2D	368637	292730	39.8433	-75.2102
Deep	MW-11DD	368189	293543	39.8421	-75.2073
Deep	MW-24D	370455	292250	39.8483	-75.2119

Table B-6. Geographic Coordinates of Proposed Sampling Locations for the Field Investigation[a]

Station Type	Well or Station Number	Coordinates Northing[b]	Coordinates Easting[b]	Coordinates Latitude[c]	Coordinates Longitude[c]
Deep	MW-3D	367862	292568	39.8412	-75.2107
Deep	MW-5DD	368062	293179	39.8418	-75.2085
Confined	MW-10X	367969	292819	39.8415	-75.2098
Confined	MW-5X	368050	293062	39.8417	-75.2090
Surface Water and Sediment Stations					
DRBC	SSI001	228929	206004	39.4575	-75.5134
DRBC	SSI002	229444	203144	39.4588	-75.5235
DRBC	SSI003	306939	206894	39.6717	-75.5133
DRBC	SSI004	308495	207386	39.6760	-75.5117
Additional Location	SSI005	313208	207700	39.6889	-75.5107
Additional Location	SSI006	314632	207686	39.6928	-75.5108
Additional Location	SSI007	316072	208068	39.6968	-75.5095
DRBC	SSI008	354418	242436	39.8031	-75.3888
DRBC	SSI009	355218	243463	39.8053	-75.3851
Airport	SSI010	373642	281691	39.8568	-75.2496
Airport	SSI011	373988	282176	39.8578	-75.2479
Airport	SSI012	374075	282609	39.8580	-75.2464
Creek	SSI013	372119	287110	39.8528	-75.2303
Creek	SSI014	372222	287319	39.8530	-75.2295
Outfall	SSI015	372707	288048	39.8544	-75.2270
Outfall	SSI016	372915	288290	39.8550	-75.2261
Outfall	SSI017	372707	288602	39.8544	-75.2250
Creek	SSI018	373170	290171	39.8557	-75.2194
Creek	SSI019	373264	290195	39.8560	-75.2193
Dredge Spoils	SSI020	376727	296815	39.8656	-75.1958
Dredge Spoils	SSI021	376983	297211	39.8663	-75.1944
Dredge Spoils	SSI022	377332	297583	39.8673	-75.1931
DRBC	SSI023	419991	335214	39.9851	-75.0600

Table B-6. Geographic Coordinates of Proposed Sampling Locations for the Field Investigation[a]

Station Type	Well or Station Number	Coordinates		Coordinates	
		Northing[b]	Easting[b]	Latitude[c]	Longitude[c]
DRBC	SSI024	420229	335444	39.9858	-75.0592
DRBC	SSI025	491642	423641	40.1829	-74.7451
DRBC	SSI026	491531	423989	40.1826	-74.7439

Notes:
DRBC = Delaware River Basin Commission

[a] These proposed station coordinates are approximate and may be adjusted in the field based on field conditions at the time of sampling.

[b] New Jersey State Plane feet (NAD83).

[c] World Geodetic System (WGS84) decimal degrees.

Table B-7. Solvay Site Wells

Count	Well ID	Zone	Ground Elevation (ft NAVD88)	TOC Elevation (ft NAVD88)	Depth to TOS (ft TOC)	Screen Length (ft)
1	M/H-4	Shallow	11.8	11.44	11.64	10
2	MW-1	Shallow	12.18	13.77	14.45	10
3	MW-10S	Shallow	11.91	14.74	14.83	10
4	MW-15S	Shallow	11.9	14.46	12.56	15
5	MW-17S	Shallow	14.59	16.74	16.16	10
6	MW-6S	Shallow	12.21	14.67	16.46	10
7	P-2S	Shallow	11.87	13.62	15.75	15
8	P-5S	Shallow	11.63	13.56	16.24	15
9	P-6S	Shallow	12.97	14.18	14.38	15
10	WCC-1	Shallow	12.42	13.68	16.76	10
11	WCC-2	Shallow	11.64	13.65	17	10
12	WCC-3	Shallow	12.59	14.31	19.72	10
13	WCC-5	Shallow	7.66	9.74	14.07	10
14	WCC-7	Shallow	12.14	13.98	17.34	10
15	M/H-4D	Intermediate	11.87	11.55	74.69	10
16	M/H-6D	Intermediate	12.36	14.39	79.03	10
17	M/H-7D	Intermediate	12.08	13.94	39.86	10
18	MW-10I	Intermediate	12.04	14.08	35.04	10
19	MW-11D	Intermediate	11.98	14.18	80.21	10
20	MW-24I	Intermediate	4.969	7.539	45.57	10
21	MW-5D	Intermediate	12.55	14.74	75.18	10
22	MW-5I	Intermediate	12.44	14.71	64.54	10
23	MW-6I	Intermediate	12.16	14.91	61.6	10
24	M/H-1D	Deep	12.53	14.5	96.47	10
25	M/H-2D	Deep	11.82	13.75	41.93	10
26	MW-11DD	Deep	12.03	13.86	94.83	10
27	MW-24D	Deep	4.97	7.62	65.65	10
28	MW-3D	Deep	12.26	14.16	114.89	10
29	MW-5DD	Deep	12.22	14.71	111.49	10
30	MW-10X	Confined	11.71	14.15	192	15
31	MW-5X	Confined	11.72	14.15	189	10

Notes:
NAVD8 = North American Vertical Datum of 1988
TOC = top of casing
TOS = top of screen

ATTACHMENT B1

SITE HEALTH AND SAFETY PLAN

SITE HEALTH AND SAFETY PLAN

Perfluorinated Compounds Work Plan

Attachment B-1 to the Field and Sampling Plan

Prepared for
Solvay Specialty Polymers USA, LLC
10 Leonard Lane
West Deptford, NJ 08086

Prepared by

200 Harry S. Truman Pkwy.
Suite 330
Annapolis, MD 21401

November 15, 2013

CONTENTS

LIST OF ATTACHMENTS

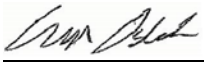
ACRONYMS AND ABBREVIATIONS

CHSM	Corporate Health and Safety Manager
CPR	cardiopulmonary resuscitation
FSP	field sampling plan
HAZWOPER	Hazardous Waste Operations and Emergency Response
HSP	health and safety plan
IDLH	immediately dangerous to life and health
Integral	Integral Consulting Inc.
MUA	Municipal Utility Authority
OSHA	Occupational Safety and Health Administration
PEL	permissible exposure limit
PFC	perfluorinated compound
PFD	personal flotation device
PFNA	perfluorononanoic acid
PPE	personal protective equipment
HSP	site health and safety plan
Site	West Deptford, New Jersey, Plant
Solvay	Solvay Specialty Polymers USA, LLC
SSO	site safety officer
STEL	short-term exposure limit
TRIS-HCL	tris hydrochloride
TRIS	tris (hydroxymethyl) aminomethane
USCG	U.S. Coast Guard

SITE HEALTH AND SAFETY PLAN APPROVAL

This site health and safety plan has been reviewed and approved for groundwater, surface water, and sediment sampling at the West Deptford, New Jersey, Plant.

Philip Goodrum, Project Manager — November 15, 2013 Date



Eron Dodak, Corporate Health and Safety Manager — November 15, 2013 Date

SITE HEALTH AND SAFETY PLAN ACKNOWLEDGMENT

In the absence of an appropriate subcontractor or consultant health and safety plan, and with the written approval of Integral Consulting Inc. (Integral) corporate health and safety manager, the subcontractor or consultant may utilize the Integral site health and safety plan (HSP), provided there is written concurrence from the subcontractor or consultant that they will directly administer the plan for their employees and assume all risks associated with any possible errors or omissions in the plan. This HSP does not cover any construction activities. The Integral HSP is a minimum standard for the site and will be strictly enforced for all Integral personnel, or its subcontractors or consultants where applicable.

I have reviewed the HSP prepared by Integral, dated November 15, 2013, for the fieldwork associated with the perfluocarbon compound work plan at the West Deptford, New Jersey, Plant. I understand the purpose of the plan, and I consent to adhere to its policies, procedures, and guidelines while an employee of Integral, or its subcontractors or consultants. I have had an opportunity to ask questions regarding this plan, which have been answered satisfactorily by Integral.

Employee signature	Company	Date
Employee signature	Company	Date
Employee signature	Company	Date
Employee signature	Company	Date
Employee signature	Company	Date
Employee signature	Company	Date
Employee signature	Company	Date

1 INTRODUCTION

It is the policy of Integral Consulting Inc. (Integral) to provide a safe and healthful work environment that is compliant with applicable regulations. No aspect of the work is more important than protecting the health and safety of all workers.

This site health and safety plan (HSP) provides general health and safety provisions to protect workers from potential hazards during field activities at and near the West Deptford, New Jersey, Plant (Site). Note that some field activities will occur outside the Site boundaries (i.e., offsite); for purposes of this HSP, references to the "Site" are understood to mean both onsite and offsite components of the overall work plan, unless specified otherwise. The state of New Jersey does not have state health and safety rules or guidance for the private sector, but complies with the federal Occupational Safety and Health Administration (OSHA) safety regulations (29 CFR [Code of Federal Regulations] 1910 and 29 CFR 1926). This HSP has been prepared in accordance with federal OSHA regulations.

Attachments to the HSP provide a site-specific map and specific routes to the hospital from the Site (Attachment 1), regulatory notices (Attachment 2), safety procedures (Attachment 3), material safety data sheets (Attachment 4), an employee exposure/injury incident report (Attachment 5), and a near-miss incident report (Attachment 6).

This HSP has been prepared to identify potential Site hazards to the extent possible based on information available to Integral. Integral cannot guarantee the health or safety of any person entering the Site. Because of the potentially hazardous nature of the Site and the activity occurring thereon, it is not possible to discover, evaluate, and provide protection for all possible hazards that may be encountered. Strict adherence to the health and safety guidelines set forth herein will reduce, but not eliminate, the potential for injury and illness at the Site. The health and safety guidelines in this plan were prepared specifically for this Site and should not be used on any other site without prior evaluation by trained health and safety personnel.

A copy of this HSP must be in the custody of the field crew during field activities. All individuals performing fieldwork must read, understand, and comply with this plan before undertaking field activities. Once the information has been read and understood, the individual must sign the Site Health and Safety Plan Acknowledgment form provided as part of this plan. The signed form will become part of the project file.

This plan may be modified at any time based on the judgment of the Integral site safety officer (SSO) in consultation with the project manager and Integral corporate health and safety manager (CHSM) or designee. Any modification will be presented to the onsite team during a safety briefing and will be recorded in the field logbook.

1.1 OBJECTIVES AND METHODS

Groundwater, surface water, and sediment will be collected at the Site. The primary objectives of the field sampling for this study are to:

- Quantitatively assess the presence or absence of perfluorinated compounds (PFCs) in municipal water supplies by collecting finished (treated) and/or raw water from accessible wells at seven identified Municipal Utility Authorities (MUAs) that are in the vicinity of the Site
- Quantitatively assess the presence or absence of PFCs in groundwater obtained from onsite monitoring wells screened in shallow, intermediate, and deep zones of the unconfined aquifer and samples from monitoring wells in the confined aquifer
- Quantitatively assess the presence or absence of PFCs in the surface water and sediment offshore of the Site as well as locations upstream and downstream from the Site
- Determine if concentrations of PFCs in surface water and sediment in the shoreline proximal to the Site are elevated with respect to locations upstream and downstream of the Site
- Conduct air modeling to assess the potential aerial extent of historic emissions of PFCs from the Site.

To meet these objectives, four types of field activities are planned: 1) groundwater or other water supply sampling at MUAs; 2) groundwater sampling at onsite monitoring wells; 3) surface water sampling from the Delaware River; and 4) sediment sampling from the Delaware River (both surface grabs and sediment cores). Additional details on the respective field methods for each type of media to be sampled are presented in the field sampling plan (FSP), Appendix B to the work plan.

1.2 ORGANIZATION

This HSP covers three of the four field activities: groundwater sampling of onsite wells, surface water sampling, and sediment sampling. Water supply sampling at each of the MUAs is addressed in separate addenda to the FSP, and each addendum includes an HSP that is tailored to the sampling effort at the MUA. Chemical and physical hazard evaluations are presented in Sections 2 and 3, respectively. Specific health and safety guidelines associated with each task, including a brief description of the work, are discussed in Section 11 (Task-Specific Safety Procedures).

1.3 ROLES AND RESPONSBILITIES

All Integral personnel, subcontractors, or consultants and visitors on the Site must comply with the requirements of this HSP. The specific responsibilities and authority of management, safety and health, and other personnel on the Site are detailed in the following paragraphs.

1.3.1 Site Safety Officer

The SSO has full responsibility and authority to implement this HSP and to verify compliance. The SSO reports to the project manager and is onsite or readily accessible to the Site during all work operations. The SSO is responsible for assessing Site conditions and directing and controlling emergency response activities. The specific responsibilities of the SSO include:

- Managing the safety and health functions on the Site
- Serving as the onsite point of contact for safety and health concerns
- Assessing Site conditions for unsafe acts and conditions and ensuring corrective action
- Ensuring that all Integral employees and subcontractors understand and follow the HSP
- Ensuring that daily work schedules and tasks are reasonable for the required levels of effort and weather conditions
- Confirming local emergency response phone numbers and locations
- Conducting and documenting the initial and daily or periodic health and safety briefings
- Evaluating and modifying the level of protective apparel and safety equipment, based on Site conditions
- Ensuring that the field team observes all necessary decontamination procedures.

If the SSO determines that Site conditions are unsafe, he or she has the authority to suspend field operations until the problem is corrected. The SSO can modify HSP procedures in the field. Any changes must be documented in the field logbook, and field staff must be immediately informed of the change. The project manager and Integral's CHSM must be notified by phone or email within 24 hours of any major changes to the HSP.

1.3.2 Project Manager

The project manager has overall responsibility to ensure that personnel conducting field work are safe. The specific responsibilities of the project manager include:

- Ensuring that the HSP is developed prior to the field work or site visit
- Reviewing and approving the HSP prior to the field work or site visit

- Ensuring employee understanding of and compliance with the HSP.

1.3.3 Corporate Health and Safety Manager

The CHSM provides guidance to the project manager and SSO on HSP preparation and reviews and approves the HSP. The CHSM also serves as an arbitrator if there is a conflict between the project manager, SSO, and field personnel. In addition, the CHSM[1] conducts periodic unannounced audits of Integral field operations to ensure compliance with the HSP.

1.3.4 Field Personnel

All Integral personnel and subcontractors on the Site are responsible for reading and complying with this HSP, using the proper personal protective equipment (PPE), reporting unsafe acts and conditions, and following the work and safety and health instructions of the project manager and SSO. All Integral personnel, subcontractors, or consultants can and are encouraged to suspend field operations if they feel conditions have become unsafe.

1.4 SITE DESCRIPTION

- **Owners/tenants:** Solvay Specialty Polymers USA, LLC (Solvay)
- **Site history:** The Site was first developed into an industrial facility by National Steel Company and manufactured steel onsite through the 1960s. In 1970, the Site was purchased by Pennwalt and chlorinated fluorocarbon propellants and refrigerants were produced onsite. In 1985, production of polyvinylidene fluoride resin began at the Site.
- **Current site use:** Currently, fluoropolymers, fluorocarbons, and fluoroelastomers are being produced onsite.
- **Hazardous waste site:** No
- **Industrial waste site:** No
- **Topography (if applicable):** Not applicable
- **Site access:** Escorted by facility compliance manager (Mitch Gertz or delegate) when onsite
- **Nearest drinking water/sanitary facilities:** Water department main office (groundwater) or onboard sampling vessel (surface water and sediment)
- **Nearest telephone:** Field personnel will carry mobile phones
- **Size of site: 243 acres/** 34 acres used for active manufacturing operations

[1] The audit task may be delegated to an office health and safety representative by the CHSM.

- Pathways for hazardous substance dispersion: None

A detailed site map for each sampling event (by media type) is provided in the FSP.

1.5 PROJECT MANAGER AND OTHER KEY CONTACTS

	Name (Affiliation)	Work Telephone	Cell Phone
Project manager	Phillip Goodrum (Integral)	(315) 446-5090	(315) 396-6655
Site safety officer	Matt Behum (Integral)	(410) 573-1982	(443) 454-1615
Corporate health and safety manager	Eron Dodak (Integral)	(503) 943-3614	(503) 407-2933
Client/facility contact	Mitch Gertz Solvay Specialty Polymers USA, LLC	(856) 251-3360	(856) 371-9318

2 CHEMICAL HAZARD EVALUATION

Potentially hazardous chemicals known to exist at the Site are primarily PFCs. While PFCs have been detected in groundwater and surface water within the Delaware River watershed, PFCs are not volatile and are not expected to enter the field crew's breathing zone.

For this investigation, sample collection equipment (for all media types) will be decontaminated using a standard detergent (e.g., Alconox® or Liquinox®) and water.

Nitric acid, sulfuric acid, and Trizma will be used as sample preservatives. Trizma will be included in the sample kit provided by the analytical laboratory for the groundwater and surface water samples. The preservative consists of tris hydrochloride (TRIS-HCL) and tris (hydroxymethyl) aminomethane (TRIS) that will be mixed in a ratio of 15.5/1 for a 1.4-g quantity per sample bottle:

Chemical	CASRN	Ratio (parts)
TRIS-HCL	1185-53-1	15.5
TRIS	77-86-1	1.0

The following table lists the historical Site maximum constituent concentrations for constituents at the West Deptford NJ Plant. There is no established OSHA permissible exposure limit (PEL), short-term exposure limit (STEL), or immediately dangerous to life and health (IDLH) level for PFCs.

Chemical Properties

Chemical of Concern	Concentration (site maximum or range expected)	Medium	OSHA PEL (ppm)	OSHA STEL (ppm)	OSHA IDLH (ppm)	IP(eV)	Carcinogen or Other Hazard
PFCs	0-0.2 μg/L	Groundwater	--	--	--	--	--
1,1,1-Trichloroethane	29,600 μg/L	Groundwater	350 ppm	NIOSH 350 ppm (15 min. ceiling)	700 ppm	11.00	Combustible liquid
1,1-Dichloroethane	1,930 μg/L	Groundwater	100 ppm	--	3,000 ppm	11.06	Flammable liquid
1,1-Dichloroethene	3,250 μg/L	Groundwater	--	--	--	10.00	Ca
Carbon Tetrachloride	<100 μg/L	Groundwater	10 ppm	25 ppm C (NIOSH ST 2 ppm)	200 (Ca)	11.47	Ca
1-Chloro-1,1-difluoroethane (142B)	8,420 μg/L	Groundwater	--	--	--	--	Vapor is heavier than air; flammable gas
1,1,1-Trifluoroethane (143A)	6,530 μg/L	Groundwater	--	--	--	--	Flammable gas
1,1-Dichloro-1-Fluoroethane (141B)	14,100 μg/L	Groundwater	--	--	--	--	Vapor is heavier than air
Arsenic	<3.0 μg/L	Groundwater	0.010 mg/m^3	NIOSH 0.002 mg/m^3, (15 min. ceiling; Ca)	5 mg/m^3 (Ca)	--	Ca
PFNA	240 ng/L	Delaware River surface water	--	--	--	--	--
Sulfuric acid	Concentrate	Preservative in all aqueous samples	1 mg/m^3	--	15 mg/m^3	--	Corrosive
Nitric acid	Concentrate	Preservative in all aqueous samples	2	4	25	11.95	Corrosive
Alconox®/ Liquinox®	Product	Powder/liquid	--	--	--	--	--

Chemical of Concern	Concentration (site maximum or range expected)	Medium	OSHA PEL (ppm)	OSHA STEL (ppm)	OSHA IDLH (ppm)	IP(eV)	Carcinogen or Other Hazard
Trizma (Tris 1 Molar Buffer)	3-10% by weight	Preservative in all aqueous samples	--	--	--	--	Irritant

Notes:

--	= none established
C	= ceiling
Ca	= carcinogen
IDLH	= immediately dangerous to life and health
IP(eV)	= ionization potential (electron volts)
Irritant	= may cause irritation to eyes, skin, respiratory tract, or digestive system
μg/L	= micrograms per liter
ng/L	= nanograms per liter
PEL	= permissible exposure limit
PFC	= perfluorinated compound
PFNA	= perfluorononanoic acid
ppm	= parts per million
ST	= short term
STEL	= short-term exposure limit

The table below summarizes the chemical characteristics and potential chemical exposure routes at the Site.

	Likely	Possible	Unlikely
Potential Chemical Exposure Routes at the Site:			
Inhalation		X (nitric and sulfuric acids)	X (site chemicals)
Ingestion			X
Skin absorption		X	
Skin contact		X	
Eye contact		X	
Chemical Characteristics:			
Corrosive		X (nitric and sulfuric acids)	X (site chemicals)
Flammable			X
Ignitable			X
Reactive			X
Volatile			X
Radioactive			X
Explosive			X
Biological agent			X
Particulates or fibers			X
If likely, describe:	Nitric and sulfuric acids are corrosive. Always wear nitrile gloves and safety glasses or goggles when filling sample containers with these acids.		

3 PHYSICAL HAZARD EVALUATION AND GUIDELINES

The following sections present general physical hazards and overwater work guidelines.

3.1 GENERAL PHYSICAL HAZARDS

The following table presents possible physical hazards that are expected to be present during field activities.

Possible Hazard	Yes	No	Proposed Safety Procedure
Heavy equipment	X		Stay back from operating equipment; for groundwater sampling wear safety vests; wear hard hats when operating heavy equipment; coordinate and maintain eye contact with equipment operator.
Material handling	X		Lift properly; seek assistance if necessary; do not overfill coolers or boxes. Seek assistance if drums must be moved.
Adverse weather	X		Seek shelter during electrical storms or extreme cold; work in adverse weather conditions only with proper training and equipment.
Work in remote areas	X		Use buddy system; carry radio and/or cellular/satellite phone; bring sufficient equipment in case of accident or injury (first aid kit, shelter if appropriate).
Plant/animal hazards	X		Know local hazards and take appropriate precautions. Use insect repellent if mosquitoes are persistent.
Uneven terrain/tripping	X		Use caution, wear properly fitting shoes or boots, and keep work area orderly.
Heat stress		X	Follow heat stress information (Attachment 3). *Note:* potential for heat stress will depend on season and location of the site.
Cold/hypothermia	X		Keep warm and dry; bring changes of clothes; do not work in extreme conditions without proper equipment and training. Follow cold stress information (Attachment 3). *Note:* potential for cold/hypothermia will depend on season and location of the site.
Falling objects	X		Wear hard hats near overhead hazards (i.e., winch).

Summary of potential physical hazards posed by proposed Site activities:

Activity	Potential Hazard
Groundwater sampling	Uneven terrain/tripping, cold/hypothermia, possible heavy equipment and vehicles moving onsite, material handling, adverse weather
Surface water and sediment sampling	Uneven terrain/tripping, cold/hypothermia, drowning, falling objects, heavy equipment, material handling, adverse weather, vessel operations (see Section 3.2)
Sample handling/mobilization	Material handling

3.2 OVERWATER WORK GUIDELINES

3.2.1 General Overwater Safety Guidelines

The overwater safety program requires the following:

- Wear U.S. Coast Guard (USCG) approved personal flotation device (PFD) at all times when working over water greater than 3 ft deep. Inspect the PFDs prior to use and do not use defective PFDs.
- The boat operator must have training in the safe operation of the boat (Section 6.1).
- No smoking is allowed on boats or near refueling activities.
- Keep sampling equipment on boats organized at all times.
- Boats are required to be equipped with a throwable life ring, fire extinguisher, first aid kit, eyewash bottle and water (if acids are taken on the boat), drinking water (for long trips), alternate propulsion mechanism (e.g., paddles), rope, and warning horn; each field member will be briefed on the storage location of this equipment on the first day of the field event.
- Use all equipment in accordance with the manufacturers' recommendations.

The following table summarizes possible physical hazards that are expected to be present during overwater work field activities.

Possible Hazard	Yes	No	Proposed Safety Procedure
Water hazards	X		Wear a USCG-approved PFD at all times when working over water greater than 3 ft deep. Inspect the PFDs prior to use and do not use defective PFDs. Keep sampling equipment on boats organized at all times. Boats are required to be equipped with a throwable life ring, fire extinguisher, and warning horn, and each field member will be briefed on the storage location of these safety items on the first day of the field event.
Vessel operations	X		Exercise prudent overwater safety.

3.2.2 Sampling Vessel Operations

The physical hazards associated with the deployment and retrieval of sampling equipment from a sampling vessel result from the equipment's weight and the method of deployment. Only trained personnel will deploy and retrieve sampling gear. Under circumstances of potentially dangerous waves or winds, the vessel pilot and field team leader will employ best professional judgment to ensure safe field operations.

To avoid injuries from heavy equipment, personnel will wear steel-toe boots when working on the work deck or loading/unloading heavy equipment from the vessel. Hard hats will be worn by personnel when present on the work deck due to the proximity of overhead gear. Sample handling equipment, containers, deck lines, hydraulic cables, and water hoses not in immediate use will be kept clear of walkways and work areas until needed. Each time sampling operations at a given location have been completed, excess sediment on the deck will be washed from the deck over the sampling location or, if specified in the field sampling plan (depending the anticipated level of contamination) will be containerized in U.S. Department of Transportation-approved 55-gallon drums to 1) prevent personnel from slipping, 2) minimize personnel exposure to potentially contaminated sediment, and 3) limit cross-contamination between sample locations.

USCG-approved PFDs will be provided for and worn by all personnel working on the deck, or as directed by the Integral SSO or vessel operator. As mentioned above, the vessel must also be equipped with throwable life rings, fire extinguishers, and warning horns, and each crewmember will be briefed on the location of this equipment prior to initiation of the sampling event.

3.2.3 U.S. Coast Guard Notification

If required for the body of water that will be sampled, the USCG will be notified of the schedule and scope of the overwater sampling work. If the USCG deems a notice to other mariners to be necessary, then information will be provided by Integral to the USCG to make barge and other river traffic aware of their sampling activities.

4 PERSONAL PROTECTIVE EQUIPMENT AND SAFETY EQUIPMENT

The following sections address PPE and safety equipment required for completing the field activities.

4.1 PERSONAL PROTECTIVE EQUIPMENT

Based on the hazards identified above in Sections 2 and 3, the following table identifies the PPE required for Site activities.

	Level of Protection	
Site Activity	Initial	Contingency[a]
Groundwater sampling	D	Leave site
Surface water and sediment sampling	MD*	Leave site
Sample handling	D	MD
Decon	D	MD

Notes:
[a] Based on unexpected change in Site conditions.

Each level of protection will incorporate the following PPE:

Level D	Long pants and work coveralls, hard hat, nitrile gloves, eye protection, and steel-toe and steel-shank boots are required. Hearing protection is required as needed.
Level MD	Same as Level D with addition of rain gear.
Level MD*	Same as Level MD with addition of a personal flotation device.

Respirator and Respirator Cartridge Information

Is there potential for a respirator to be donned during fieldwork? Yes _____ No X

4.2 SAFETY EQUIPMENT

The following safety equipment will be onsite during the proposed field activities.

First Aid Kit Mandatory, including absorbent compress, adhesive bandages, adhesive tape, antiseptic, burn treatment, medical exam gloves, sterile pad, cardiopulmonary resuscitation (CPR) shield, triangle bandage, scissors—for cutting off the PPE from an injured person (check additional items required for the Site)

- [X] Emergency blanket
- [ ] Insect repellent
- [ ] Sunscreen
- [ ] Other: _____

Other (check the items required for this project

- [X] Eyewash
- [X] Drinking water
- [ ] Stopwatch for monitoring heart rate for heat stress monitoring[2]
- [ ] Thermoscan® thermometer for heat stress monitoring
- [ ] Survival kit[3]
- [X] Personal flotation device (onboard sampling vessel)
- [ ] Cool vests
- [ ] Fit test supplies
- [X] Fire extinguisher (onboard sampling vessel)
- [ ] Windsock
- [X] Cellular phone
- [ ] Radio sets
- [ ] Global positioning system
- [ ] Other: _____

[2] Heart rate monitoring requires special training.
[3] Consult the CHSM for guidance for site-specific survival kits.

5 AIR MONITORING

Air monitoring will not be required for this investigation. No inhalation exposure to volatile chemicals or particulates is expected.

6 HEALTH AND SAFETY TRAINING AND MEDICAL MONITORING

The following sections present requirements for health and safety training and medical monitoring.

6.1 SAFETY TRAINING AND MEDICAL MONITORING

State and federal laws establish training requirements for workers at uncontrolled hazardous waste sites (including areas where accumulations of hazardous waste create a threat to the health and safety of an individual, the environment, or both). Integral and subcontractor personnel are required to complete the following training requirements prior to working at the Site.

All field personnel will be required to complete and pass both "Basic Operations Plus" and Solvay site-specific safety courses that are available through the Delaware Valley Safety Council. Field staff will also be required to complete a brief onsite pre-project safety review session as part of Solvay's internal work permit program. All field personnel will also be required to have a valid transportation worker identification credential (e.g., valid Transportation Worker Identification Credential, or a Hazardous Materials endorsement on a commercial driver's license).

6.1.1 Training Requirements

Task	No Training	24-hour	40-hour[a]	Supervisor[b]	First Aid/CPR[c]	Medical Monitoring
Integral Field Personnel						
Matt Behum (Field Lead)			X	X	X	X
Heather Summers			X		X	X
Stefan Wodzicki			X	X	X	X
Jane Sexton			X	X	X	X
Other Integral Subcontractors[d]						
David Skitt (ELM)			X	X	X	X
Vessel operator / deckhands[e]						

See Notes Next Page

Notes:

[a] Must have current OSHA 8-hour refresher if it has been more than a year since the OSHA 40-hour training.

[b] At least one person onsite must be OSHA Hazardous Waste Operations and Emergency Response (HAZWOPER) supervisor trained if this is a hazardous waste site.

[c] At least one member of each team of two or more people onsite must be first aid/CPR trained.

[d] Integral subcontractors and consultants may have requirements that are more stringent than those listed above. These are minimum training and monitoring requirements required to work on this site.

[e] To be determined once sampling vessel has been selected. It is possible that the vessel operator will not be required to have 40-hour training. If the vessel operator does not have 40-hour training, then he/she will stay out of the exclusion and contamination reduction zones during sample collection and decontamination activities. The vessel operator and deckhands are required to have USCG training.

6.1.2 Site Safety Meetings

Site safety meetings must be held before beginning new tasks or when new staff enter the Site. Site safety meetings should be held at a minimum of once a week and should be held daily on complex or high hazard projects. Tailgate safety meetings must occur every morning during review of the day's work plan, covering specific hazards that may be encountered. Additional meetings will be held at any time health and safety concerns are raised by any of the personnel. Attendance and topics covered are to be documented in the field logbook or on daily health and safety briefing forms.

6.1.3 Site Permit Requirements

Work completed onsite requires that a Site Safe Work Permit is issued. The Safe Work Permit is a general permit to allow contractors to work onsite and is issued on a daily basis. This permit is issued by Solvay plant operations personnel depending on the area of the plant in which the work will occur. The Safe Work Permit procedure will be reviewed with field personnel prior to any onsite work being initiated. Additional permits (e.g., a hot work permit) may be required depending on the area of the plant and the type of activity that will be occurring.

6.2 MEDICAL MONITORING

OSHA requires medical monitoring for personnel potentially exposed to chemical hazards in concentrations in excess of the PEL for more than 30 days per year and for personnel who must use respiratory protection for more than 30 days per year. Integral requires medical monitoring for all employees potentially exposed to chemical hazards.

Will Integral personnel working at the Site be enrolled in a medical monitoring program?	Yes X	No _____

7 EMERGENCY RESPONSE PLAN

The following sections discuss emergency recognition and prevention, emergency response and notification, emergency decontamination, site communications, and use of the buddy system.

7.1 EMERGENCY RECOGNITION AND PREVENTION

It is the responsibility of all personnel to monitor work at the Site for potential safety hazards. All personnel are required to immediately report any unsafe conditions to the SSO. The SSO is responsible to immediately take steps to remedy any unsafe conditions observed at the work site.

The following are examples of some emergency situations that could occur during the field activities:

- Slips, trips, and falls (e.g., on sloped areas, steel stairs, boat deck)
- Entrainment of clothes or objects in moving equipment or parts
- A person falls overboard
- Serious injury or illness (e.g., physical injury, heart attack)
- Severe thunderstorm with lightning.

Immediate actions will be taken by the field team under the leadership of the SSO in response to these emergencies.

7.2 EMERGENCY RESPONSE AND NOTIFICATION

If an emergency at the Site warrants it, all personnel must immediately evacuate the affected work area and report to the SSO at the predetermined emergency assembly location:

Field vehicle

In case of injury, field personnel should take precautions to protect the victim from further harm and notify local or facility emergency services. In remote areas, it will be necessary to have first aid-trained personnel on the field team. The victim may require decontamination prior to treatment if practicable—requirements will vary based on Site conditions.

Emergency medical care will be provided by:

- [X] Local emergency medical provider (i.e., fire department)
- [ ] Facility emergency medical provider
- [ ] First aid-trained field staff (for remote areas only)

Local Resources	Name	Telephone	Notified Prior to Work (Yes/No)?
Fire	West Deptford Volunteer Fire Company	911	No
Police	West Deptford Police Department	911	No
Ambulance	American Medical Response	(215) 629-2600	No
	Accurate Transport	(856) 384-4715	No
Hospital	Kennedy University Hospital	(856) 582-2500	No
Site phone	Mitch Gertz	(856) 251-6630	Yes
Directions to the hospital:	Consult attached maps		

The SSO must confirm that the hospital listed is still in operation and that it has an emergency room. **It is required that the SSO drive to the hospital so that the directions are practiced and understood prior to initiating fieldwork.**

Corporate Resource	Name	Work Telephone	Cell Phone
Integral CHSM [a]	Eron Dodak	Office: (503) 943-3614	(503) 407-2933
Integral President	Lucinda Jacobs	Office: (206) 957-0328	(206) 999-3061
Integral Human Resources Manager	Amy Logan	Office: (720) 465-3312	NA
Medical consultant	Dr. Calvin Jones (HealthForce Partners)	Office: (425) 806-5700	NA

Notes:

[a] If the CHSM cannot be reached, call Ian Stupakoff [Office: (360)705-3534, ext. 20; Cell: (360)259-2518]. If Ian Stupakoff cannot be reached, call David Livermore [Office: (503)943-3613; Cell: (503)806-4665]. If David Livermore cannot be reached, call Barbara Trenary [Office: (206) 248-9645; Cell: (206) 849-0882].

In case of serious injuries, death, or other emergency, the Integral CHSM must be notified immediately at the phone numbers listed above. The Integral CHSM will notify the project manager and Integral's President. The project manager will notify the client.

7.3 EMERGENCY DECONTAMINATION PROCEDURES

In case of an emergency, if possible, gross decontamination procedures will be promptly implemented. If a life-threatening injury occurs and the injured person cannot undergo decontamination procedures onsite, then the medical facility will be informed that the injured person has not been decontaminated and given information regarding the most probable chemicals of concern.

Decontamination procedures will only be used if practical and if they will not further injure the person or delay treatment. Decontamination procedures should not be implemented if there is not a reasonable possibility that the injured party requires such intervention. The SSO will make the determination whether or not to decontaminate the injured person. The following steps will be followed for decontaminating injured personnel while onsite:

- If it will not injure the person further, cut off PPE using scissors or scrub the gross contamination from the injured person's PPE (e.g., work boots) with a Liquinox® or Alconox® solution followed by a rinse with tap or deionized/distilled water
- Remove PPE if feasible without further injuring the person.

7.4 SITE COMMUNICATONS

Each field team will carry a cell phone or satellite phone that is in good working order. If there is any type of emergency that requires the Site to be evacuated (e.g., severe thunderstorm with lightening, chemical release), the field team leader will contact all field team personnel (either by cell phone or using an air horn if working in remote conditions). Once directed by the field team leader to evacuate, all personnel will meet at the predetermined emergency assembly location, provided the muster point is in safe territory (field vehicle or sampling vessel cabin). All other emergency notifications that do not require evacuation (e.g., a person falling overboard) will be conducted using a cell or satellite phone. Emergency phone numbers are listed above in Section 7.2.

Any injuries that require first aid or any chemical spills will be immediately reported to Solvay management.

7.5 BUDDY SYSTEM

The buddy system will be used at the Site at all times. The buddy system is a system of organizing employees into field teams in such a manner that each employee of the field team is designated to be observed by at least one other employee in the field team. The purpose of the buddy system is to provide rapid assistance to employees in the event of an emergency.

8 WORK ZONES

Work zones are defined as follows:

Exclusion zone	Any area of the Site where hazardous substances are present, or are reasonably suspected to be present, and pose an exposure hazard to personnel (e.g., approximately 15 ft around the monitoring well, or the rear deck of the sampling vessel when the winch and hydrowire/cable are in use)
Contamination reduction zone	Area between the exclusion and support zones that provides a transition between contaminated and clean zones
Support zone	Any area of the Site, so designated, that is outside the exclusion and contamination reduction zones

Site control measures in work zones are described below for each type of field activities.

8.1 GROUNDWATER SAMPLING

Exclusion zone: An approximate 15-ft radius around the monitoring well will be the exclusion zone. Only properly equipped and trained personnel (i.e., level D protective clothing) will be allowed in this area.

Contamination reduction zone: After sampling is completed at a station, the exclusion zone will become the contamination reduction zone.

Support zone: All areas outside the exclusion and contamination reduction zones.

8.2 SURFACE WATER AND SEDIMENT SAMPLING

Exclusion zone: The rear deck of the sampling vessel when the winch and hydrowire/cable are in use. Only properly equipped and trained personnel (i.e., wearing MD* protective clothing) will be allowed in this area.

Contamination reduction zone: After sampling is completed at a station, the exclusion zone will become the contamination reduction zone.

Support zone: The front deck and wheel house of the sampling vessel will be the support zone. No chemical or sample handling activities will occur in this area. Personnel will be required to wash chemicals and sediment from raingear before entering this area.

9 EQUIPMENT DECONTAMINATION AND PERSONAL HYGIENE

9.1 EQUIPMENT DECONTAMINATION PROCEDURES

After sampling is completed, the exclusion zone will be used as the contaminant reduction zone for decontamination activities, provided there is no contamination remaining after the sampling is completed. To minimize or prevent personal exposure to hazardous materials, all personnel working in the exclusion zone and contaminant reduction zone will comply with the following decontamination procedures:

- All personnel will wash sediment and chemicals from their raingear before leaving the exclusion zone.
- All gloves, rain gear, and rubber boots will be removed prior to entering the field vehicle.

Decontamination equipment required at the Site includes the following:

- Buckets or tubs
- Laboratory grade distilled/deionized water
- Site or tap water
- Scrub brushes (long-handled)
- Liquinox® or Alconox® detergent
- Garbage bags
- Clean garden sprayer.

Decontamination procedures for all non-disposable sampling equipment are provided in the FSP. No solvent rinses will be used during the proposed field events as described in the FSP.

9.2 PERSONAL HYGIENE

The following personal hygiene practices will be used at the Site to reduce exposure to chemicals.

- Long hair will be secured away from the face so it does not interfere with any activities.
- All personnel leaving potentially contaminated areas will wash their hands, forearms, and faces in the contaminant reduction zone prior to entering any clean areas or eating areas.

- Personnel leaving potentially contaminated areas will shower (including washing hair) and change to clean clothing as soon as possible after leaving the Site.
- No person will eat, drink, or chew gum or tobacco in potentially contaminated areas. Single portion drink containers and drinking of replacement fluids for heat stress control will be permitted only in support areas.
- Smoking is prohibited by Integral personnel and subcontractors in all areas of the Site because of the potential for contaminating samples and for the health of the field team.

10 VEHICLE SAFETY, SPILL CONTAINMENT, AND SHIPPING INSTRUCTIONS

10.1 VEHICLE SAFETY

Integral's vehicle safety program requires the following:

- Cell phone usage while driving is not allowed, including the use of hands-free devices. If it not feasible to wait to use the cell phone until arriving at the destination, pull off the road and park in a safe location to use the cell phone. Do not pull to the side of the road to use a cell phone because this significantly increases the risk of a rear-end collision.
- All vehicles are to be operated in a safe manner and in compliance with local traffic regulations and ordinances.
- Drivers are to practice defensive driving and drive in a courteous manner.
- Drivers are required to have a valid driver's license and liability insurance (per local state laws).
- Seat belts are to be worn by the driver and all passengers.
- No persons are allowed to ride in the back of any trucks or vans, unless equipped with seatbelts.
- Vehicles are to be driven in conformance with local speed limits.
- Personnel who are impaired by fatigue, illness, alcohol, illegal or prescription drugs, or who are otherwise physically unfit, are not allowed to drive or work on Integral field sites.
- Personnel are to avoid engaging in other distractions such as changing radio stations while driving.
- Motor vehicle accidents are to be reported to the responsible law enforcement agency, the Integral human resources manager, and the Integral CHSM on the same day of occurrence. Documentation of damage should be photographed.
- Personnel who have experienced work-related vehicle accidents or citations may be required to complete a defensive driving program.
- Drivers will comply with all posted traffic and speed signs posted at the Site.

10.2 SPILL CONTAINMENT

No bulk chemicals will be used during the proposed field events as described in the FSP.

10.3 SHIPPING INFORMATION

Federal laws and international guidelines place restrictions on what materials may be shipped by passenger and cargo aircraft. In addition, 49 CFR regulates labeling, manifesting, and shipment of all packages containing potentially hazardous materials. In the course of this field investigation, the following items will be shipped to and from the Site as shown below:

Item	Hazardous Constituent	Quantity	Packaging	How Shipped
Samples	None	31 groundwater samples	Coolers	Overnight shipping courier
		26 surface water samples		
		70 sediment samples		
Preservative	Trizma	0.1 gal	In groundwater and surface water sample bottles sent from the testing laboratory	Shipping courier
Preservative	Nitric acid	2–3 mL/bottle	Sample containers	Shipping courier
Preservative	Sulfuric acid	2–3 mL/bottle	Sample containers	Shipping courier
Other	None			

A 24-hour emergency response number (on any shipping documents such as a Uniform Hazardous Waste Manifest, Shipper's Declaration of Dangerous Goods, etc.) is required for shipments of all dangerous or hazardous goods. Integral does not have a 24-hour emergency contact number for dangerous or hazardous goods shipment. No dangerous or hazardous goods may be shipped by Integral until an account is set up with a 24-hour emergency response service such as CHEM-TEL (1-813-248-0573). If any hazardous or dangerous goods need to be shipped for a project, they must be shipped directly to the Site by the supplier. Any hazardous or dangerous goods that are not used in the course of the field effort must remain at the Site.

The samples will be prepared and labeled for shipment in accordance with the FSP developed for the Site.

Air shipment of equipment with lithium batteries is required to note the presence of these batteries. Warning labels are available from the equipment rental agency and can be copied.

11 TASK-SPECIFIC SAFETY PROCEDURE SUMMARY

11.1 GROUNDWATER SAMPLING

Field team personnel will comply with the client's safety requirements when onsite during the sampling event. A hard hat, safety glasses, and steel-toe boots are required at all times while onsite. Nitrile gloves will be worn during groundwater sample collection activities. Avoid getting groundwater or sample preservatives on clothes or skin. Exercise care when lifting equipment and coolers full of samples. Always get help when lifting heavy coolers or equipment. Keep the sampling area organized to reduce the chances of slip/trip/fall incidents. Use the buddy system at all times.

11.2 SURFACE WATER AND SEDIMENT SAMPLING

Always wear a USCG-approved PFD when doing any work on a sampling vessel or dock. Safety glasses, steel-toe boots, and nitrile gloves are required at all times without exception. A hard hat will also be required during sample collection in the exclusion zone of the larger sampling vessel; also without exception. Use hearing protection as needed.

Exercise caution when working on a boat deck. Always be aware of the surroundings and river wave action that can rock the sampling vessel without notice. Keep sampling equipment on boats organized at all times. Boats are required to be equipped with a throwable life ring, fire extinguisher, and warning horn, and each field member will be briefed on the storage location for this equipment.

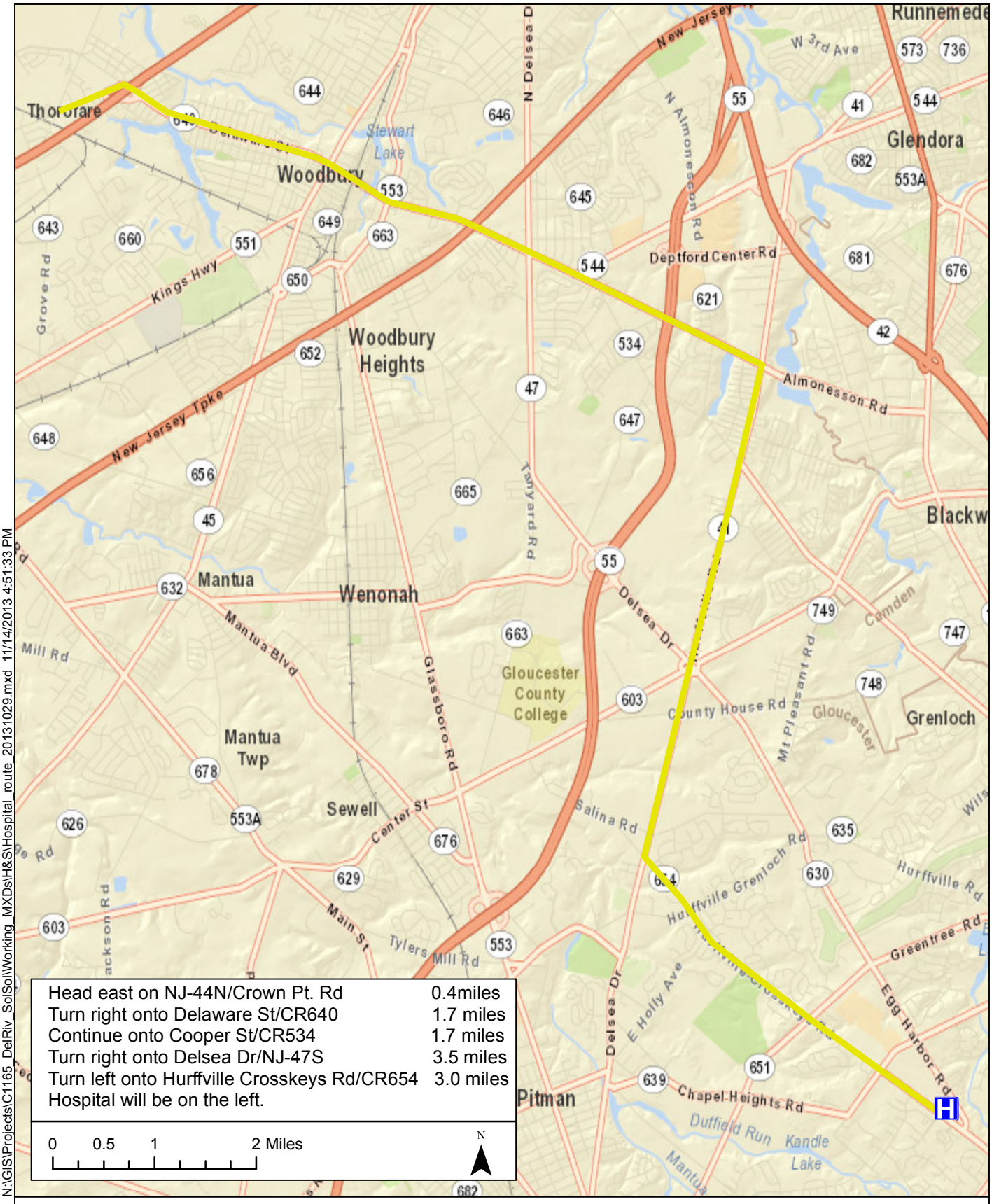
Avoid getting surface water and sediment on clothes or skin. Exercise care when lifting, assembling, and decontaminating grab samplers and coring devices. On the larger sampling vessel, always stay clear of the winch line and be aware of its location.

Exercise care when lifting equipment and coolers full of samples. Always get help when lifting heavy coolers or equipment. Keep the sampling area organized to reduce the chances of slip/trip/fall incidents. Use the buddy system at all times.

If it is necessary for personnel to enter restricted areas of the upland facility during the sampling event, then they will comply with the client's safety requirements for the Site (e.g., wear hard hats and safety glasses).

ATTACHMENT 1

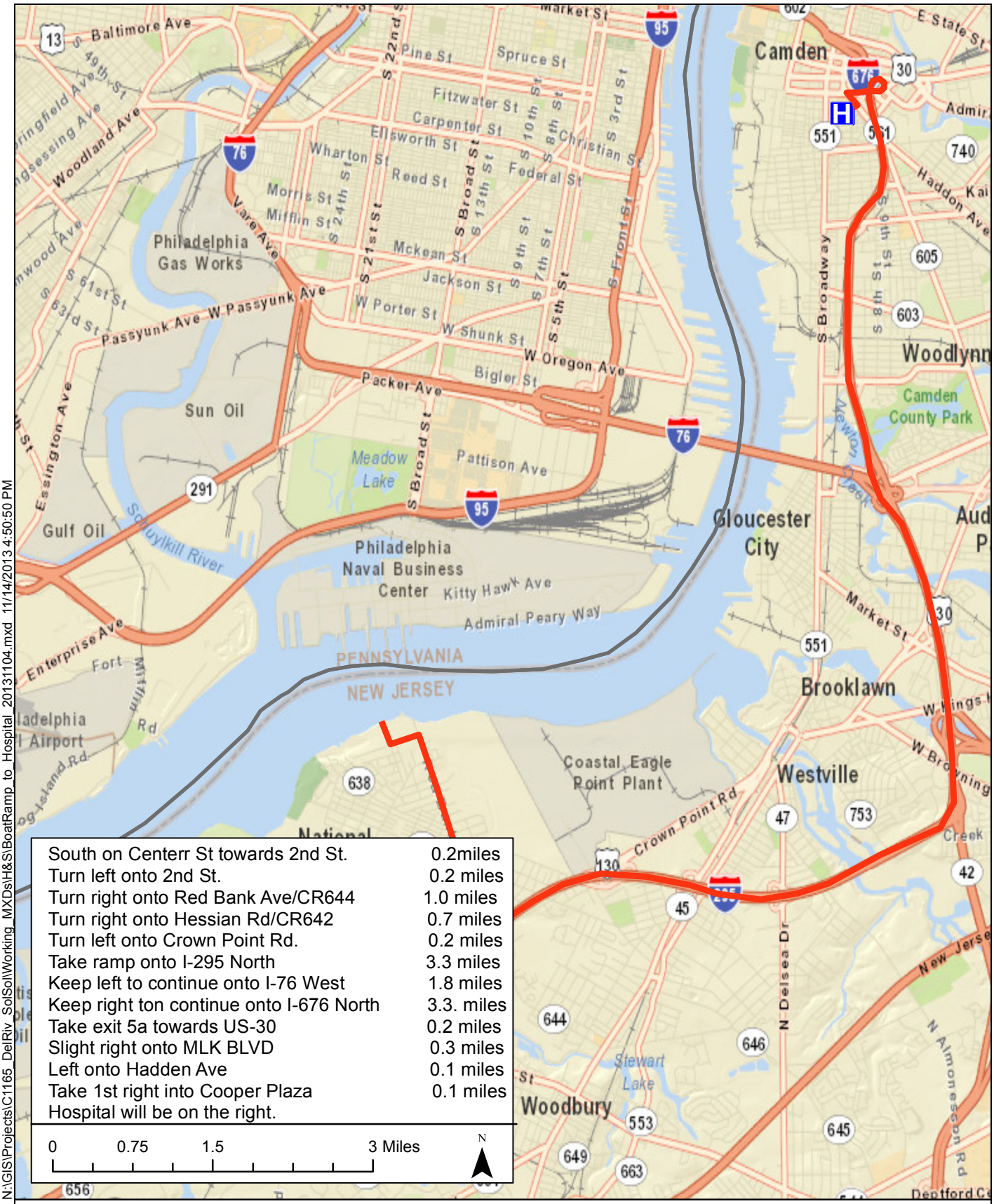
SITE MAP AND HOSPITAL ROUTE



N:\GIS\Projects\C1165_DelRiv_SolSol\Working_MXDs\H&S\Hospital_route_20131029.mxd 11/14/2013 4:51:33 PM

integral consulting inc.

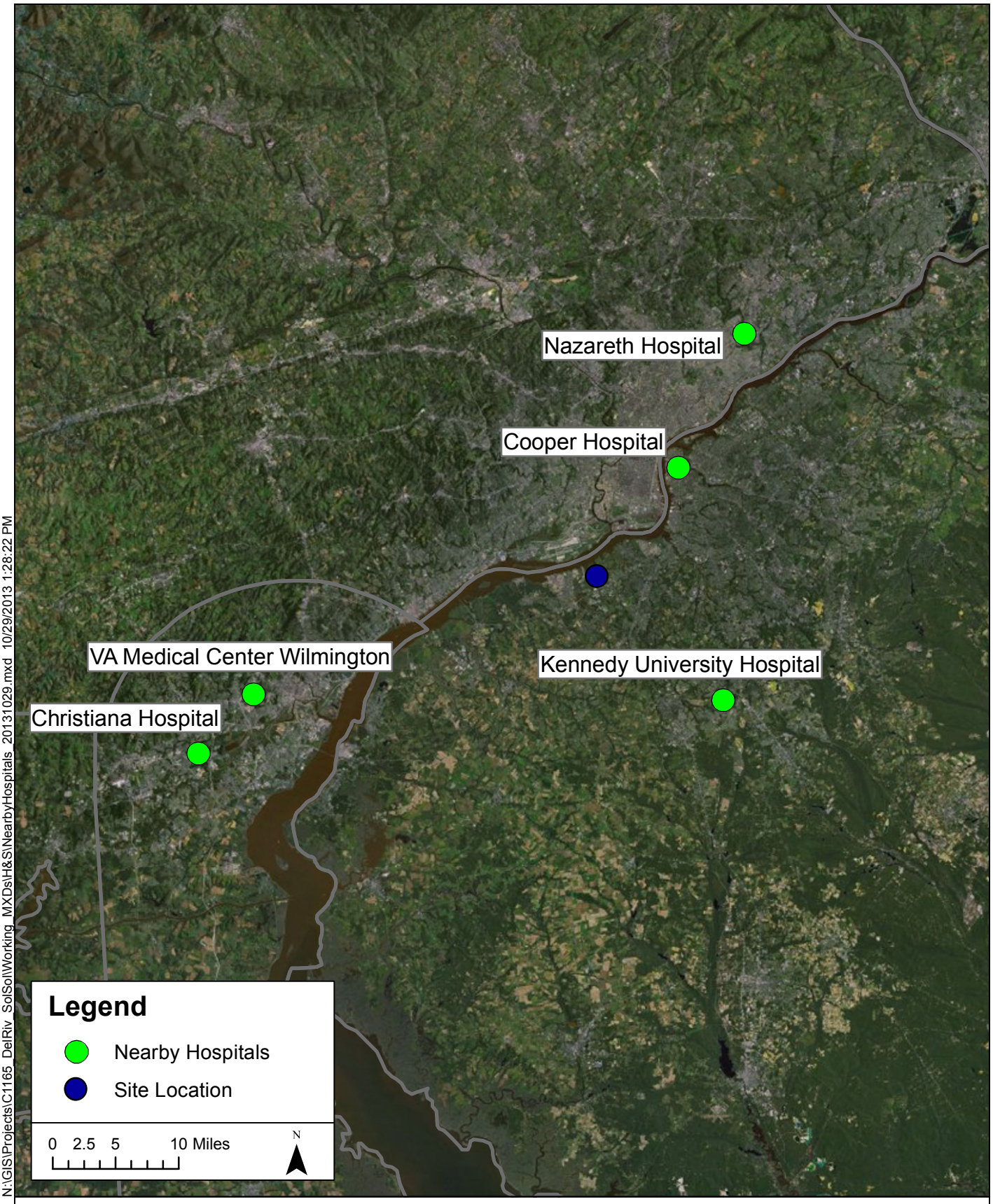
Route from Site to Nearest Emergency Room



N:\GIS\Projects\C1165_DelRiv_SolSol\Working_MXDs\H&S\BoatRamp_to_Hospital_20131104.mxd 11/14/2013 4:50:50 PM

integral consulting inc.

Route from Boat Ramp to Nearest Emergency Room - Cooper University Hospital



N:\GIS\Projects\C1165_DelRiv_SolSol\Working_MXDs\H&S\NearbyHospitals_20131029.mxd 10/29/2013 1:28:22 PM

integral consulting inc.

Emergency Rooms Upstream
and Downstream of Site

ATTACHMENT 2

REGULATORY NOTICES

You Have a Right to a Safe and Healthful Workplace.

IT'S THE LAW!

- You have the right to notify your employer or OSHA about workplace hazards. You may ask OSHA to keep your name confidential.
- You have the right to request an OSHA inspection if you believe that there are unsafe and unhealthful conditions in your workplace. You or your representative may participate in the inspection.
- You can file a complaint with OSHA within 30 days of discrimination by your employer for making safety and health complaints or for exercising your rights under the *OSH Act*.
- You have a right to see OSHA citations issued to your employer. Your employer must post the citations at or near the place of the alleged violation.
- Your employer must correct workplace hazards by the date indicated on the citation and must certify that these hazards have been reduced or eliminated.
- You have the right to copies of your medical records or records of your exposure to toxic and harmful substances or conditions.
- Your employer must post this notice in your workplace.

The *Occupational Safety and Health Act of 1970 (OSH Act)*, P.L. 91-596, assures safe and healthful working conditions for working men and women throughout the Nation. The Occupational Safety and Health Administration, in the U.S. Department of Labor, has the primary responsibility for administering the *OSH Act*. The rights listed here may vary depending on the particular circumstances. To file a complaint, report an emergency, or seek OSHA advice, assistance, or products, call 1-800-321-OSHA or your nearest OSHA office: • Atlanta (404) 562-2300 • Boston (617) 565-9860 • Chicago (312) 353-2220 • Dallas (214) 767-4731 • Denver (303) 844-1600 • Kansas City (816) 426-5861 • New York (212) 337-2378 • Philadelphia (215) 861-4900 • San Francisco (415) 975-4310 • Seattle (206) 553-5930. Teletypewriter (TTY) number is 1-877-889-5627. To file a complaint online or obtain more information on OSHA federal and state programs, visit OSHA's website at **www.osha.gov**. If your workplace is in a state operating under an OSHA-approved plan, your employer must post the required state equivalent of this poster.

1-800-321-OSHA
www.osha.gov

U.S. Department of Labor • Occupational Safety and Health Administration • OSHA 3165

ATTACHMENT 3

SAFETY PROCEDURES

FROSTBITE

What happens to the body:

Freezing in deep layers of skin and tissue; pale, waxy-white skin color; skin becomes hard and numb; usually affects fingers, hands, toes, feet, ears, and nose.

What to do: (land temperatures)

- Move the person to a warm, dry area. Don't leave the person alone.
- Remove wet or tight clothing that may cut off blood flow to the affected area.
- **Do not** rub the affected area because rubbing damaged the skin and tissue.
- Gently place the affected area in a warm water bath (105°) and monitor the water temperature to **slowly** warm the tissue. Don't pour warm water directly on the affected area because it will warm the tissue too fast, causing tissue damage. Warming takes 25-40 minutes.
- After the affected area has been warmed, it may become puffy and blister. The affected area may have a burning feeling or numbness. When normal feeling, movement, and skin color have returned, the affected area should be dried and wrapped to keep it warm. **Note:** If there is a chance the affected area may get cold again, do not warm the skin. If the skin is warmed and then becomes cold again, it will cause severe tissue damage.
- Seek medical attention as soon as possible.

How to Protect Workers

- Recognize the environmental and workplace conditions that lead to potential cold-induced illnesses and injuries.
- Learn the signs and symptoms of cold-induced illnesses/injuries and what to do to help the worker.
- Train workers about cold-induced illnesses and injuries.
- Select proper clothing for cold, wet, and windy conditions. Layer clothing to adjust to changing environmental temperatures. Wear a hat and gloves, in addition to underwear that will keep water away from the skin (polypropylene.)
- Take frequent short breaks in warm, dry shelters to allow the body to warm up.
- Perform work during the warmest part of the day.
- Avoid exhaustion or fatigue because energy is needed to keep muscles warm.
- Use the buddy system (work in pairs.)
- Drink warm, sweet beverages (sugar water, sports-type drinks.) **Avoid drinks with caffeine** (coffee, tea, or hot chocolate) **or alcohol**.
- Eat warm, high-calorie foods like hot pasta dishes.

Workers are at increased risk when…

- They have predisposing health conditions such as cardiovascular disease, diabetes, and hypertension.
- They take certain medications. Check with your doctor, nurse, or pharmacy and ask if medicines you take affect you while working in cold environments.
- They are in poor physical condition, have a poor diet, or are older.

HYPOTHERMIA - (Medical Emergency)

What happens to the body:

Normal body temperature (98.6°F/37°C) drops to or below 95°F/35°C; fatigue or drowsiness; uncontrolled shivering; cool, bluish skin; slurred speech; clumsy movements; irritable, irrational, or confused behavior.

What to do: (land temperatures)

- Call for emergency help (i.e., ambulance or 911).
- Move the person to a warm, dry area. Don't leave the person alone.
- Remove wet clothing and replace with warm, dry clothing or wrap the person in blankets.
- Have the person drink warm, sweet drinks (sugar water or sports-type drinks) if he is alert. **Avoid drinks with caffeine** (coffee, tea, or hot chocolate) **or alcohol**.
- Have the person move his arms and legs to create muscle heat. If he is unable to do this, place warm bottles or hot packs in the armpits, groin, neck, and head areas. **Do not** rub the person's body or place him in a warm water bath. This may stop his heart.

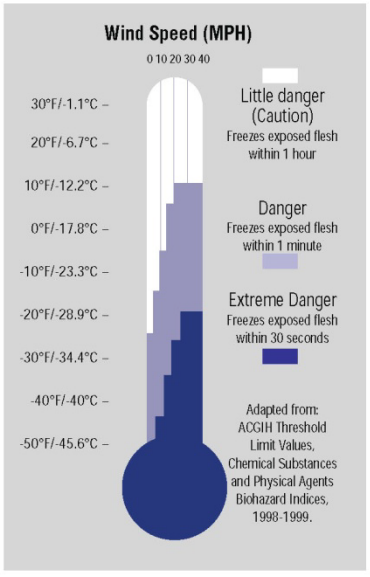
What to do: (water temperatures)

- Call for emergency help (i.e., ambulance or 911). Body heat is lost up to 25 times faster in water.
- **Do not** remove any clothing. Button, buckle, zip, and tighten any collars, cuffs, shoes, and hoods because the layer of trapped water closest to the body provides a layer of insulation that slows the loss of heat. Keep the head out of the water and put on a hat or hood.
- Get out of the water as quickly as possible or climb on anything floating. **Do not** attempt to swim unless a floating object or another person can be reached because swimming or other physical activity uses body heat and reduces survival time by about 50 percent.
- If getting out of the water is not possible, wait quietly and conserve body heat by folding arms across the chest, keeping thighs together, bending knees, and crossing ankles. If another person is in the water, huddle together with chests held closely.

THE COLD STRESS EQUATION

LOW TEMPERATURE + WIND SPEED + WETNESS = INJURIES & ILLNESS

When the body is unable to warm itself, serious cold-related illnesses and injuries may occur, and permanent tissue damage and death may result. **Hypothermia** can occur when *land temperatures* are **above** freezing or *water temperatures* are below 98.6°F/37°C. Cold-related illnesses can slowly overcome a person who has been chilled by low temperatures, brisk winds, or wet clothing.



Oregon Occupational Safety & Health Division
440-3336E S (9/03)

HEAT EXHAUSTION

What happens to the body:

Headaches, dizziness, or light-headedness, weakness, mood changes, irritability or confusion, feeling sick to your stomach, vomiting, fainting, decreased and dark-colored urine, and pale, clammy skin.

What should be done:

- Move the person to a cool shaded area. Don't leave the person alone. If the person is dizzy or light-headed, lay him on his back and raise his legs about 6-8 inches. If the person is sick to his stomach, lay him on his side.
- Loosen and remove heavy clothing.
- Have the person drink some cool water (a small cup every 15 minutes) if he is not feeling sick to his stomach.
- Try to cool the person by fanning him. Cool the skin with a cool spray mist of water or wet cloth.
- If the person does not feel better in a few minutes call for emergency help (ambulance or call 911.)

(If heat exhaustion is not treated, the illness may advance to heat stroke.)

HEAT STROKE - A Medical Emergency

What happens to the body:

Dry, pale skin (no sweating); hot red skin (looks like a sunburn); mood changes; irritability, confusion, and not making any sense; seizures or fits, and collapse (will not respond).

What should be done:

- Call for emergency help (i.e., ambulance or 911.)
- Move the person to a cool, shaded area. Don't leave the person alone. Lay him on his back and if the person is having seizures, remove objects close to him so he won't hit them. If the person is sick to his stomach, lay him on his side.
- Remove heavy and outer clothing.
- Have the person drink some cool water (a small cup every 15 minutes) if he is alert enough to drink anything and not feeling sick to his stomach.
- Try to cool the person by fanning him or her. Cool the skin with a cool spray mist of water, wet cloth, or wet sheet.
- If ice is available, place ice packs in armpits and groin area.

How to Protect Workers

- Learn the signs and symptoms of heat-induced illnesses and what to do to help the worker.
- Train workers about heat-induced illnesses.
- Perform the heaviest work during the coolest part of the day.
- Slowly build up tolerance to the heat and the work activity (usually takes up to 2 weeks.)
- Use the buddy system (work in pairs.)
- Drink plenty of cool water (one small cup every 15-20 minutes.)
- Wear light, loose-fitting, breathable (like cotton) clothing.
- Take frequent short breaks in cool, shaded areas (allow your body to cool down.)
- Avoid eating large meals before working in hot environments.
- Avoid caffeine and alcoholic beverages (these beverages make the body lose water and increase the risk of heat illnesses.)

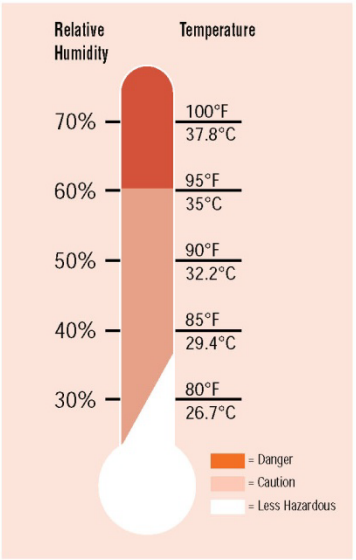
Workers are at increased risk when...

- They take certain medications. Check with your doctor, nurse, or pharmacy to see if medicines you take affect you when working in hot environments.
- They have had a heat-induced illness in the past.
- They wear personal protective equipment.

THE HEAT EQUATION

HIGH TEMPERATURE + HIGH HUMIDITY + PHYSICAL WORK = HEAT ILLNESS

When the body is unable to cool itself through sweating, **serious** heat illnesses may occur. The most severe heat-induced illnesses are **heat exhaustion** and **heat stroke**. If actions are not taken to treat heat exhaustion, the illness could progress to heat stroke and **death**.



Oregon Occupational Safety & Health Division
440-3333E-S(9/03)

ATTACHMENT 4

MATERIAL SAFETY DATA SHEETS

Klenzoid, Inc. Industrial Water Specialists
912 Spring Mill Ave. • Conshohocken, PA 19428 • 610-825-9494
P.O. Box 389 Fax -610-825-0238

MATERIAL SAFETY DATA SHEET

PRODUCT: KLENPHOS 100
APPLICATION: Drinking Water Additive

Information On Physical Hazards, Health Hazards, Pel's and TLV's For Specific Product Ingredients As Required By The OSHA Hazard Communications Standard Are Listed. Refer In Section VI For Our Assessment Of The Health Hazards Of This Formulation.

SECTION I

Manufacturer's Name: Klenzoid, Inc.
Address: 912 Spring Mill Avenue
Conshohocken, PA 19428

Emergency Telephone Number: (610) 825-9494

Telephone Number for Information: (610) 825-9494

Date Prepared: 01/12/12

SECTION II - Hazardous Ingredients/Identify Information

Tetrapotassium Pyrophosphate (30%-50%) CAS# 7320-34-5.
Zinc Pyrophosphate (<1 %) CAS # 7646-26-6
Water (50-80%) CAS # 7732-18-5

SECTION III - Physical/Chemical Characteristics

pH: 10.0-10.5
Boiling Point: 220 Degrees F
Specific Gravity (H2 0=1): 1.25
Vapor Pressure (mm Hg.): Non Volatile
Vapor Density (Air = 1): Non Volatile
Freezing Point: < 20 Degrees F
Evaporation Rate (Butyl Acetate = 1): None volatile
Solubility in Water: 100%
Appearance and Odor: Slightly Viscous, clear liquid, no odor

SECTION IV - Fire and Explosion Hazard Data

Flash Point (Method Used): Not Combustible
Special Fire Fighting Procedures: N/A
Unusual Fire and Explosion Hazards: N/A
This material is not flammable.

SECTION V - Reactivity Data

Stability:
Stable: Yes

Incompatibility (Materials to Avoid): Corrosive to Aluminum based on DOT 49 CFR
Hazardous Decomposition or Byproducts: None
Hazardous Polymerization: Will not occur.

SECTION VI - Health Hazard Data

Inhalation: No Published Data
Ingestion: No adverse health effects are expected if swallowed. Refer to Toxicology information
Skin: Practically non irritating based on toxicity of TKPP
Eyes: Will cause redness, Tearing and irritation.

Health Hazards (Acute and Chronic):
Carcinogenicity:
NTP? No Data
IARC Monographs? No Data
OSHA Regulated? No Data

Emergency and First Aid Procedures:

Skin: Wash off with water. If irritation occurs and persists, obtain medical attention.
Eyes: Flush eyes with large amounts of water for 15 minutes. Seek medical attention.
Ingestion: Rinse mouth and dilute stomach contents with water, or preferable with milk if available. Call poison control or a physician.
Inhalation: Remove from exposure. If discomfort occurs and persists, obtain medical attention.

SECTION VII - Precautions for Safe Handling and Use

Steps to Be Taken in Case Material is Released or Spilled:
Material should be salvaged or disposed of.

Waste Disposal Methods: **If material cannot be salvaged, a method of disposal is in a Landfill in accordance with all Local, State, and Federal Regulations.**

Precautions to Be Taken in Handling and Storing:
No special requirements.

SECTION VIII-Control Measures

Respiratory Protection (Specify Type):
Ventilation: No special requirements
- **Local Exhaust: No special requirements**
- **Special: None**
- **Mechanical (General): Provide natural or mechanical ventilation**

Protective Gloves: Recommended
Eye Protection: Chemical goggles
Other Protective Clothing or Equipment: Remove and wash contaminated clothing
Work/Hygienic Practices: Follow good industrial practices

Univar USA Inc.
17425 NE Union Hill Road
Redmond, WA 98052
(425) 889-3400

For Emergency Assistance involving chemicals call - CHEMTREC (800) 424-9300

==========

The Version Date and Number for this MSDS is : 09/29/2005 - #002

PRODUCT NAME: KLENPHOS 300

MSDS NUMBER: 40762

DATE ISSUED: 01/05/05

SUPERSEDES: 03/01/1998

ISSUED BY: 002590

****
****

MATERIAL SAFETY DATA SHEET

PRODUCT: KLENPHOS 300
APPLICATION: Drinking Water Additive

Information On Physical Hazards, Health Hazards, Pel's and TLV's For Specific Product Ingredients As Required By The OSHA Hazard Communications Standard Are Listed. Refer In Section VI For Our Assessment Of The Health Hazards Of This Formulation.

SECTION I
Manufacturer's Name: Klenzoid, Inc.
Address: 912 Spring Mill Avenue Conshohocken, PA 19428
Emergency Telephone Number: (610) 825-9494
Telephone Number for Information: (610) 825-9494

SECTION II - Hazardous Ingredients/Identify Information

Zinc Pyrophosphate (30%-50%) CAS# 7446-26-6
Water (50-80%) CAS # 7732-18-5

SECTION III - Physical/Chemical Characteristics

pH: 9.4-9.8
Boiling Point: 220 Degrees F
Specific Gravity (H2 0=1): 1.34

Vapor Pressure (nun Hg.): Non Volatile
Vapor Density (Air = 1): Non Volatile
Freezing Point: < 20 Degrees F
Evaporation Rate (Butyl Acetate = 1): None volatile
Solubility in Water: 100%
Appearance and Odor: Slightly Viscous, clear liquid, no odor

SECTION IV - Fire and Explosion Hazard Data

Flash Point (Method Used): Not Combustible
Special Fire Fighting Procedures: N/A
Unusual Fire and Explosion Hazards: N/A
This material is not flammable.

SECTION V - Reactivity Data

Stability:
Stable: Yes

Incompatibility (Materials to Avoid): Corrosive to Aluminum based on DOT 49 CFR

Hazardous Decomposition or Byproducts: None

Hazardous Polymerization: Will not occur

SECTION VI - Health Hazard Data

Inhalation: No Published Data

Ingestion: No adverse health affects are expected if swallowed. Refer to Toxicology information

Skin: Practically non irritating base on toxicity of TKPP

Eyes: Will cause redness, Tearing and irritation

Health Hazards (Acute and Chronic):
Carcinogenicity:
NTP? No Data
IARC Monographs? No Data
OSHA Regulated? No Data

Emergency and First Aid Procedures:
Skin: Wash off with water. If irritation occurs and persists, obtain medical attention.

Eyes: Flush eyes with large amounts of water for 15 minutes. Seek medical attention.

Ingestion: Rinse mouth and dilute stomach contents with water, or preferable with milk if available. Call poison control or a physician.

Inhalation: Remove from exposure. If discomfort occurs and persists, obtain medical attention.

SECTION VII - Precautions for Safe Handling and Use

Steps to Be Taken in Case Material is Released or Spilled:
Material should be salvaged or disposed of.

Waste Disposal Methods: If material can not be salvaged, a method of disposal is in a landfill in accordance with all Local, State, and Federal regulations.

Precautions to Be Taken in Handling and Storing: No special requirements.

SECTION VIII-Control Measures

Respiratory Protection (Specify Type):
Ventilation: No special requirements
  Local Exhaust: No special requirements
  Special: None
  Mechanical (General): Provide natural or mechanical ventilation

Protective Gloves: Recommended
Eye Protection: Chemical goggles
Other Protective Clothing or Equipment: Remove and wash contaminated clothing
Work/Hygienic Practices: Follow good industrial practices

For Additional Information:
Contact: MSDS Coordinator - Univar USA
During business hours, Pacific Time - (425) 889-3400

NOTICE
Univar USA expressly disclaims all express or implied warranties of merchantibility and fitness for a particular purpose with respect to the product or information provided herein, and shall under no circumstances be liable for incidental or consequential damages.

Do not use ingredient information and/or ingredient percentages in this MSDS as a product specification. For product specification information refer to a Product Specification Sheet and/or a Certificate of Analysis. These can be obtained from your local Univar USA Sales Office.

All information appearing herein is based upon data obtained from the manufacturer and/or recognized technical sources. While the information is believed to be accurate, Univar USA makes no representations as to its accuracy or sufficiency. Conditions of use are beyond Univar USA's control. Therefore, users are responsible to verify this data under their own operating conditions to determine whether the product is suitable for their particular purposes, and they assume all risks of their use, handling, and disposal of the product or from the publication or use of, or reliance upon, information contained herein. This information relates only to the product designated herein and does not relate to its use in combination with any other material or in any other process.

END OF MSDS

Material Safety Data Sheet
Tris 1 Molar Buffer

MSDS# 45485

Section 1 - Chemical Product and Company Identification

MSDS Name:	Tris 1 Molar Buffer
Catalog Numbers:	BP1756-100, BP1756-500, BP1757-100, BP1757-500, BP1758-100, BP1758-500
Synonyms:	Tris EDTA Buffer; Tris Buffer.
Company Identification:	Fisher Scientific One Reagent Lane Fair Lawn, NJ 07410
For information in the US, call:	201-796-7100
Emergency Number US:	201-796-7100
CHEMTREC Phone Number, US:	800-424-9300

Section 2 - Composition, Information on Ingredients

Risk Phrases:

CAS#:	77-86-1
Chemical Name:	Tris (hydroxymethyl) aminomethane
%:	10-14
EINECS#:	201-064-4
Hazard Symbols:	

Risk Phrases:

CAS#:	1185-53-1
Chemical Name:	Tris Hydrochloride
%:	3-4.5
EINECS#:	214-684-5
Hazard Symbols:	

Risk Phrases:

CAS#:	7732-18-5
Chemical Name:	Water
%:	>50.0
EINECS#:	231-791-2
Hazard Symbols:	

Text for R-phrases: see Section 16

Hazard Symbols:	None listed
Risk Phrases:	None listed

Section 3 - Hazards Identification

EMERGENCY OVERVIEW

Caution! The toxicological properties of this material have not been fully investigated. May cause eye and skin irritation. May cause respiratory and digestive tract irritation. Target Organs: None known.

Potential Health Effects

Eye: May cause eye irritation.

Skin: May cause skin irritation.

Ingestion: May cause irritation of the digestive tract. The toxicological properties of this substance have not been fully investigated.

Inhalation: May cause respiratory tract irritation. The toxicological properties of this substance have not been fully investigated.

Chronic: Prolonged or repeated skin contact may cause defatting and dermatitis.

Section 4 - First Aid Measures

Eyes: Flush eyes with plenty of water for at least 15 minutes, occasionally lifting the upper and lower eyelids. Get medical aid.

Skin: Flush skin with plenty of water for at least 15 minutes while removing contaminated clothing and shoes. Get medical aid if irritation develops or persists. Wash clothing before reuse.

Ingestion: Never give anything by mouth to an unconscious person. Get medical aid. Do NOT induce vomiting. If conscious and alert, rinse mouth and drink 2-4 cupfuls of milk or water.

Inhalation: Remove from exposure and move to fresh air immediately. If not breathing, give artificial respiration. If breathing is difficult, give oxygen. Get medical aid if cough or other symptoms appear.

Notes to Physician:

Section 5 - Fire Fighting Measures

General Information: As in any fire, wear a self-contained breathing apparatus in pressure-demand, MSHA/NIOSH (approved or equivalent), and full protective gear. During a fire, irritating and highly toxic gases may be generated by thermal decomposition or combustion.

Extinguishing Media: Use water spray, dry chemical, carbon dioxide, or chemical foam.

Autoignition Temperature: Not applicable.

Flash Point: Not applicable.

Explosion Limits: Lower: Not available

Explosion Limits: Upper: Not available

NFPA Rating: health: 1; flammability: 0; instability: 0;

Section 6 - Accidental Release Measures

General Information: Use proper personal protective equipment as indicated in Section 8.

Spills/Leaks: Absorb spill with inert material (e.g. vermiculite, sand or earth), then place in suitable container. Avoid runoff into storm sewers and ditches which lead to waterways. Clean up spills immediately, observing precautions in the Protective Equipment section. Provide ventilation.

Section 7 - Handling and Storage

Handling: Wash thoroughly after handling. Remove contaminated clothing and wash before reuse. Use with adequate ventilation. Minimize dust generation and accumulation. Avoid contact with skin and eyes. Keep container tightly closed. Avoid ingestion and inhalation. Discard contaminated shoes.

Storage: Store in a tightly closed container. Store in a cool, dry, well-ventilated area away from incompatible substances.

Section 8 - Exposure Controls, Personal Protection

Chemical Name	ACGIH	NIOSH	OSHA - Final PELs
Tris (hydroxymethyl) aminomethane	none listed	none listed	none listed
Tris Hydrochloride	none listed	none listed	none listed
Water	none listed	none listed	none listed

OSHA Vacated PELs: Tris (hydroxymethyl) aminomethane: None listed Tris Hydrochloride: None listed Water: None listed

Engineering Controls:

Use process enclosure, local exhaust ventilation, or other engineering controls to control airborne levels below recommended exposure limits. Facilities storing or utilizing this material should be equipped with an eyewash facility and a safety shower.

Exposure Limits

Personal Protective Equipment

Eyes:	Wear appropriate protective eyeglasses or chemical safety goggles as described by OSHA's eye and face protection regulations in 29 CFR 1910.133 or European Standard EN166.
Skin:	Wear appropriate protective gloves to prevent skin exposure.
Clothing:	Wear appropriate protective clothing to prevent skin exposure.
Respirators:	A respiratory protection program that meets OSHA's 29 CFR 1910.134 and ANSI Z88.2 requirements or European Standard EN 149 must be followed whenever workplace conditions warrant respirator use.

Section 9 - Physical and Chemical Properties

Physical State:	Liquid
Color:	clear, colorless
Odor:	odorless
pH:	10
Vapor Pressure:	Not available
Vapor Density:	Not available
Evaporation Rate:	Not available
Viscosity:	Not available
Boiling Point:	Not available
Freezing/Melting Point:	Not available
Decomposition Temperature:	Not available
Solubility in water:	Soluble in water.
Specific Gravity/Density:	Not available.
Molecular Formula:	Mixture
Molecular Weight:	0

Section 10 - Stability and Reactivity

Chemical Stability:	Stable at room temperature in closed containers under normal storage and handling conditions. Decarboxylates above 150C.
Conditions to Avoid:	Incompatible materials.
Incompatibilities with Other Materials	Not available
Hazardous Decomposition Products	Carbon monoxide, carbon monoxide, carbon dioxide, nitrogen oxides (NOx) and ammonia (NH3).
Hazardous Polymerization	Will not occur.

Section 11 - Toxicological Information

RTECS#:	CAS# 77-86-1: TY2900000 CAS# 1185-53-1: None listed CAS# 7732-18-5: ZC0110000
LD50/LC50:	RTECS: **CAS# 77-86-1:** Oral, rat: LD50 = 5900 mg/kg; . RTECS: **CAS# 1185-53-1:**. RTECS: **CAS# 7732-18-5:** Oral, rat: LD50 = >90 mL/kg; .
	Tris (hydroxymethyl) aminomethane - Not listed as a carcinogen by ACGIH, IARC, NTP, or CA Prop

Carcinogenicity: 65.
Tris Hydrochloride - Not listed as a carcinogen by ACGIH, IARC, NTP, or CA Prop 65.
Water - Not listed as a carcinogen by ACGIH, IARC, NTP, or CA Prop 65.

Other: See actual entry in RTECS for complete information.

Section 12 - Ecological Information

Not available

Section 13 - Disposal Considerations

Dispose of in a manner consistent with federal, state, and local regulations.

Section 14 - Transport Information

US DOT
Shipping Name: Not regulated as a hazardous material
Hazard Class:
UN Number:
Packing Group:
Canada TDG
Shipping Name: Not available
Hazard Class:
UN Number:
Packing Group:

Section 15 - Regulatory Information

European/International Regulations

European Labeling in Accordance with EC Directives

Hazard Symbols:Not available

Risk Phrases:

Safety Phrases:

S 24/25 Avoid contact with skin and eyes.

WGK (Water Danger/Protection)

CAS# 77-86-1: 1

CAS# 1185-53-1: Not available

CAS# 7732-18-5: Not available

Canada

CAS# 77-86-1 is listed on Canada's DSL List

CAS# 1185-53-1 is listed on Canada's DSL List

CAS# 7732-18-5 is listed on Canada's DSL List

Canadian WHMIS Classifications: Not available

This product has been classified in accordance with the hazard criteria of the Controlled Products Regulations and the MSDS contains all of the information required by those regulations.

CAS# 77-86-1 is not listed on Canada's Ingredient Disclosure List.

CAS# 1185-53-1 is not listed on Canada's Ingredient Disclosure List.

CAS# 7732-18-5 is not listed on Canada's Ingredient Disclosure List.

US Federal

TSCA

CAS# 77-86-1 is listed on the TSCA Inventory.

CAS# 1185-53-1 is listed on the TSCA Inventory.

CAS# 7732-18-5 is listed on the TSCA Inventory.

Section 16 - Other Information

MSDS Creation Date: 6/25/1999

Revision #7 Date 7/20/2009

The information above is believed to be accurate and represents the best information currently available to us. However, we make no warranty of merchantibility or any other warranty, express or implied, with respect to such information, and we assume no liability resulting from its use. Users should make their own investigations to determine the suitability of the information for their particular purposes. In no event shall the company be liable for any claims, losses, or damages of any third party or for lost profits or any special, indirect, incidental, consequential, or exemplary damages howsoever arising, even if the company has been advised of the possibility of such damages.

MSDS Number: **N3660** * * * * * *Effective Date:* ***07/02/02*** * * * * * *Supercedes:* ***07/13/00***

MSDS Material Safety Data Sheet

From: Mallinckrodt Baker, Inc.
222 Red School Lane
Phillipsburg, NJ 08865

Mallinckrodt CHEMICALS J.T.Baker

24 Hour Emergency Telephone: 908-859-2151
CHEMTREC: 1-800-424-9300

National Response in Canada
CANUTEC: 613-996-6666

Outside U.S. and Canada
Chemtrec: 703-527-3887

NOTE: CHEMTREC, CANUTEC and National Response Center emergency numbers to be used only in the event of chemical emergencies involving a spill, leak, fire, exposure or accident involving chemicals.

All non-emergency questions should be directed to Customer Service (1-800-582-2537) for assistance.

NITRIC ACID, 50-70%

1. Product Identification

Synonyms: Aqua Fortis; Azotic Acid; Nitric Acid 50%; Nitric Acid 65%; nitric acid 69-70%
CAS No.: 7697-37-2
Molecular Weight: 63.01
Chemical Formula: HNO3
Product Codes:
J.T. Baker: 411D, 412D, 5371, 5796, 5801, 5826, 5856, 5876, 5896, 9597, 9598, 9600, 9601, 9602, 9603, 9604, 9606, 9607, 9608, 9610, 9616, 9617, 9670
Mallinckrodt: 1409, 2704, 2716, 6623, H862, H993, H998, V077, V633, V650

2. Composition/Information on Ingredients

Ingredient	CAS No	Percent	Hazardous
Nitric Acid	7697-37-2	50 - 70%	Yes
Water	7732-18-5	30 - 50%	No

3. Hazards Identification

Emergency Overview

POISON! DANGER! STRONG OXIDIZER. CONTACT WITH OTHER MATERIAL MAY CAUSE FIRE. CORROSIVE. LIQUID AND MIST CAUSE

SEVERE BURNS TO ALL BODY TISSUE. MAY BE FATAL IF SWALLOWED OR INHALED. INHALATION MAY CAUSE LUNG AND TOOTH DAMAGE.

SAF-T-DATA(tm) Ratings (Provided here for your convenience)

Health Rating: 4 - Extreme (Poison)
Flammability Rating: 0 - None
Reactivity Rating: 3 - Severe (Oxidizer)
Contact Rating: 4 - Extreme (Corrosive)
Lab Protective Equip: GOGGLES & SHIELD; LAB COAT & APRON; VENT HOOD; PROPER GLOVES
Storage Color Code: White (Corrosive)

Potential Health Effects

Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison.

Inhalation:
Corrosive! 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.
Ingestion:
Corrosive! Swallowing nitric acid can cause immediate pain and burns of the mouth, throat, esophagus and gastrointestinal tract.
Skin Contact:
Corrosive! Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color.
Eye Contact:
Corrosive! Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Chronic Exposure:
Long-term exposure to concentrated vapors may cause erosion of teeth and lung damage. Long-term exposures seldom occur due to the corrosive properties of the acid.
Aggravation of Pre-existing Conditions:
Persons with pre-existing skin disorders, eye disease, or cardiopulmonary diseases may be more susceptible to the effects of this substance.

---

4. First Aid Measures

Immediate first aid treatment reduces the health effects of this substance.
Inhalation:
Remove to fresh air. If not breathing, give artificial respiration. If breathing is difficult, give oxygen. Call a physician.
Ingestion:
DO NOT INDUCE VOMITING! Give large quantities of water or milk if available. Never give anything by mouth to an unconscious person. Get medical attention immediately.
Skin Contact:
In case of contact, immediately flush skin with plenty of water for at least 15 minutes while removing contaminated clothing and shoes. Wash clothing before reuse. Thoroughly clean shoes before reuse. Get medical attention immediately.
Eye Contact:

Immediately flush eyes with plenty of water for at least 15 minutes, lifting lower and upper eyelids occasionally. Get medical attention immediately.

---

5. Fire Fighting Measures

Fire:
Not combustible, but substance is a strong oxidizer and its heat of reaction with reducing agents or combustibles may cause ignition. Can react with metals to release flammable hydrogen gas.
Explosion:
Reacts explosively with combustible organic or readily oxidizable materials such as: alcohols, turpentine, charcoal, organic refuse, metal powder, hydrogen sulfide, etc. Reacts with most metals to release hydrogen gas which can form explosive mixtures with air.
Fire Extinguishing Media:
Water spray may be used to keep fire exposed containers cool. Do not get water inside container.
Special Information:
Increases the flammability of combustible, organic and readily oxidizable materials. In the event of a fire, wear full protective clothing and NIOSH-approved self-contained breathing apparatus with full facepiece operated in the pressure demand or other positive pressure mode.

---

6. Accidental Release Measures

Ventilate area of leak or spill. Wear appropriate personal protective equipment as specified in Section 8. Isolate hazard area. Keep unnecessary and unprotected personnel from entering. Contain and recover liquid when possible. Neutralize with alkaline material (soda ash, lime), then absorb with an inert material (e. g., vermiculite, dry sand, earth), and place in a chemical waste container. Do not use combustible materials, such as saw dust. Do not flush to sewer! US Regulations (CERCLA) require reporting spills and releases to soil, water and air in excess of reportable quantities. The toll free number for the US Coast Guard National Response Center is (800) 424-8802.

J. T. Baker NEUTRASORB® or TEAM® 'Low Na+' acid neutralizers are recommended for spills of this product.

---

7. Handling and Storage

Store in a cool, dry, ventilated storage area with acid resistant floors and good drainage. Protect from physical damage. Keep out of direct sunlight and away from heat, water, and incompatible materials. Do not wash out container and use it for other purposes. When diluting, the acid should always be added slowly to water and in small amounts. Never use hot water and never add water to the acid. Water added to acid can cause uncontrolled boiling and splashing. Containers of this material may be hazardous when empty since they retain product residues (vapors, liquid); observe all warnings and precautions listed for the product.

8. Exposure Controls/Personal Protection

Airborne Exposure Limits:
-OSHA Permissible Exposure Limit (PEL):
2 ppm (TWA), 4 ppm (STEL)
-ACGIH Threshold Limit Value (TLV):
2 ppm (TWA); 4 ppm (STEL)

Ventilation System:
A system of local and/or general exhaust is recommended to keep employee exposures below the Airborne Exposure Limits. Local exhaust ventilation is generally preferred because it can control the emissions of the contaminant at its source, preventing dispersion of it into the general work area. Please refer to the ACGIH document, *Industrial Ventilation, A Manual of Recommended Practices*, most recent edition, for details.
Personal Respirators (NIOSH Approved):
If the exposure limit is exceeded, wear a supplied air, full-facepiece respirator, airlined hood, or full-facepiece self-contained breathing apparatus. Nitric acid is an oxidizer and should not come in contact with cartridges and canisters that contain oxidizable materials, such as activated charcoal. Canister-type respirators using sorbents are ineffective.
Skin Protection:
Wear impervious protective clothing, including boots, gloves, lab coat, apron or coveralls, as appropriate, to prevent skin contact.
Eye Protection:
Use chemical safety goggles and/or a full face shield where splashing is possible. Maintain eye wash fountain and quick-drench facilities in work area.

9. Physical and Chemical Properties

Appearance:
Colorless to yellowish liquid.
Odor:
Suffocating, acrid.
Solubility:
Infinitely soluble.
Specific Gravity:
1.41
pH:
1.0 (0.1M solution)
% Volatiles by volume @ 21C (70F):
100 (as water and acid)
Boiling Point:
122C (252F)
Melting Point:
-42C (-44F)
Vapor Density (Air=1):
2-3
Vapor Pressure (mm Hg):
48 @ 20C (68F)
Evaporation Rate (BuAc=1):
No information found.

10. Stability and Reactivity

Stability:
Stable under ordinary conditions of use and storage. Containers may burst when heated.
Hazardous Decomposition Products:
When heated to decomposition, emits toxic nitrogen oxides fumes and hydrogen nitrate. Will react with water or steam to produce heat and toxic and corrosive fumes.
Hazardous Polymerization:
Will not occur.
Incompatibilities:
A dangerously powerful oxidizing agent, concentrated nitric acid is incompatible with most substances, especially strong bases, metallic powders, carbides, hydrogen sulfide, turpentine, and combustible organics.
Conditions to Avoid:
Light and heat.

11. Toxicological Information

Nitric acid: Inhalation rat LC50: 244 ppm (NO2)/30M; Investigated as a mutagen, reproductive effector. Oral (human) LDLo: 430 mg/kg.

--------\Cancer Lists\----------

	---NTP Carcinogen---		
Ingredient	Known	Anticipated	IARC Category
Nitric Acid (7697-37-2)	No	No	None
Water (7732-18-5)	No	No	None

12. Ecological Information

Environmental Fate:
No information found.
Environmental Toxicity:
No information found.

13. Disposal Considerations

Whatever cannot be saved for recovery or recycling should be managed in an appropriate and approved waste facility. Although not a listed RCRA hazardous waste, this material may exhibit one or more characteristics of a hazardous waste and require appropriate analysis to determine specific disposal requirements. Processing, use or contamination of this product may change the waste management options. State and local disposal regulations may differ from federal disposal regulations. Dispose of container and unused contents in accordance with federal, state and local requirements.

14. Transport Information

Domestic (Land, D.O.T.)

Proper Shipping Name: NITRIC ACID (WITH NOT MORE THAN 70% NITRIC ACID)

Hazard Class: 8
UN/NA: UN2031
Packing Group: II
Information reported for product/size: 150LB

International (Water, I.M.O.)

Proper Shipping Name: NITRIC ACID (WITH NOT MORE THAN 70% NITRIC ACID)

Hazard Class: 8
UN/NA: UN2031
Packing Group: II
Information reported for product/size: 150LB

International (Air, I.C.A.O.)

Proper Shipping Name: NITRIC ACID (WITH NOT MORE THAN 70% NITRIC ACID)

Hazard Class: 8
UN/NA: UN2031
Packing Group: II
Information reported for product/size: 150LB

---

15. Regulatory Information

--------\Chemical Inventory Status - Part 1\---------------------------------

Ingredient	TSCA	EC	Japan	Australia
Nitric Acid (7697-37-2)	Yes	Yes	Yes	Yes
Water (7732-18-5)	Yes	Yes	Yes	Yes

--------\Chemical Inventory Status - Part 2\---------------------------------

Ingredient	Korea	--Canada-- DSL	NDSL	Phil.
Nitric Acid (7697-37-2)	Yes	Yes	No	Yes
Water (7732-18-5)	Yes	Yes	No	Yes

--------\Federal, State & International Regulations - Part 1\----------------

Ingredient	-SARA 302- RQ	TPQ	------SARA 313------ List	Chemical Catg.
Nitric Acid (7697-37-2)	1000	1000	Yes	No
Water (7732-18-5)	No	No	No	No

--------\Federal, State & International Regulations - Part 2\----------------

Ingredient	CERCLA	-RCRA- 261.33	-TSCA- 8(d)
Nitric Acid (7697-37-2)	1000	No	No
Water (7732-18-5)	No	No	No

```
Chemical Weapons Convention:  No     TSCA 12(b):  No     CDTA:  No
SARA 311/312:  Acute: Yes      Chronic: Yes  Fire: Yes Pressure: No
Reactivity: No          (Mixture / Liquid)
```

Australian Hazchem Code: 2PE
Poison Schedule: S6
WHMIS:
This MSDS has been prepared according to the hazard criteria of the Controlled Products Regulations (CPR) and the MSDS contains all of the information required by the CPR.

---

16. Other Information

NFPA Ratings: Health: **3** Flammability: **0** Reactivity: **0** Other: **Oxidizer**
Label Hazard Warning:
POISON! DANGER! STRONG OXIDIZER. CONTACT WITH OTHER MATERIAL MAY CAUSE FIRE. CORROSIVE. LIQUID AND MIST CAUSE SEVERE BURNS TO ALL BODY TISSUE. MAY BE FATAL IF SWALLOWED OR INHALED. INHALATION MAY CAUSE LUNG AND TOOTH DAMAGE.
Label Precautions:
Do not get in eyes, on skin, or on clothing.
Do not breathe vapor or mist.
Use only with adequate ventilation.
Wash thoroughly after handling.
Keep from contact with clothing and other combustible materials.
Do not store near combustible materials.
Store in a tightly closed container.
Remove and wash contaminated clothing promptly.
Label First Aid:
In case of contact, immediately flush eyes or skin with plenty of water for at least 15 minutes while removing contaminated clothing and shoes. Wash clothing before reuse. If swallowed, DO NOT INDUCE VOMITING. Give large quantities of water. Never give anything by mouth to an unconscious person. If inhaled, remove to fresh air. If not breathing, give artificial respiration. If breathing is difficult, give oxygen. In all cases get medical attention immediately.
Product Use:
Laboratory Reagent.
Revision Information:
MSDS Section(s) changed since last revision of document include: 3.
Disclaimer:
****

Mallinckrodt Baker, Inc. provides the information contained herein in good faith but makes no representation as to its comprehensiveness or accuracy. This document is intended only as a guide to the appropriate precautionary handling of the material by a properly trained person using this product. Individuals receiving the information must exercise their independent judgment in determining its appropriateness for a particular purpose. MALLINCKRODT BAKER, INC. MAKES NO REPRESENTATIONS OR WARRANTIES, EITHER EXPRESS OR IMPLIED, INCLUDING WITHOUT LIMITATION ANY WARRANTIES OF MERCHANTABILITY, FITNESS FOR A PARTICULAR PURPOSE WITH

RESPECT TO THE INFORMATION SET FORTH HEREIN OR THE PRODUCT TO WHICH THE INFORMATION REFERS. ACCORDINGLY, MALLINCKRODT BAKER, INC. WILL NOT BE RESPONSIBLE FOR DAMAGES RESULTING FROM USE OF OR RELIANCE UPON THIS INFORMATION.

****

Prepared by: Environmental Health & Safety
Phone Number: (314) 654-1600 (U.S.A.)

Material Safety Data Sheet

Sulfuric acid solution 69-75%

ACC# 91670

Section 1 - Chemical Product and Company Identification

MSDS Name: Sulfuric acid solution 69-75%
Catalog Numbers: NC9232054, NC9252283, NC9316478, SA170-4, XX75%SA4LI04, XX75% SASK212LI, XXSULFAC70%4
Synonyms: Hydrogen sulfate; Oil of vitriol; Vitriol brown oil; Mattling acid; Battery acid; Sulphuric acid; Electrolyte acid; Dihydrogen sulfate; Spirit of sulfur; Chamber acid.
Company Identification:

Fisher Scientific
1 Reagent Lane
Fair Lawn, NJ 07410

For information, call: 201-796-7100
Emergency Number: 201-796-7100
For CHEMTREC assistance, call: 800-424-9300
For International CHEMTREC assistance, call: 703-527-3887

Section 2 - Composition, Information on Ingredients

CAS#	Chemical Name	Percent	EINECS/ELINCS
7664-93-9	Sulfuric acid	69-75	231-639-5
7732-18-5	Water	25-31	231-791-2

Section 3 - Hazards Identification

EMERGENCY OVERVIEW

Appearance: oily liquid.
Danger! Causes eye and skin burns. Causes digestive and respiratory tract burns. May be fatal if mist inhaled. Concentrated sulfuric acid reacts violently with water and many other substances under certain conditions. Contact with metals may evolve flammable hydrogen gas. May cause lung damage. Hygroscopic (absorbs moisture from the air).
Target Organs: Lungs, teeth, eyes, skin, mucous membranes.

Potential Health Effects

Eye: Causes severe eye burns. May cause irreversible eye injury. May cause permanent corneal opacification. The severity of injury depends on the concentration of the solution and the duration of exposure.
Skin: Causes skin burns. The severity of injury depends on the concentration of the solution and the duration of exposure.
Ingestion: May cause severe and permanent damage to the digestive tract. Causes gastrointestinal tract burns.
Inhalation: May cause irritation of the respiratory tract with burning pain in the nose and throat, coughing, wheezing, shortness of breath and pulmonary edema. Causes chemical burns to the

respiratory tract. Inhalation may be fatal as a result of spasm, inflammation, edema of the larynx and bronchi, chemical pneumonitis and pulmonary edema. Causes corrosive action on the mucous membranes. Because its vapor pressure is negligible, it exists in the air only as a mist or spray.
Chronic: Prolonged or repeated skin contact may cause dermatitis. Prolonged or repeated inhalation may cause nosebleeds, nasal congestion, erosion of the teeth, perforation of the nasal septum, chest pain and bronchitis. Prolonged or repeated eye contact may cause conjunctivitis. Effects may be delayed. Workers chronically exposed to sulfuric acid mists may show various lesions of the skin, tracheobronchitis, stomatitis, conjunctivitis, or gastritis. Occupational exposure to strong inorganic acid mists containing sulfuric acid is carcinogenic to humans.

Section 4 - First Aid Measures

Eyes: In case of contact, immediately flush eyes with plenty of water for a t least 15 minutes. Get medical aid immediately.
Skin: In case of contact, immediately flush skin with plenty of water for at least 15 minutes while removing contaminated clothing and shoes. Get medical aid immediately. Wash clothing before reuse.
Ingestion: If swallowed, do NOT induce vomiting. Get medical aid immediately. If victim is fully conscious, give a cupful of water. Never give anything by mouth to an unconscious person.
Inhalation: POISON material. If inhaled, get medical aid immediately. Remove victim to fresh air. If not breathing, give artificial respiration. If breathing is difficult, give oxygen.
Notes to Physician: Monitor arterial blood gases, chest x-ray, and pulmonary function tests if respiratory tract irritation or respiratory depression is evident. Treat dermal irritation or burns with standard topical therapy. Effects may be delayed. Do NOT use sodium bicarbonate in an attempt to neutralize the acid.
Antidote: Do NOT use oils or ointments in eye.

Section 5 - Fire Fighting Measures

General Information: As in any fire, wear a self-contained breathing apparatus in pressure-demand, MSHA/NIOSH (approved or equivalent), and full protective gear. Use water spray to keep fire-exposed containers cool. Substance is noncombustible. Contact with water can cause violent liberation of heat and splattering of the material. Contact with metals may evolve flammable hydrogen gas. Runoff from fire control or dilution water may cause pollution. Strong dehydrating agent, which may cause ignition of finely divided materials on contact. Oxides of sulfur may be produced in fire.
Extinguishing Media: Use extinguishing media most appropriate for the surrounding fire. Do NOT get water inside containers. If water is used, care should be taken, since it can generate heat and cause spattering if applied directly to sulfuric acid.
Flash Point: Not applicable.
Autoignition Temperature: Not available.
Explosion Limits, Lower: Not available.
Upper: Not available.
NFPA Rating: (estimated) Health: 3; Flammability: 0; Instability: 2; Special Hazard: -W-

Section 6 - Accidental Release Measures

General Information: Use proper personal protective equipment as indicated in Section 8.

Spills/Leaks: Avoid runoff into storm sewers and ditches which lead to waterways. Clean up spills immediately, observing precautions in the Protective Equipment section. Carefully scoop up and place into appropriate disposal container. Provide ventilation. Do not expose spill to water. Cover with dry earth, dry sand, or other non-combustible material followed with plastic sheet to minimize spreading and contact with water.

Section 7 - Handling and Storage

Handling: Wash thoroughly after handling. Remove contaminated clothing and wash before reuse. Do not get in eyes, on skin, or on clothing. Keep container tightly closed. Do not allow contact with water. Discard contaminated shoes. Use only with adequate ventilation. Do not breathe spray or mist.
Storage: Do not store near combustible materials. Keep container closed when not in use. Store in a cool, dry, well-ventilated area away from incompatible substances. Keep away from water. Corrosives area. Do not store near alkaline substances. Store protected from moisture.

Section 8 - Exposure Controls, Personal Protection

Engineering Controls: Facilities storing or utilizing this material should be equipped with an eyewash facility and a safety shower. Use adequate general or local exhaust ventilation to keep airborne concentrations below the permissible exposure limits.
Exposure Limits

Chemical Name	ACGIH	NIOSH	OSHA - Final PELs
Sulfuric acid	0.2 mg/m3 TWA (thoracic fraction)	1 mg/m3 TWA 15 mg/m3 IDLH	1 mg/m3 TWA
Water	none listed	none listed	none listed

OSHA Vacated PELs: Sulfuric acid: 1 mg/m3 TWA Water: No OSHA Vacated PELs are listed for this chemical.
Personal Protective Equipment
Eyes: Wear chemical splash goggles and face shield.
Skin: Wear appropriate protective gloves to prevent skin exposure.
Clothing: Wear appropriate protective clothing to prevent skin exposure.
Respirators: Follow the OSHA respirator regulations found in 29 CFR 1910.134 or European Standard EN 149. Use a NIOSH/MSHA or European Standard EN 149 approved respirator if exposure limits are exceeded or if irritation or other symptoms are experienced.

Section 9 - Physical and Chemical Properties

Physical State: Liquid
Appearance: clear colorless - oily
Odor: odorless
pH: 0.3 (1N solution)
Vapor Pressure: < 0.001 mm Hg @ 20 deg C
Vapor Density: 3.38 (air=1)
Evaporation Rate: Slower than ether.
Viscosity: 21 mPas @ 25 C

Boiling Point: 290 - 338 deg C
Freezing/Melting Point:10 deg C
Decomposition Temperature:340 deg C
Solubility: Soluble with much heat
Specific Gravity/Density:1.6015
Molecular Formula:H2SO4
Molecular Weight:98.07

Section 10 - Stability and Reactivity

Chemical Stability: Sulfuric acid reacts vigorously, violently or explosively with many organic and inorganic chemicals and with water.
Conditions to Avoid: Excess heat, exposure to moist air or water, Note: Use great caution in mixing with water due to heat evolution that causes explosive spattering. Always add the acid to water, never the reverse..
Incompatibilities with Other Materials: Metals, reducing agents, bases, acrylonitrile, chlorates, finely powdered metals, nitrates, perchlorates, permanganates, epichlorohydrin, aniline, carbides, fulminates, picrates, organic materials.
Hazardous Decomposition Products: Oxides of sulfur.
Hazardous Polymerization: Has not been reported.

Section 11 - Toxicological Information

RTECS#:
CAS# 7664-93-9: WS5600000
CAS# 7732-18-5: ZC0110000
LD50/LC50:
CAS# 7664-93-9:
Draize test, rabbit, eye: 250 ug Severe;
Inhalation, mouse: LC50 = 320 mg/m3/2H;
Inhalation, mouse: LC50 = 320 mg/m3;
Inhalation, rat: LC50 = 510 mg/m3/2H;
Inhalation, rat: LC50 = 510 mg/m3;
Oral, rat: LD50 = 2140 mg/kg;
.

CAS# 7732-18-5:
Oral, rat: LD50 = >90 mL/kg;
.

Carcinogenicity:
CAS# 7664-93-9:

- **ACGIH:** A2 - Suspected Human Carcinogen (contained in strong inorganic acid mists)
- **California:** carcinogen, initial date 3/14/03 (listed as Strong inorganic acid mists containing sulfuric acid).
- **NTP:** Known carcinogen (listed as Strong inorganic acid mists containing s).
- **IARC:** Group 1 carcinogen

CAS# 7732-18-5: Not listed by ACGIH, IARC, NTP, or CA Prop 65.

Epidemiology: Workers exposed to industrial sulfuric acid mist showed a statistical increase in laryngeal cancer. This suggests a possible relationship between carcinogenesis and inhalation of sulfuric acid mist.
Teratogenicity: Sulfuric acid was not teratogenic in mice and rabbits, but was slightly embryotoxic in rabbits (a minor, rare skeletal variation). The animals were exposed to 5 and 20 mg/m3 for 7 hr/day throughout pregnancy. Slight maternal toxicity was present at the highest dose in both species.
Reproductive Effects: No information found
Mutagenicity: There are no mutagenicity studies specifically of sulfuric acid. However, there are established effects of reduced pH in mutagenicity testing, as would be caused by sulfuric acid. These effects are an artifact of low pH and are not necessarily due to biological effects of sulfuric acid itself.
Neurotoxicity: No information found
Other Studies:

Section 12 - Ecological Information

Ecotoxicity: Fish: Bluegill/Sunfish: 49 mg/L; 48Hr; TLm (tap water @ 20C)
Fish: Bluegill/Sunfish: 24.5 ppm; 48Hr; TLm (fresh water)

Section 13 - Disposal Considerations

Chemical waste generators must determine whether a discarded chemical is classified as a hazardous waste. US EPA guidelines for the classification determination are listed in 40 CFR Parts 261.3. Additionally, waste generators must consult state and local hazardous waste regulations to ensure complete and accurate classification.
RCRA P-Series: None listed.
RCRA U-Series: None listed.

Section 14 - Transport Information

	US DOT	Canada TDG
Shipping Name:	SULFURIC ACID	SULFURIC ACID
Hazard Class:	8	8(9.2)
UN Number:	UN1830	UN1830
Packing Group:	II	II

Section 15 - Regulatory Information

US FEDERAL

TSCA
CAS# 7664-93-9 is listed on the TSCA inventory.
CAS# 7732-18-5 is listed on the TSCA inventory.
Health & Safety Reporting List
None of the chemicals are on the Health & Safety Reporting List.

Chemical Test Rules

None of the chemicals in this product are under a Chemical Test Rule.

Section 12b

None of the chemicals are listed under TSCA Section 12b.

TSCA Significant New Use Rule

None of the chemicals in this material have a SNUR under TSCA.

CERCLA Hazardous Substances and corresponding RQs

CAS# 7664-93-9: 1000 lb final RQ; 454 kg final RQ

SARA Section 302 Extremely Hazardous Substances

CAS# 7664-93-9: 1000 lb TPQ

SARA Codes

CAS # 7664-93-9: immediate, delayed, reactive.

Section 313

This material contains Sulfuric acid (CAS# 7664-93-9, 69-75%),which is subject to the reporting requirements of Section 313 of SARA Title III and 40 CFR Part 373.

Clean Air Act:

This material does not contain any hazardous air pollutants.
This material does not contain any Class 1 Ozone depletors.
This material does not contain any Class 2 Ozone depletors.

Clean Water Act:

CAS# 7664-93-9 is listed as a Hazardous Substance under the CWA.
None of the chemicals in this product are listed as Priority Pollutants under the CWA.
None of the chemicals in this product are listed as Toxic Pollutants under the CWA.

OSHA:

None of the chemicals in this product are considered highly hazardous by OSHA.

STATE

CAS# 7664-93-9 can be found on the following state right to know lists: California, New Jersey, Pennsylvania, Minnesota, Massachusetts.

CAS# 7732-18-5 is not present on state lists from CA, PA, MN, MA, FL, or NJ.

California Prop 65

The following statement(s) is(are) made in order to comply with the California Safe Drinking Water Act:

WARNING: This product contains Sulfuric acid, listed as `Strong inorganic acid mists contain', a chemical known to the state of California to cause cancer.

California No Significant Risk Level: None of the chemicals in this product are listed.

European / International Regulations

European Labeling in Accordance with EC Directives

Hazard Symbols:

C

Risk Phrases:

R 35 Causes severe burns.

Safety Phrases:

S 26 In case of contact with eyes, rinse immediately with plenty of water and seek medical advice.
S 30 Never add water to this product.
S 45 In case of accident or if you feel unwell, seek medical advice immediately (show the label where possible).

WGK (Water Danger / Protection)

CAS# 7664-93-9: 2
CAS# 7732-18-5: No information available.

Canada - DSL / NDSL

CAS# 7664-93-9 is listed on Canada's DSL List.
CAS# 7732-18-5 is listed on Canada's DSL List.

Canada - WHMIS

This product has a WHMIS classification of D2A, D1A, E.
This product has been classified in accordance with the hazard criteria of the Controlled Products Regulations and the MSDS contains all of the information required by those regulations.

Canadian Ingredient Disclosure List

CAS# 7664-93-9 is listed on the Canadian Ingredient Disclosure List.

Section 16 - Additional Information

MSDS Creation Date: 10/12/2000
Revision #5 Date: 1/23/2007

ALDRICH CHEMICAL CO INC. -- LIQUI-NOX PHOSPHATE-FREE DETERGENT, 24302-7 -- 6810-00N016648

========== Product Identification ==========

Product ID:LIQUI-NOX PHOSPHATE-FREE DETERGENT, 24302-7
MSDS Date:01/09/1990
FSC:6810
NIIN:00N016648
MSDS Number: BQTFQ
=== Responsible Party ===
Company Name:ALDRICH CHEMICAL CO INC.
Address:1001 W. ST. PAUL AVE
Box:355
City:MILWAUKEE
State:WI
ZIP:53201
Country:US
Info Phone Num:414-273-3850/FAX -4979
Emergency Phone Num:414-273-3850
CAGE:60928
=== Contractor Identification ===
Company Name:ALDRICH CHEMICAL CO INC
Address:1001 WEST ST PAUL AVE
Box:355
City:MILWAUKEE
State:WI
ZIP:53233
Country:US
Phone:414-273-3850
CAGE:60928

========== Composition/Information on Ingredients ==========

Ingred Name:LIQUI-NOX, PHOSPHATE-FREE DETERGENT

========== Hazards Identification ==========

LD50 LC50 Mixture:NONE SPECIFIED BY MANUFACTURER.
Routes of Entry: Inhalation:YES Skin:YES Ingestion:YES
Reports of Carcinogenicity:NTP:NO IARC:NO OSHA:NO
Health Hazards Acute and Chronic:ACUTE: MAY BE HARMFUL BY INHALATION, INGESTION, OR SKIN ABSORPTION. MAY CAUSE EYE IRRITATION. MAY CAUSE SKIN IRRITATION. TO THE BEST OF OUR KNOWLEDGE, THE CHEMICAL, PHYSICAL, AND TOXICOLOGICAL PROPERT IES HAVE NOT BEEN THOROUGHLY INVESTIGATED.
Explanation of Carcinogenicity:NOT RELEVANT
Effects of Overexposure:SEE HEALTH HAZARDS.
Medical Cond Aggravated by Exposure:NONE SPECIFIED BY MANUFACTURER.

========== First Aid Measures ==========

First Aid:EYE: IMMEDIATELY FLUSH EYES WITH COPIOUS AMOUNTS OF WATER FOR AT LEAST 15 MIN. SKIN: IMMEDIATELY WASH SKIN WITH SOAP AND COPIOUS AMOUNTS OF WATER. INHAL: REMOVE TO FRESH AIR. IF NOT BREATHING GIVE ART F RESP. IF BREATHING IS DIFFICULT, GIVEOXYGEN. CALL A PHYSICIAN. WASH CONTAMINATED CLOTHING BEFORE REUSE. INGEST: GET MD IMMEDIATELY .

========== Fire Fighting Measures ==========

Extinguishing Media:WATER SPRAY, CARBON DIOXIDE, DRY CHEMICAL POWDER, ALCOHOL OR POLYMER FOAM.
Fire Fighting Procedures:WEAR NIOSH/MSHA APPROVED SCBA AND FULL PROTECTIVE EQUIPMENT TO PREVENT CONTACT WITH SKIN AND EYES.
Unusual Fire/Explosion Hazard:NONE SPECIFIED BY MANUFACTURER.

========== Accidental Release Measures ==========

Spill Release Procedures:WEAR NIOSH/MSHA APPROVED RESP, CHEMICAL SAFETY GOGGLES, RUBBER BOOTS AND HEAVY RUBBER GLOVES. ABSORB ON SAND OR VERMICULITE AND PLACE IN CLOSED CONTAINERS FOR DISPOSAL. VENTILATE AREA AND WASH SPILL S ITE AFTER MATERIAL PICKUP IS COMPLETE.
Neutralizing Agent:NONE SPECIFIED BY MANUFACTURER.

========== Handling and Storage ==========

Handling and Storage Precautions:KEEP TIGHTLY CLOSED. STORE IN A COOL DRY PLACE. AVOID INHALATION. AVOID CONTACT WITH EYES, SKIN AND CLOTHING. AVOID PROLONGED OR REPEATED EXPOSURE.
Other Precautions:NONE SPECIFIED BY MANUFACTURER.

========== Exposure Controls/Personal Protection ==========

Respiratory Protection:NIOSH/MSHA APPROVED RESPIRATOR.
Ventilation:MECHANICAL EXHAUST REQUIRED.
Protective Gloves:COMPATIBLE CHEMICAL-RESISTANT GLOVES.
Eye Protection:CHEMICAL SAFETY GOGGLES.
Other Protective Equipment:SAFETY SHOWER AND EYE BATH.
Work Hygienic Practices:WASH THOROUGHLY AFTER HANDLING.
Supplemental Safety and Health
WASTE DISP: AND NEUTRALIZATION REACTIONS MAY GENRATE HEAT & FUMES WHICH CAN BE CONTROLLED BY THE RATE OF ADDITION. OBSERVE ALL FEDERAL, STATE AND LOCAL LAWS.

========== Physical/Chemical Properties ==========

HCC:N1
Spec Gravity:1.051
Appearance and Odor:NONE SPECIFIED BY MANUFACTURER.

========== Stability and Reactivity Data ==========

Stability Indicator/Materials to Avoid:YES
STRONG OXIDIZING AGENTS.
Stability Condition to Avoid:NONE SPECIFIED BY MANUFACTURER.
Hazardous Decomposition Products:NATURE OF DECOMPOSITION PRODUCTS NOT KNOWN.

========== Disposal Considerations ==========

Waste Disposal Methods:SML QTYS: CAUTIOUSLY ADD TO A LRG STIRRED EXCESS OF WATER. ADJUST THE PH TO NEUTRAL, SEPARATE ANY INSOLUBLE SOLIDS OR LIQUIDS & PACKAGE THEM FOR HAZARDOUS-WASTE DISP. FLUSH THE AQUEOUS SOLN DOWN THE D RAIN W/PLENTY OF WATER. THE HYDROLYSIS (SUPP DATA)

Disclaimer (provided with this information by the compiling agencies):
This information is formulated for use by elements of the Department

of Defense. The United States of America in no manner whatsoever, expressly or implied, warrants this information to be accurate and disclaims all liability for its use. Any person utilizing this document should seek competent professional advice to verify and assume responsibility for the suitability of this information to their particular situation.

ALCONOX MSDS

Section 1 : MANUFACTURER INFORMATION

Product name: Alconox

Supplier: Same as manufacturer.

Manufacturer: Alconox, Inc.
30 Glenn St.
Suite 309
White Plains, NY 10603.

Manufacturer emergency phone number: 800-255-3924.
813-248-0585 (outside of the United States).

Manufacturer: Alconox, Inc.
30 Glenn St.
Suite 309
White Plains, NY 10603.

Supplier MSDS date: 2005/03/09

D.O.T. Classification: Not regulated.

Section 2 : HAZARDOUS INGREDIENTS

C.A.S.	CONCENTRATION %	Ingredient Name	T.L.V.	LD/50	LC/50
25155-30-0	10-30	SODIUM DODECYLBENZENESULFONATE	NOT AVAILABLE	438 MG/KG RAT ORAL 1330 MG/KG MOUSE ORAL	NOT AVAILABLE
497-19-8	7-13	SODIUM CARBONATE	NOT AVAILABLE	4090 MG/KG RAT ORAL 6600 MG/KG MOUSE ORAL	2300 MG/M3/2H RAT INHALATION 1200 MG/M3/2H MOUSE INHALATION
7722-88-5	10-30	TETRASODIUM PYROPHOSPHATE	5 MG/M3	4000 MG/KG RAT ORAL 2980 MG/KG MOUSE ORAL	NOT AVAILABLE
7758-29-4	10-30	SODIUM PHOSPHATE	NOT AVAILABLE	3120 MG/KG RAT ORAL 3100 MG/KG MOUSE ORAL >4640 MG/KG RABBIT DERMAL	NOT AVAILABLE

Section 2A : ADDITIONAL INGREDIENT INFORMATION

Note: (supplier).
CAS# 497-19-8: LD50 4020 mg/kg - rat oral.
CAS# 7758-29-4: LD50 3100 mg/kg - rat oral.

Section 3 : PHYSICAL / CHEMICAL CHARACTERISTICS

Physical state: Solid

Appearance & odor: Almost odourless.
White granular powder.

Odor threshold (ppm): Not available.

Vapour pressure (mmHg): Not applicable.

Vapour density (air=1): Not applicable.

By weight: Not available.

Evaporation rate (butyl acetate = 1): Not applicable.

Boiling point (°C): Not applicable.

Freezing point (°C): Not applicable.

pH: (1% aqueous solution).
9.5

Specific gravity @ 20 °C: (water = 1).
0.85 - 1.10

Solubility in water (%): 100 - > 10% w/w

Coefficient of water\oil dist.: Not available.

VOC: None

Section 4 : FIRE AND EXPLOSION HAZARD DATA

Flammability: Not flammable.

Conditions of flammability: Surrounding fire.

Extinguishing media: Carbon dioxide, dry chemical, foam.
Water
Water fog.

Special procedures: Self-contained breathing apparatus required.
Firefighters should wear the usual protective gear.

Auto-ignition temperature: Not available.

Flash point (°C), method: None

Lower flammability limit (% vol): Not applicable.

Upper flammability limit (% vol): Not applicable.

Not available.

Sensitivity to mechanical impact: Not applicable.

Hazardous combustion products: Oxides of carbon (COx).
Hydrocarbons.

Rate of burning: Not available.

Explosive power: None

Section 5 : REACTIVITY DATA

Chemical stability: Stable under normal conditions.

Conditions of instability: None known.

Hazardous polymerization: Will not occur.

Incompatible substances: Strong acids.
Strong oxidizers.

Hazardous decomposition products: See hazardous combustion products.

Section 6 : HEALTH HAZARD DATA

Route of entry: Skin contact, eye contact, inhalation and ingestion.

Effects of Acute Exposure

Eye contact: May cause irritation.

Skin contact: Prolonged contact may cause irritation.

Inhalation: Airborne particles may cause irritation.

Ingestion: May cause vomiting and diarrhea.
May cause abdominal pain.
May cause gastric distress.

Effects of chronic exposure: Contains an ingredient which may be corrosive.

LD50 of product, species & route: > 5000 mg/kg rat oral.

LC50 of product, species & route: Not available for mixture, see the ingredients section.

Exposure limit of material: Not available for mixture, see the ingredients section.

Sensitization to product: Not available.

Carcinogenic effects: Not listed as a carcinogen.

Reproductive effects: Not available.

Teratogenicity: Not available.

Mutagenicity: Not available.

Synergistic materials: Not available.

Medical conditions aggravated by exposure: Not available.

First Aid

Skin contact: Remove contaminated clothing.
Wash thoroughly with soap and water.
Seek medical attention if irritation persists.

Eye contact: Check for and remove contact lenses.
Flush eyes with clear, running water for 15 minutes while holding eyelids open: if irritation persists, consult a physician.

Inhalation: Remove victim to fresh air.
Seek medical attention if symptoms persist.

Ingestion: Dilute with two glasses of water.
Never give anything by mouth to an unconscious person.
Do not induce vomiting, seek immediate medical attention.

Section 7 : PRECAUTIONS FOR SAFE HANDLING AND USE

Leak/Spill: Contain the spill.
Recover uncontaminated material for re-use.
Wear appropriate protective equipment.
Contaminated material should be swept or shoveled into appropriate waste container for disposal.

Waste disposal: In accordance with municipal, provincial and federal regulations.

Handling procedures and equipment: Protect against physical damage.
Avoid breathing dust.
Wash thoroughly after handling.
Keep out of reach of children.
Avoid contact with skin, eyes and clothing.
Launder contaminated clothing prior to reuse.

Storage requirements: Keep containers closed when not in use.
Store away from strong acids or oxidizers.
Store in a cool, dry and well ventilated area.

Section 8 : CONTROL MEASURES

Precautionary Measures

Gloves/Type:

Neoprene or rubber gloves.

Respiratory/Type:

If exposure limit is exceeded, wear a NIOSH approved respirator.

Eye/Type:

Safety glasses with side-shields.

Footwear/Type: Safety shoes per local regulations.

Clothing/Type: As required to prevent skin contact.

Other/Type: Eye wash facility should be in close proximity.
Emergency shower should be in close proximity.

Ventilation requirements: Local exhaust at points of emission.

ATTACHMENT 5

EMPLOYEE EXPOSURE/INJURY INCIDENT REPORT

Employee Exposure/Injury Incident Report

(completed by the CHSM or designee)

Employee: _____

Office or field location: _____

Incident:

Potential or known exposure (describe): _____

Physical injury or illness (describe): _____

Location (city and state): _____ Project and Contract No. _____

Date of incident: _____ Time of incident: _____

Date incident reported: _____ Person to whom incident was reported: _____

Weather condition during incident: Temperature: _____ Precipitation: _____

Wind speed and direction: _____ Cloud cover: _____

Name of materials potentially encountered (chemical exposure):

Chemical and phase (i.e., liquid, solid, gas, vapor, fume, mist), radiological, etc.: _____

Describe the exposure/injury in detail and the parts of the body affected (attach extra sheets if necessary):

Describe exact onsite or offsite location where the incident occurred:

What was the employee doing when the exposure/injury occurred? (Describe briefly as site reconnaissance, soil sampling, etc.):

How did the incident occur? Describe fully the factors that led to or contributed to the incident:

_____

_____

Was medical treatment given? ☐ Yes ☐ No If yes, when? _____

By whom? Name of paramedic: _____

Name of physician: _____

Other: _____

Where? Onsite _____ Offsite _____

If offsite, name of hospital or clinic: _____

Length of inpatient stay (dates): _____

Was Integral Consulting management notified? ☐ Yes ☐ No If yes, when? _____

Name and title of manager(s) notified: _____

Did the exposure/injury result in permanent disability or death? ☐ Yes ☐ No

If yes, explain: _____

Number of days away from work _____ Number of days of restricted work activity: _____

Has the employee returned to work? (Yes / No) If yes, date: _____

Names of other persons affected during the incident:

_____ _____

Names of persons who witnessed the incident:

_____ _____

Name and title of field team leader or immediate supervisor at the site:

_____

Was the operation being conducted under an established safety plan? ☐ Yes ☐ No

If yes, attach a copy. If no, explain: _____

_____

Was personal protective equipment (PPE) used by the employee? ☐ Yes ☐ No

If yes, list items: _____

Did any limitations in safety equipment or PPE affect or contribute to exposure? ☐ Yes ☐ No

If yes, explain: _____

_____

Attachments to this report: ____ Medical report(s) (if not confidential) ____ Site safety plan

____ Other relevant information

_____ _____

Employee's signature Date

_____ _____

Site safety officer's signature Date

_____ _____

Project manager's signature Date

Corporate health and safety manager review and comments

Corrective action/procedure changes carried out on the project:

_____

_____

_____

Corrective actions to be taken to prevent similar incidents at other sites:

_____

_____

_____

_____ _____

Corporate Health and Safety Manager's signature Date

ATTACHMENT 6

NEAR-MISS INCIDENT REPORT

Near-Miss Incident Report

(completed by field staff)

Employee: _____

Office or site location: _____

Near-Miss Incident (check one or more): Exposure ☐ Physical injury ☐ Property damage ☐

Location (city and state): _____ Project and Contract No. _____

Date of incident: _____ Time of incident: _____

Fully describe the incident, including how it happened, persons involved, if chemicals were involved in the incident, etc.:

_____

_____

_____

_____

_____

_____

_____

Was the operation being conducted under an established safety plan? ☐ Yes ☐ No

If yes, attach a copy. If no, explain: _____

_____

_____

_____ _____

Employee's signature Date

_____ _____

Project Manager's signature Date

_____ _____

Site safety officer's signature Date

Corporate health and safety manager review and comments

Corrective action/procedure changes carried out at the site:

Corrective actions to be taken to prevent similar incidents at other sites:

Corporate Health and Safety Manager's signature

Date

ATTACHMENT B2

STANDARD OPERATING PROCEDURES

INTEGRAL CONSULTING SOPS

STANDARD OPERATING PROCEDURE (SOP) AP-01

SAMPLE PACKAGING AND SHIPPING

SCOPE AND APPLICATION

This SOP describes specific requirements for sample packaging and shipping to ensure the proper transfer and documentation of environmental samples collected during field operations. Procedures for the careful and consistent transfer of samples from the field to the laboratory are outlined herein. This SOP also presents the method to be used when packing samples that will either be hand delivered or shipped by commercial carrier to the laboratory.

EQUIPMENT AND SUPPLIES REQUIRED

Make sure that you have the equipment and supplies necessary to properly pack and ship environmental samples, including the following:

- Project-specific sampling and analysis plan (SAP)
- Project-specific field logbook
- Sealable airtight bags in assorted sizes (e.g., Ziploc®)
- Wet ice in doubled, sealed bags; frozen Blue Ice®; or dry ice
- Cooler(s)
- Bubble wrap
- Fiber-reinforced packing tape, clear plastic packing tape, and duct tape
- Scissors or knife
- Chain-of-custody (COC) forms
- COC seals
- Large plastic garbage bags (preferably 3 mil [0.003 in.] thick)
- Paper towels
- "Fragile," "This End Up," or "Handle With Care" labels
- Mailing labels
- Air bills for overnight shipment

PROCEDURE

Customize the logistics for sample packaging and shipping to each study. If necessary, transfer samples from the field to a local storage facility where they can be frozen or refrigerated. Depending on the logistics of the operation, field personnel may transport samples to the laboratory or use a commercial courier or shipping service. In the latter case, Integral field personnel must be aware of any potentially limiting factors to timely shipping, such as availability of overnight service and weekend deliveries to specific areas, and shipping regulations regarding "restricted articles" (e.g., dry ice, formalin) prior to shipping the samples.

SAMPLE PREPARATION

Take the following steps to ensure the proper transfer of samples from the field to the laboratories:

At the sample collection site:

1. Document all samples using the proper logbooks or field forms (see SOP AP-02), required sample container identification (i.e., sample labels with tag numbers), and COC form (example provided in SOP AP-03). Fill out the COC form as described in SOP AP-03, and use the sample labeling techniques provided in SOP AP-04.
2. Make all applicable laboratory quality control sample designations on the COC forms. Clearly identify samples that will be archived for future possible analysis. Label these samples as follows: "Do Not Analyze: Hold and archive for possible future analysis." Some laboratories interpret "archive" to mean that they should continue holding the residual sample after analysis.
3. Notify the laboratory contact and the Integral project quality assurance/quality control (QA/QC) coordinator that samples will be shipped and the estimated arrival time. Send copies of all COC forms to Integral's project QA/QC coordinator or project manager, as appropriate.
4. Keep the samples in the possession of the sampling personnel at all times. Lock and secure any temporary onsite sample storage areas to maintain sample integrity and COC requirements.
5. Clean the outside of all dirty sample containers to remove any residual material that may lead to cross-contamination.
6. Complete the COC form as described in SOP AP-03, and retain the back (pink) copy for project records prior to sealing the cooler. Check sample containers against the COC form to ensure all the samples that were collected are in the cooler.

7. Store each sample container in a sealed plastic bag that allows the sample label (example provided in SOP AP-03) to be read. Before sealing the bags, ensure that volatile organic analyte (VOA) vials are encased in a foam sleeve or in bubble wrap.
8. If the samples require storage at a specific temperature, place enough ice in the sample cooler to maintain the temperature (e.g., 4°C) throughout the sampling day.

At the sample processing area (immediately after sample collection) take the following steps:

1. If the samples require a specific storage temperature, then cool the samples and maintain the temperature prior to shipping. For example, place enough ice in each sample cooler to maintain the temperature at 4°C until processing begins at the testing laboratory.
2. Be aware of holding time requirements for project-specific analytes and arrange the sample shipping schedule accordingly.
3. Place samples in secure storage (i.e., locked room or vehicle) or keep them in the possession of Integral sampling personnel before shipment. Lock and secure any sample storage areas to maintain sample integrity and COC requirements.
4. Store samples in the dark (e.g., keep coolers shut).

At the sample processing area (just prior to shipping), do the following:

1. Check sample containers against the COC form to account for all samples intended for shipment.
2. Choose cooler(s) of appropriate size and make sure they are clean of gross contamination inside and out. If the cooler has a drain, close the drain and secure it with duct tape.
3. Line the cooler with bubble wrap and place a large plastic bag (preferably with a thickness of 3 mil), open, inside the cooler.
4. Individually wrap each glass container (which was sealed in a plastic bag at the collection site) in bubble wrap and secure with tape or a rubber band. Place the wrapped samples in the large plastic bag in the cooler, leaving room for ice to keep the samples cold (i.e., 4°C).
5. If temperature blanks have been provided by the testing laboratory, place one temperature blank in each sample cooler.
6. If the samples require a specific storage temperature, add enough wet ice or Blue Ice® to maintain that temperature during overnight shipping (i.e., 4°C). Always overestimate the amount of ice that will be required. Keep ice in a sealed plastic bag, which is placed in a second sealed plastic bag to prevent leakage. Avoid separating the samples from the ice with excess bubble wrap because it may insulate the samples from the ice. After adding all samples and ice to the cooler, use bubble wrap (or other

available clean packing material) to fill any empty space and prevent the samples from shifting during transport.

7. If possible, consolidate all VOA samples in a single cooler and ship them with (a) trip blank(s) if the project-specific QA project plan calls for them.
8. Sign, date, and include any tracking numbers provided by the shipper on the COC form. Remove the back (pink) copy of the original COC form and retain this copy for the project records.
9. Seal the rest of the signed COC form in a bag and tape the bag to the inside of the cooler lid. Each cooler should contain an individual COC form for the samples contained inside it. If time is short and it becomes necessary to combine all the samples onto a single set of COC forms and ship multiple coolers together, then indicate on the outside of the appropriate cooler, "Chain-of-Custody Inside."
10. After the cooler is sufficiently packed to prevent shifting of the containers, close the lid and seal it with fiber-reinforced packing tape. Tape the cooler around the opening, joining the lid to the bottom, and around the circumference of the cooler at both hinges.
11. As security against unauthorized handling of the samples, apply two COC seals across the opening of the cooler lid (provided with example field forms). Place one seal on the front right portion of the cooler and one on the back left. Be sure the seals are properly affixed to the cooler to prevent removal during shipment. Additional tape across the seal may be necessary if the outside of the cooler is wet.

SAMPLE SHIPPING

Hand Delivery to the Testing Laboratory

1. Notify the laboratory contact and the Integral project QA/QC coordinator that samples will be delivered to the laboratory and the estimated arrival time.
2. When hand-delivering environmental samples, make sure the testing laboratory receives them on the same day that they were packed in the coolers.
3. Fax or scan and e-mail copies of all COC forms to the Integral project QA/QC coordinator. Note: It may be necessary to photocopy the COC form on a slightly darker setting so the form is readable after it has been faxed. Never leave the original COC form in the custody of non-Integral staff.

Shipped by Commercial Carrier to the Laboratory

1. Apply a mailing label to the cooler with destination and return addresses, and add other appropriate stickers, such as "This End Up," "Fragile," and "Handle With Care." If the shipment contains multiple coolers, indicate on the mailing label the number of coolers that the testing laboratory should expect to receive (e.g., 1 of 2; 2 of 2). Place clear tape over the mailing label to firmly affix it to the cooler and to protect it from the weather. This is a secondary label in case the air bill is lost during shipment.
2. Fill out the air bill and fasten it to the handle tags provided by the shipper (or the top of the cooler if handle tags are not available).
3. If samples must be frozen (–20°C) during shipping, make sure that dry ice has been placed in the sample cooler. Be aware of any additional shipping, handling, and special labeling requirements that the shipper may require.
4. Make sure that benthic infauna samples have been preserved with formalin in the field prior to shipping. Be aware of any additional shipping, handling, and special labeling requirements that the shipper may require for these samples.
5. Notify the laboratory contact and the Integral project QA/QC coordinator that samples will be shipped and the estimated arrival date and time. If environmental samples must be shipped at 4°C or –20°C, choose overnight shipping for delivery next morning. Fax or scan and e-mail copies of all COC forms to the Integral project QA/QC coordinator. Note: It may be necessary to photocopy the COC form on a slightly darker setting so the form is readable after faxing. Never leave the original COC form in the custody of non-Integral staff.

STANDARD OPERATING PROCEDURE (SOP) AP-02

FIELD DOCUMENTATION

SCOPE AND APPLICATION

This SOP describes the Integral procedure for accurate record-keeping in the field for the purposes of ensuring that samples can be traced from collection to final disposition.

Document all information relevant to field operations properly to ensure that activities are accounted for in written records to the extent that someone not present at the site could reconstruct the activity without relying on the memory of the field crew. Several types of field documents are used for this purpose and should be consistently used by field personnel. Field documentation should include only a factual description of site-related activities and observations. Field personnel should not include superfluous comments or speculation regarding the field activities or observations.

FIELD LOGBOOKS

During field sampling events, field logbooks must be used to record all daily activities. The purpose of the field logbook is to document events and record data measured in the field to the extent that someone not present at the site could reconstruct the activity without relying on the memory of the field crew. The project manager (or designee) should issue a field logbook to the appropriate site personnel for the direction of onsite activities (e.g., reconnaissance survey team leader, sampling team leader). It is this designee's responsibility to maintain the site logbook while it is in his or her possession and return it to the project manager or turn it over to another field team.

Make entries in the field logbook as follows:

1. Document all daily field activities in indelible ink in the logbook and make no erasures. Make corrections with a single line-out deletion, followed by the author's initials and the date. The author must initial and date each page of the field logbook. The author must sign and date the last page at the end of each day, and draw a line through any blank space remaining on the page below the last entry.

2. Write the project name, dates of the field work, site name and location (city and state), and Integral job number on the cover of the field logbook. If more than one logbook is used during a single sampling event, then annotate the upper right-hand corner of the logbook (e.g., Volume 1 of 2, 2 of 2) to indicate the number of logbooks used during the field event. Secure all field logbooks when not in use in the field. The following is a list of the types of information that is appropriate for entry in the field notebook:
   - Project start date and end date
   - Date and time of entry (24-hour clock)
   - Time and duration of daily sampling activities
   - Weather conditions at the beginning of the field work and any changes that occur throughout the day, including the approximate time of the change (e.g., wind speed and direction, rain, thunder, wave action, current, tide, vessel traffic, air and water temperature, thickness of ice if present)
   - Name and affiliation of person making entries and other field personnel and their duties, including what times they are present
   - The location and description of the work area, including sketches, map references, and photograph log, if appropriate
   - Level of personal protection being used
   - Onsite visitors (names and affiliations), if any, including what times they are present
   - The name, agency, and telephone number of any field contacts
   - Notation of the coordinate system used to determine the station location
   - The sample identifier and analysis code for each sample to be submitted for laboratory analysis, if not included on separate field data sheets
   - All field measurements made (or reference to specific field data sheets used for this purpose), including the time of collection and the date of calibration, if appropriate
   - The sampling location name, date, gear, water depth (if applicable), and sampling location coordinates, if not included on separate field data sheets
   - For aquatic sampling, the type of vessel used (e.g., size, power, type of engine)
   - Specific information on each type of sampling activity
   - The sample type (e.g., groundwater, soil, surface sediment), sample number, sample tag number, and any preservatives used, if not included on separate field data sheets
   - Sample storage methods

- Cross-references of numbers for duplicate samples
- A description of the sample (source and appearance, such as soil or sediment type, color, texture, consistency, presence of biota or debris, presence of oily sheen, changes in sample characteristics with depth, presence/location/thickness of the redox potential discontinuity [RPD] layer, and odor) and penetration depth, if not included on separate field data sheets
- Estimate of length and appearance of recovered cores, if not included on separate field data sheets
- Photographs (uniquely identified) taken at the sampling location, if any
- Details of the work performed
- Variations, if any, from the project-specific sampling and analysis plan (SAP) or standard operating protocols and reasons for deviation
- Details pertaining to unusual events that might have occurred during sample collection (e.g., possible sources of sample contamination, equipment failure, unusual appearance of sample integrity, control of vertical descent of the sampling equipment)
- References to other logbooks or field forms used to record information (e.g., field data sheets, health and safety log)
- Any field results not appearing on the field data sheets (if used), including station identification and location, date, and time of measurement
- Sample shipment information (e.g., shipping manifests, chain-of-custody (COC) form numbers, carrier, air bill numbers, time addresses)
- A record of quantity of investigation-derived wastes (if any) and storage and handling procedures.

3. During the field day, as listed above, record in the logbook a summary of all site activities. Provide a date and time for each entry. The information need not duplicate anything recorded in other field logbooks or field forms (e.g., site health and safety officer's logbook, calibration logbook, field data sheets), but should summarize the contents of the other logbooks and refer to the pages in these logbooks for detailed information.
4. If measurements are made at any location, record the measurements and equipment used, or refer to the logbook and page number(s) or field forms on which they are recorded. All maintenance and calibration records for equipment should be traceable through field records to the person using the instrument and to the specific piece of instrumentation itself.

5. Upon completion of the field sampling event, the sampling team leader will be responsible for submitting all field logbooks to be copied. A discussion of copy distribution is provided below.

FIELD DATA FORMS

Occasionally, additional field data forms are generated during a field sampling event (e.g., groundwater monitoring form, sediment core profile form, water quality measurement form) to record the relevant sample information collected. For instructions regarding the proper identification of field data forms, sampling personnel should consult the project-specific SAP.

Upon completion of the field sampling event, the sampling team leader will be responsible for submitting all field data forms to be copied. A discussion of copy distribution is provided below.

PHOTOGRAPHS

In certain cases, photographs (print or digital) of sampling stations may be taken using a camera-lens system with a perspective similar to the naked eye. Ensure that photographs include a measured scale in the image, when practical. If you take photographs of sample characteristics and routine sampling activities, avoid using telephoto or wide-angle shots, because they cannot be used in enforcement proceedings. Record the following items in the field logbook for each photograph taken:

1. The photographer's name or initials, the date, the time of the photograph, and the general direction faced (orientation)
2. A brief description of the subject and the field work shown in the picture
3. For print photographs, the sequential number of the photograph and the roll number on which it is contained
4. For digital photographs, the sequential number of the photograph, the file name, the file location, and back-up disk number (if applicable).

Upon completion of the field sampling event, the sampling team leader is responsible for submitting all photographic materials to be developed (prints) or copied (disks). Place the prints or disks and associated negatives in the project files (at the Integral project manager's location). Make photocopies of photo logs and any supporting documentation from the field logbooks, and place them in the project files with the prints or disks.

EQUIPMENT CALIBRATION RECORDS

Record in the field logbook all equipment calibration records, including instrument type and serial number, calibration supplies used, calibration methods and calibration results, date, time, and personnel performing the calibration. Calibrate all equipment used during the investigation daily, at a minimum, in accordance with the manufacturers' recommendations.

DISTRIBUTION OF COPIES

When the field team has returned from the sampling event, the field team leader is responsible for making sure that the field documentation is 1) scanned and placed into the project file on the portal (in a subfolder named Field under Working_Files), and 2) a copy of all field logbooks and additional field data forms is made and placed into the project file. Both the scanned copy and the hard copy will be available for general staff use.

The original field logbooks and forms will be placed in a locked file cabinet for safekeeping. One file cabinet at each Integral office will contain the original field documentation for multiple projects. The original field documentation will be filed at the Integral office where the project manager is located.

SET-UP OF LOCKING FILE CABINET

Place each project in its own file folder in a locking file cabinet. On the folder label, include the project name and contract number. Each project folder will include up to six kinds of files:

- Field logbook(s)
- Additional field data forms
- Photographs
- COC forms
- Acknowledgment of Sample Receipt forms
- Archive Record form (to be completed only if samples are archived at an Integral field storage facility or Integral laboratory).

STANDARD OPERATING PROCEDURE (SOP) AP-03

SAMPLE CUSTODY

SCOPE AND APPLICATION

This SOP describes Integral procedures for custody management of environmental samples.

A stringent, established program of sample chain-of-custody will be followed during sample storage and shipping activities to account for each sample. The procedure outlined herein will be used with SOP AP-01, which covers sample packaging and shipping; SOP AP-02, which covers the use of field logbooks and other types of field documentation; and SOP AP-04, which covers sample labeling. Chain-of-custody (COC) forms ensure that samples are traceable from the time of collection through processing and analysis until final disposition. A sample is considered to be in a person's custody if any of the following criteria are met:

1. The sample is in the person's possession
2. The sample is in the person's view after being in his or her possession
3. The sample is in the person's possession and is being transferred to a designated secure area
4. The sample has been locked up to prevent tampering after it was in the person's possession.

At no time is it acceptable for samples to be outside of Integral personnel's custody unless the samples have been transferred to a secure area (i.e., locked up). If the samples cannot be placed in a secure area, then an Integral field team member must physically remain with the samples (e.g., at lunch time one team member must remain with the samples).

CHAIN-OF-CUSTODY FORMS

The COC form is critical because it documents sample possession from the time of collection through final disposition. The form also provides information to the laboratory regarding what analyses are to be performed on the samples that are shipped.

Complete the COC form after each field collection activity and before shipping the samples to the laboratory. Sampling personnel are responsible for the care and custody of the samples until they are shipped. The individuals relinquishing and receiving the samples must sign the

COC form(s), indicating the time and date of the transfer, when transferring possession of the samples.

A COC form consists of three-part carbonless paper with white, yellow, and pink copies. The sampling team leader keeps the pink copy. The white and yellow sheets are placed in a sealed plastic bag and secured inside the top of each transfer container (e.g., cooler). Field staff retain the pink sheet for filing at the Integral project manager's location. Each COC form has a unique four-digit number. This number and the samples on the form must be recorded in the field logbook. Integral also uses computer-generated COC forms. If computer-generated forms are used, then the forms must be printed in triplicate and all three sheets signed so that two sheets can accompany the shipment to the laboratory and one sheet can be retained on file. Alternatively, if sufficient time is available, the computer-generated forms will be printed on three-part carbonless paper.

Record on the COC form the project-assigned sample number and the unique tag number at the bottom of each sample label. The COC form also identifies the sample collection date and time, type of sample, project name, and sampling personnel. In addition, the COC form provides information on the preservative or other sample pretreatment applied in the field and the analyses to be conducted by referencing a list of specific analyses or the statement of work for the laboratory. The COC form is sent to the laboratory along with the sample(s).

PROCEDURES

Use the following guidelines to ensure the integrity of the samples:

1. Sign and date each COC form. Have the person who relinquishes custody of the samples also sign this form.
2. At the end of each sampling day and prior to shipping or storage, make COC entries for all samples. Check the information on the labels and tags against field logbook entries.
3. Do not sign the COC form until the team leader has checked the information for inaccuracies. Make corrections by drawing a single line through any incorrect entry, and then initial and date it. Make revised entries in the space below the entries. After making corrections, mark out any blank lines remaining on the COC form, using single lines that are initialed and dated. This procedure will prevent any unauthorized additions.

   At the bottom of each COC form is a space for the signatures of the persons relinquishing and receiving the samples and the time and date of the transfer. The time the samples were relinquished should match exactly the time they were received by another party. Under no circumstances should there be any time when custody of the samples is undocumented.

4. If samples are sent by a commercial carrier not affiliated with the laboratory, such as FedEx or United Parcel Service (UPS), record the name of the carrier on the COC form. Also enter on the COC form any tracking numbers supplied by the carrier. The time of transfer should be as close to the actual drop-off time as possible. After signing the COC forms and removing the pink copy, seal them inside the transfer container.
5. If errors are found after the shipment has left the custody of sampling personnel, make a corrected version of the forms and send it to all relevant parties. Fix minor errors by making the change on a copy of the original with a brief explanation and signature. Errors in the signature block may require a letter of explanation.
6. Provide a COC form and an Archive Record form for any samples that are archived internally at Integral.

Upon completion of the field sampling event, the sampling team leader is responsible for submitting all COC forms to be copied. A discussion of copy distribution is provided in SOP AP-02.

CUSTODY SEAL

As security against unauthorized handling of the samples during shipping, affix two custody seals to each sample cooler. Place the custody seals across the opening of the cooler (front right and back left) prior to shipping. Be sure the seals are properly affixed to the cooler so they cannot be removed during shipping. Additional tape across the seal may be prudent.

SHIPPING AIR BILLS

When samples are shipped from the field to the testing laboratory via a commercial carrier (e.g., FedEx, UPS), the shipper provides an air bill or receipt. Upon completion of the field sampling event, the sampling team leader will be responsible for submitting the sender's copy of all shipping air bills to be copied at an Integral office. A discussion of copy distribution is provided in SOP AP-02. Note the air bill number (or tracking number) on the applicable COC forms or, alternatively, note the applicable COC form number on the air bill to enable the tracking of samples if a cooler becomes lost.

ACKNOWLEDGMENT OF SAMPLE RECEIPT FORMS

In most cases, when samples are sent to a testing laboratory, an Acknowledgment of Sample Receipt form is faxed to the project QA/QC coordinator the day the samples are received by the laboratory. The person receiving this form is responsible for reviewing it, making sure that the laboratory has received all the samples that were sent, and verifying that the correct analyses were requested. If an error is found, call the laboratory immediately, and document

any decisions made during the telephone conversation, in writing, on the Acknowledgment of Sample Receipt form. In addition, correct the COC form and fax the corrected version to the laboratory.

Submit the Acknowledgment of Sample Receipt form (and any modified COC forms) to be copied. A discussion of copy distribution is provided in SOP AP-02.

ARCHIVE RECORD FORMS

On the rare occasion that samples are archived at an Integral office, it is the responsibility of the project manager to complete an Archive Record form. This form is to be accompanied by a copy of the COC form for the samples, and will be placed in a locked file cabinet. The original COC form remains with the samples in a sealed Ziploc® bag.

STANDARD OPERATING PROCEDURE (SOP) AP-04

SAMPLE LABELING

SCOPE AND APPLICATION

This SOP describes the general Integral procedures for labeling samples, and the three kinds of labels that can be used on a project (i.e., sample labels, sample tags, and internal sample labels). Consult the project-specific sampling and analysis plan (SAP) to determine the exact sample identifiers and sample labels that are required for a given project. If they are not specified in the SAP, then follow the designations below.

SAMPLE IDENTIFIERS

Before field sampling begins, establish sample identifiers to be assigned to each sample as it is collected. Sample identifiers consist of codes designed to fulfill three purposes: 1) to identify related samples (i.e., replicates) to ensure proper data analysis and interpretation, 2) to obscure the relationships between samples so that laboratory analysis will be unbiased by presumptive similarities between samples, and 3) to track individual sample containers to ensure that the laboratory receives all material associated with a single sample. To accomplish these purposes, each container may have three different codes associated with it: the sample identifier, the sample number, and the sample tag number. These codes and their use are described as follows:

- **Sample Identification Code**—The sample identification code (Sample ID) is a unique designation that identifies where and how the sample was collected. The sample identifier is recorded in the field logbook *only* and is not provided on the sample label or chain-of-custody (COC) form. The sample identifier is a multiple-part code. The first component begins with the letter abbreviation; for example, "SWNS" or "SWNB" to designate the surface water sample was collected from the near-surface or near-bottom of the water column. The second part could identify the sampling event; for example, "1" to designate Round 1 sampling. The third part could contain an abbreviation for whether the station is a single point (SP), a transect (TR), a composite (CO), or a vertically integrated station (VI). The station number would be the final component of the sample identifier. Use leading zeros for stations with numbers below 100 for ease of data management and correct data sorting.

  If appropriate, add a supplemental component to the sample identifier to code field

duplicate samples and splits. Use a single letter (i.e., a suffix of "A" and "B") to indicate field duplicates or splits in the final component of the sample identifiers. For equipment decontamination blanks, assign sequential numbers starting at 900 instead of station numbers. Use a sample type code that corresponds to the sample type for which the decontamination blank was collected. Additional codes may be adopted, if necessary, to reflect sampling equipment requirements (see project-specific SAP).

Examples of sample IDs are as follows:

- SWNS-1-SP-002: Surface water sample collected from the near-surface at a single point during Round 1 from Station 2.
- SWNB-1-TR-010-A: Duplicate surface water sample from the near-bottom transect during Round 1 from Station 10.

- **Sample Number**—The sample number is an arbitrary number assigned to each distinct sample or split that is shipped to the laboratory for separate analysis. The sample number appears on the sample containers and the COC forms. Each sample will be assigned a unique sample number. All aliquots of a composited field sample will have the same sample number. In cases where samples consist of multiple bottles from the same location, assign each bottle the same sample number and time. However, assign replicates from the same location different sample numbers and times. Sample numbers of related field replicates will not necessarily have any shared content.

  Each field split of a single sample will also have a different sample number and time. The sample number is generally a unique six-digit number that includes a two-digit media code and a four-digit number. The media code may be site-specific, but the Integral default codes are as follows:

  - SS—Surface soil
  - BH—Subsurface soil or rock (typically from borehole)
  - GW—Groundwater
  - SW—Surface water
  - PW—Pore water
  - SD—Sediment
  - BT—Biota or biological tissue

    The exact sample numbering scheme may vary from project to project. Variances in the sample numbering scheme will be described in the project-specific SAP for the field event. Example sample numbers are PW0001, PW0002, PW0003, etc.

- **Tag Number—**Attach a different tag number to each sample container. If the amount of material (i.e., everything associated with a single sample number) is too large for a single container, assign each container the same sample number and a different sample tag. A sample will also be split between containers if a different preservation technique is used for each container (i.e., because different analyses will be conducted).

  The sample tag number is a unique five- or six-digit number assigned to each sample label (or "tag") for multiple bottles per sample. Integral sample labels come with a preprinted sample tag number. The tag number provides a unique tracking number to a specific sample bottle. This allows for greater flexibility in tracking sample bottles and assists in field quality control when filling out documentation and shipping. Sample tags are not used by many other consultants, and there may be resistance from such firms during teaming situations. However, experience has shown that tags can be very valuable, both in the field and while processing data from field efforts.

  Record tag numbers on the COC form. Laboratories use tag numbers only to confirm that they have received all of the containers that were filled and shipped. Data are reported by sample number.

Assign sample numbers sequentially in the field; sample labels are preprinted with sequential tag numbers.

SAMPLE LABELS

Integral sample labels are designed to uniquely identify each individual sample container that is collected during a sampling event. Field sampling teams are provided with preprinted sample labels, which must be affixed to each sample container used. Fill out the labels at the time the samples are collected, documenting the following information:

- Sample number
- Site name or project number
- Date and time sample is collected
- Initials of the samplers
- Preservatives used, if any
- A unique number (commonly referred to as the "Tag Number") that is preprinted on the label consisting of five or six digits; used to identify individual containers.

SAMPLE TAGS

Integral sample tags are designed to be affixed to each container that is used for a sample. Sample tags are required only for environmental samples collected in certain U.S.

Environmental Protection Agency (EPA) regions (e.g., EPA Region 5). Field crews are provided with preprinted sample tags. Attach sample tags to each individual sample container with a rubber band or wire through a reinforced hole in the tag. Mark all sample tag entries with indelible ink. Fill out the tags at the time the samples are collected, documenting the following information:

- Sample number
- Site name or project number
- Date and time sample is collected
- Initials of the samplers
- Preservatives used, if any
- Type of analysis.

A space for the laboratory sample number (provided by the laboratory at log-in) will also be provided on the sample tag.

INTERNAL SAMPLE LABELS

For benthic infaunal samples, wash away the sediment from the sample and collect the remaining benthic infauna into a sample container. Affix sample label (as discussed above) to the outside of the sample container. In addition, place an internal sample label inside the sample container. This internal sample label is made of waterproof paper; be sure to make all internal sample label entries with pencil. Fill out the internal sample labels at the time the samples are collected, documenting the following information:

- Sample number
- Site name or project number
- Date and time sample is collected
- Initials of the samplers
- Preservative used (e.g., formalin).

STANDARD OPERATING PROCEDURE (SOP) AP-05

INVESTIGATION-DERIVED WASTE HANDLING

SCOPE AND APPLICATION

This SOP presents the method to be used for handling wastes generated during field sampling activities that could be hazardous. These wastes are referred to as investigation-derived waste and are subject to specific regulations.

All disposable materials used for sample collection and processing, such as paper towels and gloves, are not considered investigation-derived wastes and will be placed in heavyweight garbage bags or other appropriate containers. Disposable supplies will be removed from the site by sampling personnel and placed in a normal refuse container for disposal at a solid waste landfill.

EQUIPMENT AND REAGENTS REQUIRED

- 55-gallon drums (or appropriately sized waste container)
- Paint markers
- Tools (to open and close drum)
- Ziploc® bags
- Drum labels.

PROCEDURES

1. Place solid wastes that need to be containerized in properly labeled, DOT- approved, 55-gallon drums.
2. Properly close, seal, label, and stage all filled or partially filled drums before demobilization. Properly profile full drums and have them shipped off site to a RCRA Subtitle C facility.

3. Sampling activities generate personal protective equipment and miscellaneous debris that require disposal. Remove gross contamination from these items, and place the items in plastic bags. It is acceptable to store these items in plastic bags as an interim measure. At the end of each day, dispose of the bags at an appropriate solid waste facility dumpster.

STANDARD OPERATING PROCEDURE (SOP) AP-06

NAVIGATION AND STATION POSITIONING

SCOPE AND APPLICATION

This SOP describes procedures for accurate station positioning required to ensure quality and consistency in collecting samples and in data interpretation and analysis. Station positioning must be both absolutely accurate in that it correctly defines a position by latitude and longitude, and relatively accurate in that the position must be repeatable, allowing field crew to reoccupy a station location in the future (e.g., for long-term monitoring programs).

This SOP describes the most commonly used station positioning method, differential global positioning system (DGPS). Integral uses a Trimble Pathfinder™ Pro XRS DGPS for station positioning for many field efforts. The Pro XRS offers the submeter accuracy often required for documenting sampling station locations and for re-locating previously sampled stations. A comprehensive discussion of the Trimble Pathfinder™ Pro XRS DGPS is provided in Attachments 1, 2, and 3 of this SOP.

SUMMARY OF METHOD

Global positioning system (GPS) navigation is used to position the sampler at the desired location. GPS is a satellite-based system that receives positioning data at 1-second intervals from multiple satellites at known positions in space. Standard GPS is calculated to an accuracy of about 10 m.

One can obtain a higher accuracy of approximately 2 m by applying differential corrections to the standard GPS positioning data using DGPS. These differential corrections are applied by sending GPS differential corrections to the GPS receiver via radio transmission. If the sampling location is near the coastal U.S, the U.S. Coast Guard generates differential corrections that are transmitted via radio link to the GPS receiver. If a Coast Guard station is out of range of the sampling area, then a receiver may be set up at a known (i.e., surveyed) reference point on land, or real-time satellite differential signals can be purchased from a private company (e.g., OmniSTAR).

With the Pro XRS, GPS data can be gathered to submeter accuracy using a choice of differential correction sources (i.e., free beacon differential signals such as Coast Guard beacons or OmniSTAR) without establishing a reference station. Data must be corrected to gain submeter accuracy. Free beacon or base station signals allow differential corrections to be

performed after data collection by using a nearby beacon or base station logging data files. (Note: The station must be within 300 miles of the data collection location.) For satellite-based signals, a built-in virtual base station allows for real-time data correction, eliminating the need for post-processing data in some cases. However, postprocessing data corrections can obtain accuracies in the range of 30–50 cm. These accuracies are for the horizontal (northing and easting) component only. The vertical component (elevation) accuracy ranges from submeter to 3 times larger than the horizontal accuracy.

The GPS receiver displays and transmits differentially corrected positioning data to the computer using an integrated navigation software package (e.g., HYPACK, Terrasync). The computer data are typically displayed and recorded in World Geodetic System of 1984 (WGS-1984) geographic coordinates (latitude/longitude). However, the integrated navigation system can display and record information in other datums (e.g., UTM, NAD83). The integrated navigation system, acting as a data manager, displays the sampler's position relative to a target station location in plan view on a video screen. The resulting pictorial screen presentation, as well as numeric navigation data (e.g., range and bearing to the target sampling location) assists the vessel operator (when sampling on-water) in approaching and maintaining the station position while sampling.

SUPPLIES AND EQUIPMENT

- Cable
- GPS antenna
- Telemetry antenna (for differential corrections)
- GPS receiver
- Differential corrections receiver
- Computer and monitor
- Navigation software (e.g., Terrasync)
- Logbook or log sheets.

PROCEDURES

Obtain latitude and longitude coordinates at the locations where samples are collected. An average positioning objective is to accurately determine and record the positions of all sampling locations to within 2 m. Positioning accuracies on the order of 1–3 m can be achieved by avoiding the few minutes per day when the satellites are not providing the same level of signal. The GPS provides the operator with a listing of the time intervals during the

day when accuracies are decreased. Avoiding these times allows for better positioning accuracy.

On-Land Sampling Event

A backpack DGPS unit may be used to direct the sampling team to the proposed sampling location. To expedite field activities, enter the target station coordinates into the navigation system database prior to beginning sampling. Place the DGPS antenna as close as possible to where the sampling will occur. Once the sample(s) have been collected at the appropriate location, record the horizontal coordinates of the station in the field logbook.

On-Water Sampling Event

Mount the GPS antenna vertically at the outboard end of the vessel's boom, with the GPS antenna cable extended along the boom into the cabin. Mount the telemetry antenna for receiving differential corrections on a convenient fixture outside the cabin. Locate the GPS receiver, the differential corrections receiver, and the computer in the cabin. Orient the video screen for the computer to allow the vessel operator to observe on-screen positioning data from the helm.

Alternatively, use a backpack DGPS unit to position the sampling vessel (e.g., barge) over a proposed sampling location. Place the DGPS beacon as close as possible to where the drilling will occur (i.e., moon pool). Using the DGPS unit, direct the sampling vessel operator to the sample station location.

Once the sampling vessel is anchored at the appropriate location, record the horizontal coordinates of the station in the field logbook. To expedite field activities, enter the target station coordinates in the navigation system database prior to beginning sampling.

Positioning System Verification

GPS requires no calibration, as all signal propagation is controlled by the U.S. government (the Department of Defense for satellite signals and the U.S. Coast Guard for differential corrections). Verifying the accuracy of the GPS requires coordinates to be known for one (or more) horizontal control point within the study area. The GPS position reading at any given station can then be compared to the known control point. Verify the GPS accuracy at the beginning and end of each sampling day.

Station Positioning Activities

Use a consistent routine for each day's positioning activities. After confirming successful reception of differential signals, turn on the computer on, and the boot the software. Verify the accuracy of the system at a horizontal control point, as described in the previous section.

The sampling team proceeds to a target station location selected by the team leader. That station location is then selected from a number of preselected station locations that have been entered into the integrated navigation system database. Once the station has been selected, the positioning data are displayed on the computer screen or hand-held unit to assist in proceeding to the station and in maintaining the station position during sampling. A confirmed position is recorded electronically each time a sample collection is attempted. (This means that during sediment grab sampling and coring, the locations of both accepted and rejected grabs or cores are recorded.) Upon recovery of the sampling device, read the station position northing (y) and easting (x) coordinates from the archived computer file and record them in the field logbook or on log sheets as a backup to the computer record. Also record time and water depth, if applicable. Ancillary information recorded in the field logbook may include personnel operating the GPS, tidal phase, type of sampling activity, and time when coordinates were collected.

REFERENCES

Trimble Navigation Limited. 2001. TSC1 Asset Surveyor operation manual. Version 5.20. http://trl.trimble.com/dscgi/ds.py/Get/File-8145/Oper.pdf.

Trimble Navigation Limited. 2007. GPS tutorial. Accessed on January 12, 2007. http://www.trimble.com/gps/index.shtml.

ATTACHMENT 1
PRO XRS DESCRIPTION

The Pro XRS combines a high-performance GPS receiver and antenna, beacon differential receiver, and satellite differential receiver in one compact unit. It also includes Trimble's advanced Everest™ technology, which allows users to collect accurate position data near walls, water, vehicles, or other surfaces that reflect satellite signals. Reflected signals, also called multipath signals, make it difficult for GPS receivers to accurately determine position. Everest™ uses a patented technique to remove multipath signals before measurements are used to calculate position.

Equipment Required

The GPS Pathfinder™ Pro XRS consists of the following:

- GPS receiver in backpack casing (with system batteries and cables)
- Hand-held data logger (TSC1) and cable, *or* laptop computer with Terrasync software installed and cable. (Note: Terrasync procedures are described under separate cover.)
- Pro XRS antenna, range poles, and cable
- Compass and tape measure
- Spare 12-volt camcorder and 9-volt batteries (minimum of two each) (use only Kodak, Duracell, or Energizer 9-volt batteries)
- Battery charger and power cord.

Pro XRS Setup

Follow these procedures for the proper setup of the Pro XRS:

1. Ensure that connections between batteries, receiver, and data logger are correct and secure. The coaxial antenna cable connects from the GPS receiver port "ANT" to the base of the antenna. The TSC1 cable (a "pig-tail"-type cable) connects from the bottom or top of the TSC1 to the receiver port "B," where a 9-pin serial port dongle is attached. The dual Y-clip cables should be connected from the receiver to the batteries. Alternatively, if AC power is available (e.g., aboard a vessel), then the power cable for the battery charger can be attached directly to the receiver on some models.
2. Screw the three long antenna poles together (the shorter pole may be added if necessary for taller users). Screw on the antenna and connect its cable.
3. Put backpack and/or shoulder strap on. The pouch for the data logger should be in place around the waist strap or in the backpack.

4. Screw antenna to the attachments on the top of the backpack. Wind cord around pole, and ensure the antenna is secure. Please be aware of overhead hazards, especially if working near low-hanging power lines. Severe injury or death can result.

Basic Operation of the Pro XRS

Recording a Feature

Before beginning field use, ensure that all GPS configurations and settings are set correctly for the particular use of the Pro XRS and that an appropriate data dictionary is loaded onto the TSC1 (see Attachments 2 and 3 for typical settings). These steps outline the basic use of the GPS to document a sample position or any other defined "feature." Note that the TSC1 has both hard and soft keys that allow for its operation. The hard keys comprise all of the keys (e.g., letters and numbers) on its surface. The soft keys are the F1 through F5 hard keys. The function of these changes depending upon the context. These keys will be referred to with brackets around them (<soft-key>).

1. Turn data logger on outside in an open area. Wait for antenna to receive satellite signals. The display will read Recording Almanac, Too Few SVs, and PDOP Too High. Continue to wait until enough satellites (four) are acquired and the PDOP is below 5.0.
2. Ensure that the real-time settings are correct according to the parameters listed in Attachment 2.
3. Select **Data Collection**, and create a new rover file or open an existing file. This file should be named according to the format specified by the project GIS analyst. Note: If opening an existing file, press **<NEW>** to access the *Antenna Options* menu and *Start Feature* menu.
4. Enter the height of the antenna from the ground to the *Measurement Method* reference point shown in the *Antenna Options* menu and then press **ENTER** to bring up the *Start Feature* menu.
5. Pick the appropriate data dictionary to use with the rover file. Only one dictionary can be used with a rover file. Consult with the project GIS analyst to formulate the most appropriate data dictionary for the type of sampling you wish to perform. The data dictionary titled *Generic* contains only a comment field and is appropriate for simple navigation tasks. If using a data dictionary, make sure to become familiar with its attributes before recording information in the field.

6. Move to the location of the first feature for which you want to record the GPS position. Select the appropriate feature and press **ENTER** to begin logging. Log data points in accordance with the feature type. Point features should have at least 10 points collected at a stationary location. Line features should be collected while moving. If movement is stopped, press the **<PAUSE>** key. When movement starts again, press the **<RESUME>** key. Area features should be collected with enough points to define the outline of the area (e.g., a square building would have four single points, collected on each corner, and the **<PAUSE>** key would be used between each of the points).
7. Depending on the setup of the data dictionary, each feature may have one or more feature attributes. An attribute is used to record additional data associated with the feature. For example, the attributes assigned to a sediment sampling station could be the sample number, station ID, sampling gear, sediment color, odor, etc.
8. Use the **<PAUSE>** key while recording feature attributes to avoid too many data points being collected at one point feature. (Body movements while logging attributes for an extended time can decrease the accuracy of collection.) The **<PAUSE>** key must be used when recording attributes of a line or area feature because only one data point should be collected in a single location.
9. Once all attributes are entered and the feature data points are logged, press **ENTER** to complete and save the feature and move on to a new feature. Pressing **ESC** instead of **ENTER** will allow the user to abandon the logged feature without saving.
10. When all features in a given area have been recorded, from the *Data Collection* menu, press **ESC** to exit data capture and then press **<YES>** to close the file. Features are appended and saved to the file after each collection, so there is no need to "save" the file. When the Pro XRS is not in use, it should be turned off. If you need to come back to the same rover file later in the day, the rover file may be reopened at that time. Rover files may not be edited after 7 days from the first feature was created. Please consult the project GIS analyst for the best way to handle multi-week sampling projects.
11. At the end of each day, download the rover file to a PC using Pathfinder Office software.

Feature Collection Options

Offsets—The Pro XRS can collect a point or line feature while standing at a set distance away from the feature. This option may be necessary because of obstructions such as tree cover, buildings, or car traffic. For a point feature, measure the distance between the object you want recorded and the Pro XRS antenna. Use the compass to determine the bearing (e.g., west is 270°). The bearing is the direction the point should be moved for it to be located in the correct place (e.g., if you are due north of the feature, the bearing is south, or 180°; i.e., the position you want recorded is south of where you are standing). Estimate the inclination from the

feature to the GPS antenna (if altitude determination is critical, a clinometer should be used). The inclination is the degree angle up from the feature to the antenna (e.g., if the feature is 5° below the antenna position, enter −5°). During data capture, from within the feature, press the **<OFFSET>** button, and enter the distance, bearing, and inclination. Press **OK** to complete the feature. Note: This procedure describes an offset of a single feature. A constant offset may be applied to all features collected as well.

Nesting—While recording a line feature or an area feature, a point feature may be collected to avoid backtracking. While recording the line or area feature, press **<PAUSE>** and then **<NEST>**. The Pro XRS will prompt for collection of a new feature. Move to the feature, and collect data as for any other point feature. When the feature is complete, press **OK**. The Pro XRS is ready to resume collecting data as part of the line/area feature: press **<RESUME>**. (Remember to continue moving before pressing resume to avoid having multiple positions recorded in the same place in the line or area feature.)

Segmenting—While moving along a line feature, changing the attributes of that line may be necessary (e.g., because of a change in surface type from paved to dirt road). This change may be done without having to begin a new feature by pressing **<PAUSE>** and then ****. Change the appropriate attributes and then press **<RESUME>** to continue recording.

Repeat—This function allows the collection of a new feature with the same feature attributes as the previous feature. If features are not exactly the same, it also allows editing of the attributes.

Quickmark—Allows collection of point features while moving (e.g., from a car or a boat) by estimating the exact location. The use of this feature will not result in positionally accurate locations and is not recommended for most sampling operations.

Reviewing and Editing Features

It is possible to review or edit features collected in the field while still in the data capture mode. For example, it may be necessary to document the GPS location in the field logbook or to edit one of the feature's attributes. Without exiting data capture, press **<REVIEW>**. (If data capture is already complete, just press **<REVIEW>** and then select the appropriate rover file.) This step will display a list of data points including each feature collected. Scroll to the appropriate feature, and follow the steps below depending on the required action:

- To view the GPS location (e.g., lat/lon), press **<POS>.**
- To edit the attributes, press **ENTER**. Make any necessary edits to the attributes by scrolling through.
- To change or add an offset, press **<POS>** and then **<OFFSET>**. Make any necessary changes.
- To delete a feature collected in error, press **<DEL>**.

Navigating to an Existing Location

Waypoints

To use the Pro XRS to navigate to a previously established position, this position must be loaded into the data logger as a waypoint, present as a feature position in the data files, or generated in the field using the GPS unit. Waypoints may be entered into the TSC1 by:

- Entering coordinates manually
- Choosing previously recorded locations and importing them into the TSC1 by using Pathfinder Office
- Defining a location stored in a rover file saved to the TSC1 as a waypoint (see *Reviewing/Editing Features*, above)
- Creating a way point from the current position being shown by the operating GPS unit in the field.

Navigating

Usually you will use the *Navigation* module (accessed by pressing **MENU** followed by **Navigation**) to guide yourself to a target (waypoint or feature). You can also use the *Map* module (accessed by pressing **MENU** followed by **Map**) to:

1. Orient yourself in the area where you are working.
2. Get a general indication of the location of a feature or waypoint that you want to find.
3. Find or select features or waypoints to which you wish to navigate toward.
4. Plot a course from one place to another.
    a. While in the Map screen, the GPS cursor x shows the current position reported by the receiver and is always shown on the Map screen (Note: it may not always be within the visible part of the screen when panning or scrolling). The **<OPTIONS>** key can be used to hide or display the GPS trail (line of dots showing up to 60 previous positions), the heading showing the direction of travel, and other options on the map display.
    b. Select a feature by pressing **MENU**, Data Collection to reach the *Start Feature* screen, and then **<REVIEW>** to access all features contained in the data file. Highlight and select the desired feature by pressing the **<Target>** key, which adds a crossed flag to the feature. Reaccess the *Map* screen by selecting **MENU**, then **Map**, which will now show the highlighted feature with a crossed flag symbol on the Map screen. You can then start moving toward the feature, and the current position (shown by the x) will move closer to the target position as the user approaches.

c. There are two graphical modes of navigation with the Pro XRS in the TSC1 *Navigation* module. On both modes, text information appears on the right of the screen in the *Info* panels, which can be configured by the user. The graphical modes available are the *Directional Dial* screen or the *Road* screen, which can be toggled between using the **<Mode>** key.

d. To navigate, select a target and then a start position. Each of these positions can be features from an open data file or a waypoint. Access a list of available features or waypoints by pressing **<TARGET>** or **<START>**. Once the item has been chosen as a target, it will show the crossed flags symbol in the list. Once a target has been selected, *Distance to Go* appears at the bottom of the *Navigation* screen, which indicates the distance from the current GPS position to the target. Select a start position (not required but useful for calculating crosstrack error and other navigation information) by pressing **<START>**. A waypoint of the current GPS position can be created for use as the Start point by selecting **<CREATE>**. Once the Start position is selected, a flag symbol will appear next to the item in the list.

e. In the *Directional Dial* mode, an arrow will appear that will always point at the target. This is the bearing to go. (Note: You need to be moving for this to be accurate, as it will lock if you are moving too slowly or have stopped.) The triangle at the top represents the direction that you are going or heading. This triangle never moves, but by changing directions, you can line up the arrow with the triangle. When the two are aligned, you are heading in the direction of the target. When you are close to the target, a bull's-eye (two concentric circles) will appear at the edge of the screen. This is warning you that the unit will be switching to the close up screen. A proximity alarm will sound and the directional arrow will be replaced by the bull's-eye on the close up screen. Your current position will be shown by an x and the target by the bull's-eye. Move so that the x is in the same location as the bull's-eye.

f. In the *Road* mode, navigate by walking down a road. Your position is shown by a stick figure and is always positioned in the center of the screen. The target (crossed flags) shows the point to which you are navigating toward. Your heading is shown by the top center of the screen and the bearing to go is shown by the direction of the road, which will rotate as you change your heading. Change your heading until the road is pointing at the top of the screen (*Target* is also at the top of the screen) and the edges are parallel to the sides of the screen. As you move toward the target the screen zooms in, so the road appears to get wider.

Downloading Rover Files

Upon returning to the office, download all rover files from the TSC1 to a PC for post-processing. You will need the Trimble Pathfinder software installed on your computer. If you

are not using a field laptop that already has the program installed, contact your project GIS analyst for instructions on how to install the software.

Connect the TSC1 to your computer using the appropriate cables. In addition to the "pigtail" cable, you will also need a null modem (a 9-pin female-to-female cable) to plug into a PC serial port. Once connected, power up the TSC1 unit and navigate to *MENU>File Manager>File Transfer*. Then, open the Pathfinder software and navigate to the *Utilities>Data Transfer…* window from the menu bar. Select **GIS Datalogger** on COM1 (for most computer systems), and press the green **Connect** button. Download files from the TSC1 by selecting the **Receive** tab and choosing the data file type from the *Add* pulldown menu (Figure 1).

After downloading, remove all rover files and waypoints from the TSC1 to conserve memory. Rover files may be deleted from the *File Manager* menu as follows:

1. Select **MENU>File Manager>Delete File(s)**
2. Select the rover file to be deleted, and press **<ENTER>**
3. Confirm the deletion of this file by pressing **<YES>**.

Delete data dictionaries in the same manner by selecting **Data Dictionaries** from the *File Manager* menu. Delete waypoints by selecting **Utilities** from the *Main* menu and then by selecting **Waypoints,** followed by **<DEL>**.

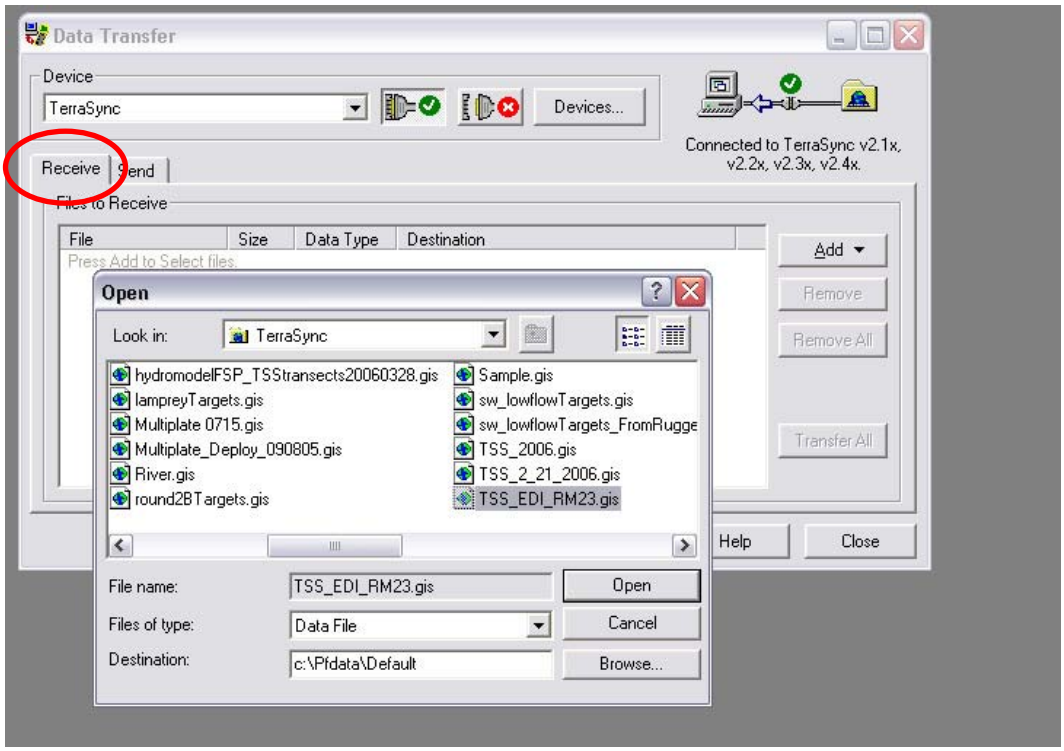


Figure 1. Transferring File from Terrasync

ATTACHMENT 2
TSC1 SETTINGS

The following are lists of menus that can be accessed through the TSC1 keypad. Please ensure that settings are correct before proceeding. Do not make changes to the settings unless necessary. Each menu will list all available subheadings, the correct setting, and the available **<soft-keys>** to access additional menus. Comments are included only where necessary.

GPS Rover Options

To access this menu, select **Configuration** from the main menu and then select **GPS Rover Options**. The table below lists logging options and settings.

Logging Options	Setting	Comment
Logging intervals		
Point feature	1s	
Line/area feature	2s–5s	depending upon speed of movement
Not in feature	None	
Velocity	None	
Confirm end feature	No	
Minimum pos	10	
Carrier Mode	Off	
Carrier phase min. time	10 minutes	
Dynamics code	Land	May be changed to sea or air, as appropriate
Audible click	Yes	
Log DOP data	Yes	
Log PPRT data	Yes	
Log QA/QC data	Yes	
Allow GPS update	Warn First	
Warning Distance	Any	
Position Mode Manual	3D	
Elevation Mask	15°	Should not go below 13° (accuracy decreases)
SNR Mask	6.0	Can raise to 7 if multi-path filtering is poor
PDOP Mask	5.0	Can be raised up to 8; reduces accuracy
PDOP Switch	6.0	

Real-Time Input Options

Access this menu from the GPS Rover *Options* menu by selecting **Real-Time Input**. The table below shows options and settings for real-time input.

Options	Setting	Comment
Preferred Correction Source		
	Choice 1	Integrated Beacon
	Choice 2	Integrated WAAS
	Choice 3	Use uncorrected GPS
Correction Age Limit	20s	

Antenna Options

Access this menu from the GPS rover *Options* menu by selecting **Antenna Options**. The table below shows antenna options and settings.

Option	Setting	Comment
Height	6 ft	Enter correct user antenna height using measurement method indicated below
Measure	Uncorrected	
Type	Integrated GPS/Beacon/Satellite	
Confirm	Per file	Can be changed to “Per feature” if antenna height varies and elevation is critical
Part Number	33580-50	Auto selected based on TYPE selected
Measurement	Bottom of Antenna	
Method	Mount	

ATTACHMENT 3
ADDITIONAL SETTINGS FOR THE TSC1

Additional TSC1 settings can be found in the *Configuration* menu. Items of particular importance are indicated in italics.

Configuration

This menu can be accessed by selecting **Configuration** from the main menu. The table below lists options and descriptions for the *Configuration* menu.

Options	Description
GPS base station options	For using a land base station or beacon for real time corrections
NMEA/TSIP output	Consult manual
Coordinate system	Changes coordinate system among latitude/longitude, UTM, and other coordinate systems. System can be converted, if necessary, after data capture by using Pathfinder Office software.
Map Display options	Change layers, scale, background files and items shown on the TSC1 screen during data collection
Navigation options	Changes Navigation parameters
Units and display	Changes various units, for example: length (e.g., feet, meters), altitude reference (e.g., MSL), *North reference* (i.e., true or magnetic). Units can be converted, if necessary, after data capture by using Pathfinder Office software.
Time and date	Changes to *local time*, 24-hour clock, date format, and other options
Quickmarks	Set-up parameters for use with Quickmarks.
Constant offset	Set-up parameters for use with a constant offset.
External sensors	Connections with external sensors.
Hardware (TSC1)	TSC1 settings such as beep volume, contrast, *internal and external battery status*, software version, free space.

Contrast and Backlighting

The TSC1 display can be viewed in various light settings. Press **FUNC,** then **L** to turn on the display backlight for viewing in dim lighting. Adjust the contrast by pressing **FUNC**, then **E** or **F**.

ATTACHMENT 4
PRE-SAMPLING ACTIVITIES BEFORE USE OF THE PRO XRS

Determination of Optimal Satellite-Use Time

Positioning accuracies on the order of ±1 to 3 m can be achieved by avoiding the few minutes per day when the satellites are not providing the same level of signal. The GPS unit provides the operator with a listing of the time intervals during the day when accuracies are decreased. Avoiding these time intervals permits the operator to maintain better positioning accuracy.

ATTACHMENT 5
MANAGING GPS DATA FROM TERRASYNC—A TUTORIAL

Currently, positional data collected in the field is most often done with a Trimble GPS unit (usually rented) interfaced with a laptop via Trimble's Terrasync software. The Terrasync software sometimes exhibits quirks that interfere with the smooth operation of data collection in otherwise stressful field conditions. This tutorial is meant to supplement the Terrasync software documentation and serve as a guide to field personnel to help them retrieve and collect geographic data as efficiently as possible with existing software.

Scope

This document is intended to be a reference for procedures involving the following:

- Fixing files that are more than 7 days old so that they can be updated
- Adding features in GPS Pathfinder software (companion to Terrasync) and then importing them as base files in Terrasync..

This document is not intended to be a comprehensive manual for using Terrasync or Pathfinder software. It is assumed that the reader has received at least some training on how to use the basic features of Terrasync and is competent at using MS Windows.

The Basics

GPS data collection currently relies on two pieces of complementary software:

- Terrasync—the interface for GPS navigation and data collection.
- Pathfinder Office—a multiuse piece of software that acts as a conduit between GIS data files (shape files) and Terrasync GPS files. Pathfinder can also be used as a simple map editor.

Installing the Correct Versions of Terrasync and Pathfinder

Important Note: This tutorial uses Pathfinder Office v. 3.00 and Terrasync v. 2.50. It is very important to use the proper versions of this software to avoid compatibility issues. These software versions should be included in the same folder as this tutorial, or can be obtained from GIS staff.

http://www.trimble.com/terrasync_ts.asp?Nav=Collection-4576

Key code for TerraSync
499043-00110-05273-EDD049BC

Pathfinder v.3.00
001533-00300-04152-0ee4d11f

Initial Setup of Terrasync/Pathfinder

Certain settings and configuration setups are needed before Pathfinder can talk to Terrasync. Whether you are installing this software for the first time or have an existing installation, check to make sure that these settings are in place.

1. Open Pathfinder Office and go to the *Utilities>Data Transfer...* menu. A dialog box should appear. This is the interface for communicating with Terrasync.
2. Click the **Devices** button, and then **New…** (Figure 1).
3. Click on **GIS Folder**.
4. Browse to the Terrasync data folder on your computer, which in most cases will be *C:\My Documents\TerraSync*.
5. In the next box, *Type* will be **Terrasync**, and *Version* will be **v. 2.1x, v.2.2x, v.2.3x,** and **v2.4x**.
6. At the prompt for a name that will display in the device list, enter **Terrasync**.
7. Go back to the Data Transfer dialog box, select **Terrasync** from the dropdown menu, press the **Connect** icon, and look for a green check mark indicating success.

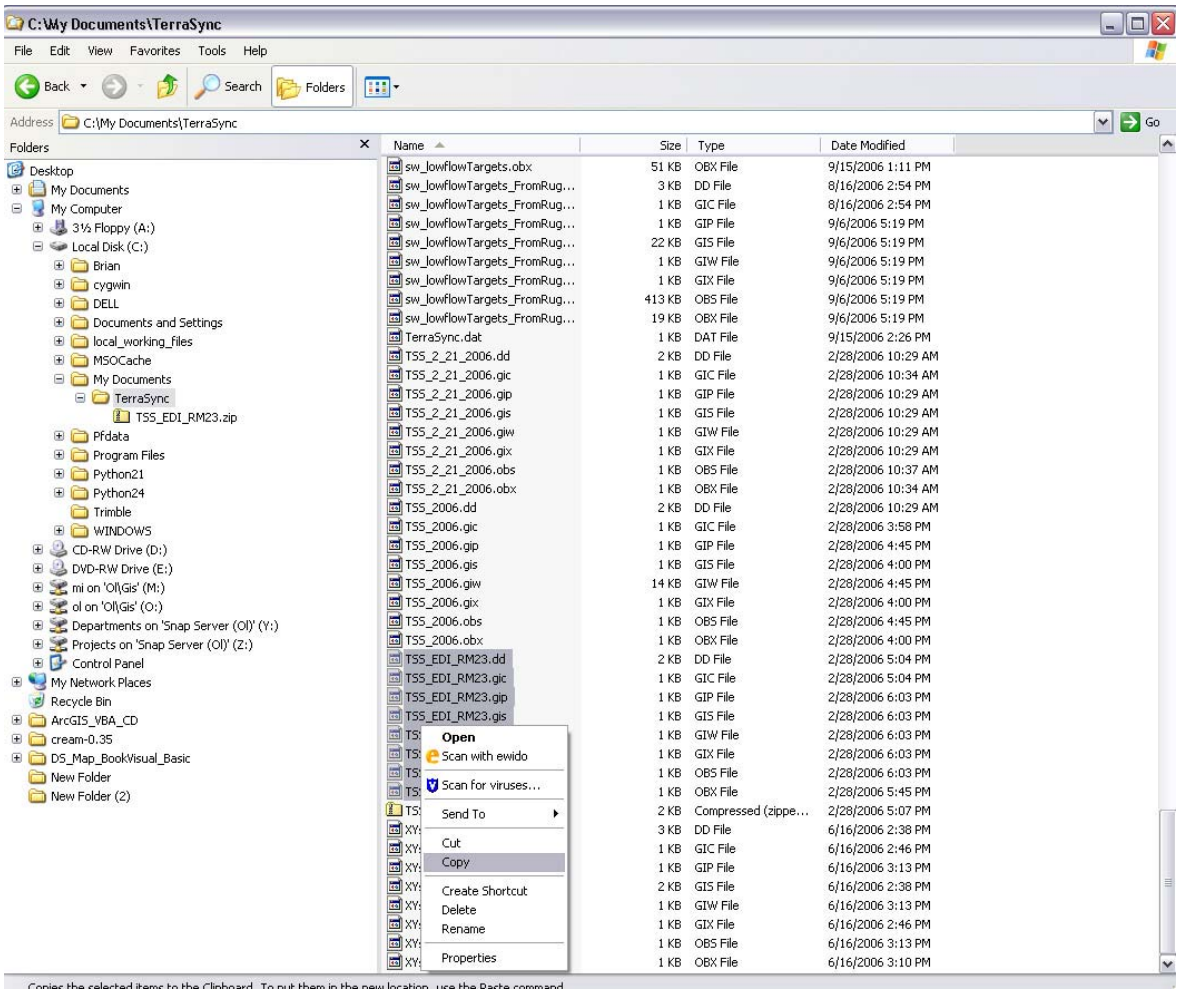


Figure 2. Selecting Files To Copy to a Different Directory

If this procedure does not work for you, you may have the wrong version of Pathfinder. For some unknown reason, with each version upgrade of Pathfinder, connectivity to older versions of Terrasync is lost. You can check what version of Pathfinder you have installed by going to the *Help>About GPS Pathfinder Office...* menu. To find out what version of Terrasync you have, go to *C:\Program Files\TerraSync*, right-click on **Terrasync.exe**, and choose the **Version** tab.

Handling Expired Files in Terrasync

One of the most common problems that field personnel will have to deal with is the 1-week expiration date when trying to collect data with Terrasync. This is a built-in function of Terrasync, and there is no simple way to work around it. The following instructions will guide you through the process to make the files usable. See Figure 3.

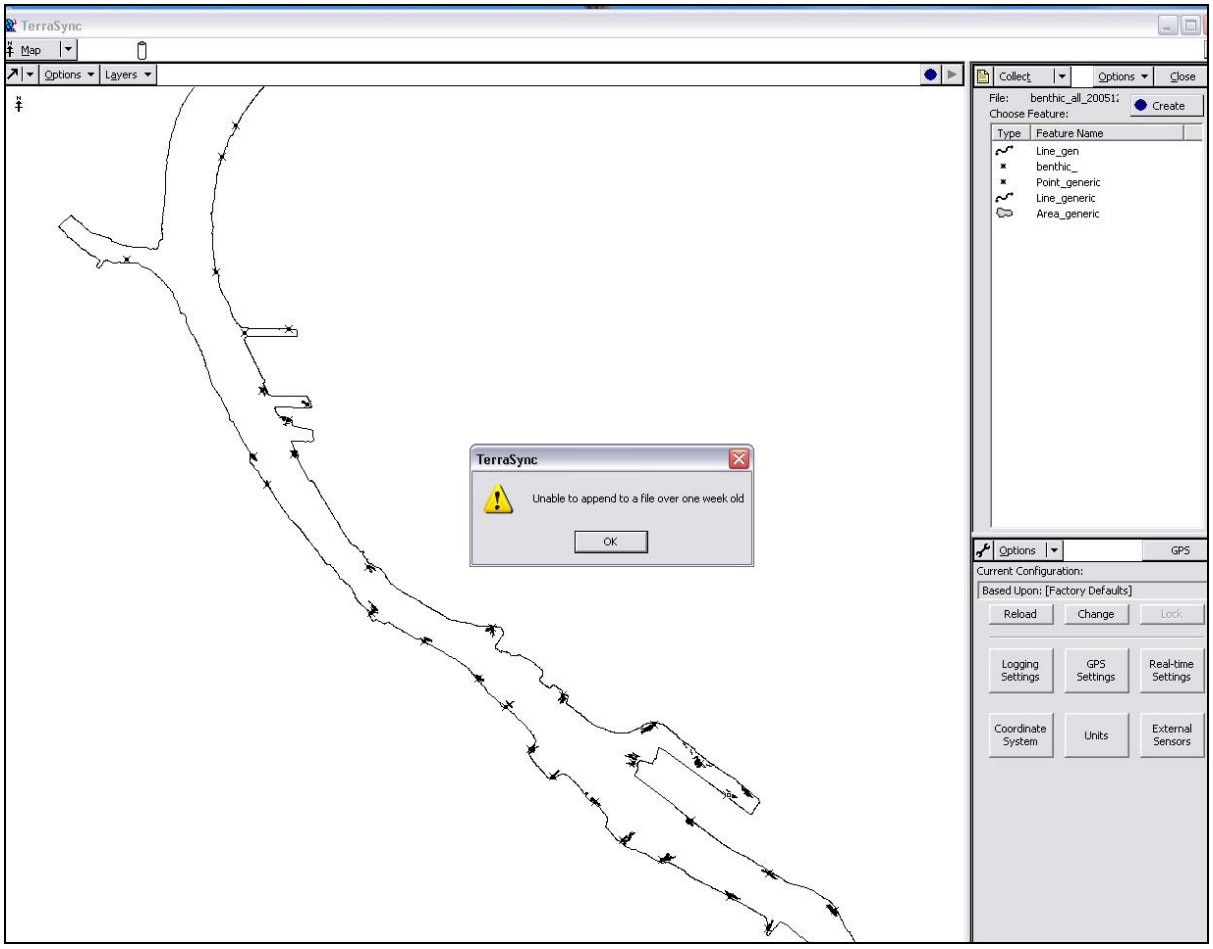


Figure 3. Notice That Terrasync File Older Than 1 Week Will Not Allow User To Collect Features (time begins to elapse when first feature is collected in the field, not when file is created)

Two options are available, depending on your needs. If you do not need to see the previously logged locations and need only to see the targets, use the original files provided by GIS staff (Option 1). If you need to see previously occupied locations in order to make decisions about where to go next, then transfer the file to Pathfinder and back again (Option 2).

Option 1: Move and replace logged files with original targets.

At the beginning of the field effort, you should receive a set of files with the target locations, most likely in a zip archive (.zip file extension). There will be six to eight files with the same name but with different extensions (Figure 4). These files will have to go into the *C:\My Documents\TerraSync* folder in order to be available to Terrasync.

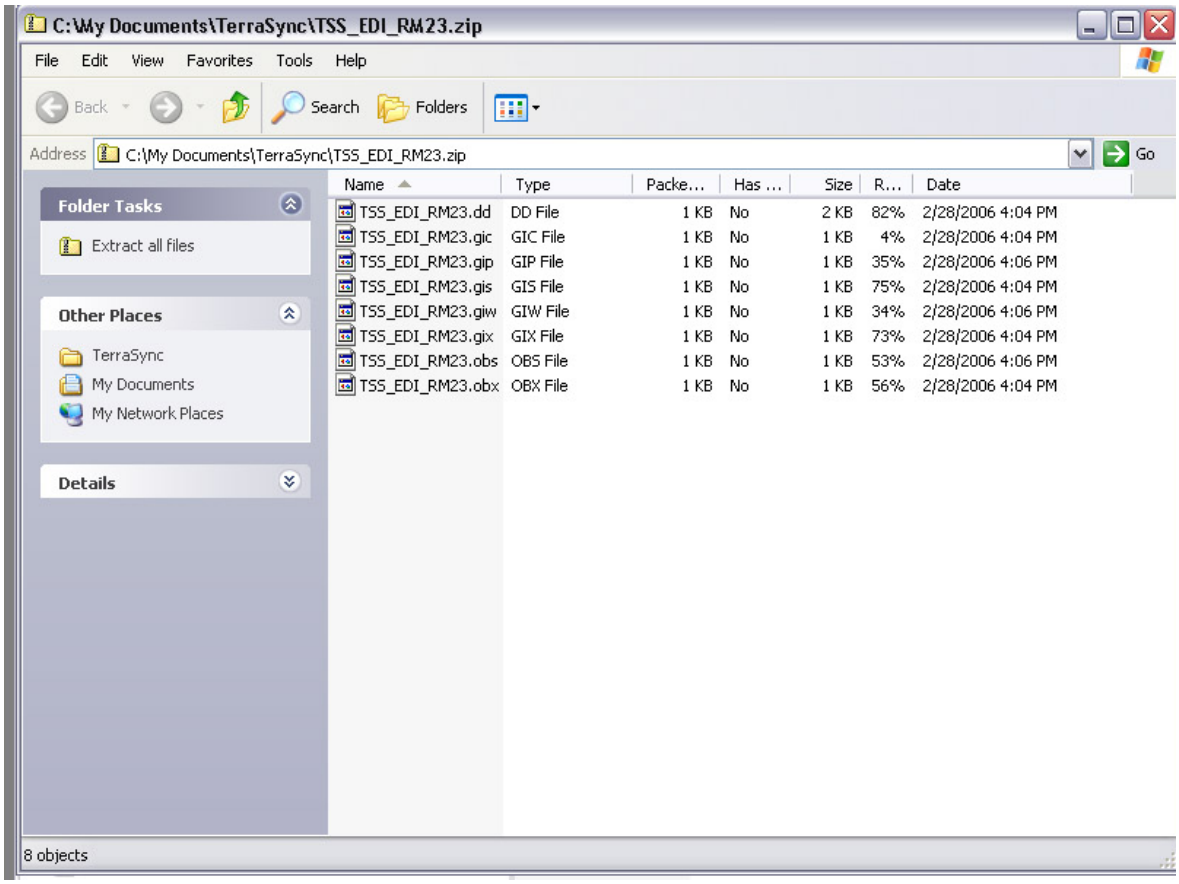


Figure 4. Example of File Set To Be Unzipped into the Terrasync Folder

After you unzip these files to Terrasync, keep this zip archive around in an easy-to-find place, such as your computer desktop, because the 1-week clock does not start until you begin collecting your first point in the field. You can use this unadulterated file again, as long as you make a copy of the work you did the previous week. The detailed steps are as follows:

1. Make sure you have the original files with the target locations available in a handy place. This will probably be the original zip archive. Also, be sure to close Terrasync while performing this process.
2. Navigate to C:\My Documents\TerraSync\ in Windows Explorer. Locate the files that you have been using the previous week. Note: It is crucial to get all of the small files associated with the data set. While it is useful to sort the files by date modified, you can miss some of the small files—it is highly recommended that you sort the files alphabetically.

3. Copy all of these files to a different directory, preferably one that is named appropriately to reflect the data and time period that you were collecting. For example: C:\Documents and Settings\bpointer\Desktop\lampreyTargets_20060925. These files contain the data you have collected the previous week and should be backed up and/or emailed to the appropriate project manager or GIS staff.
4. You can now safely replace the files you just copied with the ones from the original zip file. Right-click the zip archive, and click Extract All. When prompted to Select a folder to extract files to, browse to C:\My Documents\TerraSync. (Figure 5). If prompted about replacing existing files, select Yes to All. Note: It is crucial to make copies of the files first (see Step 3 above)—otherwise, you may lose the data.
5. You should now be able to open the file in Terrasync and begin logging as normal.

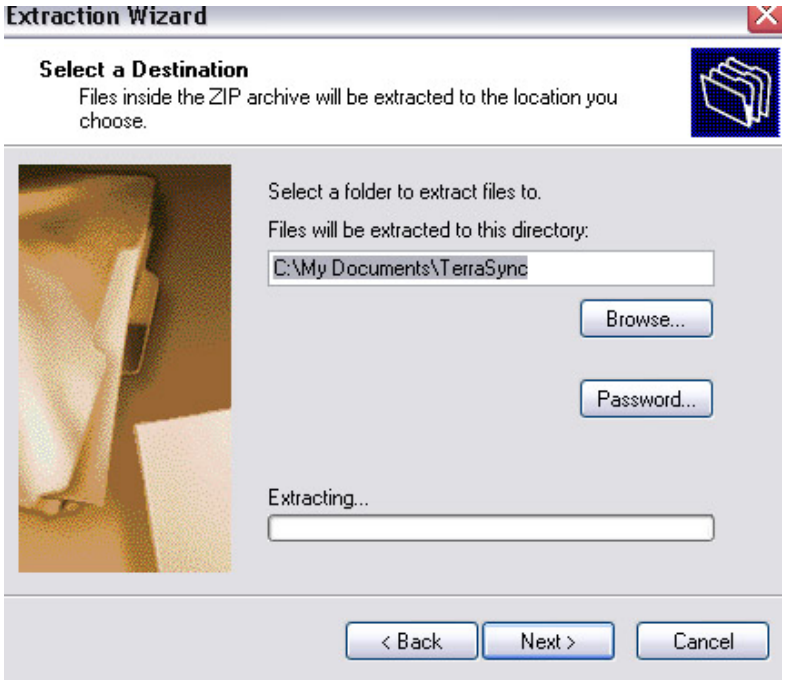


Figure 5. Extract (or copy) Original Target Files into the Terrasync Directory

Option 2: Transfer files back and forth from Terrasync.

If you need to be able to see the previously occupied positions from last week while positioning this week, you need to use Pathfinder to reset the file. This process will essentially combine the targets and actuals from last week into one file. However, this method has its drawbacks; once converted, the actuals from last week will not be able to be corrected, so a backup procedure similar to the one in the previous option should be carried out to maintain data integrity.

The steps for file transfer are as follows:

1. For good data management, back up the data files from the previous week using the procedure laid out in steps 1 through 3 in Option 1 above.
2. Close Terrasync and open up Pathfinder Office.
3. Go to the Utilities>Data Transfer menu or just click the icon on the left (Figure 6).
4. Ensure that the device listed is Terrasync. If not, follow the initial setup instructions at the beginning of this document. Most of the computers used for GPS logging are already setup for this.
5. There are two tabs, Receive and Send. Make sure that Receive is selected and then go to Add>Data File. Select the file(s) that you are using and select Open. The file should now be in the Files to Receive box. Click Transfer All and wait for the transfer to take place. If you have made the recommended backups, it is fine to replace any files.
6. Now select the Send tab (Figure 7), and go to Add>Data File. Select the file you just transferred (it will have the same name as the Terrasync file) and click Open. Now click Transfer All to move the file back to Terrasync.

   By transferring the file back and forth from Terrasync to Pathfinder, you have "reset the clock" and can now update the file for an additional 7 days. This file will have your targets and actual positions from the last week, so it is important to be aware of the features you are selecting for navigation.

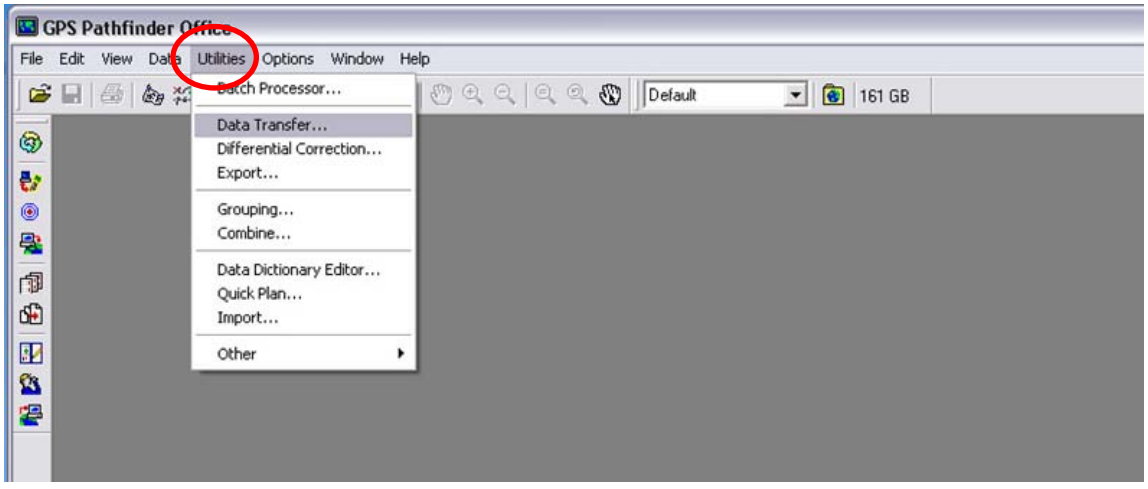


Figure 6. Data Transfer Menu

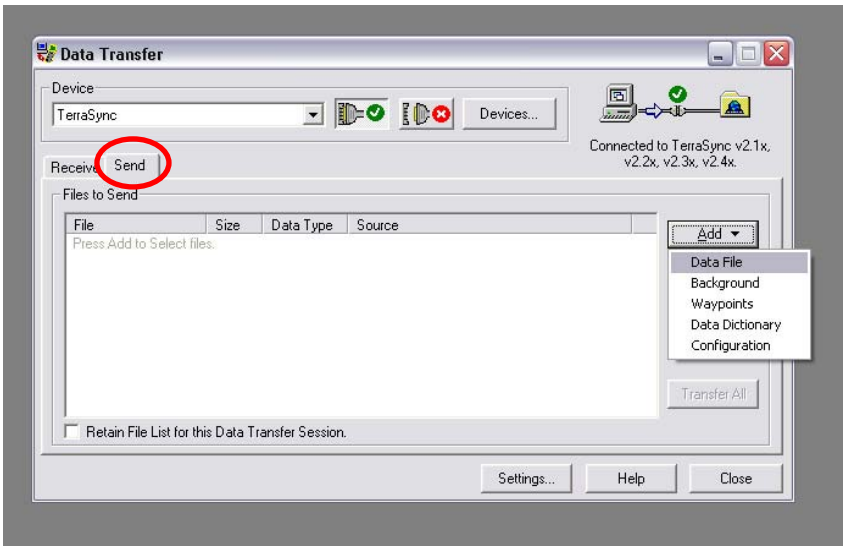


Figure 7. Sending Data File

STANDARD OPERATING PROCEDURE (SOP) SD-01

DECONTAMINATION OF SEDIMENT SAMPLING EQUIPMENT

SCOPE AND APPLICATION

This SOP describes procedures for decontaminating sampling and processing equipment contaminated by either inorganic or organic materials. To prevent potential cross contamination of samples, all reusable sediment sampling and processing equipment is decontaminated before each use. At the sample collection site, a decontamination area is established in a clean location that is upwind of actual sampling locations, if possible. All sediment sampling and processing equipment is cleaned in this location. Decontaminated equipment is stored away from areas that may cause recontamination. When handling decontamination chemicals, field personnel must follow all relevant procedures and wear protective clothing as stipulated in the site-specific health and safety plan (HSP).

Sampling equipment (e.g., van Veen, Ekman, Ponar, core tubes) may be used to collect samples that will 1) undergo a full-suite analysis (organics, metals, and conventional parameters) or 2) be analyzed for metals and conventional parameters only. Decontamination of sampling equipment used for both analyte groups should follow the order of a detergent wash, site water rinse, organic solvent rinses, and final site water rinse. Sample processing equipment (e.g., bowls, spoons) has a final rinse with distilled/deionized water rinse instead of site water. If the surface of stainless steel equipment appears to be rusting (possibly due to prolonged contact with organic-rich sediment), it should undergo an acid rinse and a site-water rinse at the end of each sampling day to minimize corrosion.

EQUIPMENT AND REAGENTS REQUIRED

Equipment required for decontamination includes the following:

- Polyethylene or polypropylene tub (to collect solvent rinsate)
- Plastic bucket(s) (e.g., 5-gal bucket)
- Tap water or site water
- Carboy, distilled/deionized water (analyte-free; received from testing laboratory or other reliable source)
- Properly labeled squirt bottles

- Funnels
- Alconox®, Liquinox®, or equivalent industrial detergent
- Pesticide-grade acetone and hexane (consult the project-specific field sampling plan [FSP], as the solvents may vary by EPA region or state)
- 10 percent (v/v) nitric acid (reagent grade) for inorganic contaminants
- Baking soda
- Long-handled, hard-bristle brushes
- Extension arm for cleaning core liners
- Plastic sheeting, garbage bags, and aluminum foil
- Core liner caps or plastic wrap and rubber bands
- Personal protective equipment as specified in the health and safety plan.

PROCEDURES

Decontamination Procedures for Full Suite Analysis (Organic, Metal, or Conventional Parameters)

Two organic solvents are used in this procedure. The first is miscible with water (e.g., ethanol) and is intended to scavenge water from the surface of the sampling equipment and allow the equipment to dry quickly. This allows the second solvent to fully contact the surface of the sampler. Make sure that the solvent ordered is anhydrous or has a very low water content (i.e., < 1 percent). If ethanol is used, make sure that the denaturing agent in the alcohol is not an analyte in the samples. The second organic solvent is hydrophobic (e.g., hexane) and is intended to dissolve any organic chemicals that are on the surface of the equipment.

The exact solvents used for a given project may vary by EPA region or state (see project-specific FSP). Integral uses ethanol and hexane as preferred solvents for equipment decontamination. If specified in the project-specific FSP, isopropanol or acetone can be substituted for ethanol, and methanol can be substituted for hexane in the decontamination sequence. The choice of solvents is also dependent on the kind of material from which the equipment is made (e.g., acetone cannot be used on polycarbonate), and the ambient temperature (e.g., hexane is too volatile in hot climates). In addition, although methanol is sometimes slightly more effective than other solvents, its use is discouraged due to potential toxicity to sampling personnel.

The specific procedures for decontaminating sediment sampling equipment and sediment compositing equipment are as follows:

1. Rinse the equipment thoroughly with tap or site water to remove visible sediment. Perform this step onsite for all equipment, including core liners that will not be used again until the next day of sampling. After removing visible solids, set aside sampling equipment that does not need to be used again that day; this equipment should be thoroughly cleaned in the field laboratory at the end of the day.
2. Pour a small amount of concentrated laboratory detergent into a bucket (i.e., about 1–2 tablespoons per 5-gal bucket) and fill it halfway with tap or site water. If the detergent is in crystal form, make sure all crystals are completely dissolved prior to use.
3. Scrub the equipment in the detergent solution using a long-handled brush with rigid bristles. For the polycarbonate core liners, use a round brush attached to an extension arm to reach the entire inside of the liners, scrubbing with a back-and-forth motion. Be sure to clean the outside of core liners, bowls, and other pieces that may be covered with sediment.
4. Double rinse the equipment with tap or site water and set right-side-up on a stable surface to drain. The more completely the equipment drains, the less solvent will be needed in the next step. Do not allow any surface that will come in contact with the sample to touch any contaminated surface.
5. If the surface of stainless steel equipment appears to be rusting (this will occur during prolonged use in anoxic marine sediments), passivate[1] the surface as follows (if no rust is present, skip to next step). Rinse with a 10 percent (v/v) nitric acid solution using a squirt bottle, or wipe all surfaces using a saturated paper towel. Areas showing rust may require some rubbing with the paper towel. If using a squirt bottle, let the excess acid drain into the waste container (which may need to be equipped with a funnel). Double-rinse equipment with tap or site water and set right-side-up on a stable surface to drain thoroughly.
6. Carefully rinse the equipment with ethanol from a squirt bottle, and let the excess solvent drain into a waste container (which may need to be equipped with a funnel). Hold core liners over the waste container and turn them slowly so the stream of solvent contacts the entire surface. Turn the sample apparatus (e.g., grab sampler) on its side and open it to wash it most effectively. Set the equipment in a clean location and allow it to air dry. Use only enough solvent to scavenge all of the water and flow off the surface of the equipment (i.e., establish sheet flow) into the waste container. Allow equipment to drain as much as possible. Ideally, the equipment will be dry. The more thoroughly it drains, the less solvent will be needed in the next step.

[1] Passivation is the process of making a material less reactive relative to another material. For example, before sediment is placed in a stainless-steel container, the container can be passivated by rinsing it with a dilute solution of nitric acid and deionized water.

7. Carefully rinse the drained or air-dried equipment with hexane from a squirt bottle, and let the excess solvent drain into the waste container (which may need to be equipped with a funnel). If necessary, widen the opening of the squirt bottle to allow enough solvent to run through the core liners without evaporating. (Hexane acts as the primary solvent of organic chemicals. Ethanol is soluble in hexane but water is not. If water beading occurs, it means that the equipment was not thoroughly rinsed with acetone or that the acetone that was purchased was not free of water.) When the equipment has been rinsed with hexane, set it in a clean location and allow the hexane to evaporate before using the equipment for sampling. Use only enough solvent to scavenge all of the acetone and flow off the surface of the equipment (i.e., establish sheet flow) into the waste container.
8. Do a final rinse with site water for the sampling equipment (i.e., van Veen, Ekman, Ponar, core tubes) and with distilled/deionized water for processing equipment (i.e., stainless-steel bowls and spoons). Equipment does not need to be dried before use.
9. If the decontaminated sampling equipment is not to be used immediately, wrap small stainless-steel items in aluminum foil (dull side facing the cleaned area). Seal the polycarbonate core liners at both ends with either core caps or cellophane plastic and rubber bands. Close the jaws of the Ekman and Ponar grab samplers and wrap in aluminum foil.

   If the sample collection or processing equipment is cleaned at the field laboratory and transported to the site, then the decontaminated equipment will be wrapped in aluminum foil (dull side facing the cleaned area) and stored and transported in a clean plastic bag (e.g., a trash bag) until ready for use, unless the project-specific FSP lists special handling procedures.
10. Rinse or wipe with a wetted paper towel all stainless-steel equipment at the end of each sampling day with 10 percent (v/v) normal nitric acid solution. Follow with a freshwater rinse (site water is okay as long as it is not brackish or salt water).
11. After decontaminating all of the sampling equipment, place the disposable gloves and used foil in garbage bags for disposal in a solid waste landfill. When not in use, keep the waste solvent container closed and store in a secure area. The waste should be transferred to empty solvent bottles and disposed of at a licensed facility per the procedures listed in the project-specific FSP. When not in use, keep the waste acid container closed and store in a secure area. The acid waste should be neutralized with baking soda and disposed of per the procedures listed in the project-specific FSP.

Decontamination Procedures for Metals and Conventional Parameters Only

The specific procedures for decontaminating sediment sampling equipment and sediment processing equipment are as follows:

1. Rinse the equipment thoroughly with tap or site water to remove the visible sediment. Perform this step onsite for all equipment, including core liners that will not be used again until the next day of sampling. Set aside pieces that do not need to be used again that day; these pieces should be and thoroughly cleaned in the field laboratory at the end of the day.
2. Pour a small amount of concentrated laboratory detergent into a bucket (i.e., about 1–2 tablespoons per 5-gal bucket) and fill it halfway with tap or site water. If the detergent is in crystal form, make sure all crystals are completely dissolved prior to use.
3. Scrub the equipment in the detergent solution using a long-handled brush with rigid bristles. For the polycarbonate core liners, use a round brush attached to an extension arm to reach the entire inside of the liners, scrubbing with a back-and-forth motion. Be sure to clean the outside of core liners, bowls, and other pieces that may be covered with sediment.
4. Double-rinse the equipment with tap or site water and set right-side-up on a stable surface to drain. Do not allow any surface that will come in contact with the sample to touch any contaminated surface.
5. If the surface of stainless steel equipment appears to be rusting (this will occur during prolonged use in anoxic marine sediments), passivate[2] the surface as follows (if no rust is present, skip to next step). Rinse with a 10 percent (v/v) nitric acid solution using a squirt bottle, or wipe all surfaces using a saturated paper towel. Areas showing rust may require some rubbing with the paper towel. If using a squirt bottle, let the excess acid drain into the waste container (which may need to be equipped with a funnel). Double-rinse sampling equipment with tap or site water and set right-side-up on a stable surface to drain. Double-rinse processing equipment with distilled/deionized water and allow to drain.
6. If the decontaminated sampling equipment is not to be used immediately, wrap small stainless-steel items in aluminum foil (dull side facing the cleaned area). Seal the polycarbonate core liners at both ends with either core caps or cellophane plastic and rubber bands. Close the jaws of the Ekman and Ponar grab samplers and wrap in aluminum foil.

   If the sample collecting or processing equipment is cleaned at the field laboratory and transported to the site, then the decontaminated equipment will be wrapped in aluminum foil (dull side facing the cleaned area) and stored and transported in a clean plastic bag until ready for use, unless the project-specific FSP lists special handling procedures.

[2] Passivation is the process of making a material less reactive relative to another material. For example, before sediment is placed in a stainless-steel container, the container can be passivated by rinsing it with a dilute solution of nitric acid and deionized water.

7. After decontaminating all of the sampling equipment, place the disposable gloves and used foil in garbage bags for disposal in a solid waste landfill. When not in use, keep the waste acid container closed and store in a secure area. The acid waste should be neutralized with baking soda and disposed of per the procedures listed in the project-specific FSP.

STANDARD OPERATING PROCEDURE (SOP) SD-02

PREPARATION OF FIELD QUALITY CONTROL SAMPLES FOR SEDIMENTS

SCOPE AND APPLICATION

This SOP describes the purpose, preparation, and collection frequency of field duplicate samples, field replicate samples, matrix spike/matrix spike duplicates, equipment rinsate blanks, bottle blanks, trip blanks, temperature blanks, environmental blanks, and reference materials (i.e., a standard reference material, a certified reference material, or other reference material; for the purposes herein, all types of reference materials are referred to as standard reference material, or SRM) for sediment sampling efforts. Not all of the field quality control samples discussed in this SOP may be required for a given project. The specific field quality control samples will be identified in the project-specific field sampling plan (FSP) and quality assurance project plan (QAPP). For most projects, Integral's recommended field quality control samples are an equipment rinsate blank, a field duplicate, and trip blanks if samples are to be analyzed for volatile organic compounds (VOCs). Definitions of all potential quality control samples are described below.

As part of the quality assurance/quality control (QA/QC) program, all field quality control samples will be sent to the laboratories "blind." To accomplish this, field quality control samples will be prepared and labeled in the same manner as regular samples, with each quality control sample being assigned a unique sample number that is consistent with the numbering for regular samples. All of the containers with preservatives that are required to complete the field quality control sample for the applicable analyte list shall be labeled with the same sample number. The sample ID for field quality control samples should allow data management and data validation staff to identify them as such and should be recorded only in the field logbook. Under no circumstances should the laboratory be allowed to use reference materials, rinsate blanks, or trip blanks for laboratory quality control analysis (i.e., duplicates, matrix spike, and matrix spike duplicates). To prevent such an occurrence, regular samples should be selected and marked on the chain-of-custody/sampling analysis request (COC/SAR) form or the laboratory should be instructed to contact the project QA/QC coordinator to select appropriate samples for each sample group.

Field quality control samples will be prepared at least once per sampling event, and certain types will be prepared more often at predetermined frequencies. If the number of samples taken does not equal an integer multiple of the intervals specified in this SOP, the number of

field quality control samples is specified by the next higher multiple. For example, if a frequency of 1 quality control sample per 20 is indicated and 28 samples are collected, 2 quality control samples will be prepared. Field quality control samples for sediment sampling activities should be prepared consistent with the requirements discussed below and at the frequency indicated unless different frequency requirements are listed in the FSP and QAPP.

The following table lists the quality control sample types and suggested frequencies for sediment sampling programs. Because sediment quality control sampling may require assessment of site cross-contamination, additional blanks may be required. A detailed explanation of each quality control sample type with the required preparation follows.

Table 1. Field Quality Control Sample Requirements

Quality Control Sample Name	Abbreviation	Preparation		Frequency[a]
		Location	Method	
Duplicate	DUP	Sampling site	Additional natural sample	One per 20 samples. May not be applicable if REP is being collected.
Replicate	REP	Sampling site	Additional natural sample	One replicate per 20 samples. May not be applicable if DUP is being collected.
Matrix spike/matrix spike duplicate	MS/MSD	Sampling site	Additional sample bottles filled for laboratory quality control requirements	One per 20 samples.
Equipment rinsate blank	ER	Sampling site	Deionized water collected after pouring through and over decontaminated equipment	Minimum of one per sampling event per type of sampling equipment used and then 1 per 20 thereafter.
Filter wipe	FW	Sampling site	Whatman filter papers (organic analysis) and Ghost Wipes (metals/mercury analysis) will be wiped over decontaminated equipment	Minimum of one per sampling event per type of sampling equipment used and then 1 per 20 thereafter.
Filter paper blank	FB	Sampling site	Clean, unused Whatman filter papers (organic analysis) and Ghost Wipes (metals/mercury analysis) will be sent to the analytical laboratory	Minimum of one for each lot number of filter papers used.
Bottle blank	BB	Field	Unopened bottle	One per sample episode or one per bottle type.
Trip blank	TB	Laboratory	Deionized water with preservative	One pair per each VOC sample cooler shipment.

Quality Control Sample Name	Abbreviation	Preparation		Frequency[a]
		Location	Method	
Temperature blank	TMB	Laboratory	Deionized water	One per sample cooler.
Environmental blank	EB	Field	Bottle filled at sample site with deionized water	One per 20 samples.
Standard reference material	SRM	Field laboratory or sampling site	SRM ampules or other containers for each analyte group	One set per 50 samples or one per episode.

[a] Frequencies provided here are general recommendations; specific frequencies should be provided in the project-specific FSP or QAPP.

FIELD DUPLICATE SAMPLES

Field duplicate (or split) samples are collected to assess the homogeneity of the samples collected in the field and the precision of the sampling process. Field duplicates will be prepared by collecting two aliquots for the sample and submitting them for analysis as separate samples. Field duplicates will be collected at a minimum frequency of 1 per 20 samples or once per sampling event, whichever is more frequent. The actual number of field duplicate samples collected during a sampling event will be determined on a case-by-case basis by the project QA/QC coordinator (consult the project-specific FSP and QAPP, as the requirements on frequency of field duplicate collection may vary by EPA region or state).

FIELD REPLICATE SAMPLES

Field replicate samples are co-located samples collected in an identical manner over a minimum period of time to provide a measure of the field and laboratory variance, including variance resulting from sample heterogeneity. Field replicates will be prepared by collecting two completely separate samples from the same station and submitting them for analysis as separate samples. Field replicates will be collected at a minimum frequency of 1 per 20 samples or once per sampling event, whichever is more frequent. If field duplicate samples are collected, then it is unlikely that field replicate samples will also be collected during a sampling event. The actual number of field replicate samples collected during a sampling event will be determined on a case-by-case basis by the project QA/QC coordinator (consult the project-specific FSP and QAPP, as the requirements on frequency of field duplicate collection may vary by EPA region or state).

MATRIX SPIKE/MATRIX SPIKE DUPLICATES

The matrix spike/matrix spike duplicate (MS/MSD) analyses provide information about the effect of the sample matrix on the design and measurement methodology used by the laboratory. To account for the additional volume needed by the laboratory to perform the analyses, extra sample volumes may be required to be collected from designated sediment stations. MS/MSDs may be collected at a minimum frequency of 1 per 20 samples or once per sampling event, whichever is more frequent. The actual number of extra bottles collected during a sampling event will be determined on a case-by-case basis by the project QA/QC coordinator (consult the project-specific FSP and QAPP, as the requirements may vary by analyte group).

EQUIPMENT RINSATE BLANKS

Equipment rinsate blanks will be used to help identify possible contamination from the sampling environment and/or from decontaminated sampling equipment. Equipment rinsate blanks will be prepared by pouring laboratory distilled/deionized water through, over, and into the decontaminated sample collection equipment, and then transferring the water to the appropriate sample containers and adding any necessary preservatives. Equipment rinsate blanks will be prepared for all inorganic, organic, and conventional analytes at least once per sampling event per the type of sampling equipment used. The actual number of equipment rinsate blanks prepared during an event will be determined on a case-by-case basis by the project QA/QC coordinator (consult the project-specific FSP and QAPP, as the requirements on frequency of equipment rinsate blank collection may vary by EPA region or state).

FILTER WIPES

Filter wipe samples will be used to help identify possible contamination from the sampling environment or from the decontaminated sediment sampling equipment (e.g., sediment grab sampler, stainless-steel bowls and spoons, shovel, trowel).

Filter wipe samples will be prepared by grasping a piece of clean, ashless filter wipe/paper with decontaminated tongs and/or tweezers and wiping down all surfaces of dry, decontaminated equipment that comes into contact with the sediment sample (e.g., stainless-steel spoon, inside of sediment grab sampler). Whatman filter papers will be used for organic analysis and Ghost Wipes will be used for metals/mercury analysis. The filter wipes/papers will be from the same lot used to prepare the filter paper blanks (see below), and the filter lot number will be clearly noted in the field logbook. One filter wipe/paper will be used for each equipment type, solid matrix type, and analysis type. For example, if two pieces of equipment were used for sediment sampling (trowel and stainless-steel spoon) and both metals and

organic compounds were being analyzed, then a total of four filter wipes/papers would be sent to the analytical laboratory.

Tongs and/or tweezers will be used to handle the filter wipe/paper, and all sediment sample-exposed surfaces will be thoroughly wiped down using one piece of filter wipe/paper (per equipment type and for each analysis). The filter wipe sample will then be placed into a labeled certified pre-cleaned sample jar provided by the analytical laboratory. The filter wipe/paper box will be stored in a clean glass container and must NOT be stored in a plastic bag. In moist environments, the filters should be wrapped thoroughly in aluminum foil to protect them from moisture.

Filter wipe samples will be prepared for all inorganic and organic analytes at least once per sampling event per the type of sampling equipment used. The actual number of filter wipe samples prepared during an event will be determined on a case-by-case basis by the project QA/QC coordinator (consult the project-specific FSP and QAPP, as the requirements on frequency of filter wipe sample collection may vary by EPA region or state).

FILTER PAPER BLANKS

Whenever a filter wipe sample is prepared in the field, a filter paper blank will also be prepared in the field to evaluate potential background concentrations present in the filter paper used for the equipment filter wipe sample.

Filter paper blanks will be prepared by using tongs and/or tweezers to remove the clean ashless filter paper from its box. Whatman filter papers will be used for organic analysis and Ghost Wipes will be used for metals/mercury analysis. The filter papers will be from the same lot used to prepare the filter wipe samples (see above), and the filter lot number will be clearly noted in the field logbook. One filter wipe/paper will be sent to the analytical laboratory for each type of analysis to be performed (i.e., inorganic or organic analytes). The filter paper blank will be placed into a labeled certified pre-cleaned sample jar provided by the analytical laboratory.

Filter paper blanks will be collected at a minimum frequency of one for each filter lot number. The actual number of filter paper blanks prepared during an event will be determined on a case-by-case basis by the project QA/QC coordinator (consult the project-specific FSP and QAPP, as the requirements on frequency of filter paper blank collection may vary by EPA region or state).

BOTTLE BLANKS

The bottle blank is an unopened sample bottle. Bottle blanks are submitted along with sediment samples to ensure that contaminants are not originating from the bottles themselves because of improper preparation, handling, or cleaning techniques. If required, one bottle

blank per lot of prepared bottles will be submitted for analysis. If more than one type of bottle will be used in the sampling (e.g., high-density polyethylene or glass), then a bottle blank should be submitted for each type of bottle and preservative. The actual number of bottle blanks analyzed during a project will be determined on a case-by-case basis by the project QA/QC coordinator (consult the project-specific FSP and QAPP as the requirements on frequency of bottle blank analysis may vary by EPA region or state).

To prepare a bottle blank in the field, set aside one unopened sample bottle from each bottle lot sent from the testing laboratory. Label the bottle as "Bottle Blank" on the sample label (and in the "Remarks" column on the COC/SAR form), and send the empty bottle to the laboratory with the field samples.

TRIP BLANKS

Trip blanks will be used to help identify whether contaminants may have been introduced during the shipment of the sediment samples from the field to the laboratory for VOC analyses only. Trip blanks are prepared at the testing laboratory by pouring distilled/ deionized water into two 40-mL VOC vials and tightly closing the lids. Each vial will be inverted and tapped lightly to ensure no air bubbles exist.

The trip blanks will be transported unopened to and from the field in the cooler with the VOC samples. A trip blank is labeled and placed inside the cooler that contains newly collected VOC samples and it remains in the cooler at all times. A trip blank must accompany samples at all times in the field. One trip blank (consisting of a pair of VOC vials) will be sent with each cooler of samples shipped to the testing laboratory for VOC analysis.

TEMPERATURE BLANKS

Temperature blanks will be used by the laboratory to verify the temperature of the samples upon receipt at the testing laboratory. Temperature blanks will be prepared at the testing laboratory by pouring distilled/deionized water into a vial and tightly closing the lid. The blanks will be transported unopened to and from the field in the cooler with the sample containers. A temperature blank shall be included with each sample cooler shipped to the testing laboratory.

ENVIRONMENTAL BLANKS

The environmental (field) blank is prepared in the field to evaluate potential background concentrations present in the air and in the distilled/deionized water used for the final decontamination rinse. If unpreserved bottles are to be used, then the appropriate preservative (i.e., for metals samples use a 10 percent nitric acid solution to bring sample pH

to 2 or less) must be added, as may be required. Environmental blanks should be collected at a minimum frequency of 1 in 20 samples. The actual number of environmental blanks analyzed during a project will be determined on a case-by-case basis by the project QA/QC coordinator (consult the project-specific FSP and QAPP, as the requirements on frequency of environmental blank analysis may vary by EPA region or state).

To prepare an environmental blank in the field, open the laboratory-prepared sample bottle while at a sample collection site, fill the sample bottle with distilled/deionized water, and then seal it. Assign the environmental blank a unique sample number, label the bottle, and then send the bottle to the laboratory with the field samples.

REFERENCE MATERIALS

SRMs are samples containing known analytes at known concentrations that have been prepared by and obtained from EPA-approved sources. The SRMs have undergone multi-laboratory analyses using a standard method that provides certified concentrations. When available for a specific analyte, SRMs provide a measure of analytical performance and/or analytical method bias (i.e., accuracy) of the laboratory. Several SRMs may be required to cover all analytical parameters. For all analytes where available, one SRM will be analyzed at a frequency of one per 50 samples. The actual number of SRMs analyzed during a project will be determined on a case-by-case basis by the project QA/QC coordinator (consult the project-specific FSP and QAPP, as the requirements on frequency of SRM analysis may vary by EPA region or state).

STANDARD OPERATING PROCEDURE (SOP) SD-04

SURFACE SEDIMENT SAMPLING

SCOPE AND APPLICATION

This SOP defines and standardizes the methods for collecting surface sediment samples from freshwater or marine environments. Surface sediments are defined as those from 0 to at most 10 cm below the sediment-water interface. The actual definition of surface sediments is typically program-specific and depends on the purpose of the study and the regulatory criteria (if any) to which the data will be compared.

This SOP utilizes and augments the procedures outlined in USEPA (1996) and ASTM (2003) guidelines. A goal of this SOP is to ensure that the highest quality, most representative data are collected, and that these data are comparable to data collected by different programs that follow the USEPA (1996) guidelines.

SUMMARY OF METHOD

Sediment samples for chemical and toxicity analysis are collected using a surface sediment sampling device (e.g., grab sampler) or hand implements (i.e., spoons, scoops, shovels, or trowels). If a sample meets acceptability guidelines, overlying water is carefully siphoned off the surface in a grab sampler, and the sediment is described in the field logbook. Depending upon the type of analysis to be performed, sediment samples for chemical analysis may be collected directly from an undisturbed surface (e.g., volatile organic compounds and sulfides), or may be homogenized using decontaminated, stainless-steel containers and utensils prior to being placed in sample jars. Sediment from several sampler casts or exposed sediment locations may also be composited and homogenized prior to being placed in sample jars.

SUPPLIES AND EQUIPMENT

A generalized supply and equipment list is provided below. Additional equipment may be required depending on project requirements.

- Sampling device
  - Grab sampler or box corer (see examples below in procedures for "Sediment Sample Collection")

  - Stainless-steel spoon, scoop, shovel, or trowel
- Field equipment
  - Siphoning hose
  - Stainless-steel bowls or containers
  - Stainless-steel spoons, spatulas, and/or mixer
  - Stainless-steel ruler
  - Project-specific decontamination supplies (e.g., Alconox™ detergent, 0.1 N nitric acid, methanol, hexane, distilled/deionized water)
  - Personal protective equipment for field team (e.g., rain gear, safety goggles, hard hats, nitrile gloves)
  - First aid kit
  - Cell phone
  - Camera
  - Sample containers
  - Ziploc® bags
  - Bubble wrap
  - Sample jar labels
  - Clear tape
  - Permanent markers
  - Indelible black-ink pens
  - Pencils
  - Coolers
  - Ice
- Documentation
  - Waterproof field logbook
  - Field sampling plan
  - Health and safety plan
  - Correction forms
  - Request for change forms
  - Waterproof sample description forms.

PROCEDURES

Sediment Sample Collection with a Grab Sampler

Use a sampler that obtains a quantifiable volume of sediment with minimal disturbance of the surrounding sediments to collect sediment for chemical and biological analyses. The sampler should be composed of a material such as stainless steel or aluminum, or have a noncontaminating coating such as Teflon™. Samplers capable of providing high-quality sediment samples include grab-type samplers (e.g., van Veen, Ekman, Smith-McIntyre, Young grab, Power Grab and modified-ponar grab) and box cores (Soutar, mini-Soutar, Gray-O'Hara, spade core). Some programs require a sampler that collects from a specific area (e.g., 0.1 m^2). Most sampling devices are typically a standard size; however, some non-standard sizes are available to meet the requirements of specific programs. Grab samplers, especially van Veen grab and Ekman grab, are the most commonly used samplers to collect surface sediment. Power Grab samplers are often used for programs requiring collection of sediment deeper than 10 cm (4 in.) or in areas with debris.

Depending on grab weight and water depth, use a hydraulic winch system to deploy the heavier samplers at a rate not exceeding 1 m/second to minimize the bow wake associated with sampler descent. Once the sampler hits the bottom, close the jaws slowly and bring the sampler to the deck of the vessel at a rate not exceeding 1 m/second to minimize any washing and disturbance of the sediment within the sampler. At the moment the sampler hits the bottom, record the time, water depth, and location of sample acquisition in the field logbook.

Retrieve and secure the sampler, and carefully siphon off any overlying water. Inspect the sample to determine acceptability using the criteria detailed in USEPA (1996), except when noted in the project-specific field sampling plan. These criteria include but are not limited to the following:

- There is minimal or no excessive water leakage from the jaws of the sampler
- There is no excessive turbidity in the water overlying the sample
- The sampler is not over-penetrated
- The sediment surface appears to be intact with minimal disturbance
- There is no anthropogenic (i.e., man-made) debris in the sampler
- The program-specified penetration depths are attained.

If the sample meets acceptability criteria, record the sample collection location using a global positioning system (GPS) and enter observations onto a sample collection form or the field logbook. Depending on programmatic goals, remove the sampling interval specified in the field sampling plan. Use a decontaminated stainless-steel ruler to measure the sample collection depth (0 to 10 cm) within the sampler. To prevent possible cross-contamination, do not use sediments touching the margins of the sampler.

Take a photograph of the sediment in the grab sampler and in the stainless-steel bowl in the field. Verify that the station number or sample ID, time, and date are shown in the photograph.

Typically, sediment from a minimum of three separate casts of the sampler is composited at each station (see project-specific field sampling plan). Once the sample has been characterized, subsample the sediment for chemical and biological analyses using a decontaminated stainless-steel spoon.

Sediment Sample Collection with Hand Implements

Obtain a quantifiable volume of sediment with minimal disturbance of the surrounding sediments to collect sediment for chemical and biological analyses. Hand implements (e.g., spoons, scoops, shovels, or trowels) must be composed of stainless steel.

Use GPS to locate the sampling site and approach the location carefully to avoid disturbing the area of sediment to be sampled. Prior to sample collection, describe and characterize the undisturbed surface sediment in the field logbook. If necessary, expose the sediment surface by clearing an approximately 1-ft^2 area at the sampling site of any rocks greater than approximately 5 in. Remove any anthropogenic (i.e., man-made) debris and organic material on the sediment surface. Note any material removed from the sampling site in the field logbook.

Using a decontaminated, stainless-steel hand implement (i.e., spoon, scoop, shovel, or trowel), excavate the sediment to 10 cm. Place the sediment in a decontaminated stainless-steel bowl and use a decontaminated stainless-steel ruler to confirm that the correct sampling interval has been collected. If the full sample collection interval (i.e., 10 cm) has not been reached, collect additional sediment, place it in the stainless-steel bowl, and reconfirm the sampling interval. Continue this process until the full sample collection interval (0 to 10 cm) has been reached.

Take a photograph of the excavated hole from where the sediment sample was removed. Verify that the station number or sample ID, time, and date are shown in the photograph.

Sample Processing

Complete all sample collection forms, labels, custody seals, and chain-of-custody forms, and record sample information in the field logbook.

Collect samples for volatile compounds (either organics or sulfides) using a decontaminated stainless-steel spoon while sediment is still in the grab sampler or, if the sample is collected using a hand implement, in the stainless-steel bowl. Sediments for volatile analysis are not homogenized. Tightly pack the volatile organics sample jar with sediment (to eliminate obvious air pockets) and fill it so that no headspace remains in the jar. Alternatively, if there is adequate water in the sediment, fill the container to overflowing so that a convex meniscus forms at the top, and then carefully place the cap on the jar. Once sealed, the jar should contain no air bubbles.

Place the remaining sediment in the grab sampler in a precleaned, stainless-steel bowl; sediment collected using hand implements are already in a stainless-steel bowl. Once a sufficient amount of sediment has been collected, mix the sediment using a decontaminated stainless-steel spoon until it is of uniform color and texture throughout.

If required for analysis, collect samples for grain-size tests before any large rocks are removed from the homogenized sediment. Identify any rocks that are greater than 0.5 in. in diameter. Determine their percentage contribution to the homogenized sediment volume, note it on the sediment field collection form or in the field logbook, and then discard the rocks.

Dispense the sediment into precleaned sample jars for the various chemical or biological analyses. For toxicity testing, fill sample jars to the top with sediment to minimize available headspace. This procedure will minimize any oxidation reactions within the sediment. For chemical analysis, sample containers may be frozen for storage. Leave enough headspace to allow for sediment expansion.

After dispensing the sediment, place the containers into coolers with ice and either ship them directly to the analytical laboratories or transport them to a storage facility.

REFERENCES

ASTM. 2003. *Standard Practice for Collecting Benthic Macroinvertebrates with Ekman Grab Sampler*. ASTM Standards on Disc, Volume 11.05.

USEPA. 1996. Puget Sound Estuary Program: Recommended protocols for measuring selected environmental variables in Puget Sound. Prepared for U.S. Environmental Protection Agency, Region 10, and Puget Sound Estuary Program, Seattle, WA. Tetra Tech and HRA, Inc., Bellevue, WA.

STANDARD OPERATING PROCEDURE (SOP) SD-06

HOLLOW-STEM AUGER DRILLING/SEDIMENT SAMPLING

SCOPE AND APPLICATION

Soil/sediment cores are collected to evaluate sediment at depths that greatly exceed those achieved by grab or other surface samplers. The purpose of this standard operating procedure (SOP) is to define and standardize procedures for core collection using split-spoon and Shelby tube samplers advanced through hollow-stem auger borings, following American Society for Testing and Materials (ASTM) Method D1586 and Method D1587, respectively. The use of Shelby tube samplers or split-spoon samplers is specified in the Slip 4 Pre-Design Sampling and Analysis Plan Addendum (Integral 2006). Shelby tubes will be used to recover relatively undisturbed soil samples suitable for laboratory tests of engineering properties such as strength, compressibility, permeability, and density.

REQUIRED EQUIPMENT

- Sampling and Analysis Plan (SAP).
- Health and Safety Plan (HSP).
- Site logbook and boring log.
- Indelible black-ink pens and markers.
- Camera.
- Hollow-stem auger drill rig.
- Driller and helper.
- Split-spoon samplers (typically 2-in. diameter; a larger 3-in. diameter, 2-ft-length split-spoon may be used to obtain more material from each depth interval).
- Shelby tube samplers conforming to thin-walled tube specifications outlined in ASTM D1587 with a 2- to 5-in. O.D and 5 to 10 times the diameter in length. Wax and end caps will also be provided for proper field sealing.
- Photoionization detector (PID).
- Plastic sheeting.
- 55-gallon drums (if required).
- Insulated cooler(s), chain-of-custody seals, Ziploc® bags.
- Sample labels and appropriate documentation.

- Assorted geology supplies (e.g., hand lens, grain-size card, scales, etc.).
- Decontamination equipment (SOP-10).

Typical Procedures

1. Ensure underground utilities in vicinity of each boring location have been marked prior to mobilizing drill rig to site.
2. Conduct daily site activity/health and safety briefing.
3. Calibrate field instrumentation, if applicable.
4. Record necessary data in field logbook.
5. Obtain photograph(s) of site before drilling.
6. Place plastic sheeting and/or drums at drilling location to collect cuttings (if necessary).
7. Move equipment and supplies to drilling location.
8. Set up decontamination and sampling stations.

Split-Spoon Sampling

1. Obtain surface soil samples, if required.
2. Drill to first sampling depth, as described in the SAP.
3. Place decontaminated split-spoon sampler on center rods.
4. Drive split-spoon sampler, as described in ASTM Method D-1586. Drive sampler to 18 inch or to refusal (no progress for 50 blows). Record blow counts on boring log form. Retrieve sampler.
5. Screen sampler with PID (if required).
6. Describe soil in accordance with ASTM D2488 on the boring log form.
7. Composite soil sample, as necessary. If volatile organic compound (VOC) samples are to be collected, collect VOC sample prior to describing soil.
8. Continue drilling at next sample location. Collect samples as outlined above.
9. Label and manage sample containers in accordance with the site-specific SAP section for shipping and handling of samples.
10. Decontaminate sampling equipment in accordance with the site-specific SAP.
11. Document activities in site logbook.
12. Backfill or grout borehole, as required.

13. Move to next location.

Shelby Tube Sampling

1. Obtain surface soil samples, if required.
2. Drill to first sampling depth, as described in the SAP.
3. Place decontaminated Shelby tube sampler on center rods.
4. Drive Shelby tube sampler, as described in ASTM Method D1587. Retrieve the sampling tube and remove the disturbed material from the top of the tube. In addition, remove 1 inch of soil from the base of the tube. Place an impervious disk at both ends of the tube seal with a wax plug prior to shipment to the laboratory.
5. If Shelby tubes are to be extruded in the field for composite sampling, the driller will use a hydraulic extruder to obtain the sample. The core is then described in accordance with ASTM Method D2488 on the boring log form. Samples will then be composited, as necessary, for analysis.
6. Screen sampler with PID (if required).
7. Label and manage sample containers in accordance with the site-specific SAP section for shipping and handling of samples. The sample tube should be packed in Styrofoam™ plugs or other cushioning material to prevent disturbance of the sample.
8. Continue drilling to next sample location. Collect samples as outlined above.
9. Decontaminate sampling equipment in accordance with the site-specific SAP section.
10. Document activities in site logbook.
11. Backfill or grout borehole, as required.
12. Move to next location.

Reference

Integral. 2006. Lower Duwamish Waterway Slip 4 Early Action Area: Sampling and Analysis Plan for Boundary Definition Addendum: Pre-Design Investigation Sampling. Prepared for Seattle City Light and King County, Seattle, WA. Integral Consulting Inc., Mercer Island, WA.

STANDARD OPERATING PROCEDURE (SOP) SD-08

SUBSURFACE SEDIMENT CORE COLLECTION USING A VIBRACORER

SCOPE AND APPLICATION

This SOP describes the procedure for collecting and processing sediment core samples using a vibracore system, which collects continuous and relatively undisturbed sediment cores. This method of sediment coring is performed from a boat and uses high-frequency low-amplitude vibration to break down the frictional resistance of the sediment and allow the core tube to penetrate into the sediment with minimal distortion. It is best used for sampling coarse, consolidated sediment and very cohesive sediment, where static weight (e.g., piston-type or conventional gravity corers) will not produce adequate penetration into the sediment. In addition, the vibracorer offers a high rate of production, superior retention of shallow samples, and a greater sample volume compared to conventional drilling equipment.

Vibracorers generally consist of a metal corer barrel (usually a 4-in.-outside-diameter, aluminum core barrel) with a location-dedicated polycarbonate or Lexan®-lined core tube, and a vibrator mechanism attached to the top of the barrel. The vibration is created either by an electric motor, a hydraulic system, or a pneumatic piston attached to the top of the barrel. Therefore, a generator or air compressor is needed on board to power the corer. The pneumatic piston does not have the same function as a piston in a piston corer. Because vibracorers generally do not have a piston in the corer, some compaction and/or bypass will occur, and recovery will be less than 100 percent.

A continuous sediment sample is retained within the tubing with the aid of a stainless-steel core cutter/catcher or nosecone attached to the bottom of each aluminum tube.

It is always best to keep the core in a vertical position to prevent the top layers of sediment (i.e., the top 5 to 15 cm) from slumping. However, in many cases, it is not feasible to process the core in a vertical position because the tripod needs to be at least twice the height of corer, and sectioning and logging the sample would have to be performed from a ladder. For studies that specify sectioning the sample into coarse intervals (>20 cm), processing the core in a horizontal position will generally not significantly disrupt the stratigraphy. For studies that specify shorter intervals (<5–10 cm), processing the core in a horizontal position is likely to disrupt stratigraphy. In this case, the top layers of sediment that have high water content should be sectioned while the core is in a vertical position, and when the sediment becomes thicker, the corer can be laid horizontally.

PROCEDURES

Decontamination

To prevent potential cross-contamination of samples, all reusable sediment sampling equipment must be decontaminated prior to use at each station and between field replicates.

Before each station is sampled, decontaminate the inner surfaces of the corer or core tube liner and all stainless-steel sample compositing equipment. Prior to sampling, all core liners will be washed in sequence with a standard detergent (e.g., Alconox®), rinsed with site water, and then air-dried. During storage and transport, decontaminated core liners will be capped at both ends to prevent contamination. Details on correct decontamination procedures can be found in SOP SD-01, *Decontamination of Equipment—Sediment*. The project-specific field sampling plan (FSP) should also be consulted to determine any project-specific decontamination procedures. The personnel performing the decontamination procedures will wear protective clothing as specified in the site-specific health and safety plan.

All solvent rinsates (if used) will be collected into a bucket or tub and allowed to evaporate over the course of the day. Any rinsate that has not evaporated by the end of the sampling event will be containerized and disposed of in accordance with applicable regulations.

Vibracorer Deployment and Retrieval

The following procedures are based on using the vibracorer aboard a boat equipped with a tripod or A-frame of sufficient height to allow recovery of the core (see project-specific FSP for information on target coring depth), and a power winch. On pontoon boats, the tripod is centered over a hole in the floor, whereas on other boats, the corer may be lowered over the side or stern. To obtain cores of high quality, the boat must be anchored with at least three anchors so the boat will not drift during the coring process.

1. Maneuver the sampling vessel to the targeted sampling location using the positioning procedures and minimum water depth restrictions.
2. Deploy 3- or 4-point anchor system to maintain position; record and monitor position throughout core acquisition.
3. Once on location, measure the water depth (depth to top of sediment) using the onboard depth sounder (fathometer) or lead line and record measured depth in the field logbook. If the water level is affected by tides, obtain tide level measurements and calculate tidal height in feet above mean low water. The date, time, weather, and water conditions (e.g., high wave activity, strong currents, turbidity, tidal flux) should also be recorded in the field logbook.

4. Assemble the decontaminated core tube, liner, core catcher, and cutter heads (or nose cone depending on the model of vibracorer used), using care to not contact decontaminated surfaces. Attach assembled vibracorer to winch cable. Note that several decontaminated catchers and cutterheads will be on hand, in case of loss. Core catchers and cutter heads can be decontaminated and reused for subsequent core collection.
5. Attach a tape measure to the vibracorer or mark the winch cable in 1/2-ft increments to measure penetration depth.
6. Inspect connections of winch cable and electrical or pneumatic lines to confirm they are secure.
7. Signal the winch operator to slowly raise the vibracorer into a vertical position and guide the vibracorer (with core liner, valve, core catcher, and cutterhead in place) overboard until it is clear of the vessel.
8. Using the winch, slowly lower the vibracorer through the water column at a speed of about 1 ft/s to avoid creating a bow wake or overturning of the vibracore. Stop lowering the corer a few feet above the sediment and confirm that the boat has not drifted.
9. Continue lowering the vibracorer until the tip of the core is resting on the sediment or to the depth recorded by the fathometer, depending on the consistency of the sediment. Record the vibracorer depth as derived from the attached tape measure or marked winch cable. Measurements will serve as a basis for determining penetration depth.
10. Resume lowering the corer at about 1 ft/s. When the nosecone or core catcher contacts the sediment, turn on the vibracorer motor. The vibracorer is then allowed to slowly penetrate the sediments. Initially, light tension should be maintained on the cable to keep the corer from tipping over.
11. Lower the vibracorer to the target penetration depth as measured by the attached tape measure or marked winch cable. If the targeted penetration depth is met, proceed to the next step; if refusal is met, retrieve the vibracorer, perform gross decontamination (i.e., rinse with river water and brush off visible sediment from the outside of the core barrel) and re-attempt at new location offset at least 3–5 ft from original location.
12. When the target penetration depth is reached, or refusal occurs, turn off the vibracorer and record the time, penetration depth, angle of the cable relative to the boat, and any other observations.
13. The vibracorer is slowly withdrawn from the sediments at a constant rate, to keep it upright and not dislodge any sediment from within the core barrel, and raised to the surface.

14. With the corer hanging in a vertical position, clean the vibracorer assembly by hosing down the equipment with site water prior to being brought on board. If the corer is not plugged, care should be taken not to direct water into the open end of the core barrel.
15. After collection of the core sample, the vibracorer is slowly guided onboard the vessel; use care to avoid jostling that might disturb the integrity of the core. Care must be taken to keep the top end of core elevated to prevent sediment from "pouring" out. Use a sawhorse or equivalent to elevate the top of the core. If necessary, as soon as the nosecone clears the water surface, the bottom of the corer may be plugged with a rubber stopper to prevent loss of sediment.
16. Before the polycarbonate or Lexan®-lined core tube is removed from the vibracorer, the nosecone or core cutter/catcher is visually inspected to ensure that proper penetration has been attained and that there is no obvious loss of sediment from the tube. Any presence of noticeable odors, the core penetration depth, and physical characteristics (e.g., color, texture, odor) of the sediment sample as observed at the ends of the tube will be recorded in the field logbook or on the Field Sediment Core form by an experienced geologist. In addition, any sheen in the water will also be noted in the field logbook.
17. If the core will be processed horizontally, slowly lay the corer down. Unscrew the cutter head (or nosecone) and carefully remove the core catcher, while retaining as much sediment as possible.
18. While removing the core catcher (or nosecone), be ready to immediately seal the end of the core liner by placing a plastic cap over the open end.
19. Carefully remove the core liner that contains the sample by lifting the lower end from the deck as needed to provide clearance. Affix core cap, wrap with tape, label core liner and end of core, remove valve from top of core liner, stand core upright, and place in a processing rack or tray to allow the sediment at the top of the core to settle. Avoid sudden movements to the core that would disrupt the sediment interface.
20. While waiting for sediment to settle, prepare the Field Sediment Core form. Identify any debris and note its depth in the core and what the debris is, if possible.
21. Once resuspended sediment has settled, measure the length of the recovered core, calculate percent recovery (100 x recovered length/penetration depth), and record in the logbook or on the Field Sediment Core form.
22. Check the core for acceptability. The following acceptability criteria should be satisfied:
    - The core tube is not overfilled with sample so that the sediment surface presses against the bottom of the vibracorer head.
    - Overlying water is present (indicates minimal leakage).

- The overlying water is not excessively turbid (indicates minimal disturbance).
- The desired penetration depth (see project-specific FSP for required penetration depth) or refusal has been reached.

Depending on requirements of the project-specific FSP, a core may be rejected based on percent recovery. Commonly, a core is deemed unacceptable if recovery is less than 80 percent. If recovery is less than 80 percent, the core sample will be retained for possible processing, while additional sampling attempts are made to collect a core with greater than 80 percent recovery. If subsequent attempts result in recoveries of less than 80 percent, then the sample with the highest percent recovery may be used for analysis. The number of attempts to collect an acceptable sample will be specified in the project-specific FSP. If recovery is less than 80 percent, the core may be acceptable if the penetration depth is deeper than the target core length. In this case, the recovered length should be equal to the target length.

23. Once sufficient time has been allowed for the sediment to settle (i.e., no sediment is suspended in the overlying water), use a decontaminated saw to cut a drain-slit or a decontaminated drill bit to drill in the side of the core liner approximately 1 to 2 in. above the sediment–water interface; allow excess water to drain. Cut excess polycarbonate liner with decontaminated blade and use a siphon to decant off the overlying water. Ensure that the saw blade, drill bit, or siphon does not contact the sediments and that fine-grained suspended sediment is not removed.
24. Cut cores into manageable sections (3–4 ft) aboard the vessel immediately after their retrieval. Cap each section with aluminum foil and plastic caps, and seal with duct tape. Mark the core with permanent marker using a unique number or alphanumeric code identifying sampling location, core number, core section, and segment orientation (i.e., which end is up). Following sectioning, store the cores in an upright position onboard the vessel in a core box and have them transported periodically throughout each field day by small boat to a field processing area where they are to be stored upright under custody on ice or refrigerated at 4°C to await processing.
25. In preparation for next core, thoroughly rinse the interior of the core barrel until all loose sediment has been washed off. Repeat process at next sampling location. Continue coring until requirements are met.

In situations where there is significant surface water depth and/or water current that could cause the vibracorer setup to lean at an unacceptable angle, a buoyant frame or rigid frame configuration should be used.

With the buoyant frame, the vibracorer is maintained in proper vertical position by two guidelines held taut between a float package and a weight stand. The larger weight stand is provided with ballast boxes so that easy-to-find ballasting material such as lead bags or scrap

metal can be used in the field. For deployment, the vibracorer is lowered with the weight stand hanging on its guidelines from the vibrahead. The float package is hooked up to the guidelines when the vibrahead reaches the deck level.

After coring and pull-up, the system is retrieved in the reverse manner. In case of limited deck space or overhead clearance, or to further accelerate the procedure on the water, the weight stand can be left in as overboard cradle.

Sample Handling, Storage, and Processing

Cores should be processed concurrently with core collection, and every effort should be made to ensure cores are processed within 24 hours of collection. Cores awaiting processing will be sealed tightly at both ends and stored upright in ice or in a refrigerator. If core collection outpaces processing such that significant delays in core processing appear likely, core collection will be suspended to allow the core processing to catch up.

As mentioned above, once coring has been completed at a given location, the cores will be transported in an upright position on ice to a designated field processing area, where they will be logged and processed. The field processing area will be equipped with a core-cutting table, core-processing tables, a decontamination area, and a storage area with appropriate refrigeration. Appropriate lighting will be installed in the field processing area so that consistent, high quality photographs can be taken of the opened cores. Care should be taken to create a field processing area that minimizes the potential for outside contamination.

Sample processing includes removing the sample from the liner, recording observations of sample characteristics, mixing subsamples, and distributing the sample to containers for shipping to the testing laboratory. Vibracore processing most often consists of the following steps:

1. Cut each core tube along the long axis using decontaminated hook blade. Rotate the tube 180^{o} and cut again.
2. After each core is cut, move the entire core tube to an aluminum foil-covered table and open it so that it can be systematically logged, described, and photographed.

However, depending on the project-specific FSP, the core may be extruded from the liner and cut into the specified intervals as it emerges or the core liner may be cut into sections, sealed, and shipped intact to the testing laboratory.

Core Observations

1. Verify that the length of the core, water depth, and all required position data have been recorded in the field logbook together with all pertinent observations and communications with the field team leader.

2. After each core is cut open, describe the sediment on a Field Sediment Core form in the field processing area notebook. When recording the information for each core, follow the guidelines below:
   - Physical sediment description (i.e., sediment type [e.g., silt, sand], density/consistency, color)
   - Odor (e.g., hydrogen sulfide, petroleum, creosote)
   - Visual stratification, laminations, and lenses
   - Presence/location/thickness of the redox potential discontinuity layer (a visual indication of black is often adequate for documenting anoxia)
   - Approximate percentage of moisture
   - Vegetation
   - Approximate percentage of vegetation
   - Debris
   - Approximate percentage of debris
   - Presence of biological structures (e.g., detritus, shells, tubes, bioturbation, live or dead organisms, chironomids)
   - Approximate percentage of biological structures
   - Presence of a sheen
   - Other distinguishing characteristics or features.

   The visual observations of sediment lithology (dominant grain sizes) will be the primary criteria for determining sample intervals (i.e., lithologic units) in the cores. For consistency, core descriptions and terms used will follow the criteria below, which are modified from methods presented in ASTM D 2488-00 (ASTM 2000):

3. Record visual estimates of the grain-size percentages of sediment units within each core on the Field Sediment Core form so that the total sum will add up to 100 percent. Make estimates of gravel, sand, and fines (silt and clay) content generally to the nearest quartiles:
   - 0 to 25 percent
   - >25 to 50 percent
   - >50 to 75 percent
   - >75 to 100 percent.

   If appropriate, describe the sediment narratively on the log based on the estimated grain-size percentages. Use the dominant constituent grain size as the primary unit

descriptor, and describe the abundance of other grain sizes present using the following terms:

- The grain-size adjective (e.g., gravelly, sandy, silty, or clayey), if estimated to constitute more than 25 percent of the sediment
- *With*, for example, sand with silt, silt with sand, etc., if estimated to constitute less than 25 percent of the sediment
- *Trace*, if estimated at less than 5 percent of the sediment (and not included in the total 100 percent).

For other features observed, such as organic matter or debris, use the following additional descriptive terms as appropriate:

- *Mostly*, if estimated to constitute 50 percent or more of the unit
- *Some*, if estimated to constitute more than 25 to 50 percent of the unit
- *Little*, if estimated to be 25 percent of the unit or less
- *Trace*, if estimated at less than 5 percent (and not included in the total 100 percent).

4. Describe density using the following terms:
   - *Loose*, if easily penetrated with a sampling spoon
   - *Dense*, if penetration is more difficult.
5. Describe consistency using the following terms:
   - *Very soft*, if present as an ooze that holds no shape
   - *Soft*, if saggy
   - *Stiff*, if it holds a shape
   - *Very stiff*, if penetration with a spoon is low
   - *Hard*, if no penetration with a spoon is possible.
6. Use other observations (e.g., obvious anthropogenic material, dramatic color changes) to define or help define sample intervals (check project-specific FSP for sample interval definition; depending upon the project-specific requirements the sample interval could be based on lithology or it could be set to a specific interval [e.g., 1 ft]).
7. Determine the boundaries of lithologic units primarily by changes in the top two dominant grain sizes estimated visually (e.g., a change from a silty sand to a gravelly sand or to a sandy silt).

8. Photograph the cores after they have been described and before any sediment is removed for processing. It is important for each core section to be photographed with adequate lighting from a standard measured distance from the core. Digital photographs will be used later in the production of digital core logs.

Mixing and Sample Preparation

1. After the sample is characterized and the core observation logged on the Field Sediment Core form, remove the specified sample interval using a stainless-steel spatula or spoon (see project-specific FSP for correct sampling interval). Exercise care to not include sediment that is in direct contact with the core tube. With the approval of the field team leader, and using a decontaminated stainless-steel instrument, carefully remove unrepresentative material (e.g., large shells, stones). Exercise care not to touch the sediment during this process. Note any unrepresentative material removed from the sample in the field processing area notebook.
2. Remove subsamples for analysis of unstable constituents (e.g., volatile organic compounds, acid- volatile sulfides), and place them directly into sample containers without homogenization. Completely fill the sample container so that there is no headspace or entrapped bubbles.
3. Transfer the remainder of the sample interval to a decontaminated stainless-steel bowl for homogenization. If additional sediment volume is required to fill all sample bottles (see project-specific FSP) and multiple cores need to be collected at a given station, cover the compositing bowl covered with aluminum foil (dull side down) to prevent sample contamination (e.g., from precipitation, engine exhaust, splashing water) and place in a cool dark place until the next core from that location is processed.
4. After all the sediment is transferred to the compositing bowl, homogenize the contents of the bowl using stainless-steel spoons until the texture and color of the sediment appears to be uniform.
5. Distribute subsamples to the various containers specified in the project-specific FSP and preserve the samples as specified in the project-specific FSP. Briefly stir the sediment in the compositing bowl between each spoon transfer to the sample containers.
6. After all subsamples have been placed in the sample containers, if it is suspected that there is clay in a sample, perform a "ribbon test." Perform a separate ribbon test for each interval within the core where clay is suspected to be present. To perform this test, remove a small piece of sediment from the sampled interval using a decontaminated stainless-steel spoon and roll it between the fingers while wearing protective gloves. If the piece easily rolls into a ribbon it is clay; if it breaks apart, it is silt. Note this information in the field processing area notebook.
7. Subsequent intervals should be processed in the same way.

Field Quality Control Samples

If additional volumes of sediment are required to perform all analyses including quality control analyses, an additional core may need to be collected from the same location and subsampled and homogenized accordingly. Details on collection of field quality control samples (e.g., field duplicates) will be specified in the project-specific FSP. Details on collection of field quality control samples and preparation of the certified reference materials can be found in SOP SD-02, *Preparation of Field Quality Control Samples—Sediment,* and SOP SD-03, *Preparation of Reference Materials—Sediment*. Not all of the field quality control samples discussed in this SOP may be required for a given project. The specific field quality control samples will be described in the project-specific FSP and quality assurance project plan.

Field Measurements

A water depth measurement must be collected at every sampling location. Depending on the specific project objectives, it may be necessary to perform field measurements of the *in situ* environment. Possible field measurements include temperature and pH of the sediment at the sediment-water interface and concentration of dissolved oxygen, salinity, or conductivity in the overlying water. Details on collection of field measurements can be found in SOP SD-11, *Field Analyses for Sediment.* The specific field measurements, if any, will be specified in the project-specific FSP.

Station Location Coordinates

Station locations for all field sampling will be determined using a differential global positioning system (DGPS) or by surveying. The accuracy to which the latitude and longitude of a station location is determined will be specified in the FSP. At a minimum, a DGPS capable of providing latitude and longitude coordinates with an accuracy of approximately 3 m is recommended. The DGPS consists of two satellite receivers linked to each other by a VHF telemetry radio system. The receiver will be on the sampling vessel. Details on collection of very accurate station coordinates can be found in SOP AP-06, *Navigation.*

Sample Custody and Shipping

Sample custody will be maintained in accordance with procedures outlined in SOP AP-03, *Sample Custody*. All samples will be packaged and shipped with other samples in accordance with procedures outlined in SOP AP-01, *Sample Packaging and Shipping*.

Troubleshooting

Insufficient Sample

The corer may not collect enough sediment because of 1) inadequate penetration, 2) adequate penetration but poor recovery due to compaction, 3) adequate penetration but poor recovery as a result of bypass, or 4) adequate penetration but loss of sample during retrieval. Compaction and bypass are two different artifacts that are difficult to distinguish and quantify. Following is an approach to identifying the causes and remedies of insufficient sample length. Keep in mind that a combination of these causes may occur:

- **Inadequate Penetration**—Allow more vibration time at the refusal depth, or increase the vibrator frequency.
- **Poor Recovery Due to Compaction**—Compaction is the process of rearranging the sediment particles, so that less volume is occupied by pore water, which results in a shorter column of sediment in the corer than *in situ*. Compaction occurs only in clean coarse silt, sand, and gravel sediments that have a high hydraulic conductivity and are not terminally compacted *in situ*. Fine-grained cohesive sediment (i.e., low hydraulic conductivity) does not compact. The key feature of compaction is that all of the solids ahead of the nosecone are collected as the corer penetrates. So, although the calculated recovery is less than 10 percent, 100 percent of the sediment solids were recovered. Therefore, if the sample has poor recovery, is composed of clean coarse-grained materials, and there is no evidence of sediment falling out the bottom, then the sample is likely to have been compacted. Depending on the project-specific FSP, the specified sample intervals may be shortened proportional to recovery. Because compaction of the solids displaces pore water, minimal compaction is needed for cores that are intended for porewater studies, or cores that will be analyzed for substances that have low K_d values. Vibration in vibracorers is known to rearrange particles, which leads to compaction, so another type of corer may be appropriate if compaction is a problem.
- **Poor Recovery as a Result of Bypass**—Bypass is the process of pushing sediment out of the path of the nosecone/corer as it penetrates the sediment. This is caused by the friction of sediment inside the core liner making it difficult for more sediment to enter the tube. This is most pronounced in fine-grained sediments that have low hydraulic conductivity, or layers of hard and soft sediment, or long cores. The low hydraulic conductivity prevents porewater from being displaced, so compaction cannot occur. Fine-grained sediments in this context are those in which particles cannot be felt between the thumb and forefinger of an ungloved hand. These are generally "sticky" or cohesive sediments. Therefore, if a sample has poor recovery, is fine grained and cohesive, and there is no evidence of sediment falling out the bottom, then some of the sediment column has likely been bypassed.
- **Poor Recovery Because of Loss of Sample during Retrieval**—This is often diagnosed by observing some of the core falling out the bottom as the corer approaches the water surface during retrieval, or a core liner that is empty near the bottom. Sample slipping

out the bottom of the corer can be caused by a loss of suction or noncohesive sediment that does not stick to the liner wall. Depending on the specific design of the vibracorer, there are several places at which suction can be lost. These may include the valve seat, the valve assembly, the nose piece, and couplings between the barrel and extensions. To prevent loss of suction, Teflon® plumber's tape should be used on all the threaded connections, and the valve assembly should be clean. For coarse-grained sediment (e.g., clean coarse sand and gravel, and shells) that is non-cohesive and falls out the bottom of the corer, it is sometimes possible to penetrate to a lower layer that is finer grained and will effectively plug the bottom of the core. As mentioned above, core catchers may are used to retain sediment in a vibracorer, although they should not be used if the surface sediments have high water contents and are to be sectioned at less than about 2 inch intervals.

Because recovery can be an important indicator of corer performance, sediment characteristics, and sample quality, some simple tests can be performed as a diagnostic tool. Penetration of the corer can be measured by putting Velcro® tape on the outside of the corer. Velcro® tape can also be used on the inside of the liner during testing to see how far up inside the liner the sediment interface moves, how much sample slips out the bottom, and how much compaction or bypass occurs.

Notes

1. For long cores that require more than one piece of liner, squarely cut the ends of both pieces with a plastic pipe cutter, butt the ends of the two pieces of liner squarely together and tape them securely so no leaks occur. Do not use too many layers of tape or the liner will not fit into the barrel. Do not use duct tape for this process. Use a high quality tape (i.e., 3M 3750) and dry the tubes before applying.
2. Sometimes tripods are not tall enough to lift the corer so that the barrel will clear the top edge of the liner when removing the liner. To remove the liner in this case, upon unscrewing the cutter head (or nose piece), lower the cutter head (or nose piece) and liner into a pail that has a rope securely tied to the handle. While the corer is raised by the winch, lower the pail through the hole in the deck and into the water (if necessary) until the top edge of the liner clears the bottom edge of the barrel. Then lift it back onto the deck.
3. If the vibracorer does not penetrate significantly or if the cable is let out too quickly, the vibracorer will contact the bottom, tip over, and fall sideways. When this happens, the line will initially go slack, then quickly snap to the side and take up the slack. In this case, reject the core and begin again.

4. A good measure of whether the vibracorer collected the sediment-water interface is to inspect the interface for a thin layer (about 1 mm) of olive green benthic or detrital algae. Also, if the core liner is rotated back and forth gently, the top centimeter will appear to have a gelatinous response.
5. It is sometimes impossible to collect an intact interface because gas bubbles are commonly released from sediment when the corer contacts the sediment. The released gas bubbles entrain surface sediment and cause the overlying water to become turbid. If this is the case, gas bubbles in the sediment can likely be observed through the liner wall.

REFERENCES

ASTM. 2000. Standard practice for description and identification of soils (visual-manual procedure). ASTM Standard Method No. D 2488-00. In: ASTM Book of Standards, Volume 04.08. American Society for Testing and Materials, West Conshohocken, PA.

STANDARD OPERATING PROCEDURE (SOP) SD-12

LOGGING OF SEDIMENT CORES

SCOPE AND APPLICATION

The following procedures for completing the Field Sediment Core Form establish the minimum information that must be recorded in the field to adequately document sediment coring activities. The field sediment core form must be filled out completely. Depending upon project specific requirements, some of the items listed below can be recorded in the observing scientist's field logbook and/or on the Station Core Log. All field forms must be filled out completely.

All of the information addressed in this standard operating procedure (SOP) should be included in the observing scientist's field documentation. Additionally, standards presented may need to be supplemented with additional technical descriptions or field test results (see project specific field sampling plan [FSP]).

ACTIVITIES OF THE OBSERVING SCIENTIST DURING CORING

1. Record the name of the coring contractor and personnel performing the coring (lead person and any support staff)
2. Record the type and make of the coring equipment being used
3. Note the weather or any special external conditions that influence the coring
4. Be certain that the coring contractor is informed about the nature of the daily records that the contractor will keep
5. Check the coring contractor's daily records to verify their accuracy
6. Note date and time of all activities associated with the coring
7. Make certain that the coring contractor follows all required procedures
8. The observing scientist's daily record shall include, but may not be limited to, the following items:

    - Date and depth of core
    - Depth of start and finish of each sampled interval
    - Depth and size of any casing or core tubing used
    - Time required to advance the core
    - Loss of water, mud, or air during sample retrieval

- Depth of overlying water
- Simplified description of strata
- Total sample recovery (in inches or centimeters)
- Details of delays and breakdowns.

The observing scientist should also record the coring start and finish dates and times. For consecutive sheets, provide, at a minimum, the project number, the station number, and the sheet number. This list excludes any special items that may be required for contractual record purposes or for special tests (see project-specific FSP).

Data on Field Sediment Core Form

Core Type/Method: Provide the sampler type (e.g., GC = gravity corer, PC = piston corer, DRCV = drive rod check valve corer, VC = vibracorer, BC = box corer).

Sample Number/Tag Number: Provide the sample number. The sample numbering scheme should be established before sampling begins. Consult the project-specific FSP for the sample numbering scheme. The depth of the sample is the depth to the top of the recovered sample to the nearest centimeter. Samples should be obtained from the entire recovered core (depending upon the sampling intervals specified in the project-specific FSP). The tag number(s) and respective sample number(s) of the sample container(s) should also be recorded in the field logbook.

Photograph Number: Provide the number of the film roll and the photograph number.

Odor: Provide information on presence of any odor associated with the sediment. Document each interval in the core at which an odor is present. Describe the odor in the *Sediment Description* section of the field sediment core form.

Sheen: Provide information on presence of any sheen associated with the sediment. Document each interval in the core at which sheen is present. Also note if sheen is present on the water surface during coring activities.

Blank Columns: Two blank columns are provided on the field sediment core form. These columns can be used for site-specific information, usually related to the contaminants of concern (e.g., sheen, air quality measurements).

Water Breaks: Record the location of any observed breaks in the sediment core.

Depth Scale: Enter the depth of the core below sediment surface. Match the sediment descriptions with the depth scale.

Unified Symbol: If a geologist is providing the sediment descriptions of the core, then the unified symbol code (USC) for different sediment types (e.g., silt, clay, sand) should be placed in this column. The USC name should be identical to the ASTM D-2488-84 Group Name with the appropriate modifiers.

Table SD-12(1) presents the USC classification system. The USC system is an engineering properties system that uses grain size to classify soils, it can however also be used by a geologist to characterize the sediment in a core.

Table SD-12(1). USC Classification System

Major Divisions			Group Symbol	Group Name
Coarse-grained soils	**Gravel**	Clean	GW	Well-graded gravel, fine to coarse gravel
	More than 50 percent of	Gravel	GP	Poorly graded gravel
	coarse fraction retained on	Gravel with	GM	Silty gravel
More than 50 percent	No. 4 sieve	fines	GC	Clayey gravel
	Sand	Clean	SW	Well-graded sand, fine to coarse sand
retained by	More than 50 percent of	Sand	SP	Poorly graded sand
No. 200 sieve	coarse fraction passes	Sand with	SM	Silty sand
	No. 4 sieve	fines	SC	Clayey sand
Fine-grained soils	**Silt and clay**	Inorganic	ML	Silt
			CL	Clay
	Liquid limit < 50	Organic	OL	Organic silt, organic clay
More than 50	**Silt and clay**	Inorganic	MH	Silt of high plasticity, elastic silt
percent passes			CH	Clay of high plasticity, fat clay
No. 200 sieve	Liquid limit ³ 50	Organic	OH	Organic clay, organic silt
Highly organic soils			PT	Peat

Note: Field classification is based on visual examination of soil in general accordance with ASTM D-2488-84.

Soil classification using laboratory tests is based on ASTM D-2487-83.

Descriptions of soil density or consistency are based on interpretation of blow count data, visual appearance of soils, and/or test data.

Liquid limit is the water content of soil-water where the consistency changed from plastic to liquid.

Sediment Description: The sediment description should follow the format described in SOP SD-13, *Field Classification of Sediment*. Information on sediment should include sediment type, percent moisture with depth through the core, color, and presence or absence of vegetation or biota. The surface conditions within the core (i.e., overlying water is present, undisturbed sediment/water interface, presence of any vegetation or biota) should also be described. The project-specific FSP should be consulted for any special descriptive items that may be required.

Comments: Include all pertinent observations. Coring observations might include coring chatter, core-bounce (hard object hit by corer during penetration), sudden differences in

coring speed, damaged coring equipment, and malfunctioning equipment. Information provided by the coring contractor should be attributed to the coring contractor.

Data on Station Core Log

Cast Number: Record the number of coring attempts at each station.

Start/End Time: The time should be recorded during coring to determine coring speed. Time should be recorded in 24- hour mode (e.g., 3:00 p.m. = 1500 hours).

Water Depth: Record the overlying water depth at the station. Note: The overlying water depth can change between coring attempts and therefore must be measured prior to each attempt.

Core Penetration Depth: Record the depth that the core was pushed into the sediment. Note: If this information is not readily apparent, it can be obtained from the coring contractor.

Retrieved Core Length: While the sediment core is vertical, record the length of the retrieved core.

Overlying Water: Record whether or not there is water on top of the sediment core once the core has been retrieved. This is necessary to determine measurable sediment/water interface.

Coordinates: Record the latitude and longitude (or geographic) of the station location. The datum used to collect the station location coordinates (e.g., WGS84) must also be recorded in the field notes.

STANDARD OPERATING PROCEDURE (SOP) SD-13

FIELD CLASSIFICATION OF SEDIMENT

SCOPE AND APPLICATION

This SOP presents the field classification of sediments to be used by Integral field staff. Sediment descriptions should be precise and comprehensive without being verbose. Assumptions and personal comments should not be included in the sediment descriptions. These descriptions will be used to interpret environmental conditions and other potential properties, rather than the exact mineralogy or tectonic environment.

Sediment descriptions should be recorded in either the observing scientist's field logbook, or if subsurface sediment is collected, then the sediment description column of the Field Sediment Core Form should be completed for each core collected. If no difference between consecutive sediment samples exists, subsequent descriptions can be noted as "same as above," or minor changes such as "increasing sand" or "becomes dark brown" can be added.

After the overlying water is removed, characterize the sediment. Sediment characteristics that are often recorded in the field logbook or the Field Sediment Core Form if subsurface sediment is collected, include:

- Sediment type (e.g., silt, sand)
- Texture (e.g., fine grain, coarse, poorly sorted sand)
- Color
- Presence/location/thickness of the redox potential discontinuity layer (a visual
- indication of black is often adequate for documenting anoxia)
- Approximate percentage of moisture
- Presence of biological structures (e.g., chironomids, tubes, macrophytes) and the
- approximate percentage of these structures
- Presence of organic debris (e.g., twigs, leaves) and the approximate percentage of
- debris
- Presence of shells and the approximate percentage of shells
- Stratification, if any
- Presence of a sheen
- Odor (e.g., hydrogen sulfide, oil, creosote).

In addition, the project-specific field sampling plan should be reviewed to determine if there are any project-specific reporting requirements.

In general, the similarities of consecutive sediment samples should be noted. Examples of surface sediment descriptions are provided in Table SD-13(1). The minimum elements of the sediment descriptions are discussed below. The format of sediment descriptions for each sample should be consistent throughout the logbook.

Table SD-13(1). Example of Surface Sediment Desctiptions

Station No.	Grab No.	Example Descriptions
TC01	1	SILT, mottled dark gray (10YR 4/1) with thin layer < 1 cm of very pale brown (10YR 7/4) on surface. Occasional roots, some twigs, and leaves on surface. Slight reducing odor. Sheen on overlying water in grab.
TC02	1	Sandy SILT, fine sand, dark gray (10YR 4/1) throughout grab, with 10 percent medium to coarse sand, trace woody debris. Chironomid on surface.
TC02	2	Same description as first grab at Station TC02.
TC02	3	Same description as first grab at Station TC02, but no sand (SILT only) and color is very dark gray (10YR 3/1) with no chironomid present.

Definition of Sediment Types

Fine- grained sediments are classified as either silts or clays. Field determinations of silts and clays are based on observations of dry strength, dilatancy, toughness, and plasticity. Field procedures for these tests are included in ASTM D-2488-84. If these tests are used, the results should be included in the sediment description. Sediments with high plasticity can be emphasized by describing them as "silty CLAY with high plasticity." Plasticity is an important descriptor because a sediment can be dilatant/nonplastic and serve as a transport pathway, or it can be highly plastic and very impervious.

Coarse-grained sediments are classified as predominantly sand. The gradation of a coarse grained sediment is included in the specific sediment name (i.e., fine to medium SAND with silt). Estimating the percentage of size ranges following the group name is encouraged for mixtures of silty sand and sand. If applicable, use the modifiers "poorly graded" or "well graded" when describing the sand component of the sediment.

Color

The basic color of a sediment, such as brown or gray, must be provided in the description. The color term can be modified by adjectives such as light, dark, or very dark. Especially

note streaking or mottling. The color chart designations provided in either the *Globe Soil Color Book* or the Munsell color guide can be used.

Moisture Content

The degree of moisture present in the sediment should be defined as moist, wet, or very wet. The percent moisture content should be estimated.

Other Components

Other components, such as organic debris and shell fragments, should be preceded by the appropriate adjective reflecting relative percentages: trace (0–5 percent), few (5–10 percent), little (15–25 percent), and some (30–45 percent). The word "occasional" can be applied to random particles of a larger size than the general sediment matrix (i.e., occasional stone, large piece of wood).

Additional Descriptions

Features such as sloped surface in the grab, root holes, odor, and sheen should be noted if they are observed. Anything unusual should be noted. Additional sediment descriptions may be made at the discretion of the project manager or as the field conditions warrant.

STANDARD OPERATING PROCEDURE (SOP) SW-01

DECONTAMINATION OF SURFACE WATER SAMPLING EQUIPMENT

SCOPE AND APPLICATION

This SOP defines and standardizes Integral's methods for decontamination of field sampling equipment for collecting surface water samples to ensure sample integrity and minimize contamination during sample handling.

This SOP utilizes and augments the procedures outlined in the San Francisco Estuary Institute's *Field Sampling Manual for the Regional Monitoring Program for Trace Substances* (David et al. 2001), *Interagency Field Manual for the Collection of Water-Quality Data* (USGS various dates), and U.S. Environmental Protection Agency (EPA) Method 1669, *Sampling Ambient Water for Trace Metals at EPA Water Quality Criteria Levels* (USEPA 1996). Clean sampling techniques designed for trace metals will be used for the collection of filtered and unfiltered water samples.

Samples may be analyzed for organic compounds, metals, nutrients, and conventional analytes for the surface water sampling events, according to the project-specific sampling and analysis plan (SAP).

To prevent cross-contamination of samples, all reusable surface water sampling equipment will be decontaminated before each use. Decontamination of field sampling equipment can be done in the field or in a commercial laboratory. Depending on the project's complexity and analytical reporting limits (see project-specific SAP), sampling equipment may need to be decontaminated at a qualified laboratory. It is strongly discouraged to decontaminate sampling equipment in the field due to the high risk of contamination. Thorough decontamination procedures should be followed under controlled conditions at the laboratory. However, it is necessary to perform certain decontamination steps in the field.

Set up a decontamination station onsite in a clean location upwind of sampling locations, or perform decontamination in the field office, under a laboratory hood if available. Store decontaminated equipment away from contaminated areas and in a manner that will prevent recontamination prior to use.

When handling decontamination chemicals, follow all relevant procedures outlined in the site-specific health and safety plan.

EQUIPMENT AND REAGENTS REQUIRED

Equipment required for decontamination includes the following, depending on the target analyte and sampling equipment:

- Plastic brushes with rigid bristles
- Properly labeled squirt bottles
- 5-gal plastic bucket
- Tap water
- Alconox®, Liquinox® detergent, or equivalent
- Pesticide-grade decontamination solvents (e.g., ethanol and methanol, according to the project-specific SAP, as the solvents may vary by EPA region or state)
- Nitric acid (5 percent)
- Hydrochloric acid (10 percent) if nutrients are being analyzed
- Deionized water (analyte-free; received from testing laboratory)
- Sealable waste container equipped with a funnel
- 1 gal sealable plastic bags
- 2.5 L amber glass bottles.

DECONTAMINATION PROCEDURES

Decontamination methods vary depending on whether the samples collected will be analyzed for conventional analytes, organic chemicals, or trace metals.

Conventional Analytes and First Use

The following procedure is used when sampling for conventional analytes such as chloride, sulfate, sodium, and calcium. It is also used for new equipment and for equipment that is being used for the first time at a site. Conventional analytes have the simplest decontamination procedure because they tend to be highly soluble in water and detergent solutions, and do not tend to sorb significantly to the surface of the sampling equipment.

For collection of lake water samples at different depths from the same location, equipment needs to be rinsed only with site water three times between stations following an initial decontamination. Similarly, for collection of samples from rivers where stations are close to one another spatially and temporally, only a site-water rinse is necessary. The steps are as follows:

1. Rinse the equipment thoroughly with tap water.

2. Pour a small amount of Alconox® (or similar product) into a 5 gal bucket and fill it with tap water. Using a plastic brush with rigid bristles, scrub each piece in the detergent solution.
3. Rinse the equipment with tap water to remove all detergent (some detergents contain surfactants that are analytes) and set aside to drain.
4. Rinse the equipment three times with site water immediately prior to collecting the sample.

Organic Chemicals

The following procedure is used for decontaminating equipment (e.g., Kemmerer sampler) used to collect surface water that will be analyzed for organic chemicals. Two organic solvents are used in the procedure. The first is miscible with water (e.g., ethanol) and is intended to scavenge water from the surface of the sampling equipment and allow the equipment to dry quickly. Make sure that the solvent ordered is anhydrous or has a very low water content (i.e., <1 percent). The second organic solvent is hydrophobic (e.g., methanol) and is intended to dissolve any organic chemicals on the surface of the equipment.

The exact solvents used for a given project may vary by EPA region or state (see project-specific SAP). The choice of solvents also depends on the material the equipment is made from (e.g., acetone cannot be used on polycarbonate), and the ambient temperature (e.g., hexane is too volatile in hot climates). In addition, although methanol and hexane are sometimes slightly more effective than other solvents, their use is discouraged because of toxicity to sampling personnel. The decontamination procedure is as follows:

1. Rinse the equipment thoroughly with tap or site water.
2. Pour a small amount of Alconox® (or similar product) into a 5 gal bucket and fill it with tap or site water. Using a plastic brush with rigid bristles, scrub each piece in the detergent solution.
3. Rinse the equipment with tap or site water and set aside to drain.
4. Rinse the equipment with ethanol dispensed from a squirt bottle and let the excess solvent drain into a waste container equipped with a funnel (ethanol acts primarily as a drying agent, but also works as a solvent for some organic contamination). Rinse the inside of the sampling equipment that comes in contact with sample water. Set the equipment in a clean location and allow it to air dry. In cold temperatures, it may take a long time for equipment to dry. In this case, it is important to remove all water from the surface by thoroughly rinsing with a more volatile solvent such as acetone. In hotter temperatures, use a less volatile water solvent (e.g., isopropanol).

5. Rinse the air-dried equipment with methanol dispensed from a squirt bottle and let the excess solvent drain into the waste container. Methanol acts as the primary solvent, but it is insoluble with water. If water beading occurs, it means that the equipment was not thoroughly rinsed with ethanol or the equipment was not given sufficient time to dry completely. Rinse the inside of the sampling equipment that comes in contact with site water. In hotter climates, use a less-volatile solvent such as methanol. When the equipment has been rinsed thoroughly, set it in a clean location and allow the solvent to evaporate before storing or using it.
6. Close the solvent waste container when not in use and store it in a secure place.
7. Transfer the waste to empty solvent bottles and dispose of it at a licensed facility.

Trace Metals

In addition to the following decontamination procedures, personnel collecting water samples must be aware of other sources of contamination. Sources commonly encountered in the field include lead batteries used to power pumps, metal objects such as tools, and gasoline cans. To the extent possible, these items should be removed from the sample collection area and the sampling equipment, and anyone collecting the samples should avoid handling these items beforehand. Wear vinyl clean-room gloves (e.g., Oak class 100, powder free) when handling sampling equipment that will be used to collect surface water samples for trace metals analysis. Discard gloves between stations or if they come into contact with any materials known or likely to be contaminated.

The following procedures should be used for decontaminating equipment used to collect surface water samples for trace metals (e.g., Teflon™ tubing, Teflon™ churn splitter, connectors and adapters made of Teflon™ or other similar material, or plastic stands used for holding sample tubing). This procedure is not intended for containers in which samples will be stored and/or shipped to the laboratory for analysis.

1. Rinse the equipment thoroughly with tap or site water.
2. Pour a small amount of Alconox® (or similar product) into a 5 gal bucket and fill it with tap or site water. Using a plastic brush with rigid bristles, scrub each piece in the detergent solution. Fill bottles about halfway with detergent solutions and shake for a few minutes. Pump the detergent solution through any tubing for a few minutes. Small parts can be placed in large-mouth jars that have tight lids and shaken with the detergent solution.
3. Rinse the equipment with tap water to remove all detergent (detergents will neutralize the nitric acid) and set it aside to drain.

4. Clean all equipment surfaces that come into contact with water samples using a 5 percent nitric acid solution for at least 30 minutes. Place small items, such as Teflon™ water intakes, in plastic containers filled with 5 percent nitric acid. Fill sampling containers/bottles with 5 percent nitric acid solution and allow to stand. Cover the containers and keep them away from potential contamination sources.
5. Either pump acid solution through tubing, or leave it static in the tubing for the same duration.
6. Drain all equipment thoroughly and flush with at least three volumes of laboratory deionized water (not deionized water from the grocery store).
7. Drain thoroughly and flush with at least three volumes of site water before collecting a sample.

PROCEDURES USED TO DECONTAMINATE SAMPLING DIAPHRAGM PUMPS

The following procedure is used for samples to be analyzed for trace metals and conventional analytes. Two types of pumps are commonly used for collecting water samples, peristaltic and diaphragm. For peristaltic pumps, only the tubing needs to be cleaned according to the above procedure. It is best to keep precleaned short lengths of tubing for each station when using the peristaltic pump. For diaphragm pumps, the procedure is as follows:

1. Using two short pieces of tube on the pump, place both ends in a 1-gal container with detergent solution and circulate the solution through the system for 2 minutes.
2. Purge the system with about 1 gal of laboratory deionized water, keeping the outflow tubing over a waste bucket. Do not recirculate this solution. Repeat the 1 gal deionized water purge.
3. Connect the two ends of the short tubes with a decontaminated plastic coupler and keep it sealed until sampling time.
4. When ready to sample, remove the short tubing protecting the inlet of the pump, connect the tubing used for sampling to the pump, and purge the system with site water for 2 minutes, or with enough water to rinse the entire system (i.e., pump head and tubing) immediately before collecting the sample.

REFERENCES

David, N., D. Bell, and J. Gold. 2001. Field sampling manual for the regional monitoring program for trace substances. San Francisco Estuarine Institute, San Francisco, CA.

USEPA. 1996. Method 1669 – Sampling ambient water for trace metals at EPA water quality criteria levels. U.S. Environmental Protection Agency, Office of Water Engineering and Analysis Division (4303). Washington, DC.

USGS. [various dates]. National field manual for the collection of water-quality data: U.S. Geological Survey techniques of water-resources investigations, Book 9, Chaps. A1-A9. http://pubs.water.usgs.gov/twri9A. Accessed February 5, 2008. http://water.usgs.gov/owq/FieldManual/index.html#Citation. U.S. Geological Survey.

STANDARD OPERATING PROCEDURE (SOP) SW-04

SURFACE WATER SAMPLING USING A PERISTALTIC PUMP

SCOPE AND APPLICATION

This SOP defines and standardizes the methods for collecting surface water samples from freshwater or marine environments using a peristaltic pump and Teflon™ tubing.

This SOP utilizes and augments the procedures outlined in the *San Francisco Estuary Institute's Field Sampling Manual for the Regional Monitoring Program for Trace Substances* (David et al. 2001), the *Interagency Field Manual for the Collection of Water-Quality Data* (USGS various dates), and *U.S. Environmental Protection Agency (EPA) Method 1669, Sampling Ambient Water for Trace Metals at EPA Water Quality Criteria Levels* (USEPA 1996). The goal of this SOP is to ensure that the highest quality, most representative data be collected, and that these data are comparable to data collected by different programs that follow EPA guidelines.

By following this SOP, surface water can be collected with a high level of sample integrity and minimal contamination during sample handling. The trace clean sampling method described in this SOP can be used to collect surface water for filtered and unfiltered water analysis, trace metals analysis, analysis of organic compounds, and analysis of conventional analytes, such as total suspended solids, dissolved organic carbon, and total dissolved solids.

STATION ACCESS

Prior to entering select areas such as private beaches or embayments, or nearing docks, it may be necessary to acquire property access permission from the landowner. Be sure to secure such permission, including any written agreements, in advance of the sampling program.

STATION LOCATION

When collecting near-bottom surface water samples, take care to avoid resuspending sediments in the water column, which could affect the sample being collected or samples to be collected at other downstream stations. To avoid resuspended sediment interference in the sample being collected, always approach stations from downstream. Avoid sampling near eddies that may circulate water from the sampling location to upstream of the sampling location. To avoid interference from resuspended sediment at other stations, begin collecting samples with the most downstream station and continue upstream.

Collect near-surface water samples at least 6 in. below the surface–air interface or surface water microlayer to avoid collecting non-representative compounds such as transient dust particles and thin oil films, unless otherwise instructed.

Collect water samples from areas that are representative of the surface water body conditions. A station that is located away from immediate point sources (e.g., tributaries and industrial and municipal effluents) is preferred for collecting surface water samples unless sampling is designed to assess these sources. Representative samples can usually be collected in portions of the surface water body that have a uniform cross section and flow rate. Because mixing is influenced by turbulence and water velocity, select a site immediately downstream of a riffle area (e.g., fast flow zone) to ensure good vertical mixing.

Sample tributaries as near the mouth as is feasible. However, consider the impact of the downstream receiving water body on the tributary flow and sediments. The downstream body may decrease water velocity (causing suspended solids to settle) or create eddies (causing mixing of the two waters). The downstream water body may change the water quality (e.g., salinity), temperature, or turbidity in the tributary near its mouth. It is important to determine how far upstream the tributary is influenced by the downstream water body, and then establish a sampling point with a reasonable distance upstream from that boundary.

Pay attention to intakes and outflows within lagoons or settling ponds, which may cause localized concentrations that are not representative of general conditions. Sample locations adjacent to structures (e.g., banks, piers) may also have biased characteristics as a result of flow or release of substances from the structure. Note these kinds of confounding factors in the field logbook. For ponds and lakes that may be vertically stratified, use a multi-parameter water quality meter to collect depth profiles throughout the water body to aid in selecting appropriate sampling points and depths.

SUMMARY OF METHOD

To collect surface water samples for standard chemical and conventional analyses, use a peristaltic pump with an extended sampling tube lowered to the desired depths (see project-specific sampling and analysis plan [SAP]). Two kinds of sampling devices may be used to obtain the water samples, depending upon the project's needs. The near-surface water polyvinyl chloride (PVC) sampling structure (water sampler) has a polyurethane-coated weight suspended from the bottom of the structure to maintain it in an upright position (Figure 1a).

A near-bottom water sampler has a weighted landing base designed to keep the sampling tube at a fixed distance from the bottom (e.g., 30 cm, or 12 in.) and prevent the intake from coming in contact with the sediment (Figure 1b). Both types of water samplers keep the tubing intake pointing into the current with the help of a vane. The vane can be removed if the water is

quiescent. Additional equipment, such as a multi-probe or underwater video camera, may be mounted on the PVC structure.

At each station, when either a near-surface or a near-bottom water sampler is deployed, attach the Teflon™ tubing to the vane with zip ties and place the water intake approximately 10 ft from the bow of the boat with the aid of an A-frame or davit. Keeping the boat facing the current, lower the water sampler unit to the appropriate depth with the help of a hydraulic or electric winch. Using a peristaltic pump, direct the outflow from the sampling tube into either a polycarbonate (for inorganic analyses) or glass or stainless-steel (for organic analyses) composite mixing container (Figure 2). Pump equal volumes of water into each large, pre-cleaned 10- or 20 L mixing container (depending on the project-specific needs) that is equipped with a Teflon™-coated magnetic stirring bar, and place them over a magnetic stir table. Use the containers for mixing and compositing samples for subsequent chemical analysis.

Following sample compositing in the mixing container, fill appropriate sample bottles (see project-specific SAP) using a second peristaltic pump, with the outflow directed into the sample bottle. If enough water volume is available, hold the sample bottle near the pump outlet, rinse the sample container one or two times, discard the rinsate, and then fill the sample bottle. Be aware that laboratory bottles are pre-cleaned and this rinsing option is not mandatory if water volume is an issue. Collect field rinsate blanks to ensure that sampling containers are not a source of contamination. If preservatives are present in the sample bottle, then omit the rinsing step. Cap and label the sample containers, and place them inside a cooler to store at approximately 4±2 °C.

Two types of surface water samples may be collected: unfiltered and filtered. For filtered metals and dissolved organic carbon samples, place the 0.45-µm filter (or project-specific pore size filter; see project-specific SAP) in-line near the tubing outlet to filter samples immediately before the water is discharged into the sample bottle (Figure 2). In general, filter samples for total suspended solids (TSS) and total dissolved solids (TDS) at the laboratory (see project-specific SAP).

Use the same technique described above to collect water for compositing surface water collected at horizontally integrated near-surface and near-bottom stations.

EQUIPMENT AND REAGENTS REQUIRED

This section describes the general types of required equipment and reagents. Attachment 1 provides a detailed supply and equipment list. Additional equipment may be required depending upon project-specific needs.

Use one or two peristaltic pumps at each sampling station (near-surface and near-bottom) for collecting surface water samples. To collect unfiltered and filtered split samples from the mixing containers, use the same pump that is used to fill the mixing containers. Use a sample

processing and preservation chamber (i.e., workbox) made of PVC pipes and 6-mil plastic sheeting to house stir plate(s), a peristaltic pump, sampling bottles, and ancillary equipment. Place a polycarbonate, glass, or stainless-steel mixing container (10 or 20L) on the stir plate. Each mixing container is equipped with a 3-in.-long Teflon™-coated stir bar at the bottom and a lid containing inflow, outflow, and vent Teflon™ spouts (Figure 2). For each sampling station, assemble a filtering kit (laboratory precleaned 0.45-µm filter with C-Flex™ and Teflon™ tubing placed in a double Ziploc™ bag) and attach it to a peristaltic pump and mixing containers. If necessary, attach a precleaned 10-µm prefilter inline to prolong the life of the 0.45-µm filter. You will need the following equipment:

- Peristaltic pump
- Surface water parameter multimeter capable of measuring pH, reduction/oxidation (redox) potential, temperature, specific conductance, turbidity, and dissolved oxygen
- PVC pipes and plastic sheeting
- Polycarbonate (inorganic analyses) and/or glass or stainless-steel (organic analyses) mixing containers (see project-specific SAP for analyte list)
- Sample tubing (type and length are site-dependent)
- Stir plate with Teflon™-coated stir bar
- 0.45-µm filter with C-Flex™ and Teflon™ tubing (if needed; see project-specific SAP to determine if filtered samples are required)
- Water Sampling Log forms (attached)
- Sample tags/labels and appropriate documentation (e.g., chain-of-custody forms)
- Insulated cooler(s), chain-of-custody seals, Ziploc® bags
- Sample containers with preservative, coolers, and blue ice or equivalent.

PROCEDURES

The sampling team should comprise three people. Two are needed to conduct the sampling and a third must keep track of sample logging and processing. In addition, the third person may be responsible for collecting the surface water quality parameters.

Equipment Preparation

Bring enough decontaminated sampling tubing and filtering kits to the field to avoid performing decontamination procedures between stations. Each participating laboratory is responsible for preparing its equipment prior to the sampling cruise. Predesignated commercial laboratories will decontaminate sample tubing, mixing containers, and sampling bottles according to their specific SOPs.

Note: Decontamination of large amounts of sampling equipment requires several days, if not weeks, to be ready for sampling. Contract agreements with commercial laboratories and scheduling decontamination work may require several weeks to months. Initiate this critical step as early as possible.

The main components of the peristaltic pump sample collection system are as follows:

- **Processing and Preservation Chamber**—Build a workbox with ¾-in. PVC tubing and cover it with a 6-mil plastic sheet in order to contain the peristaltic pump sampling equipment and conduct the subsampling from the carboys. Leave one side of the workbox open for placing sampling equipment and sample containers. Wash all components with Alconox™ and rinse with tap water. To secure the receiving Teflon™ tubing and filter cartridge, use stands and clamps made of non-metallic components or resin-coated stainless steel, which have been washed with soap, rinsed in tap water, washed in acid, and rinsed with distilled/deionized water.
- **Water Sampler**—The water sampler device for near-surface sampling should be made of PVC tubing with a polyurethane-coated 50-lb weight at the bottom to keep the sampler in the vertical position (Figure 1a) (Note: To reduce potential drag at the water surface, do not include a base on the near-surface sampling device.) The near-bottom sampling device should also be made of PVC tubing and have a polypropylene vane, constructed with a weighted base (Figure 1b). Both sampling devices should be attached to the boat by a Technora™ or Kevlar™ rope. Figure 1b shows the sampling device with a YSI water quality multimeter and underwater camera attached to it. Figure 1b also shows how the Teflon™ tubing is positioned on the vane and the relationship of the inlet to the water sampler. The vane works to keep the water intake into the flow and elevated at a constant height from the bottom. Prior to commencement of sampling activities, wash all components with Alconox™ and rinse with tap water.
- **Water Quality Meter**—Use a YSI 650/6600 multi-probe (newer model or similar) for measuring surface water parameters, such as temperature, pH, dissolved oxygen, conductivity, oxidation-reduction potential (ORP), and turbidity. Attach it to the water samplers as shown in Figures 1a and 1b. The unit will come pre-calibrated from the laboratory and will be checked daily for proper functioning and drift. However, the must be calibrated daily for certain parameters such as pH, conductivity, ORP, and dissolved oxygen If possible, install a YSI unit on each water sampler (i.e., near-surface and near-bottom) if both are deployed at the same time. A YSI unit installed on the near-bottom water sampler can also take an initial near-surface measurement at the beginning and at the end of the sampling event, therefore avoiding the cost of having to install an additional YSI unit on the near-surface water sampler. The proper handling of the multi-probe is described in detail in SOP SW-06. Except for the probe sensor, wash all components with soap (Alconox™) and rinse with tap water. Because

this equipment will not be in the pathway with the surface water being collected, there is no need for a thorough decontamination.

Take the following steps to set up the surface water collection system:

1. Assemble and secure the water samplers to either the A-frame or a davit.
2. Determine the correct position of the sampling station, have the captain anchor the vessel into the current at the sample site, and switch off the engines. If anchoring is not possible and the engine must be on, make sure the water intake tubing is always facing into the current.
3. Set up a clean area for the workboxes. Set workboxes on a secure table or bench top onboard the sampling vessel to house stir plate(s) and a small peristaltic pump in each workbox. Provide enough space inside the workboxes for a stand to hold the outlet tubing and filter (if necessary; see project-specific SAP) and to collect surface water and processing sample bottles (Figure 3).
4. Place stirring plate(s) inside the workbox and the mixing container(s) on top of the plate. Check each mixing container (a polycarbonate container for inorganic analytes and a glass or stainless-steel container for organics) to ensure:
   - Containers were properly wrapped by the laboratories and are free of rips or holes that may have occurred during shipment to the field.
   - Each container contains a 3-in. stir bar at the bottom.
   - All components such as inflow and outflow tubing have been properly assembled in the laboratory (e.g., one end of the outflow tubing should be touching the bottom inside the container), and that they are intact and securely placed on the cap.
5. Attach the outlet tubing "kits" (i.e., Unit # 3) to the mixing containers (Figure 2). The kits are composed of 10-cm C-Flex™ tubing, 0.5-m Teflon™ tubing, 30-cm C-Flex™ tubing, and 30-cm Teflon™ tubing, placed sequentially.
6. Place the small peristaltic pump inside each of the workboxes.
7. Place a stand inside the workboxes and secure each tubing outlet from both mixing containers with clamps (Figure 3).

8. Attach Teflon™ tubing (collecting end) to 30-cm C-Flex™ tubing and 1-m Teflon™ tubing, sequentially, and then connect these interconnected pieces of tubing to a mixing container (polycarbonate for inorganics and glass or stainless steel for organics). Clamp the C-Flex™ tubing section firmly into place inside the large peristaltic pump head, which is placed outside the workbox. (Note: The length of the Teflon™ tubing will vary depending on project-specific requirements and water depth at a given station. For example, 4 m of Teflon™ tubing could be used for near surface sampling and 25 m could be used for near-bottom sampling)
9. Attach the intake part of the Teflon™ tubing to the vane of the near-surface sampler (Figure 1a) or to the vane of the near-bottom sampler (Figure 1b). Take care not to remove the protective cap from the tip of the sample collection tube until the sampling device is ready for submersion.
10. Secure the pump and pump speed controller, and connect them to the vessel's power source with an extension cord. If vessel power is not available, use the pump's battery power supply.
11. To limit sediment suspension during near-bottom sampling, tether a submersible, underwater video camera to the boat and attach it to the sampling device vane to reveal when the sampling device touches bottom.

Sample Collection

Take the following steps to collect and process the surface water samples:

1. Remove the protective cap from the sampling tube and lower the sampler gently below the water surface.
2. To sample water near the surface, submerge the sample tubing inlet approximately 1 m (3 ft) below the surface of the water column (consult project-specific SAP for exact sampling water depth).
3. To sample water near the bottom, submerge the sample tubing inlet approximately 3 m (9 ft) above the bottom (consult project-specific SAP for required distance) with the help of the A-frame or davit. If it is necessary to sample surface water at a fixed depth from the bottom, adjust the vane height on the sampler while the sampler rests on the bottom surface. The vane will maintain the sample tubing inlet into the current at a constant depth between 30 cm (12 in.) and 1 m (3 ft) above the sediment–water interface.

4. Begin collecting measurements of water quality parameters at each depth using the water quality meter (e.g., YSI, Hydrolab, Horiba). Set data collection intervals according to data needs. If a vertical water column profile is needed, set the multi-probe to collect data every 1 second for a high-resolution profile. If sampling a vertically integrated water column with several round trips to the bottom, reset the multi-probe to collect data at time intervals relative to the sampling time period after the initial high resolution profile. For example, if sampling a vertically integrated water column during a period of 2 hours, set the multi-probe to record data every 1 second for the first roundtrip to the bottom and then reset to record data every 5 minutes for the subsequent roundtrips until sampling is complete. If collecting surface water at a stationary location for more than 1 hour, and no major changes in water quality are expected, set the multi-probe to collect data every 15 minutes.
5. Note: Failure to adjust the multi-probe for data collection according to sampling periods can result in data loss. That is, if the multi-probe memory bank is quickly filled early in a long sampling period, no additional data will be stored in the memory bank for the remaining sampling time.
6. Record the water quality measurements on the Water Sampling Log forms every 15 minutes during sample collection. If the surface water sample collection is completed within 15 minutes, then collect water quality parameters at least three times: at the beginning, middle, and end of sample collection.
7. Switch the pump on and pump surface water through the sample tubing and into the mixing containers. Once the water reaches one-third of the container's volume, turn on the stir plates.
8. Turn off the pump once the mixing containers have been filled to 1 in. below the inflow spout or when sufficient volume has been collected to fill all of the sample bottles at a given station.
9. Place the C-Flex section of the outflow tubing kit from the first container to be sampled inside the small peristaltic pump head and clamp firmly.
10. Before turning on the small pump, make final adjustments to the stand, holding the outflow spout as close as possible to the sample bottle opening, but without touching the inside of the bottle.
11. Fill container to the "neck" with unfiltered sample water.
12. After collecting the unfiltered samples, attach the 0.45-µm filter cartridge (or appropriate pore size filter) to the sample tubing outlet and secure it to the stand with a clamp (consult project-specific SAP to determine if filtered samples are required). Drain the storage solution inside the filter, and flush the entire sample tubing and filter assembly with sample water. Discard this first "rinse" of sample water.

13. After rinsing the filter and sample tubing, fill the sample bottle to the "neck" with filtered sample water. (Note: If dissolved constituents are being analyzed [per the project-specific SAP], then discard the 0.45-µm filtration cartridge after each sampling site.)
14. As soon as a sample container is filled, turn off the peristaltic pump and label the container. Include the date, time, project name or number, sample ID, type of analysis required, and sampler initials on the label (see SOP AP-04).

Once a surface water sample container is properly closed and labeled, place it inside a cooler containing wet or blue ice and store it at approximately 4°C. Store all samples in coolers with ice on board the vessel and transfer them to the field laboratory (if applicable) at the end of the sampling day.

Water Quality Measurements

If specified in the project-specific SAP, measurements of physical and chemical water parameters may need to be collected at surface water stations. Several physical and chemical water parameters are best measured in the field because of the unstable nature of the parameter or because the information is needed to direct further sampling. It is frequently preferable to perform these analyses in the field, especially if the samples will not be immediately transported to the analytical laboratory (pH, in particular, should be measured in the field if feasible). In addition, measurements of temperature and transparency can be accurately collected only in the field.

It is always best to place the water quality meter directly into the surface water body at the station location at the desired water depth instead of collecting a sample and measuring parameters in a container. However, if this is not possible, use a plastic bucket to collect samples for water quality analyses (e.g., pH, temperature, and conductivity). Rinse a clean bucket twice with the water from the station prior to measuring water quality parameters.

The name(s) of the person(s) making the measurement and the field equipment used to make that measurement must be recorded in the field logbook and on any field forms used during the sampling event. Equipment maintenance and calibration records must be kept in logbooks and field records so that the procedures are traceable.

Sample Handling

Gloved hands are required for sample collection and handling, as described above. Field staff will wear appropriate non-contaminating, disposable, powderless nitrile gloves during the entire sampling operation. Change gloves frequently, usually with each change in task (wearing multiple layers of gloves allows rapid glove changes).

Gloved hands are required for all operations that involve equipment that comes into contact with the sample, including the following activities:

- Handling the sample bottle
- Handling the discharge end of the sample tube or line
- Setting up working space inside the processing and preservation chambers
- Setting up the equipment (i.e., the sample bottles, mixing containers, and the filtration and preservation equipment) inside the chambers
- Working inside the chambers during collection, processing, and preservation
- Handling the filter (if needed)
- Changing the chamber covers as needed.

Ungloved hands take care of all operations that involve contact with potential sources of contamination, including the following activities:

- Working exclusively exterior to the processing and preservation chambers
- Preparing a clean workspace (inside boat)
- Preparing and operating the sampling equipment, including the pumps and discrete samplers, peristaltic pump switch, pump controller, and manifold system
- Handling the generator or other power supply for samplers
- Handling the tools, such as hammers, wrenches, keys, locks, and sample-flow manifolds
- Handling the single or multi-parameter instruments for field measurements
- Setting up and checking the field-measurement instruments
- Measuring and recording the water depths and field measurements.

Store all samples in coolers with ice at approximately 4°C on board the vessel and transfer them to the field laboratory (if applicable) at the end of the sampling day. The sampling team leader is responsible for maintaining sample integrity throughout the sampling event.

If storage freezers or refrigeration units are available at the field laboratory, monitor these units daily to ensure temperature compliance. Each unit will have a separate log form containing date, time, and temperature information.

Avoid contaminating samples by handling the sample containers with clean gloves and transferring the samples into clean refrigerators (or clean coolers) immediately after the samples have been brought back from the field. Always wear disposable, powderless nitrile gloves when handling samples. This includes any and all sample handling that may occur during sample packing and shipping (see SOP AP-01).

RELATED SOPS

- Pack and ship all surface water samples in accordance with procedures outlined in SOP AP-01.
- Record field activities in accordance with procedures outlined in SOP AP-02.
- Maintain sample custody in accordance with procedures outlined in SOP AP-03.

REFERENCES

David, N., D. Bell, and J. Gold. 2001. Field sampling manual for the Regional Monitoring Program for Trace Substances. San Francisco Estuarine Institute, San Francisco, CA.

USEPA. 1996. Method 1669 – Sampling ambient water for trace metals at EPA water quality criteria levels. U.S. Environmental Protection Agency, Office of Water Engineering and Analysis Division (4303). Washington, DC.

USGS. [various dates]. National field manual for the collection of water-quality data: U.S. Geological Survey techniques of water-resources investigations, Book 9, Chap. A1-A9. Available online at http://pubs.water.usgs.gov/twri9A. U.S. Geological Survey. Accessed February 5, 2008, at http://water.usgs.gov/owq/FieldManual/index.html#Citation.

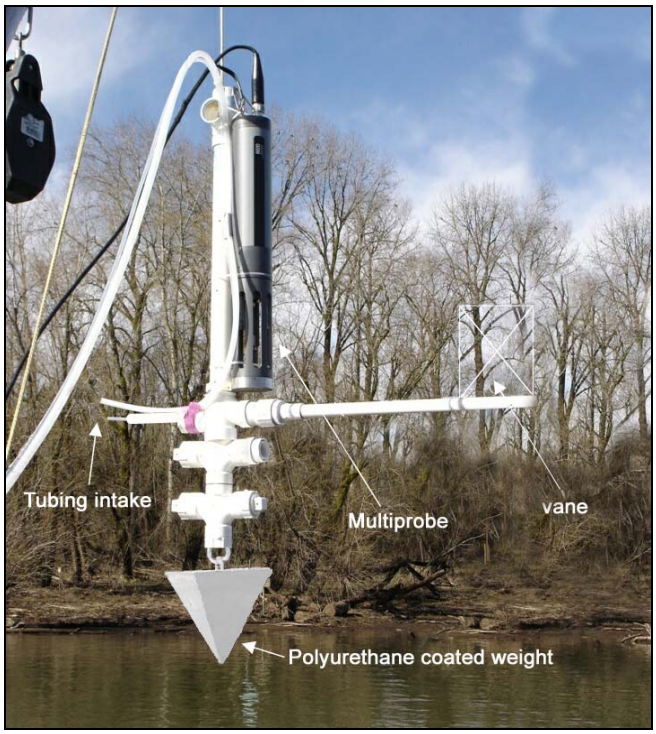


Figure 1a. Near-Surface Water Sampler

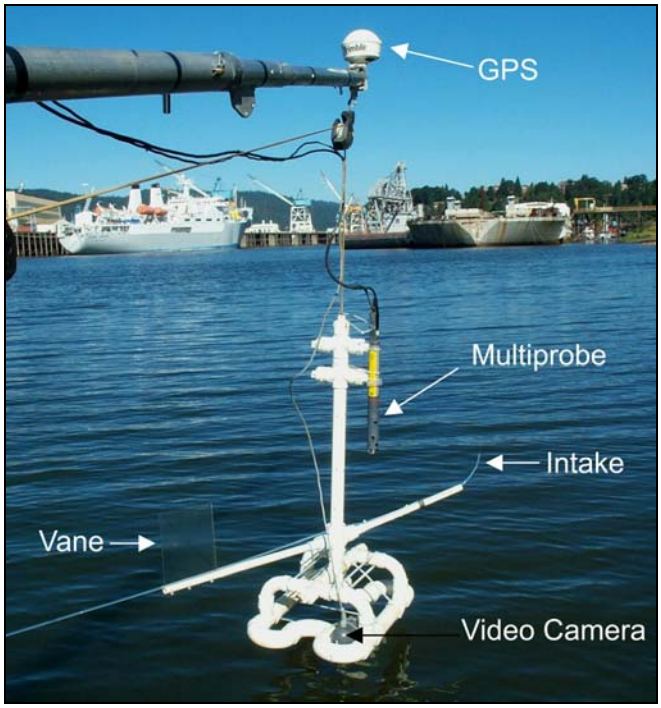


Figure 1b. Near-Bottom Water Sampler

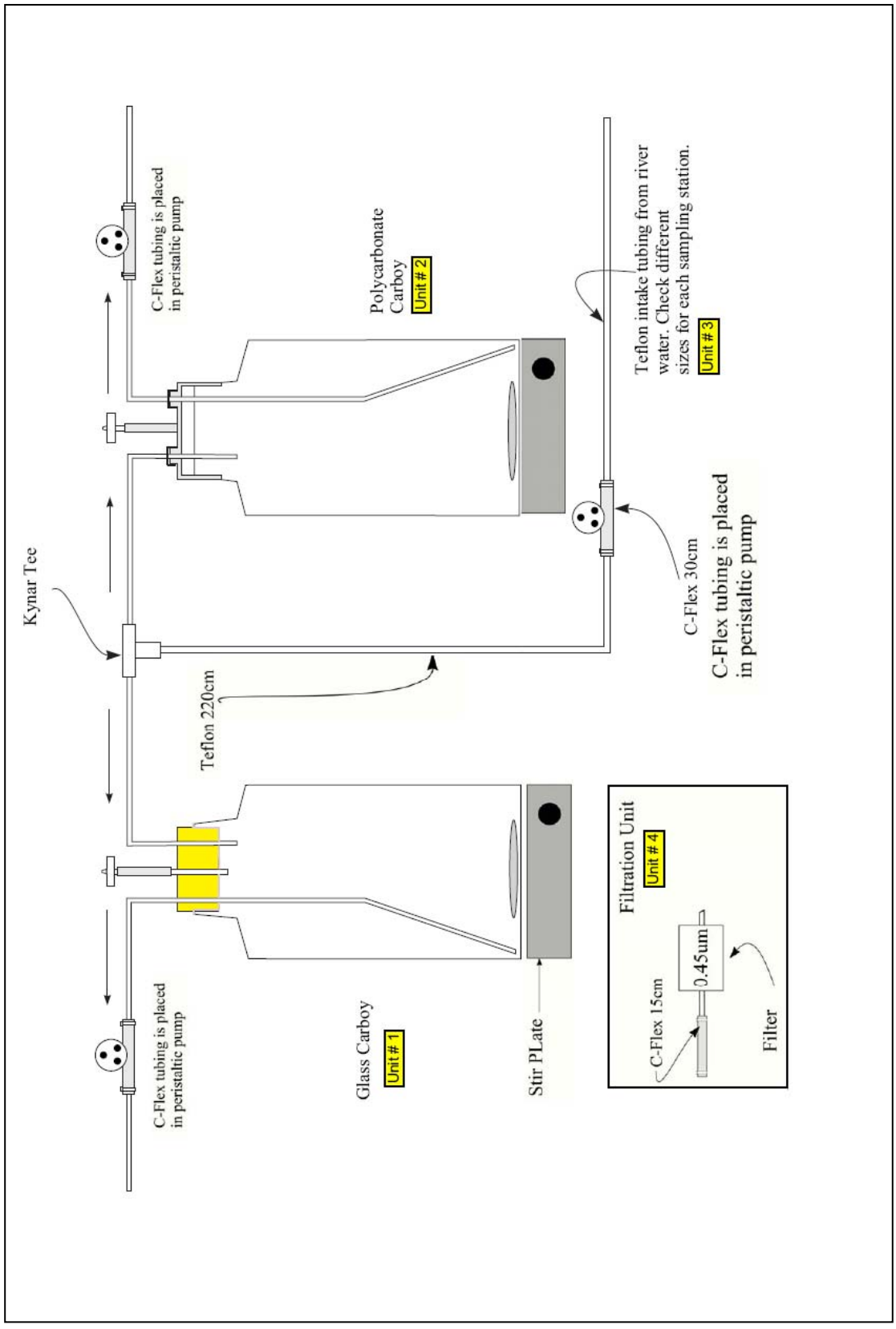


Figure 2. Peristaltic Pump Sampling Apparatus

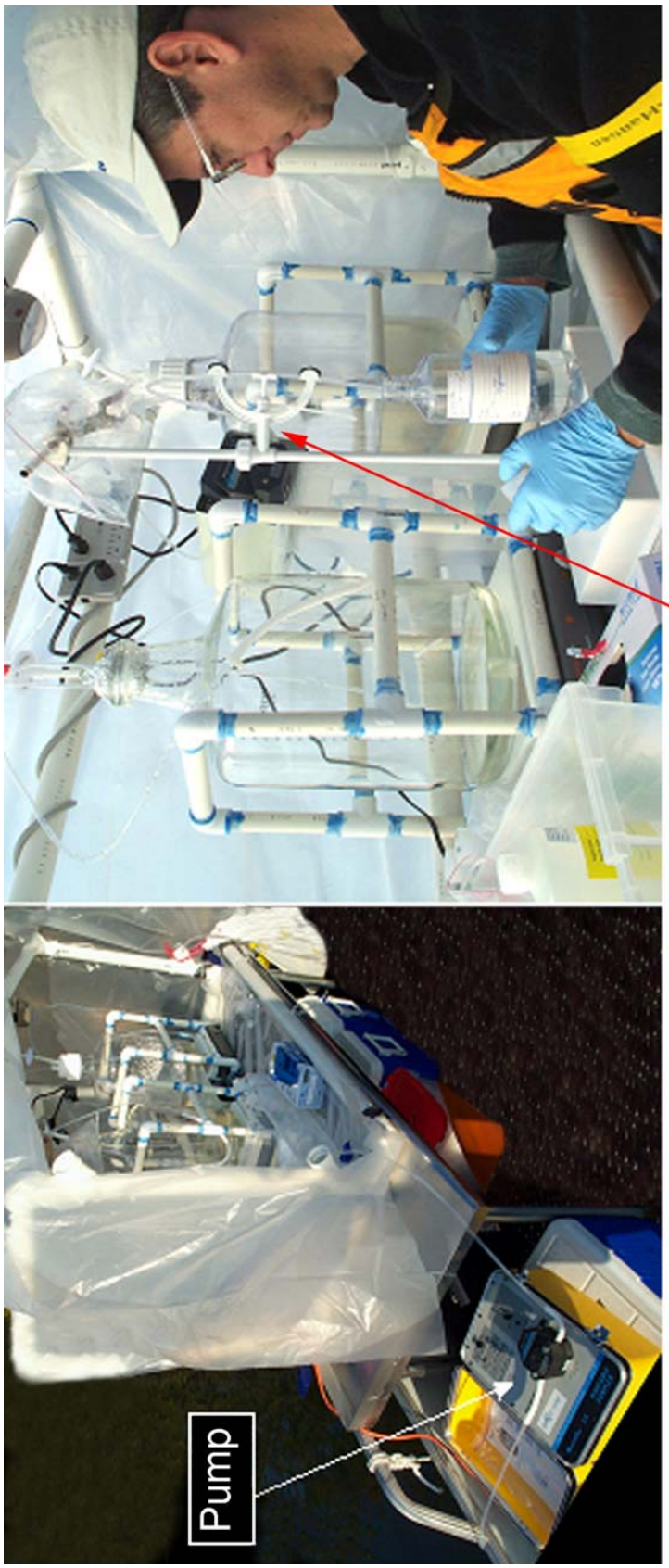


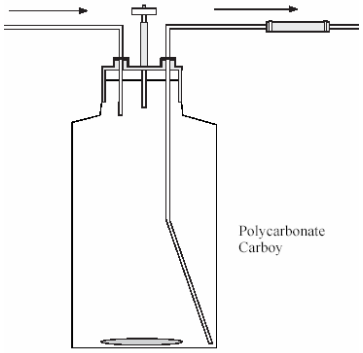
Figure 3. Peristaltic pump setup in workbox.

ATTACHMENT 1. CHECKLIST OF SUPPLIES FOR SURFACE WATER SAMPLING WITH PERISTALTIC PUMP

All sampling equipment described here will be sent to Battelle Marine Laboratories at Sequim, Washington, or other approved laboratory for decontamination and assembly prior to sampling. Each unit below shall be wrapped in plastic bags and clearly labeled on the outside in large letters.

UNIT #1

For polycarbonate carboys



Teflon

50 cm	for inflow from Kynar tee into carboy
140 cm	for outflow from carboy to small peristaltic pump
60 cm	for outflow from small peristaltic pump to sample bottle

C-Flex

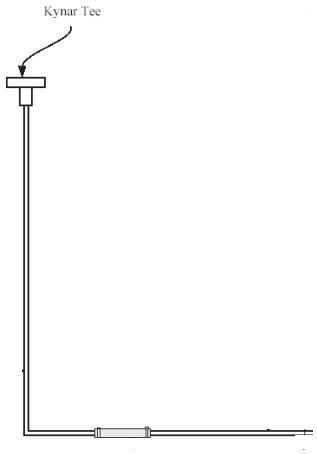
6 cm	for connecting air filter on carboy (additional internal tubing is not needed)
30 cm	for connecting outflow tubing from carboy to tubing for filling sample bottles

Other

3-in. stir bar
Vacu-guard filter
2 small plastic zip-ties for C-Flex tubing

The total number of bags labeled UNIT #1 will depend on the number of sampling stations per specific sampling event.

UNIT #2



From sample intake tubing to large peristaltic pump to carboys

Teflon

220 cm — for inflow from large peristaltic pump to Kynar tee

C-Flex

30 cm — for connecting Kynar tee and inflow tubing to variable lengths of sampling intake tubing

Other

Kynar tee — for connecting carboys to intake tubing

Variable lengths of sampling intake tubing (station dependent) to large peristaltic pump

Teflon

40 to 100 m — for near-bottom sampling at transect stations

8 m — for near-surface water sampling at any station

15 m — for near-bottom sampling at shallow stations

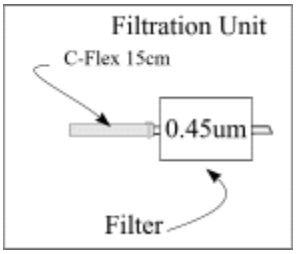
The total number of bags labeled UNIT #2 will depend on the number of sampling stations per specific sampling event.

UNIT #3

Set of one filter in line

C-Flex

15 cm — for connecting the filter to the outflow from small peristaltic pump to sample bottle



Filter

0.45 μm — Whatman POLYCAP 36 TF

Other

Small plastic zip-tie for C-Flex tubing

Loose small plastic zip-tie (extra zip-tie to be placed in bag to connect to carboy outflow)

The total number of bags labeled UNIT #3 will depend on the number of sampling stations per specific sampling event.

YSI WATER QUALITY PARAMETERS SAMPLE LOG

Survey _____ Crew _____ Date _____

Station	Time	Temp (C)	Conduc-tivity (µS/cm)	DO (mg/L)	Depth (m)	pH	ORP (mV)	Turbidity (NTU)	Comments/Observations

STANDARD OPERATING PROCEDURE (SOP) SW-05

SURFACE WATER SAMPLING USING GRAB SAMPLERS

SCOPE AND APPLICATION

This SOP describes methods for sampling surface water from freshwater bodies (e.g., creeks, rivers, lakes, ponds) or from estuarine and marine systems (e.g., estuaries, embayments, open ocean). These methods were developed based on Greenberg et al. (1985) and USEPA (1991). Not all of the sampling methodologies discussed in this SOP may be required for a given project. The specific sample collection techniques and associated sampling equipment will be detailed in the project-specific sampling and analysis plan (SAP).

STATION ACCESS

Prior to entering select areas, it may be necessary to acquire property access permission from the landowner. Access permission must be acquired in advance of the sampling program and may require a written agreement. Surface water stations may be accessed by boat, wading, or standing on the shore or other structure (e.g., bridge) and extending a sampling device into the water body.

STATION LOCATION

When sampling in proximity to the bottom of a water body, surface water samples must be collected in a manner that avoids resuspending sediments into the sample being collected or samples that will be collected at other downstream or downcurrent stations. Therefore, if it is necessary to enter the water to sample (i.e., wade), care must be taken to avoid resuspending sediment into the water column. To avoid resuspended sediment interference in the sample being collected, stations should always be approached from downstream. Avoid sampling near eddies that may circulate water from the sampling location to upstream of the sampling location. To avoid interference from resuspended sediment at other stations, samples should be collected beginning with the most downstream station and continuing in an upstream direction.

Water samples should be collected in areas that are representative of the surface water body conditions. A station that is located away from immediate point sources (e.g., tributaries and industrial and municipal effluents) is preferred for collecting surface water samples unless

sampling is designed to assess these sources. Representative samples can usually be collected in portions of the surface water of a river that have a uniform cross-section and flow rate. Because mixing is influenced by turbulence and water velocity, the selection of a site immediately downstream of a riffle area (e.g., fast flow zone) will ensure good vertical mixing.

Whenever possible, a depth-integrated sample (as well as a width-integrated sample) should be collected from flowing water bodies. This is particularly true for data that will be used in mass balance calculations. When sampling is performed to assess the effects of a tributary or discharge on receiving water chemistry, some calculations may be needed prior to sampling to estimate appropriate distances downstream and across the stream to characterize concentrations. It is common for streams to be incompletely laterally mixed for downstream distances of 50 times the stream width, or more. Guidance on mixing rates can be found in *Mixing in Inland and Coastal Waters* (Fischer et al. 1979).

Tributaries should be sampled as near the mouth as is feasible. However, it is important to select the sample location taking into consideration the impact that the downstream receiving water body has on the tributary flow and sediments. The downstream body may affect the tributary by decreasing water velocity (causing suspended solids to settle) or by eddies (causing mixing of the two waters). The downstream water body may change the water quality (e.g., salinity), temperature, or turbidity in the tributary near its mouth.

Attention must be given to identifying intakes and outflows within lagoons or settling ponds, which may cause localized concentrations that are not representative of general conditions. Sample locations adjacent to structures (e.g., banks, piers) may also have biased characteristics as a result of flow or release of substances from in-water structures. These kinds of possible confounding factors should be noted in the field logbook. For ponds, lakes, and large rivers that may be vertically stratified, a multi-parameter water quality meter can be used to collect depth profiles throughout the water body to aid in the selection of appropriate sampling points and depths.

SURFACE WATER SAMPLE COLLECTION

Appropriate surface water sampling techniques and equipment are designed to minimize effects on the chemical and physical integrity of the sample. Different kinds of surface water sampling techniques and equipment are discussed in the following sections. The project-specific SAP should be consulted to determine the appropriate surface water sampling techniques and associated sampling equipment.

In general, if both surface water and sediment samples are to be collected from the same location, the surface water samples should be collected first. Every attempt should be made to keep floating debris from entering the sample bottles, which could result in unrepresentative analytical data. Sample collection when the flow depth is minimal (i.e., less than a few inches) will require special consideration to prevent sediment disturbance. If water depth is shallow

and sampling equipment will come into contact with the sediment surface, then a small excavation in the stream bed to create a "sump" for sample collection may be permissible, but it should be prepared well in advance of the sample collection event to allow sediment to settle. This technique should be considered very carefully because digging a depression may expose the surface water to other possible contaminants and natural compounds such as sulfides, phosphates, and ammonium. In addition, certain sediment types will crumble as a hole is dug up and will not support enough depth for bottle dipping. A peristaltic pump may be needed (consult project-specific SAP and field conditions).

As mentioned above, surface water samples shall be collected moving in an upstream or upcurrent direction, in accordance with the following procedure:

1. Immediately after collecting the sample, record the temperature, dissolved oxygen, pH, turbidity, and specific conductance using a water quality meter (e.g., YSI, Horiba®, or equivalent) and following the manufacturer's specifications.
2. If stipulated in the project-specific SAP, collect a water depth measurement at every surface water station.
3. Target analytes, container types, and preservatives are specified in the project-specific SAP. In general, when collecting surface water samples for a variety of analyses, collect them in the following order:
   - Samples for analysis for volatile organic compounds (VOCs)

     Note: When collecting samples for VOC analysis, let the water flow down the side of the sample container to minimize aeration. Hold caps in hand to minimize contamination of sample. Fill all VOC sample containers to the top. A positive meniscus at the top of the container will help ensure that no air is trapped inside when cap is screwed down on the container. No air bubbles should be trapped in the sample when the container is sealed. VOC sample bottles must be checked after filling to ensure no air bubbles are present. Invert the bottle and lightly tap it to release any bubbles beneath the cap. If an air bubble is present, the VOC sample must be retaken using a fresh bottle.
   - Samples for analysis for dissolved gases and total organic carbon
   - Samples for analysis of semivolatile organic compounds
   - Samples for analysis of metals and cyanide
   - Samples for analysis of major water quality cations and anions
   - Samples for radionuclides.

4. Collect quality assurance and quality control (QA/QC) samples (i.e., duplicate, equipment rinsate, trip blank, laboratory matrix spike, and laboratory matrix spike duplicate, as applicable) at the same time by filling all bottles from the same flow. The number and types of QA/QC samples are specified in the project-specific SAP.
5. Label sample bottles with the date, sample number, time, sampler's name, and type of preservative, as described in the project-specific SAP and in accordance with SOP AP-04. Sample bottles must be placed in a cooler and on ice to keep the sample cool (4°C). Samples must be cooled continuously from time of collection to the time of receipt at the laboratory, as described in SOP AP-01.
6. Complete sample logs, labels, custody seals, and chain-of-custody forms. Record sample information in the field notebook. The depth in the water column where the sample is collected *must* be recorded in the field logbook.

Dipping Using Sample Analysis Bottle

In some cases, water is collected directly from the water body into the bottle that is sent to the laboratory for analysis. This surface water sampling technique is appropriate only if a composite sample is not required for analysis (i.e., only filling one sample bottle per station). If compositing is required, then a decontaminated churn splitter or mixing container will need to be used (see discussion below).

When collecting samples in a riverine environment, approach the station from downstream of the sampling location and face upstream to collect the sample without disturbing the sediment. Using a bottle attached to a dip stick or wearing nitrile gloves to hold the bottle, quickly immerse the inverted sample bottle through the surface of the water to the desired sampling depth and then tilt the opening of the bottle upstream to fill. If possible, samples should be collected approximately one-third of the distance from the surface to the bottom, and the sample bottle should be completely submerged. Note: If collection is done too slowly, the film at the surface of the water will be collected; water must be collected from below the air–water interface.

Be careful not to displace the preservative from a pre-preserved sample container (e.g., VOC vial). If water is needed to fill bottles that contain preservative, then collect water with a clean bottle and pour this water into the sample bottle that contains the preservatives.

Specific Depth Interval Sampler

If surface water samples are required from a specific depth, a standard Kemmerer, GO-FLO™, Niskin bottle, or Van Dorn sampler, or plastic tubing with a peristaltic pump may be used. The Kemmerer, GO-FLO™, and Niskin bottle samplers are stainless steel or acrylic cylinders with closures at each end that leave the ends of the sampler open while being lowered in a vertical position through the water column to allow free passage of the water through the

cylinder. The Van Dorn sampler is similar in construction, but is lowered in a horizontal position through the water column. In each case, the sampler is lowered to the desired depth and a messenger is sent down a rope or cable that causes the sampler to close. The sampler is then raised to the surface. Water is removed through a valve to fill sample bottles. If the sampler needs to be reused during the sampling event (e.g., at different stations), then the sampler must be decontaminated between stations.

Note: The analyte list in the project-specific SAP should be reviewed prior to sample collection to determine if a Teflon™-coated sampler is required.

GO-FLO™ Bottle Sampler

GO-FLO™ bottle samplers are fabricated by General Oceanics Inc. 1295 N.W. 163rd Street, Miami, Florida 33169 USA. The specific model depicted here is a 20 L GO-FLO™ 1080 series (General Oceanics, http://www.generaloceanics.com/genocean/1080.htm)**.**

Description: Close-open-close operation. Opens automatically (hydrostatic pressure activated) at approximately 10 m (33 ft), and then flushes until closed by standard GO Devil messengers (Model 1000-MG) individually, serially, or sequentially by remote command with Model 1015 Rosette multi-bottle array, or with Model AR1015 Acoustic Command Control. (See data sheet 1015-12/85.) Inert gas can be injected into bottle to force retrieved sample out of sampling valve, directly through filter system. The GO-FLO™ sampling bottle avoids sample contamination at the surface, internal spring contamination, loss of sample on deck (internal seals), and exchange of water from different depths.

Material: Rigid PVC tube section, ball valve, handles and cable clamp blocks. Delrin stopcocks and push rod. Stainless steel cable clamp bolts. Latex external spring, Viton and silicone O-ring seals. Monofilament nylon and Kevlar lanyards.

Inert Gas Connection: Dual-purpose air vent pressure release valve with standard 9/16-18 thread for connecting inert gas line. (Operation requires additional fitting - part S1080-AFIT.)

Closure: Ball valve with Viton and silicone seals.

Health and Safety: All hands on deck must be wearing appropriate personal protective equipment (i.e., hard hats, personal flotation device, steel-toed boots, and gloves) (Figure 1). People in charge of handling the GO-FLO™ bottle must have read the instruction manual and be familiar with the proper use of the sampling equipment, must have read the project-specific health and safety plan, and must have participated in a safety meeting debrief on the hazards associated with sampling equipment of the specific research vessel used for that project.

Figure 1. Proper Personal Protective Equipment

Trace Metal Analyses: If trace metal analyses are required, the interior of the GO-FLO™ bottle must be Teflon™ coated. Decontamination procedures for trace metals are described in Mason and Sullivan (1996) and are outlined in SOP SW-15 for laboratory decontamination and SOP SW-01 for field decontamination. Decontaminated GO-FLO™ bottles must be wrapped in plastic bags to prevent any contamination from airborne dust particles or exhaust fumes from power generators and boat engines. Additional plastic bags or clean nitrile gloves must be wrapped around the sampling spigots and removed only at the last minute before deployment. A non-metallic wire (e.g., Technora™, Kevlar™) must be spooled on a winch that will deploy the GO-FLO™ bottle. The non-metallic wire must be rated to safely support the weight of a bottle filled with water plus ancillary equipment such as bottom weight, water quality multiprobe meter, underwater video camera, and stainless steel frame if multiple bottles are deployed at the same time. Prior to the collection of water samples, GO-FLO™ bottles are checked for defects and to ensure the opening and closing mechanisms are operational.

Pre-deployment Checks: The bottle must be secured in a transport box or shipping crate that can also be used as a secure platform for deployment and retrieval of the unit. The GO-FLO™ bottles are pre-cocked before deployment. During the cocking procedure, the GO-FLO™ bottle is either placed on a clean plastic sheet/bag on the floor or handheld if it is a small volume bottle (i.e., less than 20 L). The cocking procedure consists of the following:

- Rotate the bungee cord attached to the ball valve so that the plastic ball string is loose. Figure 2 shows how the bungee cord is wrapped around the ball valve wheel in order to loosen tension on plastic ball string. In this instance, residual water flows out of the bottle.

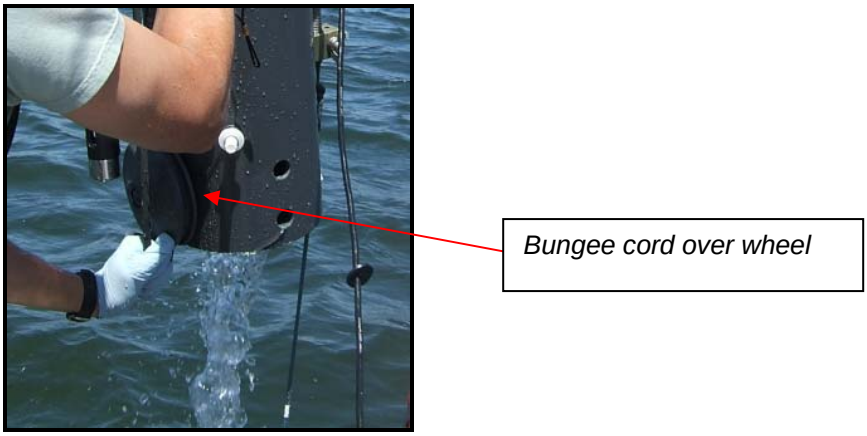


Figure 2. Release Tension on Ball Valve

- Position the plastic balls on the string around the pressure release valve and pull the pressure release valve outward locking the two plastic balls between the valve and the U-shaped stainless steel wire located on the middle of the bottle just below one of the PVC handles (Figure 3).

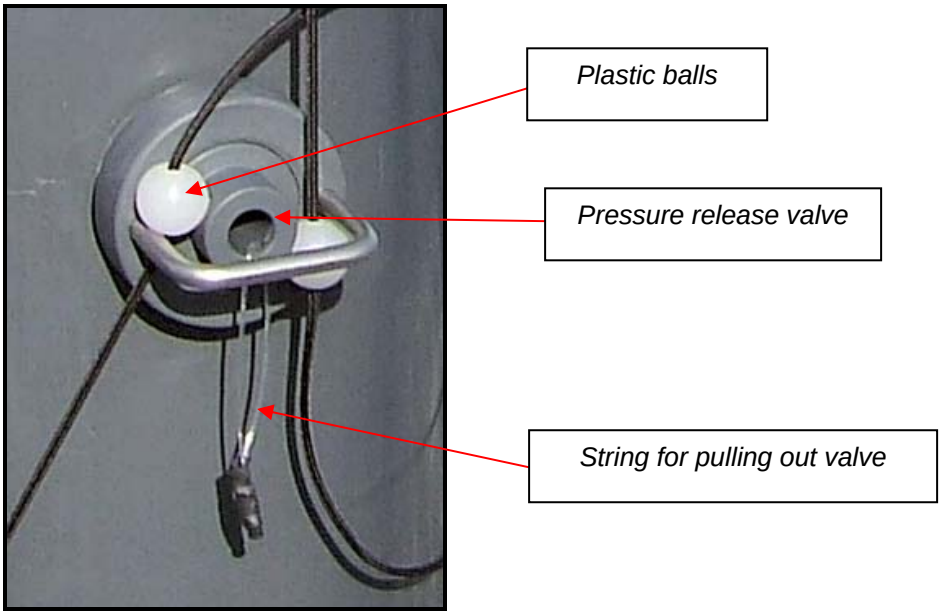


Figure 3. Pressure Release Valve in Cocked Position

- Rotate the bungee cord attached to the ball valve back so that both the string and the cord are under tension (Figure 4).

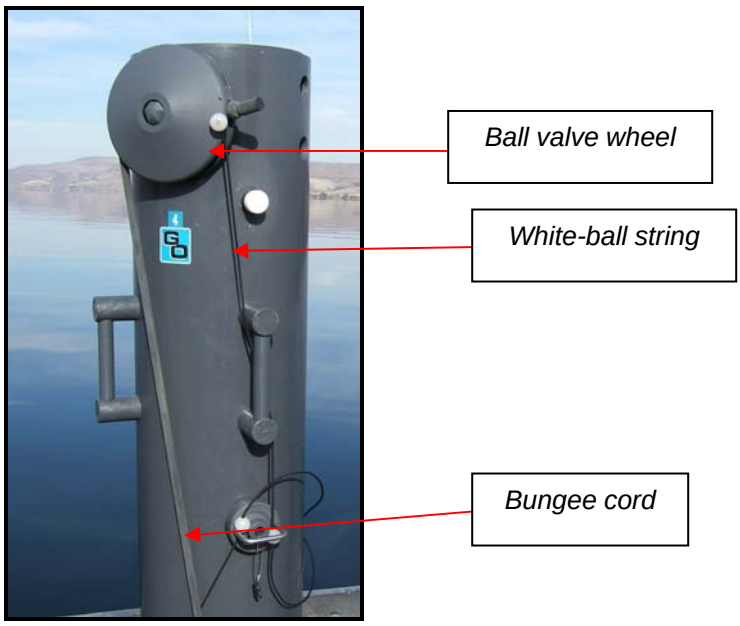


Figure 4. Bungee Cord on Cocked Position

- Perform the following checks to ensure the GO-FLO™ sampler has been properly cocked.
  - Push the pressure release valve and ensure the balls' valves move to the open position.
  - Press the push rod release mechanism to release the string, which should cause the bottle to close (Figure 5).

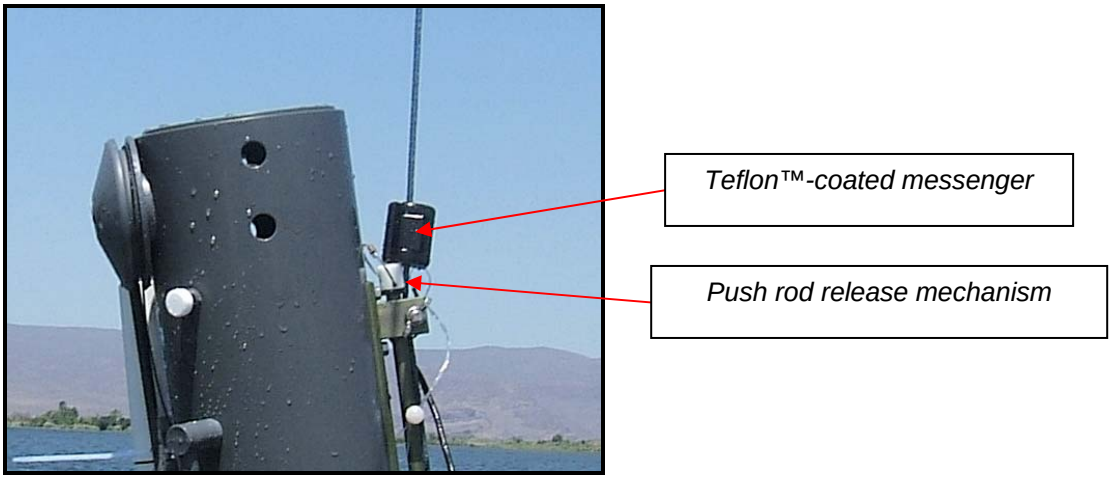


Figure 5. Push Rod Triggering Mechanism with Messenger Deployed

- Recock the bottle after this check as described earlier.
- Check that vent valve is turned all the way in and that sampling spigots are pulled out and white flange twisted away from release pin before deployment (Figure 6).

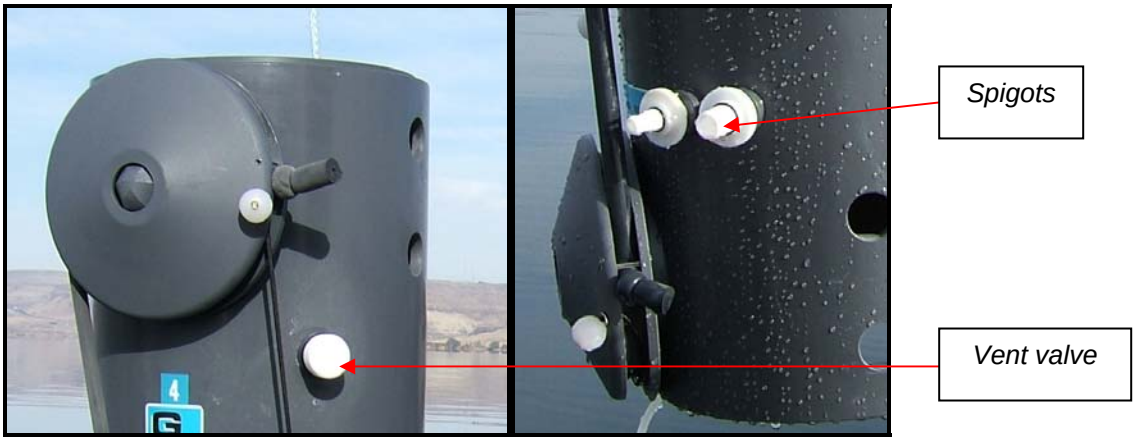


Figure 6. Vent Valve at Top End of Bottle and Sampling Spigots at Bottom End of Bottle

Deployment: Before deploying the sampler, make sure any ancillary equipment, such as multiprobe (Figure 7) and underwater video camera, is attached to the bottle and a non-metallic or polyurethane-coated weight (approximately 100 lb) is attached to the end of the Kevlar line. The weight is lifted overboard and at least 10 m of line is let out prior to the GO-FLO™ bottle attachment depending on water depth. If near bottom sampling is required, the distance between the GO-FLO™ bottle and the bottom weight must be adjusted.

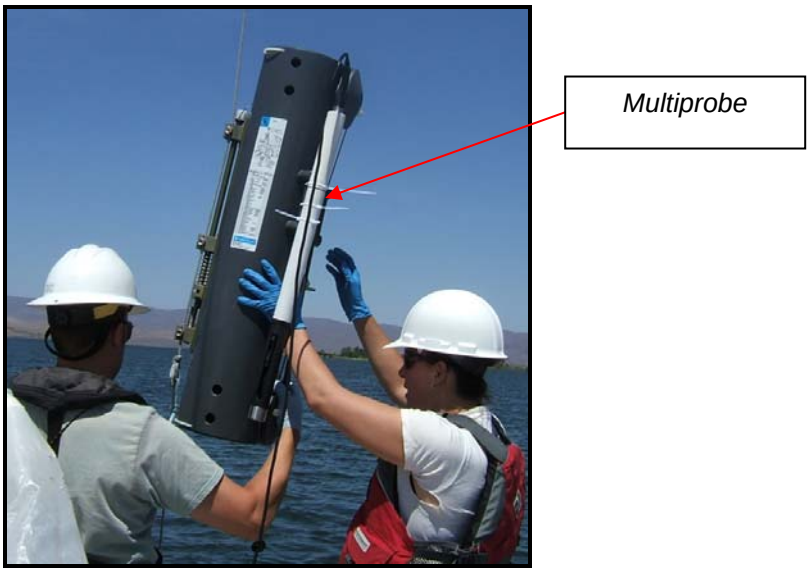


Figure 7. GO-FLO™ Bottle with Multiprobe Attached to Handle prior to Deployment

Quickly lower the bottle down into the water to about 15 m so that the pressure valve is activated to open the two ball valves at each end of the bottle when the bottle reaches a water depth of 10 m. If the bottle is lowered too slowly, water will seep into the bottle and the pressure gradient between the inside of the bottle and the outside water column will not be sufficient to trigger the valves open. Bubbles should rise to the surface as the pressure release valve opens the GO-FLO™ underwater. Bubbles rising to the surface are indicative that the bottle is in the open position. If bubbles are not seen, it is possible that the bottle has not opened, although bubbles sometimes cannot be seen because of light scattering or choppy water surface. The bottle can be raised slowly to just beneath the water surface so that personnel looking over the side can see if the bottle has opened. If the bottle is not visible, do not bring it above the surface. If it is in the open position, contact with air or surface water oily microlayer can contaminate the lining of the bottle. If the water is rough or turbid, it is better to assume the bottle is open. After verifying that the bottle is open, lower it to the desired sampling depth. Attach the messenger to the line and release it. The messenger will trigger the bottle closed. Wait for the messenger to reach the bottle, before retrieving the GO-FLO™ bottle. When the bottle is retrieved to deck level, the person who attached the bottle to the line will disengage it, carry or slide it with the help of the winch, and secure it to the designated clean workspace. Once in the clean workspace, the GO-FLO™ bottle is placed upright and the air release valve is opened and the sample is decanted or pumped into the sample containers (Figure 8). Trace metal clean sampling procedures will be used that follow the EPA clean-hands technique (USEPA 1996) and SOP SW-04 instructions.

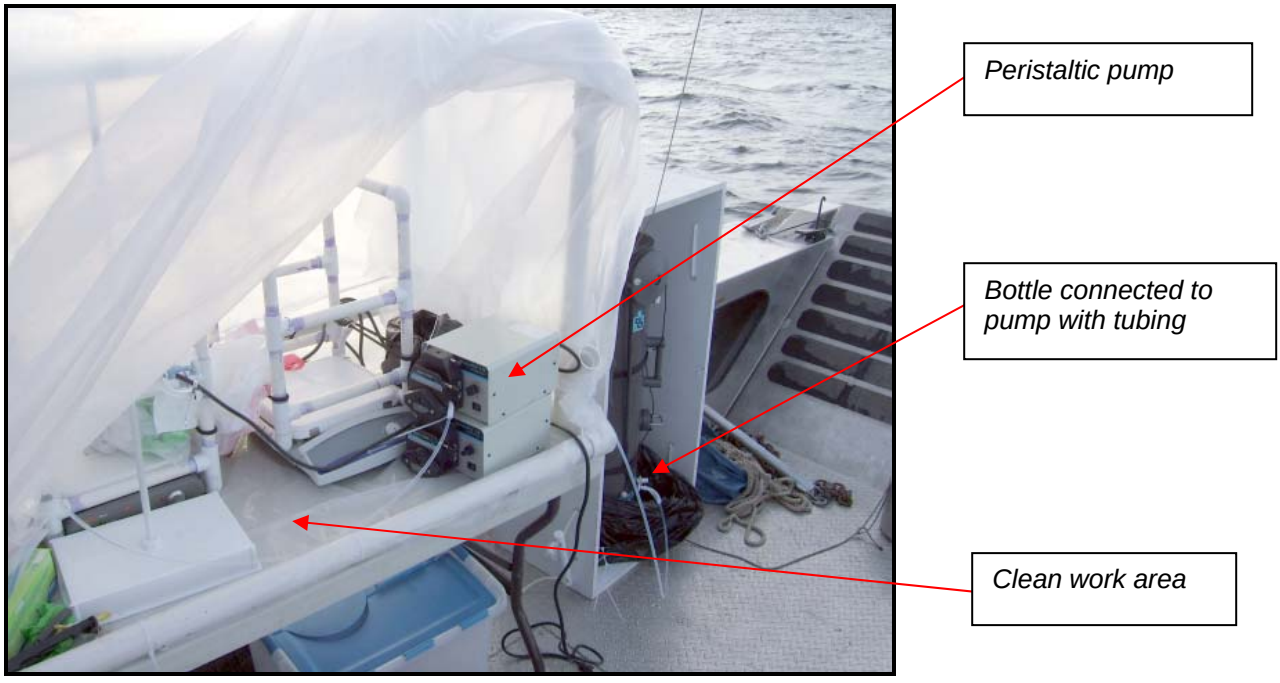


Figure 8. Typical Setup for Sampling with Peristaltic Pump

FIELD FILTRATION

In some cases, field filtration may be required (recommended for inorganic compound analysis). If applicable, attach a new, disposable filter cartridge (typically 0.45 μm) to the discharge line. Filtered water should be introduced directly into the appropriate sample container. Alternate field filtration methods may be specified in the project-specific SAP. Note: Although not recommended, the laboratory can filter the samples if the samples are NOT preserved and are filtered within 24 to 48 hours of collection.

WATER QUALITY MEASUREMENTS

If specified in the project-specific SAP, physical and chemical water parameters may need to be collected at surface water stations. Several physical and chemical water parameters are best measured in the field because of the unstable nature of the parameter or because the information is needed to direct further sampling. It is frequently preferable to perform these analyses in the field, especially if the samples will not be immediately transported to the analytical laboratory (pH, in particular, should be measured in the field if feasible). In addition, measurements of temperature and transparency can be collected accurately only in the field.

It is always best if the water quality meter is placed directly into the surface water body at the station location at the desired water depth instead of being placed into the container in which the sample is collected. However, if this is not possible, a clean plastic bucket can be used to collect samples for water quality analyses (e.g., pH, temperature, and conductivity). The bucket should be rinsed twice with the water from the station prior to measuring water quality parameters.

The name(s) of the person(s) making the measurement and the field equipment used to make that measurement must be recorded in the field logbook. Equipment maintenance and calibration records must be kept in logbooks and field records so that the procedures are traceable.

RELATED SOPS

- Record all field activities in accordance with procedures outlined in SOP AP-02.
- Package and ship surface water samples in accordance with procedures outlined in SOP AP-01.
- Maintain sample custody in accordance with procedures outlined in SOP AP-03.

REFERENCES

Fischer, H.B., E.J. List, R.C.Y. Koh, J. Imberger, and N.H. Brooks. 1979. *Mixing in inland and coastal waters*. Academic Press. New York.

Greenberg, A.E., R.R. Trussell, and C.S. Clesceri (eds). 1985. *Standard methods for the examination of water and wastewater*. 16th Edition. American Public Health Association, Washington, DC. p. 37.

Mason, R.P., and K.A. Sullivan. 1996 Standard operating procedure for site selection and sampling for mercury in Lakewater. Chesapeake Biological Laboratory University of Maryland. Solomans, MD. June 26, 1996.

USEPA. 1991. Environmental investigations standard operating procedures and quality assurance manual. U.S. Environmental Protection Agency, Region 4, SESD, Athens, GA. 424 pp.

STANDARD OPERATING PROCEDURE (SOP) SW-06

MEASUREMENT OF SURFACE WATER FIELD PARAMETERS

This SOP is based on the procedures outlined in *Field Measurements: U.S. Geological Survey Techniques of Water-Resources Investigations* (Wilde various dates).

SCOPE AND APPLICATION

Information and general instructions for field measurement of water quality parameters (pH, Eh [oxidation-reduction potential, ORP], specific conductance, dissolved oxygen, and temperature) are presented below. Because the types and complexity of water quality meters available vary widely, calibration and measurement procedures should be conducted in accordance with manufacturer's recommendations for the specific meters used. The following information describes general procedures for the measurement of water quality parameters. Where possible, sampling should be conducted first in areas expected to be least affected by constituents of interest, followed by increasingly affected areas.

EQUIPMENT AND REAGENTS REQUIRED

- Water quality parameter multimeter or meters specific to parameters of interest (i.e., temperature, dissolved oxygen, pH, transparency, turbidity, salinity, specific conductance, and ORP)
- Calibration solutions and deionized distilled water.

PROCEDURES

Before any calibration takes place, allow the probe and all calibration solutions to acclimate to the ambient field temperature along for at least 1 hour.

Calibrate the meter(s) in the field at the beginning of each day of field or laboratory work when water quality parameters will be measured. If feasible, meters must be checked for drift with calibration standards after every 4 hours of continuous use. Otherwise, a final check must be done at the end of the sampling event. If drift is evident, recalibrate.

1. Calibrate meter(s) in accordance with manufacturer's instructions using fresh (unused) calibration buffers and standards for each sensor.
2. Check slope reading with specifications (in operating manual) to verify slope is within the manufacturer's specified range.
3. Thoroughly rinse a 500-mL beaker or 8-oz jar with sample water. Discard sample water.
4. Rinse electrodes with sample water to acclimate them.
5. Fill beaker with fresh sample water.
6. Immerse electrodes in sample while swirling the sample, if needed, to provide thorough mixing. Turn on meter(s). If a flow-through cell is used, install probes and connect sample water to bottom port of flow-through cell, directing sample water up through the cell, exiting through the top port. Direct effluent tubing back in the water or into an appropriate container for storage and handling.
7. When the readings have stabilized, record the measurements displayed on the meter. It is important to determine that the correct units and unit scale are displayed on the meter and recorded for each parameter measured. Record and correct any problems encountered during measurement.
8. If available, field measurement results should be compared to previous measurements for quality control.

Several physical and chemical water parameters are best measured in the field because of the unstable nature of the parameter or because the information is needed to direct further sampling. It is frequently preferable to perform these analyses in the field, especially if the samples will not be immediately transported to the analytical laboratory (pH, in particular, should be measured in the field, if feasible). In addition, measurements of temperature and transparency can be collected accurately only in the field. Eight parameter measurements for water are described in the following sections of this SOP.

Temperature

Measure water temperature with either an alcohol or digital thermometer. It is recommended that mercury thermometers not be used to avoid possible breakage and introduction of mercury into the environment and to remove a source of possible contamination to samples collected for the analysis of mercury. Measure temperature as soon as the sample is collected to obtain a measurement that is an accurate representation of the *in situ* sample temperature. All instruments used to measure temperature should be traceable to a National Institute of Standards and Technology temperature reference. In the case of digital thermometers, follow the calibration procedure recommended by the manufacturer, if provided. Multiprobes in

general contain a temperature probe; check these probes against a calibrated thermometer before use. For more detailed procedures, see discussion in Wilde (2006).

Dissolved Oxygen

Dissolved oxygen may be measured in the field by either a dissolved oxygen polarographic-membrane type sensor or a luminescent type sensor. Dissolved oxygen can also be measured by a field-portable Winkler titration kit.

It is recommended that calibration be done at temperatures that are at least within 10ºC of the ambient water temperature. The smaller the temperature difference is between the environmental water and the calibration chamber, the more accurate the calibration will be.

When using static samples (i.e., water sample collected in a container), protect samples from absorbing oxygen from the atmosphere by using a low or zero-headspace container. If using a meter and probe, calibrate the system according to the manufacturer's procedure prior to use with a zero oxygen standard and a second standard of known oxygen content. Check the second standard by performing a Winkler titration. Other probes are calibrated by percent oxygen saturation in an enclosed container with a small amount of water. When measuring dissolved oxygen with certain polarographic-membrane probe in water samples held inside zero-headspace containers, swirl or stir samples constantly until the reading stabilizes and the measurement is recorded. Stirring the sample is not necessary if a luminescent-sensor is used. For other probes, immerse the probe in the water column and monitor a constant measurement (dynamic measurement) until the readings are stabilized. Once the readings stabilize, record the oxygen concentration readings manually or digitally. For more detailed procedures, see discussion in Lewis (2006).

pH

The pH of a water column sample can be measured in the field using a pH meter. Calibrate the meter according to manufacturer's specifications with at least two standards of known pH. The pH of these standards should bracket the expected pH at the sampling site. For example, if the pH at the sampling site is expected to be basic (pH 7 to 14), standards of pH 7.00 and 10.00 should be used to calibrate the meter. The pH of the buffer solution is temperature dependent. That is, pH 10 buffers change more per unit change in temperature than do pH 4 buffers. The temperature of buffer solutions must be measured, and temperature-correction factors must be applied before calibration adjustments are made. Calibration and operating procedures differ with instrument systems—check the manufacturer's instructions. If pH measurements at the sampling site do not fall within the initial calibration range, the meter should be recalibrated with appropriate standards and sample pH remeasured for those samples that fell outside the calibration range. For more detailed procedures, see discussion in Wilde et al. (2006).

Transparency

Water column transparency is measured with a Secchi disk, which is a weighted, black-and-white or all-white disk that is lowered into the water body on a calibrated rope or line.

Always perform these measurements from the side of the boat that faces away from the sun. Lower the disk slowly until it is no longer visible and then raise it until it is visible again. Record the depth, measured from the water surface, in feet or meters. The all-white disk may be preferable when the water transparency is high. However, either disk is acceptable to use.

Turbidity

Turbidity can be measured in the field on static water samples contained in jars with a field-portable nephelometer (turbidity meter) or *in situ* with a turbidity probe mounted in a multiprobe device. Calibrate the meter prior to use with at least two standards of different but known turbidity (in nephelometric turbidity units, or NTUs). The two standards should bracket the range of turbidity measurements expected at the sampling site.

Perform field analysis for turbidity on static water samples as soon as possible after collection. If immediate analysis is not possible, agitate the sample prior to analysis to resuspend any settled solid material. If the sample temperature increases, air bubbles may form and cause erroneous values.

When performing field analysis for turbidity *in situ*, monitor the turbidity probe constantly with a remote display and record data manually or digitally.

For more detailed procedures, see Anderson (2005).

Conductivity or Salinity

Salinity can be measured in the field with a salinometer, and conductivity with a conductivity meter. There are two types of conductivity sensors as described below.

- **Contacting-type sensors with electrodes**—Electrodes contained in a dip cell can be suspended in the sample. The cell constant is the distance between electrodes (in centimeters) divided by the effective cross-sectional area of the conducting path (in square centimeters). A cell constant is chosen on the basis of the expected conductivity. The greater the cell constant, the greater the conductivity that can be measured.
- **Electrodeless-type sensors**—Conductivity is measured by inducing an alternating current in a closed loop of solution, and measuring the magnitude of the current. Measuring errors in this type of electrode are minimized because sensors do not have issues with electrode polarization or electrode fouling.

Calibrate the conductivity meter prior to use in accordance with the manufacturer's directions using a standard of known conductivity. The conductivity of the standard should be close to the expected value at the sampling site. When measuring a sample for conductivity, swirl or stir the sample until the meter is stabilized and a measurement is recorded. For more detailed procedures, see Radtke et al. (2005).

Salinity can be automatically calculated from conductivity, temperature, and barometric pressure readings in the same multiprobe and displayed on the meter of most models. Salinity may also be calculated from the measured conductivity and temperature of a sample according to Standard Method 2520B (APHA 1998). Gross salinity measurements may also be taken with a field-portable refractometer. This instrument provides salinity measurements with an accuracy of 1 to 2 parts per thousand. For more detailed procedures, see APHA (1998).

ORP or Eh

ORP or Eh may be measured in the field with an inert metal electrode and read relative to a reference electrode that is immersed in the same medium. For most multiprobe units, the inert metal electrode is a button or ring made of platinum and the Ag/AgCl reference electrode is the same one connected to the pH probe. The readout of the sensor is a voltage (relative to the reference electrode), with positive values (e.g., + 300 mV) indicating an oxidizing environment (ability to accept electrons) and negative values (e.g., –300 mV) indicating a reducing environment (ability to donate electrons) (YSI 2005).

ORP and Eh are the same parameters in that both measure the potential of the medium to transfer electrons. However, the ORP reference electrode is made of different material than the Eh standard hydrogen electrode; therefore, a voltage offset needs to be taken into account when converting ORP measurements to Eh values.

More detailed explanation on the theoretical concept, voltage offset conversions, method limitations and interferences can be found in the attached YSI Tech Note (YSI Environmental 2005) and in Nordstrom and Wilde (2005).

REFERENCES

Anderson, C.W. 2005. Turbidity (version 2.1): U.S. Geological Survey techniques of water-resources investigations, Book 9, Chap. A6., Section 6.7. Available at: http://water.usgs.gov/owq/FieldManual/Chapter6/Section6.7_v2.1.pdf.

APHA. 1998. *Standard methods for the examination of water and wastewater.* 20th Edition. L.S. Clesceri, A.E. Greenberg, and A.D. Eaton (eds). American Public Health Association, Washington, DC.

Lewis, M.E. 2006. Dissolved oxygen (version 2.1): U.S. Geological Survey techniques of water-resources investigations, Book 9, Chap. A6, Section 6.2, Available at: http://water.usgs.gov/owq/FieldManual/Chapter6/6.2_v2.1.pdf.

Nordstrom, D.K., and F.D. Wilde. 2005. Oxidation-reduction potential (version 1.2): U.S. Geological Survey techniques of water-resources investigations, Book 9, Chap. A6, Section 6.5. Available at: http://water.usgs.gov/owq/FieldManual/Chapter6/6.5_v_1.2.pdf.

Radtke, D.B., J.V. Davis, and F.D. Wilde. 2005. Specific electrical conductance (version 1.2): U.S. Geological Survey techniques of water-resources investigations, Book 9, Chap. A6, Section 6.3. http://water.usgs.gov/owq/FieldManual/Chapter6/Final508Chapter6.3.pdf.

Wilde, F.D. (ed.). [various dates]. Field measurements: U.S. Geological Survey techniques of water-resources investigations, Book 9, Chap. A6. (Chapter sections are cited by author and date.) Available at: http://pubs.water.usgs.gov/twri9A6/.

Wilde, F.D. 2006. Temperature (version 2.0): U.S. Geological Survey techniques of water-resources investigations, Book 9, Chap. A6, Section 6.1. Available at: http://water.usgs.gov/owq/FieldManual/Chapter6/6.1_ver2.pdf.

Wilde, F.D., E. Busenberg, D.B. Radtke, and S.A. Rounds. 2006. pH (version 1.3): U.S. Geological Survey techniques of water-resources investigations, Book 9, Chap. A6, Section 6.4. Available at: http://water.usgs.gov/owq/FieldManual/Chapter6/6.4_ver1.3.pdf.

YSI Environmental. 2005. Measuring ORP on YSI 6-Series Sondes: Tips, cautions and limitations. Tech Note 0201 T608. Available at: https://www.ysi.com/DocumentServer/DocumentServer?docID=YSI_T608.

ATTACHMENT 1. MEASURING ORP ON YSI 6-SERIES SONDES: TIPS, CAUTIONS AND LIMITATIONS

Measuring ORP on YSI 6-Series Sondes: Tips, Cautions and Limitations

Introduction and Basic Theory

As described in *Standard Methods for the Examination of Water and Wastewater* (Section 2580 B.), ORP is a potentiometric measurement in which the potential (or tendency) of the medium for electron transfer is sensed by an inert metal electrode and read relative to a reference electrode that is immersed in the same medium. This determination can also be referred to as a "redox" measurement (combination of REDuction and OXidation). For most multiparameter monitoring systems, the inert metal electrode is a button or ring made of platinum and the reference electrode is the same one associated with the pH sensor, usually Ag/AgCl. The readout of the sensor is a voltage (relative to the reference electrode), with positive values (e.g., + 300 mV vs. Ag/AgCl) indicating an oxidizing environment (ability to accept electrons) and negative values (e.g. -300 mV) indicating a reducing environment (ability to furnish electrons).

The determination of ORP is particularly worthwhile in water which contains a relatively high concentration of a redox-active species, e.g., the salts of many metals (Fe^{2+}, Fe^{3+}) and strong oxidizing (chlorine) and reducing (sulfite ion) agents. Thus, ORP can sometimes be utilized to track the metallic pollution in ground or surface water or to determine the chlorine content of wastewater effluent. However, ORP is a nonspecific measurement, i.e., the measured potential is reflective of a combination of the effects of all the dissolved species in the medium. Because of this factor, the measurement of ORP in relatively clean environmental water (ground, surface, estuarine, and marine) has only limited utility unless a predominant redox-active species is known to be present. Users should thus be careful not to "over-interpret" ORP data unless specific information about the site is known.

ORP vs. Eh: Calibration of ORP Sensors

Many users of YSI 6-series sondes that make field or laboratory redox measurements have questions about the difference between ORP and Eh. In essence, the two parameters are the same in that both quantify the potential of the medium to transfer electrons -- the difference is the reference electrode (and thus the voltage offset) against which the potential of the platinum sensor is reported. Eh is defined as a voltage reading vs. the Standard Hydrogen Electrode (SHE), while ORP is a much less specific term in which the measurement can be made relative to any practical or theoretical reference electrode, such as Ag/AgCl, calomel, or SHE. Generally, it is not easy to use the SHE in laboratory or field measurements and thus redox readings are made using either the Ag/AgCl or calomel reference electrodes, with Ag/AgCl being more popular in multiparameter water quality instrumentation. Thus, Eh is usually not determined directly. However, the voltages obtained as ORP readings vs. non-SHE electrodes can easily be converted into Eh values by two mechanisms:

- Adding (or subtracting) an offset voltage to the ORP readings obtained vs. a practical reference electrode to account for the fixed difference between the SHE and the other reference system. The offset voltage can easily be obtained for several practical reference electrodes in Table 2580: II of the section of Standard Methods on ORP. For example, the potential of Zobell solution vs. the Ag/AgCl reference electrode using 4 M KCl is +228 mV while the same solution read vs. the SHE is +428 mV. Therefore to convert ORP readings taken under these conditions to Eh, simply add 200 mV to the ORP voltage. For example, ORP readings of +150 and -172 mV translate to Eh values of +350 and +28 mV, respectively.
- Using the instrument software to automatically add the offset voltage to the ORP readings as they are displayed or logged. This method is implemented during calibration of the ORP sensor in YSI 6-series sondes. For example, when calibrating an instrument with Zobell solution that has an ORP reading of 228 versus the YSI reference electrode, enter 428 mV at the calibration prompt instead of 228. After the calibration is confirmed, 200 mV (the difference between Ag/AgCl and SHE reference electrodes) will automatically be added to all displayed and logged ORP values, effectively converting them to Eh with no further correction needed. The software in YSI 6-series sondes is likely to interpret the entry of the higher voltage value as an "out of range" calibration error and provide a warning to this effect. As long as the user is knowledgeable about the procedure, the error can be "overridden" with no ill effects.

Effect of Temperature

The temperature of the water for which ORP is being determined will affect the voltage output of the sensor. This factor definitely needs to be taken into account for calibration and should be considered when reporting field ORP values.

(continued)

For calibration, the following table can be used when using Zobell solution, the YSI-recommended standard. Thus, if the Zobell calibration standard is at 15° C instead of 25° C, enter 241 mV at the calibration prompt instead of 228 mV (the 25° C value which is commonly quoted).

Temperature, C	Zobell Solution Value, mV vs. Ag/AgCl (4 M KCl)
-5	267.0
0	260.5
5	254.0
10	247.5
15	241.0
20	234.5
25	228.0
30	221.5
35	215.0
40	208.5
45	202.0
50	195.5

The user may be able to locate similar temperature-dependence data in the literature for other ORP standards such as Light's Solution and quinhydrone standards in pH buffers.

Temperature will also clearly have an effect on field readings, but, in this case, the variation is usually not definable since the temperature effect depends on the dissolved species responsible for the ORP reading, and these species are usually not known exactly for environmental water. For this reason, ORP readings on YSI 6-series sondes are **not** temperature compensated in any manner. The user must remember that ORP variation in field water could be due to temperature changes rather than analyte compensation. Usually, however, gross changes in ORP (>100 mV) are not due to the effect of temperature.

Confirming ORP Response

Unlike pH, YSI 6-series sondes only allow a single point calibration for ORP, i.e., an offset adjustment as described above. This is almost always adequate if the ORP sensor has been maintained properly. However, some users like to confirm that their ORP system tracks changes in ORP correctly, in the same way that a pH sensor responds to immersion in pH 7 and pH 10 buffers.

To check the "slope" and response characteristics of the ORP sensor, YSI recommends that the user purchase item number B125 (ORP Calibration Kit) from the manufacturer of one of our ORP sensors:

Sensorex
Tel. +1 714 895 4344
Fax. +1 714 894 4839
Email. info@sensorex.com
Web. www.sensorex.com

The kit contains solid quinhydrone which, when added to the supplied buffers, yields two solutions with well-defined, but different, ORP values.

Problems with ORP Sensors

Although based on relatively simple theory, ORP is, unfortunately, also a measurement that can show more problems than other water quality sensors with regard to consistency between different instruments and overall accuracy. In addition, these issues are further complicated in that their extent is likely to depend on both the condition of the sensor and the makeup of the water being tested. The most common problem reported with regard to ORP determination in environmental water is that readings from various instruments (sometimes with exactly the same sensor type and electronics) differ by a significant margin (50-100 mV) even though the sensors are in the same container of water. To make the problem more perplexing, all of the sensors show identical readings in an ORP standard such as Zobell solution. The exact explanation for this paradox is sometimes elusive, but there are at least three possible reasons for its occurrence.

- First, ORP sensors can show a slow response in environmental water if the platinum button of the probe has been contaminated with extraneous material. Common contaminants include hard water deposits, oil/grease, or other organic matter. If the platinum electrodes in the above example are variably contaminated, then some of them (the more contaminated) will be likely to approach potentiometric equilibrium slower. Under this scenario, if left long enough all the sensors would read the same. However, it might take days for the contaminated sensors to reach their final value, and, therefore, they appear in the time frame of a sampling experiment (< 1 hour), to be different. Naturally, if the electrode contaminant is redox-active, either in itself or because it contains redox-active impurities, the reading from that sensor will exhibit erroneous readings that may never change unless the contaminant is removed.

(continued)

- Second, in clean environmental water, there may be very few redox-active species present, and those that are present may be in very low concentration. In many cases, the concentration can be so low that the redox influence of the species is effectively below the detection limit of the method. Under these conditions, the readings will have questionable meaning and could show this type of variation described above. Note that the ORP reading variance associated by this scenario is likely to be exacerbated if any of the electrodes is also contaminated as described above.
- Third, the makeup of the surface composition of the electrode may not be ideal for the measurement in the medium under investigation. While "platinum" ORP electrodes are primarily composed of the metal itself (in a neutral state), it is well known that the surface of the electrode (where the redox action takes place) is coated to varying extents with a molecular layer of platinum oxide (PtO). The Pt/PtO ratio can change over time, depending on the medium in which the probe is stored, and thus the surface of the electrode actually possesses its own potential that can be variable. If this surface potential is similar to the ORP potential of the medium, then electrode response can be sluggish. The cleaning procedure recommended later in this document will result in a surface characterized by a low Pt/PtO ratio and one that possesses a very positive potential. This should be suitable for most environmental measurements.

The fact that similar or identical ORP sensors read differently in environmental water yet the same in Zobell solution is due to the fact that the concentration of redox-active species (ferricyanide/ferrocyanide for Zobell) is much greater in the standards. This higher concentration usually "swamps out" the inconsistencies related to detection limit problems (caused by low amounts of redox-active species) and response time issues (caused by electrode contamination), thus all sensors respond rapidly and read within the YSI specification of +/- 20 mV when in standards.

If you observe inconsistency between different sensors or experience ORP readings which seem unusual for the water being tested with your YSI 6-series multiparameter instrument, YSI recommends the following steps to identify and/or correct the problem:

First, make certain that the pH sensor is functioning properly. The reference electrode of the sonde is common to both pH and ORP sensors and, therefore, if both pH and ORP sensors are malfunctioning this is likely to be the source of the problem. Reference electrode problems usually appear as either total failure or as a slow response in both pH and ORP readings. If a reference electrode problem is suspected, test the ORP sensor in a standard and make certain that it is within 20-30 mV of the predicted value. If reference electrode performance is indicated, clean the sensor according to the instructions shown below and then retest.

Second, if the sensor performs well in the ORP standard, remove the probe from the sonde and carry out the sequential cleaning process documented in the next section.

ORP Electrode Cleaning

The following procedure will result in removal of many common contaminants from the platinum ORP electrode. Fouling of the electrode can, however, be deployment-specific, and some contaminants from polluted water may not be dissolved by this method. The use of other solvents and reagents may be possible, but they must be selected carefully so as not to damage the reference electrode or pH glass of the combination sensors nor to leach or dissolve the CPVC body of the probe itself. Consult YSI Customer Service before using cleaning methods other than those documented below.

YSI recommends that the user perform the cleaning/reconditioning operation in the order indicated. Performance can be rechecked at the conclusion of each major section (A, B, and C) and the cleaning discontinued if, at that point, the performance problem has been corrected.

Procedure A

1. Soak the probe for 10-15 minutes in clean water containing a few drops of commercial dishwashing liquid.
2. Wipe the platinum button or ring by rubbing with a cotton swab soaked in the cleaning solution. CAUTION: For 6565 probes, be certain not to damage the glass bulb of the combination sensor during this process.
3. Rinse the probe in clean water, wipe with a cotton swab saturated with clean water, and then re-rinse with clean water.

Procedure B

1. Soak the probe for 20-30 minutes in one molar (1 M) hydrochloric acid (HCl). This reagent can be purchased from most laboratory supply dealers. Be sure to follow the safety instructions supplied with the reagent.
2. Wipe the platinum button by rubbing with a cotton swab soaked in the acid. CAUTION: For 6565 probes, be certain not to damage the glass bulb of the combination sensor during this process.
3. Rinse the probe in clean water, wipe with a cotton swab saturated with clean water, then rerinse with clean water.

(continued)

Procedure C

1. Soak the probe for approximately 1-2 hours in a 1 to 1 dilution of commercially available chlorine bleach.
2. Rinse the probe with clean water and then soak for at least 1 hour in clean water to remove residual bleach from the reference junction. **CAUTION**: If removal of the chlorine bleach is incomplete, this cleaning reagent can seep into either your calibration standards or measurement media and cause erroneous ORP readings until it is dissipated. Always err on the side of caution in the chlorine bleach removal. Soaking the probe in clean water for periods of time longer than 1 hour can do no harm, however, lesser soaking times can cause problems.
3. Dry the sonde port and probe connector with compressed air and apply a very thin coat of O-ring lubricant to all O-rings before re-installation of the probe. After the probe is reinstalled, place the sensors in Zobell solution and make certain that observed ORP readings stabilize within a few minutes and remain stable for 15-20 minutes.

Typical ORP Data in Standards and Freshwater

	Zobell Reading		
Probe #	Initial	1 hour	After Testing
1	228	228	233
2	227	226	227
3	227	227	228
4	224	224	228
5	227	227	228

Table 1. ORP sensor performance in Zobell Solution.

Experiments have been performed at YSI to demonstrate the typical performance of YSI ORP sensors in both standards and in freshwater. Five (5) new 6565 sensors were taken from stock and placed directly into Zobell solution at 22° C. As shown in Table 1 below, all sensors read within 4 mV of each other. The sensors were left in the Zobell solution for 1 hour and the values recorded again. Finally, the sensors were retested in Zobell solution after the entire regimen of testing described below was completed. The values were basically unchanged, demonstrating the stability of the sensor in redox buffer.

The sensors were then rinsed and soaked in DI water and then transferred to tap water that had been diluted with deionized water to a specific conductance of 290 uS/cm and saturated with air. The ORP readings were recorded 1 minute after transfer and then again after 2.5 hours in the low-to-medium conductivity water. Note that all readings are fairly close at 1 minute, with probe 5 showing a somewhat more positive reading. Note also that the discrepancy between probe 5 and the others increased slightly after longer-term exposure to the water sample. Cleaning the ORP platinum sensor of probe 5 with clean water and a cotton swab resulted in a decrease of the reading to 207 mV -- significantly closer to the other sensors. Finally, note that all readings increased by an average of about 40 mV after longer-term exposure to the natural water. This stabilization pattern, along with some variation in probe readings, is likely to occur with all ORP sensors when used in environmental water samples. The difference in behavior between Zobell solution and the water sample is striking and demonstrates that a lower accuracy specification must be tolerated in natural water samples than in buffers. (Note that the YSI accuracy specification of +/- 20 mV refers to readings taken in redox standards.) See Table 2 for the data described in this experiment.

	ORP Reading	
Probe #	1 minute	2.5 hours
1	138	178
2	143	161
3	132	177
4	135	169
5	166	221

Table 2. ORP sensor performance in 290 uS/cm natural water.

The sensors were then cleaned using the 1 M HCl treatment described above, soaked in DI water to remove all acid traces, and then placed back into the 290 uS/cm natural water sample. ORP readings were taken 5 minutes after placing the probes in the water. The calibration of the sensors was then checked in Zobell solution the probes returned to the natural water sample, and the readings recorded after 5 minutes. Results are shown in Table 3.

Probe #	Water Sample 5 minutes post cleaning	Zobell Solution Calibration check	Water Sample 5 minutes post cal check
1	195	233	186
2	188	227	183
3	214	228	184
4	197	228	184
5	280	228	230

Table 3. ORP sensor performance in 290 uS/cm natural water after cleaning sensors with 1 M HCl.

(continued)

Note the following from the data in Table 3:

- The results in the natural water sample are about the same after the cleaning as before -- probe #5 is still significantly higher than the other 4 that are fairly tightly bunched.
- Even after multiple exposures to standards, the natural water sample, and 1 M HCl, the probes (including probe #5) all read effectively the same in Zobell solution.
- Although the effect is relatively minor, the water sample readings are somewhat dependent on the previous reagent to which the probes were exposed. Note that the results are more consistent and slightly lower overall after the probes had been in Zobell solution (column 3) than after they had been in 1 M HCl (column 1).

Most users would consider the performance of probes 1-4 in natural water acceptable in terms of their consistency with one another, but might wonder why probe 5 always seems to read somewhat more positive than the other sensors except in Zobell solution where it has the same reading. Although difficult to prove, the difference is most likely due to a different Pt/PtO ratio on the surface of probe 5. Consistent with this hypothesis, the final experiments indicate that probe 5 responds to ORP changes and that its ORP reading in natural water becomes closer to those of the other four sensors after longer-term exposure to this medium.

In the final testing, the probes were placed in a sodium sulfite solution, a reducing environment that should produce a decrease in the ORP readings. As shown in Table 4, this effect was indeed observed. The probes were then carefully cleaned and returned to the natural water sample for 18 hours and then the ORP values recorded to conclude the test protocol. These final values are found in Table 4.

Probe #	Sodium Sulfite Solution, after 5 minute exposure	Natural Water, after 18 hour exposure
1	135	196
2	125	174
3	140	207
4	120	195
5	95	218

Table 4. ORP sensor performance in sodium sulfite solution and after long-term exposure to natural water.

YSI would consider Probe 5 as an acceptable sensor for use with our 6-series sondes for the following reasons even though it reads an average of 50 mV different from the other sensors tested:

- The sensor responds quickly and shows the proper reading in Zobell solution;
- The sensor's reading in natural water is not radically different (>100 mV) from the other sensors and becomes closer after extended exposure to this medium;
- The sensor tracks changes in ORP properly.

Summary

The determination of ORP in environmental water can provide valuable insight into the sample as long as there is a significant concentration of a redox-active species present. However, in the absence of these species, ORP can be a significantly less exact measurement than for most other sensors found in YSI 6-series sondes. The inexactness is usually due to contamination of the electrode surface (either physically or chemically), but can also be due to the lack or low concentration of redox active agent in the environmental water.

The quoted accuracy specification for the YSI ORP sensor (+/- 20 mV) refers to redox- standards, such as Zobell solution, and not to environmental water of variable, and usually unknown, content. In many cases, the +/- 20 mV specification will be met in natural water, but it cannot be guaranteed.

Periodic maintenance of your YSI ORP sensor (6032 or 6565) will increase your field consistency and accuracy, but may not overcome all problems.

The value of ORP in determining the content of environmental water is greatly enhanced if the user has some knowledge or history of the site.

For additional information please contact
YSI Environmental
Tel.+1 937 767 7241
US 800 897 4151
Fax +1 937 767 1058
Email. environmental@ysi.com
Web. www.ysi.com

ELM SOPs

STANDARD OPERATING PROCEDURES

AND QUALITY CONTROL MANUAL

FOR THE

- **OAKTON WD-35615-SERIES pH/mV/TEMPERATURE METER**

- **YSI MODEL 55 DISSOLVED OXYGEN AND TEMPERATURE METER**

- **YSI MODEL 30 CONDUCTIVITY, SALINITY AND TEMPERATURE METER**

ENVIRONMENTAL LIABILITY MANAGEMENT, INC.

LABORATORY IDENTIFICATION No. 18004

SEPTEMBER 2003

TABLE OF CONTENTS

LIST OF ATTACHMENTS

1. pH METER

1.1. Introduction

The Oakton WD Meter can measure pH values from -2.00 to 16.00, with an accuracy of ± 0.01 pH. The resolution of the meter can be pre-set to 0.01 or 0.1 pH. The Oakton Operating Instructions manual is also available for review regarding all functions of this meter.

1.2. Preparation

Ensure the batteries (4 AAA) are installed properly, or insert/attach the AC adapter.

1. Insert pH electrode into top of meter. Ensure connector is dry and clean.
2. Insert the electrode into the opening of the holder until the housing touches the top of the holder.
3. Align and secure the two holders together, then attach the holders to the meter.
4. The default setting for the resolution of the meter is 0.01 pH. It can also be set to 0.1 pH.
5. Distilled water should be available for rinsing the electrode between each analyses.

1.3. Calibration

1. Each day before use, perform a **2 point calibration** using selected standard buffers solutions that bracket the expected sample range.
2. Select pH mode. Rinse the electrode with distilled water. **Do not wipe to dry the electrode**.
3. Dip the electrode into the selected buffer, and press CAL/MEAS. The primary display will show the measured reading, while the smaller display will indicate the pH of the standard buffer solution.
4. Press the MI and the MR key to display the selected buffer option.
5. When the READY indicator is displayed, the measured pH value is stabilized.

6. Press CON to confirm the calibration. Note: The OR indicator flashes if the selected buffer is not within $\pm$ 0.50 pH from the measured pH value.

7. Repeat steps 1 through 6 for the second selected buffer solution to complete the calibration.

8. Press CAL/MEAS to return to the measurement mode.

9. Do not reuse buffer solutions after calibration.

10. Each time a calibration is performed, the data must be recorded on the Laboratory Work Sheet (Attachment E), and dated and signed by the analyst performing the calibration.

11. The pH meter shall be checked after calibration by reading pH buffer standards of pH 4.00, 7.00 and/or 10.00 and recording the actual readings.

1.4. Measurements

1. The electrode can be hand held or attached to the meter holder for one-hand operations.

2. Collect each sample in a clean, quart size jar and note the time on the Laboratory Work Sheet. Samples must be analyzed within 15 minutes of collection.

3. Decant an aliquot of the sample into a clean, wide mouth container.

4. Dip the electrode into the sample and completely immerse the glass bulb. Stir the electrode gently to homogenize the sample.

5. When the reading stabilizes, the READY indicator will display.

6. To hold a measurement, simply press the HOLD key, and record the measurement and time on the Laboratory Work Sheet. Press HOLD again to release the held value.

7. Rinse the electrode and the wide mouth container with distilled water.

8. Perform a duplicate analysis for each sample collected by repeating steps 3 through 6.

9. Rinse the electrode and the wide mouth container with distilled water between each sample analyses.

1.5. Quality Control

1. Distilled water shall have at a minimum, resistivity values between 0.5 to 2.0 megohms-cm (2.0 to 0.5 micromhos/cm.) at 25 degrees C. Preferably, distilled water should have resistivity values greater than 1.0 megohms-cm (less than 1.0 micromhos/cm) at 25 degrees C. Containers of distilled water should be capped when not in use and should be capped immediately after each use.

2. Analytical reagent grade (AR) chemicals should be used for most analyses.

    - Stock and working standard solution shall be checked each day the pH meter is in use for signs of decomposition, including but not limited to discoloration, formation of precipitates, and concentration change due to evaporation.

    - All solutions shall be properly labeled with identification of the compound, concentration, solvent, date, and analyst who prepared the solution.

3. All glassware should be washed in a warm detergent solution, and thoroughly rinsed first in tap water and then in distilled water. The glassware shall be air dried and properly stored prior to use. This cleaning procedure is sufficient for most analytical needs.

4. Batteries will be checked each day when the pH analyses meter is use, and replaced when necessary.

5. The electrode and dedicated sample bottle will be rinsed with distilled water between each analyses.

6. Standard buffer solutions will be used within the appropriate expiration dates. Expired solutions will be disposed immediately in accordance with NJDEP disposal procedures.

7. A duplicate pH measurement should be taken for each sample collected to verify the accuracy of the method.

8. If the pH meter is in use for more than a three hour period, the pH of a third buffer shall be checked once every three hours. If the pH differs by more than +/- 0.2 pH units from the standard buffer value, the meter shall be recalibrated.

9. All quality control data and records shall be recorded on the Laboratory Work Sheet. Such retained data shall include but shall not be limited to, the results of and the raw data generated by Proficiency Sample analyses, and routine calibration reports.

1.6. Memory And Data Input

1. The MI key inputs data into the memory. Five sets of data can be stored for each mode.

2. Press MR each time to recall each reading.

3. To exit Memory Recall, press CAL/MEAS to return to the measurement mode.

1.7. Electrode Care and Maintenance

1. The pH electrode bulb should be stored wet by using the protective rubber cap filled with the electrode storage solution, or a buffer solution of 1/100 part of saturated KCL, with a pH value of 4. Never use distilled water for storage.

2. Rinse the electrode and reference junction in distilled water after each measurement. Never wipe dry.

3. Clean the electrode every one to three months in accordance with the Operating Instructions manual page 20, which provides specific cleaning instructions dependent on the deposits or problems.

4. If the electrode has dehydrated, soak it for 30 minutes in a 2M -4M KCL solution.

1.8. Error Messages/Troubleshooting

1. Page 21-22 of the Operating Instructions manual outlines typical problems which may arise while using the pH meter.

2. DISSOLVED OXYGEN METER

2.1. Introduction

The YSI Model 55 Handheld Dissolved Oxygen System simultaneously displays temperature in °C and dissolved oxygen in either milligrams per liter (mg/L) or percent (%) air saturation. Temperature can be measured from -5 to 45°C ± 0.4°C, dissolved oxygen mg/L from 0 to 20 mg/L ± 0.3 mg/L, and dissolved oxygen % air saturation from 0 to 200% ± 2% air saturation.

2.2. Preparation

1. Ensure that batteries are installed properly (6 AA alkaline batteries).

2. Turn on the instrument by pressing the **ON/OFF** button; the liquid crystal display (LCD) should come on.

3. Remove the probe from the calibration chamber and inspect the probe for a dry, loose, wrinkled, damaged or fouled membrane, and for air bubbles in the electrolyte reservoir. Refer to pages 8-10 of the Operating Instructions manual to replace the membrane and electrolyte solution should any abnormalities be found.

4. Put 6 to 8 drops of distilled water onto the sponge in the calibration chamber. Turn the instrument over and allow excess water to drain out of calibration chamber. The wet sponge creates a saturated-air environment for the probe which is necessary for calibration, transport, and storage of the probe. Replace the probe into the calibration chamber.

2.3. Calibration

2.3.1. Weekly/Monthly

1. The meter is tested using an EPA accepted, modified Winkler Method[1] (azide modification) at least once a month depending upon usage. If the meter is to be used in more than one

[1] LaMotte Company Water Quality Test Kit for Dissolved Oxygen; Model EDO, Code 7414

week during a month, additional Winkler Method calibrations will be performed, such that usage of the meter falls within one week of a Winkler Method testing.

2. Results are recorded on a Laboratory Work Sheet.

2.3.2. Each Use

1. Each day before use, ensure that the sponge inside the calibration chamber is wet and that the probe is functional (see Section 2.2.3 above). Insert the probe into the chamber.

2. Turn on the instrument and wait for the dissolved oxygen and temperature readings to stabilize (usually takes a few minutes).

3. Use two fingers to press the two up and down keys at the same time.

4. The LCD will prompt you to enter the local altitude in hundreds of feet. Use the arrow keys to increase or decrease the altitude (e.g., enter 2 for 200 feet).

5. When the proper altitude appears on the LCD, press the **ENTER** key once to view the calibration in the lower right of the LCD; and a second time to move to the salinity compensation procedure.

6. The LCD will prompt you to enter the approximate salinity of the water in parts per trillion. Use the arrow keys to increase or decrease the salinity compensation, then press the **ENTER** key.

After the instrument is calibrated, press the **MODE** key to move back and forth from reading dissolved oxygen in the mg/L or % air saturation display. Use the LIGHT key to activate the back-light of the instrument.

2.4. Measurements

1. Collect each sample in a clean, quart size jar and note the time on the Laboratory Work Sheet. Samples must be analyzed within 15 minutes of collection.

2. Decant an aliquot of the sample into a clean, wide mouth container.

3. Insert the probe into the sample container.

4. Stir the sample continuously; this can be achieved by rapidly moving the probe through the sample.

5. Use the **MODE** key to transfer the display between the dissolved oxygen mg/L and % air saturation readings.

6. Rinse the probe and the wide mouth container with distilled water.

7. Perform a duplicate analysis for each sample collected by repeating steps 2 through 6.

8. Rinse the probe and the wide mouth container with distilled water between each sample analyses.

2.5. Quality Control

1. Distilled water shall have at a minimum, resistivity values between 0.5 to 2.0 megohms-cm (2.0 to 0.5 micromhos/cm.) at 25 degrees C. Preferably, distilled water should have resistivity values greater than 1.0 megohms-cm (less than 1.0 micromhos/cm) at 25 degrees C. Containers of distilled water should be capped when not in use and should be capped immediately after each use.

2. Analytical reagent grade (AR) chemicals should be used for most analyses.

    - Stock and working standard solution shall be checked each day the meter is in use for signs of decomposition, including but not limited to discoloration, formation of precipitates, and concentration change due to evaporation.

    - All solutions shall be properly labeled with identification of the compound, concentration, solvent, date, and analyst who prepared the solution.

3. All glassware should be washed in a warm detergent solution, and thoroughly rinsed first in tap water and then in distilled water. The glassware shall be air dried and properly stored prior to use. This cleaning procedure is sufficient for most analytical needs.

4. Batteries will be checked each day when the meter is use, and replaced when necessary.

5. The probe and dedicated sample bottle will be rinsed with distilled water between each analyses.

6. Standard buffer solutions will be used within the appropriate expiration dates. Expired solutions will be disposed immediately in accordance with NJDEP disposal procedures.

7. A duplicate measurement should be taken for each sample collected to verify the accuracy of the method.

8. All quality control data and records shall be recorded on the Laboratory Work Sheet. Such retained data shall include but shall not be limited to, the results of and the raw data generated by Proficiency Sample analyses, and routine calibration reports.

2.6. Probe Care and Maintenance

1. Turn meter off.

2. Rinse the probe with clean water.

3. Ensure that calibration chamber sponge is moistened with distilled water and that the probe is functional. Refer to pages 8-10 of the Operating Instructions manual to replace the membrane and electrolyte solution should any abnormalities be found. Place probe into the calibration chamber.

2.7. Error Messages/Troubleshooting

Page 13-15 of the Operating Instructions manual outlines typical problems which may arise while using the meter.

3. CONDUCTIVITY METER

3.1. Introduction

The YSI Model 30 Handheld Conductivity, Salinity and Temperature System simultaneously displays temperature and one of three parameters: conductivity, temperature compensated conductivity,[2] or salinity. Conductivity can be measured from 0 to 200 microsiemens[3] per centimeter (mS/cm) ± 0.5% full scale, salinity from 0 to 80 parts per thousand (ppt) ± 0.1 ppt, and temperature from -5 to 95 degrees Celsius (°C) ± 0.1°C.

3.2. Preparation

1. Ensure that batteries are installed properly (6 AA alkaline batteries).

2. Turn instrument on by pressing and releasing the **ON/OFF** button, the display should come on. The instrument will activate all segments of the display for a few seconds. A self-test procedure follows and lasts several more seconds. The cell constant will display after the self-test. The temperature will be displayed in the lower right corner of the display when the instrument is ready to take a reading.

3.3. Calibration

3.3.1. Annually

1. The meter is calibrated using a meter with platinum electrodes.

2. A five point calibration curve is established using potassium chloride standards.

3. The cell constant is recorded on a Laboratory Work Sheet.

---

[2] The temperature compensated conductivity measurement automatically adjusts the reading to a calculated value which would have been read if the sample had been at a certain reference temperature. When this temperature is specified to be 25°C, the temperature compensated conductivity is defined as specific conductivity.

[3] siemen = unit of electrical conductance; the reciprocal of an ohm; the equivalent to a mho.

3.3.2. Each Use

1. Each day before use, select a calibration solution (potassium chloride solution) which is most similar to the sample to be measured (for fresh water, choose a 1 mS/cm conductivity standard).

2. Place at least 3 inches of solution in a clean glass container.

3. Insert probe into the container deeply enough to completely cover the oval shaped hole on the side of the probe. Suspend the probe at least ¼ inch from the bottom of the container.

4. Allow at least 60 seconds for temperature reading to become stable.

5. Move the probe vigorously from side to side to dislodge air bubbles from the electrodes.

6. Press the up and down arrow keys simultaneously.

7. The **CAL** symbol will appear at the bottom of the display, the instrument is now in the calibration mode.

8. Use the up or down arrow key to adjust the reading on the display until it matches the value of the calibration solution that was used. When the display matches the calibration solution value, press the **ENTER** key once. The word **SAVE** will flash across the display to indicate the calibration value has been accepted.

3.4. Measurements

1. Collect each sample in a clean, quart size jar and note the time on the Laboratory Work Sheet. Samples must be analyzed within 15 minutes of collection.

2. Decant an aliquot of the sample into a clean, wide mouth container.

3. Insert probe into the container deeply enough to completely cover the oval shaped hole on the side of the probe. Try to not allow the probe to touch any solid surfaces.

4. The instrument is autoranging and may take up to 5 seconds to find the correct range. During the range search, the instrument will appear to freeze on a given reading for a few seconds, then once the range is located, will pinpoint the exact reading on the display. The display may also switch to **00.0** for a second or two during a range search before it selects the proper range.

5. Shake the probe vigorously to dislodge any air bubbles around the probe before taking the measurement.

6. Press the **MODE** key to change display for the three different readings: conductivity, specific conductivity, and salinity. Conductivity and specific conductivity have the same units, however, when specific conductivity is displayed, the °C symbol will flash on and off. The symbol will not flash when conductivity is displayed.

7. Rinse the probe and the wide mouth container with distilled water.

8. Perform a duplicate analysis for each sample collected by repeating steps 2 through 7.

9. Rinse the probe and the wide mouth container with distilled water between each sample analyses.

The operations manual should be consulted for setting the reference temperature as well as other advanced operations for this instrument.

3.5. Quality Control

1. Distilled water shall have at a minimum, resistivity values between 0.5 to 2.0 megohms-cm (2.0 to 0.5 micromhos/cm.) at 25 degrees C. Preferably, distilled water should have resistivity values greater than 1.0 megohms-cm (less than 1.0 micromhos/cm) at 25 degrees C. Containers of distilled water should be capped when not in use and should be capped immediately after each use.

2. Analytical reagent grade (AR) chemicals should be used for most analyses.

3. Stock and working standard solution shall be checked each day the meter is in use for signs of decomposition, including but not limited to discoloration, formation of precipitates, and concentration change due to evaporation;

4. All solutions shall be properly labeled with identification of the compound, concentration, solvent, date, and analyst who prepared the solution;

5. All glassware should be washed in a warm detergent solution, and thoroughly rinsed first in tap water and then in distilled water. The glassware shall be air dried and properly stored prior to use. This cleaning procedure is sufficient for most analytical needs.

6. Batteries will be checked each day when the meter is use, and replaced when necessary.

7. The probe and dedicated sample bottle will be rinsed with distilled water between each analyses.

8. Standard buffer solutions will be used within the appropriate expiration dates. Expired solutions will be disposed immediately in accordance with NJDEP disposal procedures.

9. A duplicate measurement should be taken for each sample collected to verify the accuracy of the method.

10. All quality control data and records shall be recorded on the Laboratory Work Sheet. Such retained data shall include but shall not be limited to, the results of and the raw data generated by Proficiency Sample analyses, and routine calibration reports.

3.6. Electrode Care and Maintenance

ALWAYS RINSE THE CONDUCTIVITY CELL WITH CLEAN WATER AFTER EACH USE.

To clean the conductivity cell:

1. Dip the cell in cleaning solution and agitate for two to three minutes.

2. Use the nylon brush to dislodge any particulates from inside the electrode chamber.

3. Repeat steps 1 and 2 until cell is completely clean.

4. Store the conductivity cell in the meter storage chamber.

3.7. Error Messages/Troubleshooting

1. Page 19 of the Operating Instructions manual outlines typical problems which may arise while using the meter.

4. SALINITY METER

4.1. Introduction

The YSI Model 30 Handheld Conductivity, Salinity and Temperature System simultaneously displays temperature and one of three parameters: conductivity, temperature compensated

conductivity,[4] or salinity. Conductivity can be measured from 0 to 200 microsiemens[5] per centimeter (mS/cm) ± 0.5% full scale, salinity from 0 to 80 parts per thousand (ppt) ± 0.1 ppt, and temperature from -5 to 95 degrees Celsius (°C) ± 0.1°C. Salinity is a calculation done by the instrument electronics, based upon the conductivity and temperature reading.

4.2. Preparation

1. Ensure that batteries are installed properly (6 AA alkaline batteries).

2. Turn instrument on by pressing and releasing the **ON/OFF** button, the display should come on. The instrument will activate all segments of the display for a few seconds. A self-test procedure follows and lasts several more seconds. The cell constant will display after the self-test. The temperature will be displayed in the lower right corner of the display when the instrument is ready to take a reading.

4.3. Calibration

1. It is not necessary to perform a separate calibration for salinity once the instrument has been calibrated for conductivity. A check against a salinity standard (50mS/cm salinity) is sufficient.

2. Place at least 3 inches of standard in a clean glass container.

3. Insert probe into the container deeply enough to completely cover the oval shaped hole on the side of the probe. Suspend the probe at least ¼ inch from the bottom of the container.

4. Allow at least 60 seconds for temperature reading to become stable.

---

[4] The temperature compensated conductivity measurement automatically adjusts the reading to a calculated value which would have been read if the sample had been at a certain reference temperature. When this temperature is specified to be 25°C, the temperature compensated conductivity is defined as specific conductivity.

[5] siemen = unit of electrical conductance; the reciprocal of an ohm; the equivalent to a mho.

5. Move the probe vigorously from side to side to dislodge air bubbles from the electrodes.

6. Press and release the MODE key to change the display for the three different readings: conductivity, specific conductivity, and salinity. When salinity is displayed the large number will be immediately followed by ppt.

7. Rinse the probe and container with distilled water.

4.4. Measurements

1. Collect each sample in a clean, quart size jar and note the time on the Laboratory Work Sheet. Samples must be analyzed within 15 minutes of collection.

2. Decant an aliquot of the sample into a clean, wide mouth container.

3. Insert probe into the container deeply enough to completely cover the oval shaped hole on the side of the probe. Try to not allow the probe to touch any solid surfaces.

4. The instrument is autoranging and may take up to 5 seconds to find the correct range. During the range search, the instrument will appear to freeze on a given reading for a few seconds, then once the range is located, will pinpoint the exact reading on the display. The display may also switch to **00.0** for a second or two during a range search before it selects the proper range.

5. Shake the probe vigorously to dislodge any air bubbles around the probe before taking the measurement.

6. Press the **MODE** key to change display for the three different readings: conductivity, specific conductivity, and salinity. When salinity is displayed the large numbers are followed by a ppt.

7. Rinse the probe and the wide mouth container with distilled water.

8. Perform a duplicate analysis for each sample collected by repeating steps 2 through 7.

9. Rinse the probe and the wide mouth container with distilled water between each sample analyses.

The operations manual should be consulted for setting the reference temperature as well as other advanced operations for this instrument.

4.5. Quality Control

1. Distilled water shall have at a minimum, resistivity values between 0.5 to 2.0 megohms-cm (2.0 to 0.5 micromhos/cm.) at 25 degrees C. Preferably, distilled water should have resistivity values greater than 1.0 megohms-cm (less than 1.0 micromhos/cm) at 25 degrees C. Containers of distilled water should be capped when not in use and should be capped immediately after each use.

2. Analytical reagent grade (AR) chemicals should be used for most analyses.

3. Stock and working standard solution shall be checked each day the meter is in use for signs of decomposition, including but not limited to discoloration, formation of precipitates, and concentration change due to evaporation;

4. All solutions shall be properly labeled with identification of the compound, concentration, solvent, date, and analyst who prepared the solution;

5. All glassware should be washed in a warm detergent solution, and thoroughly rinsed first in tap water and then in distilled water. The glassware shall be air dried and properly stored prior to use. This cleaning procedure is sufficient for most analytical needs.

6. Batteries will be checked each day when the meter is use, and replaced when necessary.

7. The probe and dedicated sample bottle will be rinsed with distilled water between each analyses.

8. Standard buffer solutions will be used within the appropriate expiration dates. Expired solutions will be disposed immediately in accordance with NJDEP disposal procedures.

9. A duplicate measurement should be taken for each sample collected to verify the accuracy of the method.

10. All quality control data and records shall be recorded on the Laboratory Work Sheet. Such retained data shall include but shall not be limited to, the results of and the raw data generated by Proficiency Sample analyses, and routine calibration reports.

4.6. Electrode Care and Maintenance

ALWAYS RINSE THE CONDUCTIVITY CELL WITH CLEAN WATER AFTER EACH USE.

To clean the conductivity cell:

1. Dip the cell in cleaning solution and agitate for two to three minutes.

2. Use the nylon brush to dislodge any particulates from inside the electrode chamber.

3. Repeat steps 1 and 2 until cell is completely clean.

4. Store the conductivity cell in the meter storage chamber.

4.7. Error Messages/Troubleshooting

1. Page 19 of the Operating Instructions manual outlines typical problems which may arise while using the meter.

ATTACHMENT B3

FIELD FORMS

CHAIN OF CUSTODY/LABORATORY ANALYSIS REQUEST FORM

Page ______ of ______ Turn Around Requested: ________

Report to:	Phone:				Analyses Requested										Notes/Comments
Company:	Fax:														
Address:	Proj Name:														
City:	Proj Number:														
State:	Sampler:														
Zipcode:															
Sample ID	Sample Date	Sample Time	Sample Matrix	No. Con-tainers											

Relinquished: (Signature)	Received by: (Signature)	Special Instructions/Notes	
Printed name:	Printed name:		
Company:	Company:		Number of Coolers:
			Cooler Temp(s):
Date: Time:	Date: Time:		COC Seals Intact?
			Bottles Intact?

integral consulting inc.
411 S 1st Avenue, Suite 550
Seattle, WA 98104
(206) 230-9600

Log of Borehole: ____________________

Project Name: ____________________

Project Number: ____________________

Logged by: ____________________

Date: ____________________

Page of

Depth to water: Initial____________ **Completion**____________________

SAMPLE INFORMATION									
Sample ID	**Tag #**	**Time**	**Sample Depth**	**% Recov.**	**PID (ppm)**	**Sheen**	**Depth (Feet)**	**Symbol**	**Soil Description** (USCS group name, minor components, color, moisture, additional descriptions)
							--		
							--		
							--		
							--		
							--		
							--		
							--		
							--		
							--		
							--		
							--		
							--		
							--		
							--		
							--		
							--		
							--		
							--		
							--		

Drilling Contractor: ____________________

Drilling Method/Equipment: ____________________

Sampling Equipment: ____________________

Start/End Time: ____________________

Latitude: ____________________

Longitude: ____________________

Notes: ____________________

Location Sketch

	FIELD CHANGE REQUEST	Project Number:

Field Change No.
Page _____ to

Project Number:
Project Name:

CHANGE REQUEST
Applicable Reference:
Description of Change:

Reason for Change:

Impact on Present and Completed Work:

Requested by:
Date: ____/____/

(Field Scientist)

Acknowledged by:
Date: ____/____/

(Field Task Leader)

FIELD OPERATIONS MANAGER RECOMMENDATION

Recommended Disposition:

Recommendation by:
Date: ____/____/

(Sampling and Analysis Coordinator)

PROJECT MANAGER APPROVAL

Final Deposition:

Approved/Disapproved by:
Date: ____/____/

(CERCLA Coordinator)

CORRECTIVE ACTION RECORD

Page ___ of

Audit Report No. :_____ Date:

Report Originator:

Person Responsible for Response:

DESCRIPTION OF PROBLEM:

Date and Time Problem Recognized: _____ By:

Date of Actual Occurrence: _____ By:

Analyte: _____ Analytical Method:

Cause of Problem:

CORRECTIVE ACTION PLANNED:

Person Responsible for Corrective Action:

Date of Corrective Action:

Corrective Action Plan Approval: _____ Date:

DESCRIPTION OF FOLLOW-UP ACTIVITIES:

Person Responsible for Follow-up Activities:

Date of Follow-up Activity:

Final Corrective Action Approval: _____ Date:

integral consulting inc.

SURFACE SEDIMENT/SOIL COLLECTION FORM

Project Name: ______________________ **Project No.** ______________ **Page:** ______

Date: __________ **Crew:** ______________________________

Weather: ______________________________

Sampling Method: ______________________________

Time: ________ **Station:** ______________ **Replicate:** ____ **Acceptable grab:** ☐ Yes ☐ No

Bottom Depth: ________ **Penetration Depth:** ________ **RPD Depth:** ________

Analyses before homogenization: ☐ **VOC** ☐ **Sulfides** ☐ **Other**

Sample ID: ______________________________

Type: ☐ cobble ☐ gravel ☐ sand C M F ☐ silt clay ☐ organic matter ☐ wood/shell fragments

Color: ☐ drab olive ☐ gray ☐ black ☐ brown ☐ brown surface

Odor: ☐ none ☐ slight ☐ moderate ☐ strong ☐ sulfidic ☐ petroleum ☐ other

Comments:

Time: ________ **Station:** ______________ **Replicate:** ____ **Acceptable grab:** ☐ Yes ☐ No

Bottom Depth: ________ **Penetration Depth:** ________ **RPD Depth:** ________

Analyses before homogenization: ☐ **VOC** ☐ **Sulfides** ☐ **Other**

Sample ID: ______________________________

Type: ☐ cobble ☐ gravel ☐ sand C M F ☐ silt clay ☐ organic matter ☐ wood/shell fragments

Color: ☐ drab olive ☐ gray ☐ black ☐ brown ☐ brown surface

Odor: ☐ none ☐ slight ☐ moderate ☐ strong ☐ sulfidic ☐ petroleum ☐ other

Comments:

Time: ________ **Station:** ______________ **Replicate:** ____ **Acceptable grab:** ☐ Yes ☐ No

Bottom Depth: ________ **Penetration Depth:** ________ **RPD Depth:** ________

Analyses before homogenization: ☐ **VOC** ☐ **Sulfides** ☐ **Other**

Sample ID: ______________________________

Type: ☐ cobble ☐ gravel ☐ sand C M F ☐ silt clay ☐ organic matter ☐ wood/shell fragments

Color: ☐ drab olive ☐ gray ☐ black ☐ brown ☐ brown surface

Odor: ☐ none ☐ slight ☐ moderate ☐ strong ☐ sulfidic ☐ petroleum ☐ other

Comments:

Time: ________ **Station:** ______________ **Replicate:** ____ **Acceptable grab:** ☐ Yes ☐ No

Bottom Depth: ________ **Penetration Depth:** ________ **RPD Depth:** ________

Analyses before homogenization: ☐ **VOC** ☐ **Sulfides** ☐ **Other**

Sample ID: ______________________________

Type: ☐ cobble ☐ gravel ☐ sand C M F ☐ silt clay ☐ organic matter ☐ wood/shell fragments

Color: ☐ drab olive ☐ gray ☐ black ☐ brown ☐ brown surface

Odor: ☐ none ☐ slight ☐ moderate ☐ strong ☐ sulfidic ☐ petroleum ☐ other

Comments:

The ELM Group, Inc. - Groundwater Sampling Field Data Log

SITE:	MONITORING WELL ID:

PROJECT #: WELL PERMIT #: DTW (BEFORE PUMP PLACEMENT): FT BTOC

DATE: WELL DEPTH (FT): DTW (AFTER PUMP PLACEMENT): FT BTOC

SAMPLER: WELL DIAMETER (IN): COLUMN HEIGHT (FT):

WEATHER: SCREENED INTERVAL (FT): CASING VOLUME (GAL): 1X: 3X: 5X:

PUMP INTAKE DEPTH (FT): PURGE METHOD:

PUMP TYPE: SAMPLE METHOD:

PID READINGS (ppm): BACKGROUND:____ BELOW OUTER CAP:____ BELOW INNER CAP:____ Page _____ of _____

TIME	PURGING	SAMPLING	pH	SPEC COND (mS/cm)	TURBIDITY (NTU)	DISSOLVED OXYGEN (mg/L)	TEMP (°C)	ORP (mV)	PURGE RATE (mL/min)	DEPTH TO WATER (FT BTOC)	NOTES
3-5 minutes btw rdgs			*+/- 0.1*	*+/- 3%*	*+/- 10%*	*+/- 10%*	*+/- 3%*	*+/- 10 mV*	*< 500 ml/min*	*Δ < 0.3 FT*	*Low Flow Parameters*

COMMENTS:

METER SOURCE: MODEL #:

VENDOR #: SERIAL #:

SAMPLE TIME:

ANALYSIS: ELM NJDEP Lab Certification #: 18004 Checked By: Date:

APPENDIX C

AIR MODELING PLAN

APPENDIX C
AIR MODELING PLAN

Perfluorinated Compounds Work Plan

Prepared for
Solvay Specialty Polymers USA, LLC
10 Leonard Lane
West Deptford, NJ 08086

Prepared by

200 Harry S. Truman Pkwy.
Suite 330
Annapolis, MD 21401

November 15, 2013

CONTENTS

LIST OF FIGURES

LIST OF TABLES

ACRONYMS AND ABBREVIATIONS

AERMET	AMS/EPA regulatory model
AERMOD	AMS/EPA meteorological preprocessor
AMP	air modeling plan
AMS	American Meteorological Society
BPIPPRM	Building Profile Input Program
EPA	U.S. Environmental Protection Agency
GEP	good engineering practice
NJDEP	New Jersey Department of Environmental Protection
NWS	National Weather Service
PFC	perfluorinated compound
PSD	particle size distribution
PVDF	polyvinylidene fluoride
Site	West Deptford, New Jersey, Plant
Solvay	Solvay Specialty Polymers USA, LLC
USGS	U.S. Geological Survey
UTM	Universal Transverse Mercator

1 INTRODUCTION

This document presents the air modeling plan (AMP) that has been prepared as part of the voluntary program for investigation of perfluorinated compounds (PFCs) in the environment at and near the Solvay Specialty Polymers USA, LLC (Solvay) West Deptford, New Jersey, Plant (Site). This document is Appendix C to the technical work plan, which provides additional information on the Site history.

The Site is located in Gloucester County, New Jersey, on the Delaware River, across from the Philadelphia Airport near river mile 90. Approximately 34 of the 243 acres owned by Solvay are currently being used for active manufacturing operations. This active portion of the Site is referred to as the Main Plan Area. The remaining acreage is either unused or leased for agriculture.

The primary objective of the work plan is to provide information on the extent to which long carbon-chain PFCs (i.e., C8 to C13) are present in the environment at and near the Site. As presented in the work plan and corresponding field sampling plan, environmental samples will be collected from water in the distribution systems of seven municipal utility authorities identified in the vicinity of the Site, existing onsite groundwater monitoring wells, and surface water and sediment at selected locations in the Delaware River. The primary objective of the air modeling is to evaluate pathways for offsite transport of PFCs by air dispersion and subsequent deposition to soil and surface water. The results will be considered together with measurements of PFCs from the proposed environmental sampling in order to guide decisions regarding future site characterization.

1.1 OVERVIEW

The proposed air modeling will be used to evaluate environmental sampling related to atmospheric releases of long carbon-chain PFCs over the period from 1985 to 2010. Specifically, the modeling will display estimates of the spatial distribution of concentrations in air and particle deposition to surfaces in the areas surrounding the Site. These results will inform a conceptual site model for PFC fate and transport. The modeling will use estimated historical air emissions information from the period when PFC air releases were occurring to estimate the concentration in air and deposition patterns. The procedures outlined in the AMP are consistent with relevant guidance on air modeling from the New Jersey Department of Environmental Protection (NJDEP) and the U. S. Environmental Protection Agency (EPA).

1.2 DOCUMENT ORGANIZATION

This AMP describes the model inputs and assumptions, options for alternative plausible scenarios, outputs, and analyses. Section 2 provides information on the historical air emission sources and quantities. Section 3 provides details on the Site relevant to the air model inputs. Section 4 provides a discussion on the air modeling approach. Section 5 discusses how the model results will be presented to support the project goals. Section 6 provides references.

2 PROJECT DESCRIPTION

The following sections provide details regarding the facility operations and air emission sources that will be the subject of the air modeling. The facility description provides an overview of the historical operations associated with the PFC air releases. This is followed by a detailed discussion of the various PFC air sources and their respective PFC air emission contributions. A summary of the relevant air emission source parameters is provided at the end of the section.

2.1 FACILITY LAYOUT

Historically, two fluorosurfactants containing PFCs were used in the production of polyvinylidene fluoride (PVDF) at the facility. An emulsion polymerization process, incorporating the fluorosurfactant, the monomer, and water, was used at the Site for the production of PVDF. The primary air emissions were associated with the spray dryer operation at the end of the process where the PVDF product was dried. The majority of the PVDF manufacturing and handling processes were conducted in an enclosed building located in the northwestern portion of the Site (Figure C-1). A small percentage of the PFC-containing material was conveyed from the reactor vessel via closed piping to containers immediately outside the PVDF manufacturing building for offsite disposal.

2.2 AIR EMISSION ESTIMATES

Estimates of the historical air emissions were developed by Solvay based on a mass balance approach using historical Site records (Solvay 2013). An annual release was calculated by apportioning the total amount of PFC-containing fluorosurfactant used during the year for the PVDF manufacturing process to the various downstream components, including the final product and the various associated waste streams. Periodic sampling was available to estimate the PFC concentration in all of the downstream components except the air emissions. Thus, the annual PFC air emission was estimated as the difference between the amounts used in the PVDF manufacturing process and the amount in the other downstream components. These best available emissions estimates covering the period of PFC usage at the Site (1985–2010) will be used in the modeling.

2.2.1 Primary Stack Sources

The spray dryer stacks were the primary sources of PFC air emissions based on the nature of the emulsion polymerization process historically used in the PVDF manufacturing at the Site. In the spray dryer, the emulsion was introduced into a hot airstream, which evaporated the excess water, which in turn cooled the airstream. The cooler airstream was then routed through a

fabric filter to capture the dry PVDF particles before the airstream was released to the atmosphere through the spray dryer stack. During this process the PFC surfactant would sublime in the heated airstream and migrate through the dryer as a vapor. This PFC vapor would then condense to fine particles as the gas stream cooled in the stack and was released to the ambient air.

Although these are permitted stack sources, there are no stack sampling records available to characterize the emissions from the spray dryer stack during the period of interest for this modeling exercise. The uncertainty introduced by this data gap will be explored through the sensitivity analyses proposed in Section 4.3.

2.2.2 Secondary Stack and Vent Sources

There are additional locations in the PVDF manufacturing process at the Site where air emissions could occur. For convenience these sources can generally be grouped as wet or dry sources depending on whether they occurred upstream or downstream of the spray dryer. The wet sources were small stacks that vented tanks where the emulsion was processed. These wet sources are considered secondary PFC air emission sources relative to the spray dryer stacks because the flow rates were significantly lower. The concentrations in the airstream would have also been lower because the PFC surfactants tended to remain with the polymers in these portions of the PVDF manufacturing process. The dry sources were primarily related to materials handling that occurred after the PVDF product had exited the spray dryer. These stacks are also considered secondary sources relative to the spray dryer stacks for the same reasons.

There are no stack sampling records available to characterize the emissions from these secondary sources. As discussed above, based on process knowledge it is assumed that the mass released from these sources would have been a small fraction of what was released from the spray dryer stack. Furthermore, it is likely that due to the low exit velocities and/or downward angle of the emissions, they would have likely been influenced by the building wake and ultimately exerted maximum concentration and deposition impacts much nearer the release point than the spray dryer stack, akin to a fugitive emission. Thus, the spray dryer stacks are expected to have a much greater contribution to the air concentration and deposition impacts with distance from the process building and are, therefore, the critical sources for this modeling exercise to understand the spatial extent and distribution of deposition. The uncertainty introduced by this conceptualization will be explored through the sensitivity analyses proposed in Section 4.3.

2.2.3 Fugitive Emissions

The only potential fugitive emission source relevant to this modeling exercise is related to the waste material collected for offsite disposal. The containers had covers and were located

adjacent to the process building. The primary dust source would likely be the fraction of material that was on the ground immediately around the containers. Advection of this material by ambient wind would be the primary release mechanism to the atmosphere. Given the number of structures and other effective wind breaks around this potential fugitive dust source, fugitive emissions are unlikely to represent a significant air emission source of PFCs to the environment. Furthermore, like the secondary source, any fugitive emissions would likely be influenced by the building wake and ultimately exert maximum concentration and deposition impacts much nearer the release point than the spray dryer stack, making fugitive emissions an insignificant source for the purpose of this larger domain modeling exercise. Accordingly, this fugitive emission source will not be considered further in the quantitative estimates of offsite transport of PFCs.

2.3 SOURCE PARAMETERS

The air emission sources of interest for this modeling exercise are all point sources. The source parameters required for modeling point sources are the constituent emission rate, the stack height and diameter, and the temperature and velocity of the airstream at the stack exit. As discussed in Section 2.2, the spray dryer stacks are the primary emission sources for this evaluation. The source parameters for the spray dryer stacks will be developed based on the permit values for the period of PFC air emissions. The source parameters for the secondary sources (see Section 2.2.2) will be discussed in Section 4.3 as part of the proposed model sensitivity analysis.

3 SITE DESCRIPTION

The Site is located in a mixed use area with commercial/industrial facilities and rural residential and agriculture areas dominating the local land use. The Site is bounded to the north by the Delaware River. Across the river from the Site is the city of Philadelphia. This section provides an overview of the Site with respect to characteristics relevant to the proposed air modeling.

3.1 LAND USE ANALYSIS

The land use analysis is used to configure the air model to simulate the dispersion characteristics of the area over which air concentration and deposition are to be estimated. Land use in an area can influence dispersion by affecting the atmospheric turbulence. Generally, rural areas will exhibit less atmospheric turbulence than urban areas because fewer buildings and development implies lower surface roughness and heating.

EPA provides a methodology for determining whether or not the land use should be classified as urban or rural in their *Guideline on Air Quality Models* (40 CFR Part 51, Appendix W). The method is based on a scheme that classifies 12 typical land uses as either urban or rural. The classification scheme is applied to land within a 3-km radius of the source and the dominant land use type (i.e., urban or rural) is applied for the air modeling. We will apply this procedure to the Site area using topographic maps from the U.S. Geological Survey (USGS) to characterize the land use over the period of atmospheric PFC emissions from the Site. The land use analysis will be documented in the report produced to convey the basis for air modeling assumptions and sensitivity of corresponding results.

3.2 LOCAL TOPOGRAPHY

Terrain features that are high enough to intercept stack emissions before they have a chance to disperse within the plume can lead to elevated concentrations and deposition relative to flat terrain. As confirmed by topographic maps for the area, no such terrain features exist in the Site area. Accordingly, the air modeling will be conducted assuming flat terrain.

3.3 STACK HEIGHT ANALYSIS

Buildings can exert an aerodynamic influence on stack emissions that causes the plume to be quickly mixed to the ground, potentially increasing air concentrations and deposition. A good engineering practice (GEP) stack height analysis will be conducted as part of this modeling effort in order to account for building aerodynamic effects in the air model. The analysis will follow NJDEP (2009b) guidance for GEP stack height analysis. The results of the analysis will

be documented in the air modeling report. Additionally, EPA's enhanced version of the Building Profile Input Program (i.e., BPIPPRM) will be used with the building analysis to construct the necessary parameters to simulate the building effects in the air model. The focus of this effort will be on the spray dryer stack, which represents the primary air emission source in this evaluation; however, the analysis will also be relevant to the secondary sources as they are located in close proximity to the spray dyer stack.

3.4 METEOROLOGICAL DATA

A representative meteorological data set for use in the air modeling is critical to producing a reliable simulation of the dispersion and deposition of atmospheric releases from the Site. To be representative, the meteorological data must come from a monitoring station in close proximity and having similar terrain as the Site to be modeled.

The National Weather Service (NWS) collects surface weather observations at the Philadelphia International Airport, located less than 4 miles from the Site. This NWS station meets the conditions to provide representative meteorological data for modeling air emissions from the Site given the close proximity and similarity in terrain. NJDEP offers a 5-year meteorological data set generated from observations collected at the Philadelphia International Airport NWS station from 1990 to 1994. NJDEP air modeling guidance (2009b) states that 5 years of representative NWS meteorological data should be used when estimating air concentrations and deposition with an air model.

As per NJDEP (2009b) air modeling guidance, the Bureau of Technical Services in the NJDEP Division of Air Quality will be consulted to determine if this data set is available and appropriate for the proposed air modeling effort. If such is not the case, then the necessary 5-year meteorological data set for the period from 1990 to 1994 will be generated using observations from the Philadelphia International Airport NWS site paired with upper air data from the Atlantic City, New Jersey, and Brookhaven, New York, stations, as was done by NJDEP. The air modeling report will provide details of the relevant data processing steps if the NJDEP data set cannot be used.

4 PROPOSED AIR MODELING METHODOLOGY

The purpose of the proposed modeling is to estimate the potential spatial extent and distribution of PFCs transported by air associated with historical site operations. This section will provide details of the air model and proposed modeling approach for meeting the project goals. A sensitivity analysis is also proposed to evaluate the potential influence on model predictions of uncertainties inherent in such a modeling exercise.

4.1 SELECTED AIR MODEL

The air model selection process for this project followed NJDEP guidance (2009b) for modeling air quality impacts. The use of a screening model for this effort was rejected because the project goals required evaluating deposition, in addition to air concentrations, from multiple sources. The American Meteorological Society (AMS)/EPA regulatory model (AERMOD) was selected from the set of refined air models listed in NJDEP guidance. AERMOD has the capacity to model multiple sources, evaluate wet and dry deposition, and address building aerodynamic effects.

4.2 MODELING APPROACH

For the purpose of discussion, the modeling approach has been separated into two sections: 1) dispersion modeling; and 2) deposition modeling. The model will be configured to estimate both air concentration and deposition simultaneously, so the assumptions and approaches described may apply to each component.

4.2.1 Dispersion Modeling

As a refined air model, AERMOD has a significant number of options and associated inputs that allow the user to tailor the model's execution to meet individual needs. This section will describe the options and inputs that are most significant for this modeling effort.

4.2.1.1 Model Control Parameters

There are several options in AERMOD that determine the emissions scenario that the model represents. The option to operate the model in default mode, which sets all options to reflect regulatory defaults, will not be used in this evaluation. Instead, the non-default option to assume flat terrain in the modeled area of the Site will be used in conjunction with all other regulatory default options. NJDEP (2009b) air model guidance notes that this deviation from the regulatory default options is appropriate in most New Jersey locations because of the flat terrain.

4.2.1.2 Chemical Phase

The model will be configured to calculate both concentration and total deposition flux (i.e., wet and dry deposition) of particles. Gas-phase deposition will not be considered in this modeling as the PFC emissions are assumed to be exclusively in particle form. The option to include depletion will be selected for both wet and dry deposition. The model will be configured to provide annual averages of both the air concentration and deposition flux.

4.2.1.3 Source Parameters

For this model evaluation only point sources will considered in AERMOD to reflect direct emissions from stacks in the PVDF manufacturing process. The actual location of each of the stacks will be identified using Universal Transverse Mercator (UTM) coordinates. Because the modeling is being conducted assuming flat terrain, the source elevation will not be required.

For each point source the model requires the following parameters: emission rate in grams per second, release height above ground in meters, stack gas exit temperature in degrees Kelvin, stack gas exit velocity in meters per second, and stack inside diameter in meters. A preliminary review of the site historical data indicates that the annual air emission rate varied over the period during which PFC emissions would have occurred. Thus, a unit emission rate (i.e., 1 g/s) will be used to model the potential impacts for each of the spray dryer stacks alone, and the model predictions will be scaled by the respective annual emission rates to convert them to annual estimates. A multi-source analysis including the secondary stacks and vents identified in Section 2.2.2 will be conducted as part of the sensitivity analysis presented in Section 4.3.

4.2.1.4 Receptors

The receptor grid used for this analysis will begin with NDJEP (2009b) modeling guidance recommendations. Accordingly, a Cartesian grid will be specified in the model as follows:

- Receptors along the fence line, which is closer to the source than the property line, at 50-m spacing
- Receptors with 50-m spacing to a distance of 0.5 km of the fenceline
- Receptors with 100-m spacing between 0.5 and 1.5 km of the fenceline
- Receptors with 250-m spacing between 1.5 and 3 km of the fenceline
- Receptors with 500-m spacing between 3 and 5 km of the fenceline.

The model results will be inspected to ensure that concentrations and deposition flux are clearly decreasing near the edge of the receptor grid, indicating that the receptor grid was sufficient to capture the location of the point of maximum impact. Additional modeling using a fine grid (i.e., 50-m spacing) may be conducted to better characterize the potential maximum(s) if they

are predicted to occur within a region of the original grid with coarse spacing. There are no elevated open-air locations of interest for this modeling project, so "flag-pole" receptors will not be included in the modeling.

4.2.1.5 Meteorological Data

The meteorological data needed for AERMOD were discussed in Section 3.4. A 5-year data set from 1990 to 1994 will be used with surface observations from the nearby NWS station at Philadelphia International Airport, and upper air data from a combination of the Atlantic City, New Jersey, site, and the Brookhaven, New York, site. NJDEP offers to the public such a data set for use in AERMOD, and the Bureau of Technical Services in the NJDEP Division of Air Quality will be consulted to determine if this data set is appropriate for the proposed air modeling effort. For example, this modeling exercise includes deposition modeling, so the necessary precipitation data must be included in the AERMOD meteorological input. If the NJDEP data set is not adequate, then the appropriate data set will be generated using EPA's AERMET (AMS/EPA regulatory model) (USEPA 2012) program and observations from the meteorological stations identified above. Details of the AERMET processing will be provided as part of the modeling report.

4.2.2 Particle Size Distribution and Deposition Modeling

The focus of this modeling effort is the spatial distribution of historical atmospheric emissions of PFCs from the Site. As discussed earlier, the primary source for the PFC emissions were the spray dryer stacks. Based on the nature of PFCs and the manufacturing process used at the Site, the emissions from these stacks will ultimately be modeled primarily, if not exclusively, in a particle form. There are no stack tests to verify the particle size distribution (PSD) of PFCs in the spray dryer stacks, and no future measurements are possible since PFC use was discontinued in facility operations by 2010. There are stack tests of long chain PFC fluorosurfactants conducted for spray dryers from similar manufacturing processes. These data are presented in Table C-1, which shows the PSD for perfluorooctanoic acid measured either in stacks or at the property fence line. In all cases the mass fraction of the PSD is dominated by particles that are less than 1 μm in diameter. This is expected to be the case for the PFC emissions from the Site spray dryer stacks.

When conducting particle deposition modeling in AERMOD, the user has two options for specifying the PSD. When more than 10 percent of the PSD mass fraction has a diameter of 10 μm or larger, or when the PSD is well known, then Method 1 is used. Conversely, Method 2 is used when the PSD in not well known and when less than 10 percent of the PSD mass fraction has a diameter of 10 μm or larger. For this modeling exercise, Method 2 is the most appropriate option for modeling the PFC particle deposition. Assuming that the majority of the particle emissions are fine particles will tend to bias the model deposition results to predict

impacts further downwind of the source relative to larger particles which settle out of the plume faster.

The required particle inputs for AERMOD Method 2 are the fraction of mass emitted with a diameter of 2.5 μm or less, and the representative mass-mean aerodynamic particle diameter. Because of the uncertainty in these parameter values, a sensitivity analysis will be conducted using various combinations of the parameter values to determine a potential range of deposition estimates. The selected values and their justification will be provided in the modeling report.

4.3 SENSITIVITY ANALYSIS

In any modeling analysis there are numerous sources of uncertainty that will affect the model results. Several sources of uncertainty have been identified for this modeling exercise and a sensitivity analysis is proposed to evaluate their relative impacts on the model results. The three components to be evaluated in the analysis are the PFC emissions rate, the source contribution, and the particle deposition modeling. The approach for evaluating each of these components is presented below.

4.3.1 PFC Emissions Rate

The estimated annual emissions of PFC to air vary over the period of use at the Site. Generally, there is a linear relationship between the model input emission rate and the model predicted air concentrations and deposition flux. Thus a unit emission rate is proposed for model runs whereby the spray dryer stacks are the only sources evaluated. In this case the model predictions can be multiplied by the estimated emission rate for a given year to make the model predictions specific to a given year. For this modeling exercise the model predictions will be scaled by the annual emission rate for each year PFC air emissions estimates are available to develop a range of potential spatial distributions. These estimates will be used to assess the sensitivity of model results to plausible ranges of emission rates.

4.3.2 Source Contribution

As discussed in Section 2, the potential sources of PFC air emissions have very different characteristics. The spray dryer stacks will produce the greatest plume rise of the potential PFC air sources and will, therefore, produce concentrations and deposition impacts farther downwind than the others. This is an important consideration to the project goal of identifying the spatial distribution of historical PFC emissions in the vicinity of the plant. The remaining sources were constructed in such a way that vertical momentum of the exhaust is low and/or directed downwards or horizontally. Given the placement of these sources relative to buildings, these emissions were likely to be affected by the aerodynamic wake of the buildings,

leading to maximum concentration and deposition impacts much closer to the source. This hypothesis will be tested by assigning each of the secondary sources (identified in Section 2.2.2) some portion of the total annual PFC air emissions with the remainder going to the spray dryer stacks. The resulting model predictions will be evaluated to determine the contributions of secondary sources to the spatial distribution of PFC concentration and deposition estimates.

4.3.3 Particle Deposition Modeling

As no stack data are available to characterize the PSD in the historical PFC air emissions, assumptions have been made based on similar substances and processes. As discussed in Section 4.2.2, assuming the majority of the particles are less than 10 μm will tend to produce deposition impacts that are biased further from the source. A sensitivity analysis of the particle deposition modeling will be conducted by varying the input values used for the fine mass fraction and mass-median diameter in Method 2 of AERMOD. No attempt will be made to use Method 1, which assumes that the PSD is known and includes at least 10 percent of the mass fraction in particles 10 μm or larger. The range of values used in the Method 2 sensitivity analysis will be developed from best professional judgment based on the PFC properties and the manufacturing process that was used. The values will be fully documented in the modeling report along with a discussion on the model sensitivity and relevant conclusions for the project recommendations.

5 PRESENTATION OF AIR MODELING RESULTS

The air modeling results will be conveyed to NJDEP in a report that provides text, tables, and figures to support critical review of the modeling approach and conclusions. Tables will be provided that summarize each of the AERMOD inputs and their source. The model-predicted concentrations and deposition will also be summarized in both tabular and graphical format. Graphical formats will include isopleths of concentrations and deposition flux shown on maps of the Site vicinity. The AERMOD model inputs and outputs will be provided in an electronic format along with the modeling report.

6 REFERENCES

Barton, C.A., L.E. Butler, C.J. Zarzecki, J. Flaherty, and M. Kaiser. 2006. Characterizing perfluorooctanoate in ambient air near the fence line of a manufacturing facility: Comparing modeled and monitored values. *J. Air Waste Manage. Assoc.* 56:48-55.

NJDEP. 2009a. DuPont Chambers Works telomer manufacturing air dispersion and deposition of perfluorooctanoic acid. Internal memorandum from J. Jenks to S. Maybury, Bureau of Case Management, dated January 22, 2009. New Jersey Department of Environmental Protection, Division of Air Quality, Bureau of Technical Services, Air Quality Evaluation Section, Trenton, NJ.

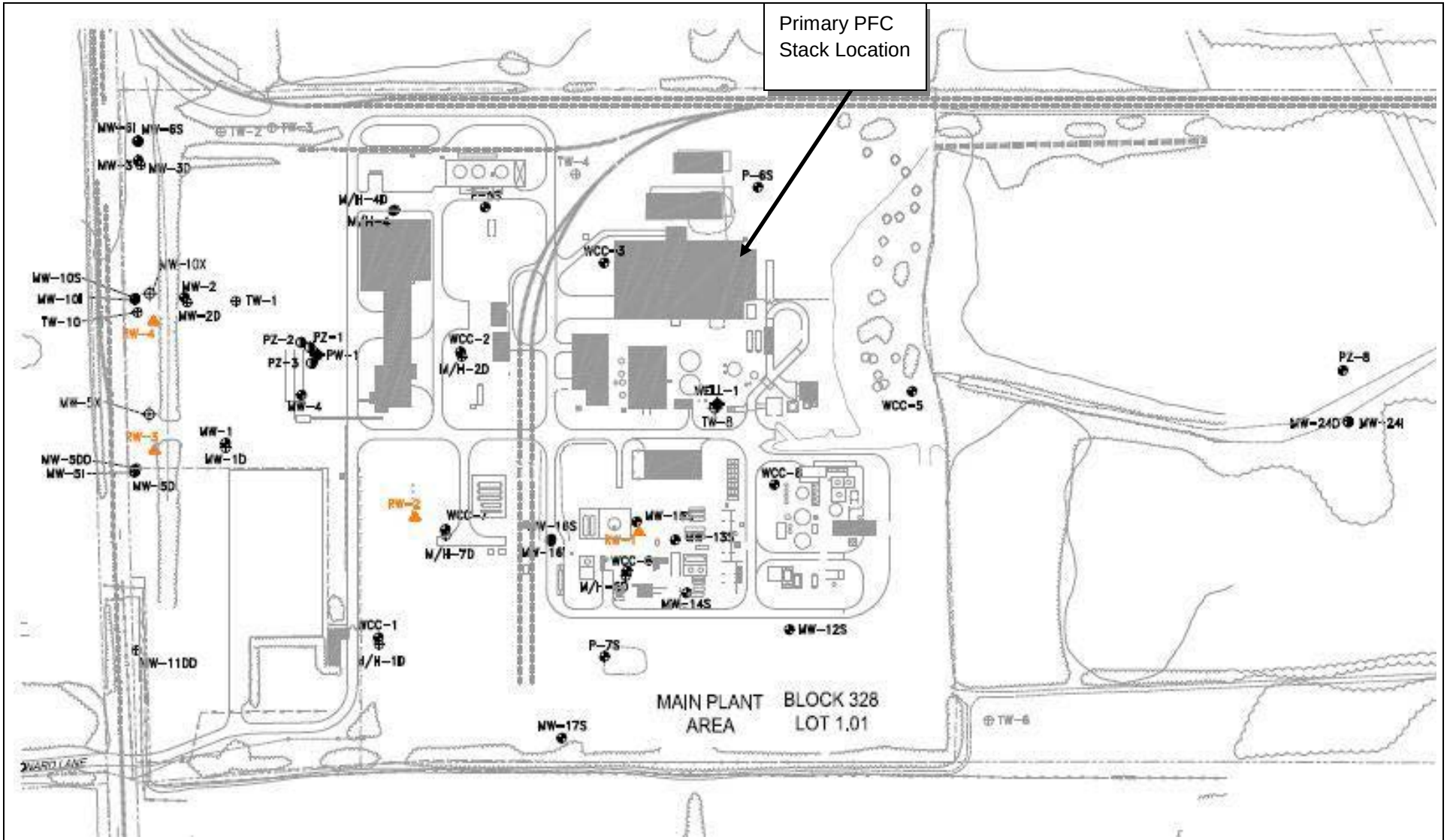
NJDEP. 2009b. Guideline on air quality impact modeling analysis. Technical Manual 1002. New Jersey Department of Environmental Protection, Division of Air Quality, Bureau of Technical Services, Trenton, NJ. November.

Paustenbach, D.J., J.M. Panko, P.K. Scott, and K.M. Unice. 2007. A methodology for estimating human exposure to perfluorooctanoic acid (PFOA): A retrospective exposure assessment of a community (1951-2003). *J. Toxicol. Environ. Health Part A.* 70:28-57.

Solvay. 2013. NJDEP PFC Meeting presentation. Solvay Specialty Polymers, NJ. 25 pp. September.

USEPA. 2012. User's guide for the AERMOD meteorological preprocessor (AERMET) (EPA-454/B-03-002, November 2004). Addendum. U.S. Environmental Protection Agency, Office of Air Quality Planning and Standards, Air Quality Assessment Division, Research Triangle Park, NC. December.

FIGURES



integral consulting inc.

Figure C-1.
Solvay West Deptford Facility Plot Plan Showing Location of PFC Primary Air Emission Source

TABLES

Table C-1. Particle Size Information for Air Emissions

Table C-1a. Particle Size Distribution for PFOA Emissions (Based on Particle Size Data Collected from the Fine Powder Packed Bed Scrubber Exhaust in 1996)

Minimum Particle Size for Category (µm)	Maximum Particle Size for Category (µm)	Mean Particle Size (µm)	Mass Fraction (%)
3.0	5.0	4.0	3.3
1.0	3.0	2.0	12.7
0.5	1.0	0.75	3.5
0.3	0.5	0.4	26.7
0.1	0.3	0.2	53.8

Source: Paustenbach et al. (2007)

Notes:
PFOA = perfluorooctanoic acid

Table C-1b. Average PSD for PFOA at Fenceline DuPont, WV

Particle Diameter (µm)	Mass Fraction (%)
>4.0	5.6
1.7	12.9
0.8	9.2
0.5	7.2
0.3	5.3
<0.28	59.8

Source: Barton et al. (2006)

Notes:
PFOA = perfluorooctanoic acid
PSD = particle size distribution

Table C-1c. Particle Size Distribution for PFOA Emissions (1995)

Particle Diameter (µm)	Mass Fraction (%)
4.0	3.3
2.0	12.7
0.75	3.5
0.4	26.7
0.2	53.8

Source: NJDEP (2009a)

Notes:
PFOA = perfluorooctanoic acid