U.S. ENVIRONMENTAL
PROTECTION AGENCY

QUALITY ASSURANCE PROJECT PLAN FOR REGION 10

Revision 0

LUST Sites in Indian Country

EPA Contract No. EP-W-12-009

June 2014

Prepared for:

U.S. Environmental Protection Agency
Office of Underground Storage Tanks
Ariel Ross Building
1200 Pennsylvania Avenue, N.W.
Washington, DC 20460

Bristol
ENVIRONMENTAL REMEDIATION SERVICES, LLC
111 W. 16th Avenue, Third Floor, Anchorage, Alaska 99501

Lust Sites in Indian Country

QUALITY ASSURANCE PROJECT PLAN FOR REGION 10
Performance Period: May 27, 2014 - April 18, 2015
Effective upon EPA Approval

Approval Signatures (required prior to project start):

Scott Ruth Bristol Environmental Remediation Services, LLC Program Manager	Date
Julie Sharp-Dahl Bristol Environmental Remediation Services, LLC Project Manager	Date
Patricia Curl Bristol Environmental Remediation Services, LLC Quality Assurance Manager	Date
Rob Rau U.S. Environmental Protection Agency Subject Matter Expert	Date
Gina Grepo-Grove U.S. Environmental Protection Agency Quality Assurance Manager	Date

TABLE OF CONTENTS

SECTION **PAGE**

ACRONYMS AND ABBREVIATIONS v
1.0 SITE INFORMATION 1
1.1 Site Descriptions 1
1.2 Site History 3
2.0 PROJECT MANAGEMENT 5
2.1 A3 – Distribution List 5
2.2 A4 – Project Organization 6
2.3 A5 – Problem Definition/Background 8
2.4 A6 – Project/Task Descriptions and Schedule 9
2.4.1 Contaminants of Concern and Analytical Methods 10
2.5 A7 – Quality Objectives and Criteria 11
2.5.1 Program Quality Objectives 12
2.5.2 Program Objectives 13
2.6 A8 – Special Training/Certifications 15
2.6.1 Policy QA Training for Management and Staff 16
2.6.2 Process for Identifying, Ensuring, and Documenting 16
2.7 A9 – Documentation and Records 18
2.7.1 Transmittal 19
2.7.2 Distribution 20
2.7.3 Retention 20
2.7.4 Access 20
2.7.5 Preservation 21
2.7.6 Traceability 21
2.7.7 Retrieval 21
2.7.8 Removal of Obsolete Documentation 22

2.7.9 Disposition 22
3.0 DATA GENERATION AND ACQUISITION 23
3.1 B1 – Sampling Process and Design....23
3.2 B2 – Sampling Methods....24
3.3 B3 – Sample Handling and Custody....25
3.3.1 Sample Custody....26
3.4 B4 – Analytical Methods 26
3.5 B5 – Quality Control Requirements 28
3.5.1 QC Samples 28
3.5.2 Analytical Data 29
3.5.3 Corrective Action 32
3.5.4 Storage and Disposal of Investigation-Derived Wastes 33
3.6 B6 – Field Instrument/Equipment Testing, Inspections and Maintenance....33
3.7 B7 – Field Instrument/Equipment Calibration and Frequency....35
3.8 B8 – Inspection/Acceptance Requirements for Supplies and Consumables 35
3.9 B9 – Data Acquisition Requirements (Non-Direct Measurements)....36
3.10 B10 – Data Management 36
4.0 ASSESSMENT AND OVERSIGHT 39
4.1 C1 – Assessments/Oversight and Response Actions 39
4.1.1 Performance System Audits 39
4.1.2 Process Ensuring Verification of Subcontractor's Conformance to Customer Requirements 39
4.1.3 Process Ensuring Verification of the Suppliers Conformance to Customer Requirements 41
4.1.4 Corrective Actions 41
4.2 C2 – Reports to Management 43

5.0 DATA VALIDATION AND USABILITY 45
5.1 D1 – Data Review, Validation, and Verification Requirements 45
5.2 D2 – Data Validation and Verification Process 46
5.2.1 Data Validation and Verification Process 46
5.2.2 Data Reporting 46
5.2.3 Data Usability 47
5.3 D3 – Reconciliation with User Requirements 48

FIGURE

Figure 1 Organization Chart

APPENDICES

Appendix A Laboratory Quality Assurance Manual, Standard Operating Procedures, and Control Limits
Appendix B QAPP Amendment Form
Appendix C Field Forms and Equipment Manuals
Appendix D Bristol Standard Operating Procedures
Appendix E All LUST Sites Covered by this QAPP

(Intentionally blank)

ACRONYMS AND ABBREVIATIONS

%	percent
°C	degrees Celsius
Bristol	Bristol Environmental Remediation Services, LLC
BTEX	benzene, toluene, ethylbenzene, and xylenes
CFR	Code of Federal Regulation
COC	contaminants of concern
DQOs	data quality objectives
EDB	ethylene dibromide
EPA	U.S. Environmental Protection Agency
FTP	file transfer protocol
IDW	investigation derived waste
km^2	square kilometer
LCS	laboratory control sample
LCSD	laboratory control sample duplicate
LUST	leaking underground storage tank
MRL	method reporting limit
MS	matrix spike
MSD	matrix spike duplicate
MTCA	Model Toxics Control Act
NWTPH	Northwest Total Petroleum Hydrocarbon
ORP	oxidation reduction potential
PARCCS	precision, accuracy, representativeness, completeness, comparability and sensitivity
PCS	petroleum-contaminated soil
pH	potential of hydrogen
PID	photoionization detector

ACRONYMS AND ABBREVIATIONS (continued)

QA	quality assurance
QAM	Quality Assurance Manual
QAPP	Quality Assurance Project Plan
QC	quality control
RCRA	Resource Conservation and Recovery Act
RPD	relative percent difference
SME	Subject Matter Expert
SOP	standard operating procedure
SW	EPA Solid Waste Test Method
TBD	To Be Determined
TCLP	Toxicity Characteristic Leaching Procedure
TERO	Tribal Employment Rights Office
TO	task order
TPH	total petroleum hydrocarbons
UST	underground storage tank
VOCs	volatile organic compounds

1.0 SITE INFORMATION

This *Quality Assurance Project Plan* (QAPP) addresses tasks planned for the Yakama and Quinault Indian Reservations in Washington. The Environmental Protection Agency (EPA) issued these task orders (TOs) to Bristol Environmental Remediation Services, LLC (Bristol) in 2014, under Contract No. EP-W-12-009. The TO include site assessments and remediation activities at two sites:

- 1006 Site Assessment and Remediation at Western Gas, Yakama Indian Reservation, EPA Facility No. 4260105
- 1009 Site Assessment and Remediation at the Former A.E. Erickson Site, Quinault Indian Reservation, EPA Facility No. 415007

1.1 SITE DESCRIPTIONS

The 1.3-million-acre **Yakama Indian Reservation** is a United States Indian reservation of the federally recognized tribe, the Confederated Tribes and Bands of the Yakama Nation. The Tribe is made up of Klikitat, Palus, Wallawalla, Wanapam, Wenatchi, Wishram, and Yakama peoples. The reservation is located in south-central Washington State, within Yakima, Lewis, and Klickitat counties. According to the United States Census Bureau, the reservation covers 2,186 square miles (5,661 km²), with approximately 31,800 inhabitants recorded in the 2000 census. The majority of the population is concentrated in the eastern third of the reservation within or near the communities of Toppenish and Wapato. The remainder of the reservation consists of several small communities (including Brownstown, Harrah, and White Swan) and sparsely settled farm land, rangeland, and forested areas.

The reservation was created in 1855 by a treaty signed by Washington Territory Governor Isaac Stevens and representatives of the Yakama tribe. Several leaders believed that those representatives did not have the authority to cede land, and had not gained consensus from the full council or tribe. A dispute over the treaty led to the Yakima War (1855-1858).

Following the Bannock War of 1878, the United States government forced the Northern Paiute people out of Nevada—although most had not been involved in the war—and into the Yakama Reservation. The more than 500 Paiute were subjected to privation for years before being allowed to return to Nevada. The Paiute returning to Nevada meant stricter competition for resources and housing by all the peoples. The Paiute did not return to Nevada until the 1880s.

The **Quinault Indian Reservation** is located on the Pacific coast of Washington, primarily in northwestern Grays Harbor County, with small parts extending north into southwestern Jefferson County. It has a land area of over 316 square miles (819.294 km²) and a population of 1,370 as of the 2000 census. This population, part of the modern Quinault Tribe, is made up of the Quinault, Hoh, Chehalis, Chinook, Cowlitz, Queets, and Quileute peoples. Linguistically, these groups belong to three language families: Chimakuan (Quileute, Hoh), Chinookan (Chinook groups), and Salishan (Chehalis, Cowlitz, Queets, and Quinault).

The Quinault people settled onto reservation lands after signing the Quinault Treaty with the former Washington Territory in 1856. About 60% of the reservation's population lives in the community of Taholah, on the Pacific coast at the mouth of the Quinault River.

It is not possible to traverse the entire reservation on Highway 109, in spite of what some online mapping services indicate. Construction of the highway north from Taholah to U.S.

Highway 101 was halted in the late 1960s. There is only limited access (for private property owners and tribe members) along the northern coast of the reservation.

Specific site descriptions are located in Appendix E.

1.2 SITE HISTORY

The LUST SITES IN INDIAN COUNTRY QAPP is designed as an overarching QAPP which describes how each of the four major QAPP elements will be addressed for specific Leaking Underground Storage Tank (LUST) activities. The four major QAPP categories include: Project Management, Data Generation and Acquisition, Assessment and Oversight, and Data Validation and Usability.

The Project Management section provides an overview of the LUST activities for each TO issued to date for work to be performed on the Yakama and Quinault Indian Reservations in Washington, including project history and objectives and contractor roles and responsibilities. The Data Generation and Acquisition section describes the specific field and analyses tasks per LUST activity (i.e., project design and implementation, sampling methods, measurement and analysis, data collection or generation, data handling). The Assessment and Oversight section describes for each LUST activity how assessment of project implementation effectiveness and quality assurance/quality control (QA/QC) activities will be performed. The Data Validation and Usability section addresses the QA activities after data collection and ensures the data conform to the project criteria and objectives for each LUST activity.

This QAPP details LUST-site related activities:

- Site Assessment
- Remediation
  - Excavation
  - Application of Chemical Oxidant
- Sampling
- Monitoring Well Installation and Development

Appendix E contains the site-specific details for the Western Gas and A. E. Erickson LUST sites (TOs 1006 and 1009), including a detailed descriptions of the planned activities, performance and data quality objectives (DQOs) and practices.

The EPA Region 10 implements the underground storage tank (UST) program in Indian Country in Alaska, Washington, Oregon, and Idaho. The EPA provides technical and financial support to tribal governments to prevent and cleanup petroleum releases from USTs. The regional office uses LUST Trust Fund moneys to directly clean up some sites within the boundaries of the reservation.

Owners and operators of USTs on tribal lands must comply with federal UST regulations. The EPA is committed to protecting human health and the environment in Indian Country while supporting tribal self-government, acting consistently with the federal trust responsibility and strengthening the government-to-government relationships between tribes and the EPA. For more than 20 years the EPA has been fulfilling these commitments by providing financial and technical assistance to tribal governments for the prevention and cleanup of UST releases, by supporting tribal governments in their capacity to build and improve tribal UST programs and by directly implementing the underground storage tank program in Indian Country. Bristol is under contract to support EPA Region 10 with the implementation of the UST program in Indian Country.

2.0 PROJECT MANAGEMENT

2.1 A3 – DISTRIBUTION LIST

Name: Title: Organization: Contact Information: Phone: Email:	Rob Rau Subject Matter Expert EPA Region 10 1200 6th Avenue, Suite 900, OCE082, Seattle, WA 98101 (206) 553-6285 rau.rob@epa.gov
Name: Title: Organization: Contact Information: Phone: Email:	Gina Grepo-Grove Quality Assurance Manager EPA Region 10 1200 6th Avenue Suite 900, Mail Stop-OEA-095, Seattle WA 98101 (206) 553-1632 Grepo-Grove.Gina@epamail.epa.gov
Name: Title: Organization: Contact Information: Phone: Email:	Scott Ruth Program Manager Bristol Environmental Remediation Services, LLC 111 West 16th Avenue, Third Floor, Anchorage, AK 99501 (907) 563-0013 sruth@bristol-companies.com
Name: Title: Organization: Contact Information: Phone: Email:	Julie Sharp-Dahl Project Manager Bristol Environmental Remediation Services, LLC 111 West 16th Avenue, Third Floor, Anchorage, AK 99501 (907) 563-0013 jsharp-dahl@bristol-companies.com
Name: Title: Organization: Contact Information: Phone: Email:	Patricia Curl Quality Assurance Manager Bristol Environmental Remediation Services, LLC 111 West 16th Avenue, Third Floor, Anchorage, AK 99501 (907) 563-0013 pcurl@bristol-companies.com

Name: Title: Organization: Contact Information: Phone: Email:	Melissa Armstrong Project Manager TestAmerica Tacoma 5755 Eighth Street East, Tacoma, WA 98466 (253) 922-2310 melissa.armstrong@testamericainc.com
Name: Title: Organization: Contact Information: Phone: Email:	Eric Orwoll Northwest Manager Emerald Services 1500 Airport Way South, Seattle, WA 98134 (206) 255-5737 eorwoll@emeraldnw.com
Name: Title: Organization: Contact Information: Phone: Email:	Neil Kranz Project Manager Pacific Soil and Water, Inc. 9790 SW Pembrook St., Tigard, OR 97224 (503) 995-4463 neil.kranz@pacificsoilandwater.com
Name: Title: Organization: Contact Information: Phone: Email:	To Be Determined (TBD) Excavation subcontractor Quinault Tribal Employment Rights Office (TERO) TBD TBD TBD

2.2 A4 – PROJECT ORGANIZATION

An organization chart is included as Figure 1.

Scott Ruth, Bristol Program Manager, will be responsible for the UST program and will resolve any project/program problems or deviations, including QA/QC issues. He will keep informed on work accomplished in this plan and resolve problems or deviations, including QA/QC issues.

Patricia Curl, Bristol QA Manager, is independent from the unit generating and using the data. She will be responsible for approving this QAPP. Ms. Curl will also ensure that the EPA QA Program is implemented, resolve any QA issues, and approve project reports.

Julie Sharp-Dahl, Bristol Project Manager, will have the responsibility for the project schedule, budget, QAPP, managing day-to-day project activities, hiring and overseeing subcontractors, ensuring QA/QC for project activities, coordination of laboratory services, and reporting. Julie Sharp-Dahl has the overall responsibility for all field activities.

Pacific Soil and Water, Inc., of Tigard, Oregon will decommission two groundwater monitoring wells, install and develop one new groundwater monitoring well, and inject chemical oxidants into the targeted subsurface treatment zone at the Western Gas site.

A local subcontractor, yet to be determined, will perform the excavation work at the A. E. Erickson site. Bristol will apply the chemical oxidant to the bottom of the excavation at the soil-groundwater interface.

TestAmerica of Tacoma, Washington will provide analytical services and technical support to both projects, following the current Laboratory QA Manual (QAM), located in Appendix A.

Emerald Service, Inc. of Seattle, Washington will dispose of the petroleum-contaminated waste soil from the A. E. Erickson removal action; they will also dispose of the waste water from monitoring well installation, development, and sampling at the Western Gas site (and potentially the A. E. Erickson site).

The subcontractor will drill, install, and develop groundwater monitoring wells at the A. E. Erickson site will be identified on the QAPP Amendment Form (Appendix B) if this follow-on work is awarded.

Bristol personnel will collect field data and samples for laboratory analysis, including groundwater samples and soil confirmation and characterization samples, and will assess the analytical data quality from the Western Gas and A. E. Erickson sites.

All reports to the EPA of project field activities will be prepared by Bristol.

2.3 A5 – Problem Definition / Background

The EPA's regulations concerning cleanup of UST releases are found in Title 40 of the Code of Federal Regulations (40 CFR) Part 280, Subpart E, F, and G. Subpart G describes the release reporting, investigation and confirmation requirements. The release response and corrective action for UST systems containing petroleum substances are included in Subpart F. Subpart G lists the requirements for out of service tanks and closure.

The purpose of the Western Gas site work effort is to decommission two onsite groundwater monitoring wells (MW-1 and MW-2); replace compliance well MW-2 (located at the southeast corner of the site and adjacent to Elm Street) with a new monitoring well (MW-2B), which will serve as a point of compliance; collect and analyze groundwater samples from the new well; dispose of all investigation derived waste; perform chemical remediation injections in accordance with the 2013 *Corrective Action Plan*; and provide a report of site activities.

The purpose of the A. E. Erickson work effort is to remove petroleum-contaminated soil (PCS), collect and analyze soil confirmation and soil waste characterization samples, treat the soil-groundwater interface (excavation bottom) with a chemical remediation product, and make recommendations for the next course of action.

This QAPP describes the quality objectives and measures necessary to achieve adequate data that can be used to support decisions and, ultimately, close both project sites. It

describes the upcoming work; sampling procedures and data collection methods for soil, groundwater, and waste samples; number of environmental samples to be collected; field parameters to be measured; methods for monitoring-well decommissioning, installation, and development at the Western Gas site; PCS removal, transportation, and disposal at the A. E. Erickson site; and the chemical remediation treatment that will be used at both sites. Potential follow-on monitoring well installation, development, and sampling at the A. E. Erickson site, which is alluded to in the TO# 1009 Performance Work Statement, is not addressed in this QAPP. If this work is awarded, the QAPP Amendment Form (Appendix B) will be used to provide specifics like the location and number of wells to install, the number of groundwater samples to collect, and the chemical analyses to perform.

Facility information for each LUST site is provided in the Site-Specific History sections of Appendix E.

2.4 A6 – Project/Task Descriptions and Schedule

Bristol and subcontractors are tasked with assessment and remediation work the Western Gas and A. E. Erickson sites:

Western Gas Site

- Decommission monitoring well.
- Install monitoring well.
- Develop monitoring well.
- Collect and analyze groundwater samples.
- Apply onsite chemical remedial treatment.
- Characterize waste.
- Dispose waste.

- Prepare a technical memorandum documenting the site assessment and remediation work.

A. E. Erickson Site

- Remove petroleum-contaminated soil.
- Field screen to guide excavation and confirmation sampling.
- Collect and analyze soil confirmation samples.
- Apply onsite chemical remediation treatment
- Characterize waste.
- Dispose waste.
- Prepare a report documenting the site assessment and remediation work; provide recommendations for any follow-up work in the report.

Appendix E provides site-specific information for both LUST sites. At a later date, TO 1009 may be expanded, in which case Bristol will install, develop, and sample groundwater monitoring wells to characterize groundwater quality and hydrology at the site. Although this QAPP addresses the possible additions, the tasks are not detailed in Appendix E because the work has yet to be scoped.

2.4.1 Contaminants of Concern and Analytical Methods

The contaminants of concern (COC) in soil and/or groundwater samples from both sites will be analyzed with the Northwest Total Petroleum Hydrocarbon Methods (NWTPH) and EPA's Solid Waste Methods (SW). Total petroleum hydrocarbons as gasoline (TPH-gas), diesel (TPH-diesel), and heavy oil (TPH-heavy oil) will be analyzed with NWTPH Methods NWTPH-Gx for gas, and NWTPH-Dx extended for diesel and heavy oil. Volatile organic compounds (VOCs) including benzene, toluene, ethylbenzene, and xylenes (BTEX) will be analyzed with EPA Method SW8260.

The waste transportation and disposal facility specified which methods to use for characterizing investigation-derived waste: VOCs by SW8260; and Resource Conservation and Recovery Act (RCRA) metals arsenic, barium, cadmium, chromium, lead, mercury, selenium, and silver (RCRA 8) plus copper, nickel and zinc by EPA Methods SW6020 and 7471 (for mercury). If Toxicity Characteristic Leaking Procedure (TCLP) metals are required for PCS disposal, EPA Method SW1311 will be used for extraction, and methods SW6020 and SW7471 will be used for analysis.

Environmental samples will be shipped to the TestAmerica Laboratory in Tacoma, Washington for analysis. TestAmerica Tacoma's QAM, applicable laboratory Standard Operating Procedures (SOPs), and laboratory limits for all referenced methods are included in Appendix A, along with the laboratory Electronic Chain-of-Custody Form.

Bristol will perform the laboratory data verification per the requirements of this QAPP.

Soil analytical results will be compared to the Washington State Department of Ecology Toxics Cleanup Program Model Toxics Control Act (MTCA)'s revised (2013) Method A Soil Cleanup Levels for Unrestricted Land Use. Groundwater analytical results will be compared to MTCA Method A Cleanup Levels for Groundwater. In all cases, the EPA will determine final cleanup goals for the site.

2.5 A7 – Quality Objectives and Criteria

Planning is conducted using established project management procedures, and the DQO process, as defined by the EPA, where applicable. The DQO process is used, where appropriate, to identify specific data and the level of QA commensurate with the intended use of the data.

Precision, bias, accuracy, representativeness, completeness, comparability and sensitivity are described in Section 3.5.2. The laboratory control criteria, reporting limits and method detection limits for the soil and groundwater analytical methods are presented in Appendix A.

Site-specific data generation and acquisition (sampling and analysis) requirements are listed at Appendix E.

2.5.1 Program Quality Objectives

One objective for this program is to develop and implement sampling, sample handling and field measurement procedures that will provide data that are adequate for decision making. The quality of data from field and laboratory data collection activities that are needed to support specific decisions or regulatory actions is described below.

Analytical results for the Western Gas site replacement-well groundwater samples will be used to monitor attenuation of petroleum hydrocarbons at the point of compliance (new monitoring well MW-2B) until Method A cleanup levels are attained. Field data collected during well purging, such as pH, dissolved oxygen, etc. will be used to determine if the groundwater coming into the well is representative of aquifer conditions and will be used to determine when to collect the groundwater sample, as well as provide information on status of the natural attenuation process.

Analytical results for soil confirmation samples from the PCS excavation at the A. E. Erickson site will be used to determine if COCs above MTCA A cleanup levels remain in the soil or if all of the PCS has been removed. A field screening method for volatile hydrocarbons and a field screening method for total petroleum hydrocarbons (TPH) will be used to guide the excavation of the PCS, and the same method will be used to determine when the PCS above MTCA A cleanup levels has been removed (or until the

excavation volume reaches contractual limits) and confirmation soil samples for laboratory analysis can be collected.

If groundwater monitoring well installation and sampling at the A. E. Erickson site is recommended and if the EPA decides to award follow-on work at this site, the analytical results from the water samples will be used to delineate the nature and extent of groundwater contamination. Field data collected during well purging, such as pH, dissolved oxygen, etc., will be used to determine if the groundwater coming into the well is representative of aquifer conditions and will be used to determine when to collect the groundwater sample, as well as provide an indication if the plume is undergoing natural attenuation. Additionally, depth to water will be measured for each well and this data will be used to determine the groundwater flow direction.

Bristol and the EPA discussed which chemical oxidation product-type would work best for each site with Regenesis, the company that will provide the chemical oxidation materials designed to remediate soil and groundwater contamination in-situ. Their recommendations are based on the highest concentrations reported in the most recent available soil and groundwater data for each site.

Appendix E details the site-specific project information for both sites addressed in this QAPP; the work design is based on identifying project objectives, specifying the data necessary to meet project objectives, and 3) describing the methods that will yield data of acceptable quality and quantity to support the required decisions.

2.5.2 Program Objectives

The program objectives for the Western Gas site are to reduce concentrations of COCs in subsurface soil in the southeast corner of the property and to subsequently restore the groundwater quality at the site to concentrations below MTCA Method A groundwater

cleanup levels to minimize the potential for exposure to humans and the environment. The technology selected for the corrective action is chemical oxidation and enhanced bioremediation achieved through injection of an oxidant into the subsurface at or around the groundwater soil interface, as described in the *Corrective Action Plan* that was developed for this site in 2013. Groundwater sampling and monitoring will be conducted at the onsite compliance well until the analytical results show that the groundwater petroleum hydrocarbon concentrations are below MTCA A cleanup levels.

The compliance well (MW-2) historically used for sampling is in poor condition and needs to be replaced. Monitoring well MW-2 will be decommissioned in accordance with State of Washington regulations and the replacement compliance well, MW-2B, will be installed and developed. Groundwater samples for laboratory analysis will be collected from MW-2B to determine current petroleum hydrocarbon concentrations in the groundwater, and to evaluate if the concentrations have decreased as a result of the remedial treatments conducted to date. After the groundwater samples are collected, another treatment will take place (described in Appendix E.1); this treatment was approved in the2013 CAP which is included as an attachment to Appendix E.1.

The program objectives for the A. E. Erickson site are to perform a site assessment and cleanup activities that will support the eventual closure of this LUST site. Petroleum contaminated soil, which is the source of any potential groundwater contamination, will be removed and a chemical oxidant will be applied to the soil-groundwater interface to enhance bioremediation. Details regarding the treatment of petroleum-contaminated zone at this site are provided in Appendix E.2.

Groundwater monitoring well installation, development, sampling, and analysis for COCs may be performed at the A. E. Erickson site in the future, depending on the results of the PCS removal.

2.6 A8 – Special Training/Certifications

Field personnel must be trained and certified as hazardous site workers per 29 CFR 1910.120(c). Bristol and their subcontractors are responsible for maintaining their own training records. Bristol receives required training in-house or through environmental training companies which offer Occupational Safety and Health Administration, Department of Transportation and EPA approved safety and hazardous materials training classes. In-house training is conducted by Bristol's corporate safety officer or other safety staff. Field staff performing environmental screening and sampling must have adequate training and experience in operating a photo-ionization detector (PID), logging borings and collecting soil samples, installing and developing ground water monitoring wells, measuring groundwater stability parameters, and measuring water levels and collecting samples from monitoring wells. Bristol has an experienced hazardous waste manager who oversees all waste disposal tasks.

All Bristol employees meet at least minimum qualifications established for their particular job and functional discipline. Project managers are responsible for assigning only qualified personnel to project activities. For the field tasks detailed in this QAPP Bristol will provide staff (or subcontractors) with special training or certifications that will include:

- Hazardous Waste Operations and Emergency Response Training
- Resource Conservation and Recovery Act Hazardous Waste Regulations
- Site-specific Health and Safety Training, Including Hazard Communication, Respiratory Protection, Personal Protective Equipment, and other topics
- Professional Licenses/Certifications

- Professional Engineer
- Registered, Certified, or Professional Geologist

2.6.1 Policy QA Training for Management and Staff

Bristol has long recognized that a constant and vigorous management and staff training program is critical for continued success. Not only is training provided to project team members to ensure technical and quality requirements are understood, an ongoing corporate training schedule is also maintained to strengthen the skills of employees and to provide for career development. Bristol's senior management and project managers assess the need for staff training and for qualifications of staff for specific projects before work starts. General training plans are included as part of ongoing personnel development and are addressed annually as an integral part of the individual performance review process.

As a professional services company, Bristol's activities often require professionally registered staff (e.g., Professional Engineer, Professional Geologist, etc.) or technically certified staff (e.g., Certified Industrial Hygienist, Certified Safety Professional, Certified Tank Inspector, etc.). Bristol encourages and supports individual employees in pursuing additional training, education, and certifications and provides incentives to employees in the form of increased compensation. Bristol also provides the resources for our professionals to maintain their current registrations.

2.6.2 Process for Identifying, Ensuring, and Documenting

2.6.2.1 Quality-Related Qualifications

Bristol maintains a file of registrations and certifications for each employee, which includes the retraining or expiration date of each license or certification. Bristol's safety staff reviews these files on a regular basis and advises employees of these dates. Prior to the assignment of any employee to a project requiring professional registration or

certifications, the file is reviewed and a copy of the required certification or registration is placed in the project file for incorporation into the planning documents.

Bristol's safety department and Senior Management maintain and review the certification/registration files. The program manager assigns only persons with the necessary qualifications to the project.

2.6.2.2 Process for Maintaining Quality-Related Qualifications

Bristol maintains training records for all its employees, and arranges for initial and refresher training as applicable to the work.

Employees are encouraged to participate in professional societies, continue their formal education, and continue to develop their professional skills. Preparation and presentation of technical papers is also encouraged by allowing the time on the job and allocating financial resources to do so. Bristol offers a comprehensive tuition reimbursement program for employees pursuing advanced degrees. Company resources, including time on the job, payment of course fees, and travel are incentives provided for professional development activities.

Bristol encourages the employee to maintain their registrations and certifications, but ultimately it is the responsibility of the employee to do so. The program manager reviews the qualifications of individuals performing projects within the program and when qualifications need renewal advises the employee in ample time to do so. It is Bristol's policy to have only qualified, certified, or registered personnel filling positions requiring such certifications, so senior management has made time and financial resources available to the employee to do so.

2.6.2.3 Process for Retraining Based on Changing Requirements

All staff working on environmental projects are required to complete a 40–hour hazardous waste and emergency response training course and an 8-hour refresher training annually. All staff are required to complete Department of Transportation Hazardous Materials Transportation training and refresher training (49 CFR 172.700-704) for the proper labeling, packaging, and shipment of hazardous materials. All Project Managers are required to complete RCRA Hazardous Materials/Waste training (40 CFR 260-272) in the proper manifesting, treating/disposal of hazardous waste.

Bristol's Project Managers maintain frequent communication with the EPA Subject Matter Expert (SME) and move quickly to train key personnel based on changing requirements.

Bristol ensures that the employees and subcontractors working on a project have the training and experience needed to meet the project requirements, and the Bristol safety requirements.

2.7 A9 – Documentation and Records

This QAPP is prepared and maintained by Julie Sharp-Dahl, the Bristol project manager for EPA Region 10 projects awarded under the UST/LUST Program. It is her responsibility to review and update the QAPP as required (at least annually) and ensure that individuals on the distribution list are provided a copy of the latest version of the revised QAPP on a timely basis. The QAPP will be reviewed before the anniversary of its approval, and the review will be documented with the EPA Region 10 QA Document Review Crosswalk; annual QAPP review documentation will be submitted to the EPA.

TestAmerica Tacoma will provide the analytical laboratory report via email directly to the Bristol project manager. Laboratory documents and records are described in the laboratory QAM located in Appendix A. Document records for this project also include information generated in the field (e.g., field forms, field log books, and Chain of Custody Forms). Field forms are included in Appendix C. The SOP for populating a field notebook is included in Appendix D. The TestAmerica Chain-of-Custody Form is located in Appendix A.

The EPA requires field-activities report upon the completion of each TO. The report will describe project objectives, site activities (when applicable), findings, and conclusions; it will also give recommendations. The draft reports will be provided to the EPA SME via email. The final reports will be provided to the EPA SME and to tribal representatives identified by the Tribe's environmental program manager, or other tribal entities as required, in hardcopy and electronic formats such as compact disk (CD).

Bristol's process for transmitting, distributing, retaining, accessing, preserving, tracking, and retrieving documents are detailed below.

2.7.1 Transmittal

Draft planning documents and reports will be submitted electronically by Bristol to the EPA, either through email or the Bristol File Transfer Protocol (FTP) site if the document is too large to be emailed. The email subject line will reference the TO number, and recipients of the draft document will include, at a minimum, the EPA SME and Project Officer. Transmittal of all final documents to the EPA and the Tribal representatives is made under a letter of transmittal identifying the contract number, TO number, name of the document, number of copies, other recipients of the document, and if electronic copies are included.

2.7.2 Distribution

Bristol will transmit the final documents to the tribe and/or other project stakeholders, at the request of the EPA SME.

2.7.3 Retention

The Bristol program manager and project managers are responsible for project document control. All completed project records will be maintained in a secure location in the Bristol Anchorage, Alaska office where they can be accessed as necessary and protected from deterioration. Hard copy and electronic data shall be archived in project files and on electronic media for the duration of the project, or 10 years, whichever is longer.

Project documentation includes paper and electronic files, field logbooks, laboratory reports, plans and reports, letters, memoranda, internal and external communications, calculations, decisions, and results.

2.7.4 Access

Any document considered to be confidential or that cannot be reproduced from public accessible files shall be maintained in a locked file cabinet with access controlled by Bristol's program or project manager.

All routine files are maintained in a secure location in Bristol's Anchorage, Alaska office where they can be accessed as necessary and protected from deterioration.

All access to Bristol's LAN/WAN system is limited to Bristol employees and password protected. All electronic data transmittals are encrypted. In rare cases, a separate secure drive is established and access controlled to client-agreed upon users.

All Bristol employees sign a confidentiality agreement upon initial hire and during each review of the Conflict of Interest Plan.

2.7.5 Preservation

Project files are stored on the Bristol network in Bristol's LAN/WAN environment and are maintained, backed up, and archived by the Bristol Information Services Department. Active file maintenance, archival, and records destruction periods are determined depending upon the type of information and client and legal requirements. Electronic (digital) records are stored for a minimum of 10 years.

All hard copy files are maintained in a temperature and humidity controlled office or storage space in steel file cabinets during the duration of the project and cardboard file boxes when archived. The archival storage space is secure, temperature and humidity controlled, and not subject to flooding or environmental hazards.

2.7.6 Traceability

Every Task Order is assigned a unique Bristol project number. That project number is placed on all documents, field logbooks, subcontract documents, and invoices, and all correspondence. All project files and documents are stored by the project number, both electronically and in hard copy. Project plans and reports are numbered sequentially with the revision number and the date of the revision on the document. During QC review and the reproduction process, a cover sheet is used to track document status as well as the person who has worked with the document. Sequential copies of the document are filed to provide the entire history of the document.

2.7.7 Retrieval

Electronic files are readily retrieved from Bristol's network server. Non-sensitive hard-copy files for current projects are located in the project manager's office. Completed project hard-copy files are archived in off-site storage.

2.7.8 Removal of Obsolete Documentation

Nonspecific and working files are often created that may in part be incorporated into the final product. These files are removed at project closeout by the project manager.

2.7.9 Disposition

All archived files are stored in file boxes with the project name, Bristol project number, contract number and date of disposal marked on the file box. The file storage area is surveyed at least annually, and files scheduled for destruction are shredded and disposed at the regional landfill.

The entities generating and using data include the Bristol project manager, Bristol field staff, subcontractors, and the EPA SME. All of these entities are independent from the Bristol QA manager.

3.0 DATA GENERATION AND ACQUISITION

All of the procedures and methods that will be used to collect environmental samples and field measurements are documented in the Bristol SOPs, listed below and included in Appendix D.

SOP Number	SOP Title, Revision Number, Date
BERS-01	Soil Sampling, Revision 2, February 17, 2010
BERS-02	Groundwater Sampling, Revision 2, February 16, 2010
BERS-03	Sample Management, Revision 1, February 23, 2010
BERS-04	Field Measurement and Test Equipment, Revision 1, February 5, 2010
BERS-05	Equipment Decontamination, Revision 2, February 23, 2010
BERS-06	Borehole Logging, Revision 2, April 1, 2010
BERS-07	Monitoring Well Installation, Revision 1, January 4, 2010
BERS-08	Water Level Measurement, Revision 1, Fberuary 23, 2010
BERS-09	Investigation-Derived Waste (IDW) Management, Revision 1, February 23, 2010
BERS-11	Field Documentation, Revision 0, January 5, 2010
BERS-12	Excavation and Trenching, Revision 1, April 20, 2010
BERS-13	General Backfill and Compaction, Revision 0, March 9, 2010
BERS-16	Photoionization Detector, Revision 1, March 14, 2013

The procedures in the SOPs are described in detail to ensure that the data represent the environmental conditions at each site. There is a variety of sample and field data collection methods detailed in select SOPs. The specific methods that Bristol will employ at the Western Gas and A. E. Erickson sites are described in Appendix E.

3.1 B1 – SAMPLING PROCESS AND DESIGN

Environmental samples and field data will be collected from groundwater monitoring wells and excavations.

Appendix E provides site-specific information for each site:

- Descriptions and histories
- Summary of the objectives
- Site maps
- Description and rationale for the sampling design

Variability in project data and outcome will be minimized by following the approved QAPP. Potential sources of variability include varying site conditions and analytical or field data quality.

3.2 B2 – Sampling Methods

Sampling will follow SOPs BERS-01 and BERS-02 (Appendix D) for soil and groundwater sampling, respectively. Site-specific sampling details are contained in Appendix E. In the event there is a conflict between the QAPP and the SOP, procedures in the QAPP will be followed. Any deviation from the QAPP will be recorded in the field notebook. No continuous monitoring will be performed. No samples will be composited, split, or filtered.

Field data will be collected according to BERS SOPs (Appendix D). Field documentation will follow SOP BERS-11. Field instruments and equipment will be operated as described in the instrument and equipment manuals (Appendix C). Monitoring well depth to water will be measured as described in SOP BERS-08 and will follow the instructions contained in the *Solonist 122 Equipment Manual*. Water quality field parameters—pH, oxidation-reduction potential (ORP), dissolved oxygen (DO), etc.—will be collected with a YSI and will follow the instructions contained in the YSI equipment manual, in accordance with SOP BERS-04. A PID will be used to field screen soil for volatile organic vapors during excavation work, in accordance with SOP BERS-16; personnel will follow the instructions contained in the MiniRAE Lite or the QRAE II equipment manual; the PID model to be

used will be determined prior to mobilization. Personnel will use the Hanby Soil Test Kit instructions included in Appendix C to field screen soil for TPH. Non-dedicated sampling equipment will be decontaminated between sample collections, as described in SOP BERS-05.

3.3 B3 – Sample Handling and Custody

TestAmerica Tacoma laboratory will analyze environmental samples. Laboratory requirements for groundwater samples, including the sample containers, preservatives, and technical hold times for the analyses, are listed in Appendix E.1 Table E.1-2 and Appendix E.2 Table E.2-4. The frequency for environmental sample QC requirements is presented in Section 3.5, Appendix E.1 Table E.1-2, and Appendix E.2 Table E.2-4. Sample containers will be labelled according to the sample identification system presented in Appendix E.1 Table E.1-4, and Appendix E.2 Table E.2-7.

Bristol prepares a Bill of Landing for waste disposal, which is signed by the EPA SME prior to shipping waste off site. The Bill of Lading will accompany the waste to the disposal facility; a copy is held by Bristol until the signed Bill of Lading is provided by the disposal company. A subcontractor transports the waste to the facility in a manner that is consistent with DOT requirements. The subcontractor hands the bill of lading to the waste disposal company representative who signs it and sends a copy back to Bristol. This process is repeated if the waste needs to be transported to another facility. Bristol will receive the final Bill of Lading, as well as the disposal paperwork that documents the waste's final resting place. Bristol will provide the documentation to the EPA in an appendix to the report.

3.3.1 Sample Custody

To ensure that samples are identified correctly and remain representative of the environment, the documentation and sample custody procedures specified in this section will be followed during sample collection and analysis. Standard sample documentation and custody procedures, as detailed in SOP BERS-03 (Appendix D), will be used to maintain and document sample integrity during collection and during transportation by a commercial carrier. The field task manager will be responsible for ensuring that proper documentation and custody procedures are initiated at the time of sample collection and that individual samples can be tracked from the time of sample collection until the samples are relinquished to the laboratory. The laboratory will be responsible for maintaining sample custody and documentation from the time the samples are relinquished to the laboratory until final sample disposition. The laboratory QAM located in Appendix A provides detail on the laboratory process for maintaining custody during sample receipt, storage, and analysis.

3.4 B4 – Analytical Methods

The SOPs that the TestAmerica Tacoma laboratory uses for the analytical methods contained in the EPA *Test Methods for Evaluating Solid Waste*, SW-846, Final Update IV, Third Edition (USEPA 2008) are included in Appendix A. Soil samples will be analyzed for TPH-gas, and TPH-diesel and heavy oil by the Washington Department of Ecology's NWTPH methods (NWTPH-Gx, and NWTPH-Dx extended) and BTEX by EPA Method SW8260B. The groundwater samples will be analyzed for TPH-gas, and TPH-diesel and heavy oil with the NWTPH methods, and BTEX or VOCs will be analyzed by EPA Method SW8260B. If EPA decides that testing for ethylene dibromide (EDB) in

groundwater is required, EDB will be analyzed by SW8011. The laboratory SOPs for the analytical methods to be used are listed below and are included in Appendix A.

SOP Number	SOP Title, Revision Number, Date
TA-QAM	TestAmerica Quality Assurance Manual, Revision 7, April 23, 2014
TA-MV-0376	Gasoline Range Organics Analysis [Methods 8015B, NWTPH-Gx], Revision 12, May 21, 2014
TA-GS-0339	Semivolatile Petroleum Products Method for Soil and Water [Method NWTPH-Dx mod], Revision 13, May 28, 2013
TA-MV-0312	Determination of Volatile Organic Compounds by GC-MS [Methods 8260B, 8260C and 624], Revision 2, February 10, 2014
TA-MT-0217	Inductively Coupled Plasma-Mass Spectrometry (ICP-MS) [Methods 6020, 6020A, 200.8], Revision 24, February 10, 2014
TA-MT-0202	Mercury Analysis by CVAA [Methods 245.1, 7470A, 7471A], Revision 2, April 23, 2014
TA-GS-0365	Determination of EDB and DBCP in Aqueous and Solid Matrices [Methods SW 8011 and EPA 504.1], Revision 10, May 10, 2013
TA-IP-0210	Toxicity Chracteristic Leaching Procedure [TCLP, Method 1311] and Synthetic Precipitation Leaching Procedure [SPL, Method 1312], Revision 13, January 28, 2013

Soil waste characterization samples will be analyzed for RCRA 8 metals plus copper, nickel, and zinc, and if a RCRA metals concentration is above the 1:20 (cut) criteria, the sample will be extracted by the TCLP and analyzed as previously described. Project data requirements for both sites do not require any modifications to the listed methods. Analytical methods, containers, sample preservation, and holding time requirements are described in the site-specific sections of Appendix E.

The laboratory turnaround time for data reporting is 21 calendar days from the time the samples are received. The laboratory report(s) will be provided to the Bristol project manager. The laboratory is responsible for sample disposal. Samples must be disposed a minimum of 30 days after the final results are reported.

3.5 B5 – QUALITY CONTROL REQUIREMENTS

3.5.1 QC Samples

The following sections describe the QC sample types that will be used to evaluate analytical data quality for the samples collected from each site. Specific QC sample types, quantities, and sampling locations for each site are detailed in Appendix E. Analytical laboratory QC samples for each method include a method blank, a laboratory control sample (LCS) and a laboratory control sample duplicate (LCSD). A 1:10 dilution is performed on one of the project samples during metal analyses to determine if a physical interference may be suppressing the internal standards signals.

3.5.1.1 Duplicate Samples

Duplicate sample results are used to assess precision of the sample collection process. A field duplicate sample is a second sample collected at the same location as the original sample. When used, duplicate samples are collected simultaneously or in immediate succession, using identical recovery techniques, and treated in an identical manner during storage, transportation, and analysis. One field duplicate for 10 environmental samples of the same matrix will be collected and analyzed.

3.5.1.2 Matrix Spike and Matrix Spike Duplicate Samples

Matrix spike (MS) and matrix spike duplicate samples (MSD) are designed to evaluate whether there are site-specific matrix (soil or water) interferences with the analytes (e.g., BTEX and naphthalene) and to determine the overall accuracy of analytical results. One MS/MSD sample will be collected for each matrix at a rate of 1 MS/MSD pair for every 20 project samples. Matrix spike samples produced in the laboratory for percent recovery studies will be conducted in accordance with the project laboratory's standard QC protocols.

3.5.1.3 Trip Blanks

Prior to any sampling, trip blanks will be prepared and provided by the laboratory. The trip blanks will consist of two 40-milliliter amber glass bottles filled with preserved reagent-grade water, so that there is no headspace, and sealed with a custody seal. One trip blank (two vials) will be placed in each cooler containing environmental samples for volatile organics analyses. The trip blanks will accompany the sample containers to and from the field; they will be stored with and transported to the laboratory in the same cooler as the samples collected for volatile organics analysis (TPH-gas, BTEX, VOCs, and—if it is determined a COC in groundwater—EDB).

3.5.1.4 Temperature Blanks

Temperature blanks are used to help determine if the samples are maintained at 4 ± 2 degrees Celsius (°C) during shipping. Temperature blanks accompany samples scheduled for analysis that require cooling as a preservation method and typically consist of a sample container filled with distilled water.

3.5.2 Analytical Data

The analytical data will be evaluated qualitatively in terms of precision, accuracy, representativeness, completeness, compatibility, and sensitivity (PARCCS) parameters. The following sections discuss how the PARCCS parameters will be used to evaluate the laboratory data and field sampling procedures. The laboratory control limits in Appendix A provide a summary of the data quality requirements for analytical data generated for the samples to be collected under this QAPP.

3.5.2.1 Precision

Precision is a measure of the size of the closeness of agreement among replicate measurements of the same property under prescribed similar conditions. The following formula will be used to calculate precision:

$$RPD = \frac{x_1 - x_2}{(x_1 + x_2)/2} x100$$

To reduce variability (lack of precision), crew will handle and ship samples according to the Sample Management SOP (Appendix D, SOP BERS-03). Precision data will be ensured in the field by daily instrument calibration. Precision will be evaluated for the analytical laboratory results by calculating the relative percent difference (RPD) for field duplicate samples. The target RPD for the analytical results from soil samples is 50%, and for water samples is 30%.

3.5.2.2 Accuracy

Accuracy is a measure of closeness to measurements to the true value. Bristol field staff will optimize the accuracy of field measurements by ensuring that field instruments/equipment will be calibrated and maintained according to the manufacturer's instructions and by following the manufacturer's instructions for operation of the equipment. Analytical data accuracy will be evaluated through comparison of the LCS and LCSD recoveries, surrogate recoveries, and MS and MSD recoveries to laboratory control limits.

3.5.2.3 Representativeness

Representativeness is a qualitative term that expresses the degree to which data accurately and precisely represents a characteristic of a population, the parameter variations of a sampling point, and a process or environmental condition. The representativeness of the

field data is determined by the design of the data collection procedures. The sampling and field measurement procedures are based on existing analytical data, hydrogeology, physical setting of the area, and land use history. Representativeness of the field sampling procedures and field measurements will be controlled by following the sampling and measurement procedures outlined in this QAPP and in Bristol SOPs. The representativeness of the analytical results for a sample is controlled by the analytical method employed, MS/MSD recoveries, and calculated RPDs. Blank sample results can be used to evaluate the representativeness of field sampling procedures. Contaminants detected in blanks indicate that procedures are not completely effective; in such cases contaminants may be attributed to cross-contamination rather than the environment.

3.5.2.4 Comparability

To ensure comparability of data, the Bristol field staff will conduct sampling, field measurements, assessments, and data management in accordance with this QAPP. Analytical and field measurement data will be evaluated by comparing the results to previous sampling records.

3.5.2.5 Completeness

Completeness of the field program will be evaluated to ensure that the appropriate numbers of samples were collected for analysis and that field data of the type and quantity outlined in this QAPP were collected appropriately. Completeness will be evaluated by comparing the actual number of samples and the actual quantity of data that met the requirements outlined in the QAPP. The completeness goal for this project is 90%. The following formula will be used to calculate completeness:

$$\% \text{ Completeness} = \frac{\text{Number of Samples Collected}}{\text{Number of Samples Planned}} \times 100$$

3.5.2.6 Bias

Bias is a measure of the closeness of agreement between an observed value and an accepted reference value, the true value. Systematic errors, termed bias, can accumulate during a measurement process. Bias may result from faults in sampling design, field screening and sampling procedures, contamination loss, interaction with containers and inaccurate instrument calibrations. Bias causes the mean value of the sample data to vary higher or lower from the true mean value. Analytical bias will be evaluated using laboratory established percent recovery criteria for the LCS, LCSD, surrogates, and the MS and MSD.

3.5.2.7 Sensitivity

Sensitivity is the capability of a method or instrument to discriminate between measurement responses representing different levels of the variable of interest. Method detection limits are typically confirmed yearly at the analytical laboratory for each extraction/analysis method and by environmental matrix, and represent the minimum concentration or attribute that can be detected by a method, or by an instrument (instrument detection limit).

3.5.3 Corrective Action

If field, laboratory, or other data indicates that problems are occurring or have occurred with sampling or field measurements, the Bristol project manager, with support from the QA Manager, will investigate if a problem is identified, its cause and corrective actions will be explored. The project manager will ensure that corrective actions are successfully implemented. Laboratory data problems and corrective actions will be documented and resolved as described in the laboratory QAM located in Appendix A. Field data problems

and corrective actions will be documented by the Bristol project manager or QA manager in a memo and the memo placed in the project file.

3.5.4 Storage and Disposal of Investigation-Derived Wastes

Investigation-derived wastes (IDW) are expected at both sites during field activities. IDW soil and groundwater will be containerized for pickup and disposal by a waste disposal company, pending analytical results.

The PCS removed from the A. E. Erickson site is likely non-regulated TPH-contaminated dirt, and it will be transported and disposed of by Emerald Services at the Rabanco permitted facility in Seattle, Washington. If analytical results from the soil stockpile sample show RCRA-regulated compounds, the contaminated soil will be transported and disposed of at Waste Management, Inc.'s Subtitle C landfill (Chemical Waste Management of the Northwest), located in Arlington, Oregon.

The pump tubing, gloves, and miscellaneous sampling and decontamination items will be disposed of locally at a municipal solid waste facility.

3.6 B6 – Field Instrument/Equipment Testing, Inspections and Maintenance

Field equipment will be operated, tested, inspected, maintained and calibrated according to the manufactures' instructions and requirements specified in the SOPs (Appendix D). The field task manager is responsible for checking field equipment prior to being mobilized for field work, for ensuring the equipment functions as designed prior to and during field activities. Field equipment inspections are also made on-site before and after each sampling event by the field task manager. If the equipment does not meet performance criteria, steps described in the instrument manual or Bristol's SOPs will be taken to identify and resolve the problem. If the problem cannot be corrected and

instrument performance criteria met, the instrument will be replaced or sent to the manufacturer for servicing and repair.

Groundwater levels, conductivity, pH, temperature, dissolved oxygen, and oxidation reduction potential (ORP) from the replacement groundwater monitoring well will be measured at the Western Gas Site. Volatile organic vapors and TPH will be measured during PCS excavation at the A. E. Erickson site; if the EPA decides that groundwater monitoring wells must be installed and sampled, personnel will measure the same field parameters listed above for the Western Gas site. Personnel will document soil lithology while installing soil borings for the wells, as described in SOP BERS-06 (Appendix D), and Bristol will obtain professional level survey data for each of the newly installed wells.

Any malfunctioning equipment will be recorded in the field log. Each piece of equipment will have its own maintenance log which is kept with the piece of equipment. The log will document any maintenance and service of the equipment. A log entry will include the following information:

- Name of person maintaining the instrument/equipment
- Date and description of the maintenance procedure
- Date and description of any instrument/equipment problem(s)
- Date and description of action to correct problem(s)
- List of follow-up activities after maintenance (i.e., system checks)
- Date the next maintenance will be needed

No spare parts for the field equipment are kept in-house. All repairs are made by the manufacturer. If the equipment problem identified in the field persists, Bristol will send the faulty piece of equipment back to the manufacturer for repair and expedite a functioning piece of equipment to the site.

3.7 B7 – Field Instrument/Equipment Calibration and Frequency

Field instruments/equipment will be calibrated according to the manufacturer's instruction and requirements. Calibration records will be included in the project field logbook and in a calibration logbook accompanying each instrument. Bristol field personnel will calibrate and test the field equipment on-site prior to each sampling event. In the event the instrument cannot be calibrated, or will not hold calibration, the instrument will be returned to the manufacturer for servicing and repair and an alternate piece of equipment will be expedited to the site.

3.8 B8 – Inspection/Acceptance Requirements for Supplies and Consumables

The Bristol field task manager is responsible for checking that all field supplies and consumables have been inspected before their use in the field. In addition to checking that all supplies and consumables have been packaged and stored appropriately, special attention will be directed toward ensuring that materials subject to expiration dates or limited shelf life will be used within their appropriate time frames. Commercial materials with a limited shelf life, or expiration date, will not be accepted for use if the shelf life or expiration date is within 30 days before intended use.

All sample containers will be provided by the TestAmerica Tacoma laboratory in certified clean condition, and they will be stored in a clean environment with a moderate temperature prior to sample collection. The laboratory will provide the trip blanks and temperature blanks. The types of sample containers and preservation methods correspond to the analytical method chosen for each type of sample; these are outlined for the Western Gas and A. E. Erickson sites in Appendix E.1 Table E.1-2, Appendix E.2, Table E.1-2, respectively. The Bristol field task manager is responsible for providing all consumables needed to conduct field activities.

3.9 B9 – Data Acquisition Requirements (Non-Direct Measurements)

Historical data furnished by the EPA (analytical data, reports, records, databases, maps, etc.) will be accepted as valid for direct use and application in the execution of the TO. Other data provided by other government entities or private authorities will not be accepted for use by Bristol without review and approval by the EPA SME or designee. Database applications that exist in the public domain or that are commercially available from private vendors will only be used within the limitations and intended use identified by the source's' instructions.

Bristol will use the historic data from each site provided by EPA at face value. The results obtained from environmental samples at each site will be compared to past data. The acceptance criteria will be based on EPA's receipt of the disposal documentation and approval of the reports by the EPA

3.10 B10 – Data Management

Field data sheets and sampling forms are reviewed and checked by the field task manager before transfer to the Bristol Project Manager. Field samples are checked by the field task manager for label completion before leaving the site and being transferred to the laboratory. The field task manager is identified on the Chain of Custody Form and is responsible for the samples until the laboratory receives them. The TestAmerica Tacoma laboratory is then responsible for testing and disposing of sample materials and recording and transferring the sample data to Bristol project manager. Upon data acceptance by the Bristol QA Manager, the project manager is responsible for ensuring the data is verified and included in the project report with appropriate data qualifiers.

Logbooks, analytical data, and other project-specific documents (such as EPA-provided document and records for the file reviews) will be maintained by the Project Manager in

project files until the project is closed. Hardcopies of project documents are scanned by the Bristol project assistants and the scans reviewed for completeness by either the Bristol project manager or the field task manager prior to being placed in the project folder. No special computer hardware or software is required for the projects covered under this QAPP.

Bristol's process for transmitting, distributing, retaining, accessing, preserving, tracking, and retrieving data, documents, and other project records is described in Section 2.7.

(Intentionally blank)

4.0 ASSESSMENT AND OVERSIGHT

4.1 C1 – ASSESSMENTS / OVERSIGHT AND RESPONSE ACTIONS

4.1.1 Performance System Audits

Oversight of field procedures will be the direct responsibility of the Bristol Project Manager, who will review all elements of the QAPP to ensure that the objectives of the TO are met. Any problems discovered relating to the sampling procedures will be communicated by the Bristol field task manager to the Bristol project manager who will consult with the Bristol QA manager for resolution.

Internal audits of field activities (sampling and measurements) will be conducted by the Bristol QA manager or designee, following each field event to verify that all of the procedures outlined in this QAPP are being followed. The audits will include examining field measurement records, field equipment calibration records, field sampling records, field instrument operation records, sample collection procedures, sample handling and shipping procedures, and chain-of-custody procedures. Follow-up audits will be conducted to correct any deficiencies identified, and to verify that QA procedures are maintained throughout the project.

4.1.2 Process Ensuring Verification of Subcontractor's Conformance to Customer Requirements

Bristol will use subcontractors to decommission, install, and develop groundwater monitoring wells; inject chemical oxidant into the petroleum-impacted zone; excavate PCS and backfill the excavation; transport and dispose contaminated soil or water; and provide analytical laboratory services.

Prior to selecting a subcontractor, Bristol contacts the Tribal Employment Rights Office (TERO) and works with the TERO to identify native-owned businesses on their list that

have the equipment, staffing, certifications, and experience to perform specific project tasks. Bristol will attempt to obtain three competitive bids from the TERO list of qualified subcontractors. If TERO cannot provide subcontractors who meet the job specific requirements, Bristol will approach at least three local subcontractors who have the skill, experience, and credentials and request their bids. A detailed scope of work will be issued to potential subcontractors to draw in competitive bids. The scope of work will include project specifics such as the required type of drilling rig, the number and depth of borings, well-construction instructions, etc. Once selected, the subcontractor will enter into a subcontract agreement with Bristol that details the scope of work, schedule, and budget for the project. The EPA contract flow-downs are also included as part of the subcontract. Since Pacific Soil and Water has successfully performed all injections to date at the Western Gas site in accordance with the CAP, Bristol selected Pacific Soil and Water to perform the injections again decommission the two existing monitoring wells on site, and install and develop one monitoring well that will be used as the (replacement) compliance well.

All subcontractors are provided the opportunity to review the project QAPP and the *Site-Specific Safety and Health Plan*. Once on site, all workers are required to sign the sheet stating they read the *Site-Specific Safety and Health Plan*, are aware of site hazards, and know the route to the nearest clinic and/or hospital.

Bristol provides subcontractor oversight, ensuring that the subcontractor perform the agreed upon scope of work from using the correct type of drilling equipment to installing monitoring wells correctly at the right location and depth. However, Bristol will not be on site when the stockpiled PCS from the A.E. Erickson site and the IDW from the

Western Gas site is picked up by the waste disposal company for transport to the disposal facility.

The Bristol field task manager will be responsible for documenting and reporting all suspected technical issues and suspected deficiencies during any field activity. Deficiencies will be documented in the field logbook and reported to the Bristol Project Manager.

4.1.3 Process Ensuring Verification of the Suppliers Conformance to Customer Requirements

All materials and deliverables are checked against project specifications and requirements specified in the EPA Performance Work Statement for each site. The Project Manager will not accept materials or deliverables that do not meet project specifications. Materials that do not meet project specifications will be returned to the supplier and new materials will be obtained and inspected prior to use, to ensure the correct materials are used on the site.

4.1.4 Corrective Actions

The field staff will be responsible for documenting and reporting all suspected technical nonconformances and suspected deficiencies during any field activity. The nonconformances and/or deficiencies will be documented in the field logbook and reported to the Bristol Project Manager. If the problem is associated with field measurements or sampling equipment, the field staff will take the appropriate steps to correct the problem. Typical field procedures to correct equipment problems include the following:

- Repeating the measurement to check for error
- Ensuring that the meters or instruments are adjusted properly for the ambient conditions, such as temperature
- Checking or replacing batteries
- Recharging batteries

- Recalibrating the instruments
- Replacing the meters used to measure field parameters
- Stopping work until the problem is corrected (if necessary)

If a nonconformance or problem requires a major adjustment to the field procedures outlined in the site-specific QAPPs located in Appendix E (e.g., changing sampling methodology), the Project Manager, in conjunction with the QA Manager, will be responsible for initiating corrective actions. The Project Manager will be responsible for the following:

- Evaluating the reported nonconformance
- Controlling additional work on nonconforming items
- Determining appropriate corrective actions
- Maintaining a log of all nonconformances and corrective actions
- Ensuring that explanations of nonconformances and corrective actions are included in an appendix of the Summary Report

The Project Manager will ensure that no additional work that is dependent on the nonconforming activity is performed until the appropriate corrective actions have been completed.

The field task manager has the authority to stop work at the site for any reason, including lack of adherence to the QAPP. Anyone on the job site has the authority to stop work for safety concerns. Issues in the field are immediately brought to the attention of the Bristol project manager, and if warranted, to the Bristol program manager for guidance and resolution. Depending on the safety issue, Bristol's corporate safety officer may be notified to provide guidance and resolution to the safety issue. Specific issues, such as lack of site access, will be brought to the attention of the EPA SME for resolution.

4.2 C2 – REPORTS TO MANAGEMENT

Data collected during field work at the Western Gas and A. E. Erickson sites will be compiled in separate reports prepared by Bristol. Upon completion of field work, Bristol will draft a report of site activities and provide recommendations for any follow-up work that may be necessary to support the eventual closure of the two LUST sites.

The reports will describe whether or not sampling protocols were followed; if proper paperwork was completed and filed; whether decontamination procedures were followed; and if samples were preserved, packed, and shipped properly. The report will include an introduction, site background, site activities, findings, conclusions and recommendations, references, definition of acronyms used, and Appendices containing figures, tabulated and verified analytical and field data (such as water quality parameter data, PID and Hanby results), site photographs, populated field forms (well diagrams, groundwater sampling forms) and field notebooks, waste disposal records, and the laboratory data package. A brief summary of the results of the data quality assessment/verification will be included in the text of the report.

The Bristol QA manager will perform a technical review of the report and confirm that the EPA specified activities were performed in accordance with this QAPP. Bristol will accomplish this by reviewing the report text, figures, and supporting data (such as analytical data, field forms and notes) and ensuring that the recommendations provided in the report are supported by the text and site data. If discrepancies are noted the QA reviewer will mark the hardcopy of the document in red pen and the report will be sent back to the author for discussion and revision, if required. Upon resolution of any issues identified by the QA reviewer, the report will be edited and formatted, and reviewed again by the author or project manager for technical accuracy, then reproduced. A final

review will be conducted by the project manager prior to submittal to ensure that the report was reproduced as specified.

The EPA will review the report and may provide comments.

Comments will be addressed by revising the report and submitting to the EPA in tracked changes for approval or additional revision. Once the EPA approves all changes, Bristol will reproduce the report for final submittal.

5.0 DATA VALIDATION AND USABILITY

5.1 D1 – DATA REVIEW, VALIDATION, AND VERIFICATION REQUIREMENTS

Field documents, measurements, and records from each site will be reviewed by the Bristol field task manager before they are transferred to the Bristol project manager. The Bristol project manager, at the conclusion of field activities, reviews the project documentation for completeness and accuracy to verify that the sampling process and design described in this QAPP were followed and met. Data will be evaluated with respect to the measurement and acceptance criteria described in Appendices E.1 and E.2 for the sampling design and process for the Western Gas and A. E. Erickson sites, respectively. Data not supported by appropriate QC data or which do not meet the defined DQOs will be reported to the EPA SME with the appropriate qualifiers.

TestAmerica Tacoma will review analytical data in accordance with the laboratory QAM and provide a case narrative describing whether or not the samples were received in good condition, properly preserved, and within the temperature criteria; TestAmerica will also identify analytical parameters that were outside of acceptance criteria such as poor surrogate, LCS, LCSD, MS, and MSD recoveries or RPDs, and they will evaluate missed hold times and review issues encountered during extraction or analysis. They will also identifying whether the GRO, DRO, and RRO results are from a mixture of the two fuel classes or if the results are actually representative of weathered gas, if applicable.

Bristol's will review field documentation, Chain of Custody receipts, and laboratory analyses:

- Field Documentation - Verify that personnel used sampling design, adhered to sample collection and handling procedures, and completed field documentation.
- Chain of Custody – Check sample records.

- Laboratory Report – Upon receipt of the laboratory report, ensure all samples were analyzed with the correct analytical procedures and that the analytical laboratory performed the method required QC.

Prior to submitting the report, the applicable portions of Bristol's Quality Assurance Review Checklist, located in Appendix D, will be completed and the checklist stored with the project records.

5.2 D2 – Data Validation and Verification Process

This section summarizes the methods to be used during data verification and reporting. Data verification procedures are performed to ensure that the sampling and analysis protocols specified in this QAPP were followed, and that the measurement systems were performed in accordance with the specified criteria. Full data validation will not be conducted on the data from either site.

5.2.1 Data Validation and Verification Process

Field data will be verified by the Bristol project manager as described in Section 5.1.

Analytical data will be verified by the Bristol chemist using the acceptance criteria specified in this QAPP. Data quality flags will be included on the data tables that will be provided in the report to the EPA of site activities.

Findings and corrective actions at the laboratory, if any, will be documented in the case narrative section of the laboratory reports.

5.2.2 Data Reporting

A Bristol chemist will verify all of the analytical data and present it in the site-activities report. Deviations from approved procedures, assumptions, data uncertainties, and QC results, as well as external performance data, will be documented in the narrative section of the laboratory data report and summarized in the site-activities report, which will be

provided to the EPA. Assumptions will be clearly explained as to validity and limitations. The laboratory will also document any known or suspected sample matrix interferences and any corrective actions taken in response to such interferences; this information will also be included in the case narrative of the analytical data report.

Analytical data that meet the criteria specified in this QAPP will be reported to the method reporting limit (MRL) without qualification. Data flags might be applied to the sample results if the QC data do not meet the QAPP criteria. The chart below defines Bristol's data and quality flags.

Flag	Definition of Data Quality Flag	Data Quality Flag Applied
J	Estimated result with no bias	One or more data quality indicators is out of control and the bias cannot be determined.
J+	Estimated result with a high bias	One or more data quality indicators were below criteria.
J-	Estimated result with a low bias	One or more data quality indicators were above criteria.
R	The result is rejected and cannot be used for any purpose	Multiple data quality indicators are out of control such that the analytical result for the sample is invalid, has too much uncertainty to report, or the percent recovery of the MS and MSD indicate the method does not work for the sample matrix.

5.2.3 Data Usability

The data generated in the field and in the laboratory must be of the quantity and quality required to make project decisions. Verified analytical data and data collected in the field will be used to evaluate the current concentrations of petroleum-contamination at each site. Verified analytical results and field attenuation data from groundwater sampling at the Western Gas site will provide the current concentrations of weathered gasoline at the point of compliance and may be used to evaluate the effectiveness of the remedial treatments. Verified analytical results from soil confirmation sampling at the A. E.

Erickson site excavation (post PCS removal) will confirm that all of the PCS has been removed or, if the volume of PCS exceeds 30 cubic yards was not removed, will provide the current concentrations of petroleum-hydrocarbons that remain in the soil. If wells are installed at the A. E. Erickson site at a later date, the groundwater data will provide information on the nature and extent of COCs in groundwater at the site post-application of the chemical oxidant to the soil-groundwater interface.

5.3 D3 – Reconciliation with User Requirements

The results obtained from the project tasks will be evaluated against the criteria presented throughout this QAPP to determine if the performance criteria specified in the EPA performance work statements and incorporated into this QAPP have been satisfied. The report for each project site will include a discussion on issues that arose during project execution, how the issue was resolved, and any limitations on the use of the data as a result of the issue.

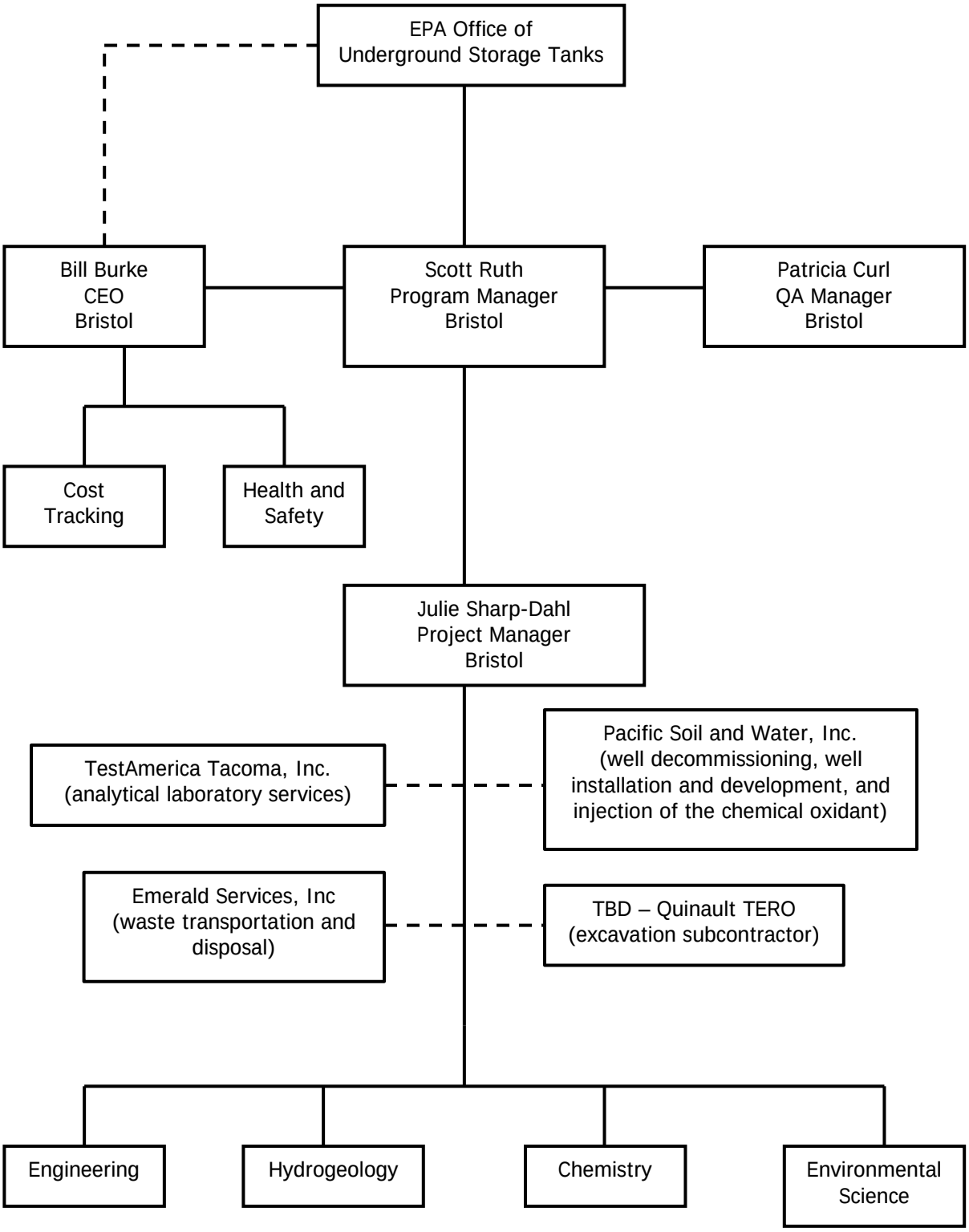
The Bristol project manager will evaluate the work conducted at both sites by reviewing field notes, field forms, boring logs and well construction diagrams, site drawings, laboratory documentation, and the results of any audits, if conducted, and compare them to the performance criteria specified in this QAPP. Data obtained for each site will be compared to available historic site data to confirm COCs, soil and groundwater concentrations, and to evaluate if the chemical oxidant treatments are accelerating the breakdown of petroleum-hydrocarbons in groundwater.

Project reports prepared by Bristol staff will be reviewed for technical content by Bristol's QA Manager, and formatted and edited by the document production team prior to submittal to the EPA.

Any limitations on the use of the data will be clearly documented in the project reports.

FIGURE

Figure 1 Organization Chart



APPENDIX A

- Laboratory Quality Assurance Manual
- Standard Operating Procedures
- Control Limits
- TestAmerica Chain of Custody

Quality Assurance Manual

TestAmerica Seattle
5755 8th Avenue East
Tacoma, WA 98424
Phone No. (253) 922-2310
Fax No. (253) 922-5047

www.testamericainc.com

Copyright Information:
This documentation has been prepared by TestAmerica Laboratories, Inc. and its affiliates ("TestAmerica"), solely for their own use and the use of their customers in evaluating their qualifications and capabilities in connection with a particular project. The user of this document agrees by its acceptance to return it to TestAmerica upon request and not to reproduce, copy, lend, or otherwise disclose its contents, directly or indirectly, and not to use it for any other purpose other than that for which it was specifically provided. The user also agrees that where consultants or other outside parties are involved in the evaluation process, access to these documents shall not be given to said parties unless those parties also specifically agree to these conditions.

THIS DOCUMENT CONTAINS VALUABLE CONFIDENTIAL AND PROPRIETARY INFORMATION. DISCLOSURE, USE OR REPRODUCTION OF THESE MATERIALS WITHOUT THE WRITTEN AUTHORIZATION OF TESTAMERICA IS STRICTLY PROHIBITED. THIS UNPUBLISHED WORK BY TESTAMERICA IS PROTECTED BY STATE AND FEDERAL LAW OF THE UNITED STATES. IF PUBLICATION OF THIS WORK SHOULD OCCUR THE FOLLOWING NOTICE SHALL APPLY:

©COPYRIGHT 2012 TESTAMERICA LABORATORIES, INC. ALL RIGHTS RESERVED.

Facility Distribution No. Uncontrolled **Distributed To:**_____

Title Page:

Quality Assurance Manual
Approval Signatures

Signatures on File

Laboratory Director – Heather Prater	Date
Quality Assurance Manager – Terri Torres	Date
Environmental Health & Safety – Sandra Langway	Date
Project Manager Supervisor – Kris Allen	Date
Volatile Organic Technical Manager – Bisrat Tadesse	Date
Inorganic Technical Manager – Stan Palmquist	Date
Semivolatile Technical Manager – Colin McKean	Date

SECTION 2. TABLE OF CONTENTS

Sec. No.	Title	2009 TNI Standard Reference	ISO/IEC 17025:2005 (E) Reference	Page No.
-	QUALITY ASSURANCE MANUAL	V1M2 Sec. 4.2.8.3		1
1.0	TITLE PAGE			2
2.0	TABLE OF CONTENTS	V1M2 Secs. 4.2.8.3-4.2.8.4		3
3.0	INTRODUCTION, SCOPE AND APPLICABILITY	V1M2 Sec. 4.2.8.4		13
3.1	Introduction And Compliance References	V1M2 Secs. 1.1; 1.2; 2.0; 3.2; 4.1.2; 4.2.4	4.1.2; 4.2.4	13
3.2	Terms And Definitions	V1M2 Secs. 3.0; 4.2.4	4.2.4	13
3.3	Scope / Fields Of Testing	V1M2 Secs. 1.2; 4.2.4	4.1.2; 4.2.4	14
3.4	Management Of The Manual	V1M2 Secs. 4.2.1; 4.2.7; 4.3.3.2; 4.3.3.3	4.2.1; 4.2.7; 4.3.3.2; 4.3.3.3	14
4.0	MANAGEMENT REQUIREMENTS	V1M2 Sec. 4		15
4.1	Overview	V1M2 Secs. 4.1.1, 4.1.3; 4.1.5	4.1.1; 4.1.3; 4.1.5; 4.2.6	15
4.2	Roles And Responsibilities	V1M2 Secs. 4.1.4; 4.1.5; 4.1.6; 4.2.1; 4.2.6; 5.2.4	4.1.3; 4.1.5; 4.1.6; 4.2.1; 4.2.6; 5.2.4	15
4.3	Deputies	V1M2 Secs. 4.1.5; 4.1.7.2; 4.2.7	4.1.5; 4.2.7	23
5.0	QUALITY SYSTEM			27
5.1	Quality Policy Statement	V1M2 Secs. 4.1.5; 4.2.2; 4.2.3; 4.2.8.3	4.1.5; 4.2.2; 4.2.3	27
5.2	Ethics And Data Integrity	V1M2 Secs. 4.1.5; 4.16; 4.2.2; 4.2.8.1; 5.2.7	4.1.5; 4.2.2	27
5.3	Quality System Documentation	V1M2 Secs. 4.1.5; 4.2.2; 4.2.5	4.2.2; 4.2.5	28
5.4	QA/QC Objectives For The Measurement Of Data	V1M2 Sec. 4.2.2	4.1.5; 4.2.2	29
5.5	Criteria For Quality Indicators			31
5.6	Statistical Quality Control			31
5.7	Quality System Metrics			31
6.0	DOCUMENT CONTROL	V1M2 Secs. 4.2.7; 4.3.1; 4.3.2.2 ; 4.3.3.3; 4.3.3.4	4.2.7; 4.3.1; 4.3.2.2; 4.3.3.3; 4.3.3.4	32
6.1	Overview			32
6.2	Document Approval And Issue	V1M2 Secs. 4.3.2; 4.3.2.1-4.3.2.3; 4.3.3.1	4.3.2.1; 4.3.2.2; 4.3.2.3; 4.3.3.1	32

Sec. No.	Title	2009 TNI Standard Reference	ISO/IEC 17025:2005 (E) Reference	Page No.
6.3	Procedures For Document Control Policy	V1M2 Secs. 4.3.2.1–4.3.2.2; 4.3.3.1	4.3.2.1; 4.3.2.2; 4.3.3.1	33
6.4	Obsolete Documents	V1M2 Secs. 4.3.2.1–4.3.2.2	4.3.2.1; 4.3.2.2	33
7.0	SERVICE TO THE CLIENT	V1M2 Secs. 4.4.1 - 4.4.4	4.4.1; 4.4.2; 4.4.3; 4.4.4	33
7.1	Overview	V1M2 Secs. 4.4.5; 4.5.5; 5.7.1	4.4.5; 5.7.1	33
7.2	Review Sequence And Key Personnel	V1M2 Sec. 4.4.5	4.4.5	34
7.3	Documentation	V1M2 Sec. 5.7.1	5.7.1	35
7.4	Special Services	V1M2 Secs. 4.7.1-4.7.2	4.7.1; 4.7.2	36
7.5	Client Communication	V1M2 Secs. 4.7.1-4.7.2	4.7.1; 4.7.2	37
7.6	Reporting	V1M2 Secs. 4.7.1-4.7.2	4.7.1; 4.7.2	37
7.7	Client Surveys	V1M2 Secs. 4.7.1-4.7.2	4.7.1; 4.7.2	37
8.0	SUBCONTRACTING OF TESTS	V1M2 Secs. 4.4.3; 4.5.4	4.4.3; 4.5.4	37
8.1	Overview	V1M2 Secs. 4.5.1 - 4.5.3; 4.5.5; 5.3.1	4.5.1; 4.5.2; 4.5.3; 5.3.1	37
8.2	Qualifying And Monitoring Subcontractors	V1M2 Secs. 4.5.1; 4.5.2; 4.5.3; 4.5.5	4.5.1; 4.5.2; 4.5.3	38
8.3	Oversight And Reporting	V1M2 Sec. 4.5.5		39
8.4	Contingency Planning			40
9.0	PURCHASING SERVICES AND SUPPLIES	V1M2 Sec. 4.6.1	4.6.1	42
9.1	Overview	V1M2 Secs. 4.6.2; 4.6.3; 4.6.4	4.6.2; 4.6.3; 4.6.4	42
9.2	Glassware	V1M2 Sec. 5.5.13.1		42
9.3	Reagents, Standards & Supplies	V1M2 Secs. 4.6.2; 4.6.3; 4.6.4	4.6.2; 4.6.3; 4.6.4	42
9.4	Purchase Of Equipment / Instruments / Software			44
9.5	Services			45
9.6	Suppliers			45
10.0	COMPLAINTS	V1M2 Sec. 4.8	4.8	46
10.1	Overview			46
10.2	External Complaints			46
10.3	Internal Complaints			47
10.4	Management Review			47
11.0	CONTROL OF NON-CONFORMING WORK	V1M2 Secs. 4.9.1; 5.10.5	4.9.1; 5.10.5	47
11.1	Overview	V1M2 Secs. 4.9.1; 4.11.3; 4.11.5	4.9.1; 4.11.3; 4.11.5	47

Sec. No.	Title	2009 TNI Standard Reference	ISO/IEC 17025:2005 (E) Reference	Page No.
11.2	Responsibilities And Authorities	V1M2 Secs. 4.9.1; 4.11.3; 4.11.5; 5.2.7	4.9.1; 4.11.3; 4.11.5	48
11.3	Evaluation Of Significance And Actions Taken	V1M2 Secs. 4.9.1; 4.11.3; 4.11.5	4.9.1; 4.11.3; 4.11.5	48
11.4	Prevention Of Nonconforming Work	V1M2 Secs. 4.9.4; 4.11.2	4.9.2; 4.11.2	49
11.5	Method Suspension / Restriction (Stop Work Procedures)	V1M2 Secs. 4.9.1; 4.9.2; 4.11.5	4.9.1; 4.9.2; 4.11.5	49
12.0	CORRECTIVE ACTION	V1M2 Sec. 4.11		50
12.1	Overview	V1M2 Secs. 4.9.2; 4.11.1; 4.11.2	4.9.2; 4.11.1; 4.11.2	50
12.2	General	V1M2 Sec. 4.11.2; 4.11.3	4.11.2; 4.11.3	50
12.3	Closed Loop Corrective Action Process	V1M2 Sec. 4.11.2; 4.11.3; 4.11.4; 4.11.6; 4.11.7; 4.12.2	4.11.2; 4.11.3; 4.11.4; 4.12.2	51
12.4	Technical Corrective Actions	V1M2 Sec. 4.11.6		53
12.5	Basic Corrections	V1M2 Secs. 4.11.1; 4.13.2.3	4.11.1; 4.13.2.3	53
13.0	PREVENTIVE ACTION / IMPROVEMENT	V1M2 Secs. 4.10; 4.12.1; 4.12.2	4.10; 4.12.1; 4.12.2	58
13.1	Overview	V1M2 Secs. 4.15.1; 4.15.2	4.15.1; 4.15.2	58
13.2	Management Of Change			59
14.0	CONTROL OF RECORDS	V1M2 Secs. 4.2.7; 4.13.1.1; 4.13.3	4.2.7; 4.13.1.1	59
14.1	Overview	V1M2 Secs. 4.13.1.1; 4.13.1.2; 4.13.1.3; 4.13.1.4; 4.13.2.1; 4.13.2.2; 4.13.2.3; 4.13.3	4.13.1.1; 4.13.1.2; 4.13.1.3; 4.13.1.4; 4.13.2.1; 4.13.2.2; 4.13.2.3	59
14.2	Technical And Analytical Records	V1M2 Sec. 4.13.2.2 - 4.13.2.3	4.13.2.2; 4.13.2.3	62
14.3	Laboratory Support Activities			63
14.4	Administrative Records			64
14.5	Records Management, Storage And Disposal	V1M2 Sec. 4.13.3		64
15.0	AUDITS			65
15.1	Internal Audits	V1M2 Sec. 4.2.8.1; 4.14; 4.14.1; 4.14.2 ; 4.14.3; 4.14.5; 5.9.1; 5.9.2	4.14.1; 4.14.2; 4.14.3; 5.9.1; 5.9.2	65
15.2	External Audits	V1M2 Secs.4.14.2; 4.14.3	4.14.2; 4.14.3; 4.14.4	67

Sec. No.	Title	2009 TNI Standard Reference	ISO/IEC 17025:2005 (E) Reference	Page No.
15.3	Audit Findings	V1M2 Secs. 4.14.2; 4.14.3; 4.14.5		68
16.0	MANAGEMENT REVIEWS	V1M2 Sec. 4.1.6; 4.15; 4.15.1; 4.15.2	4.1.6; 4.15.1; 4.15.2	68
16.1	Quality Assurance Report			68
16.2	Annual Management Review	V1M2 Sec. 4.2.2; 4.15.3	4.2.2	69
16.3	Potential Integrity Related Managerial Reviews			70
17.0	PERSONNEL	V1M2 Secs. 5.2; 5.2.1	5.2.1	70
17.1	Overview	V1M2 Secs. 5.2.2; 5.2.3; 5.2.5	5.2.2; 5.2.3; 5.2.5	70
17.2	Education And Experience Requirements For Technical Personnel	V1M2 Secs. 5.2.1; 5.2.3; 5.2.4	5.2.1; 5.2.3; 5.2.4	71
17.3	Training	V1M2 Sec. 5.2.5	5.2.5	72
17.4	Data Integrity And Ethics Training Program	V1M2 Sec. 4.2.8.1; 5.2.7		73
18.0	ACCOMMODATIONS AND ENVIRONMENTAL CONDITIONS	V1M2 Sec. 5.3		74
18.1	Overview	V1M2 Secs. 5.3.1; 5.3.3; 5.3.4; 5.3.5	5.3.1; 5.3.3; 5.3.4; 5.3.5	74
18.2	Environment	V1M2 Secs. 5.3.1; 5.3.2; 5.3.3; 5.3.4; 5.3.5	5.3.1; 5.3.2; 5.3.3; 5.3.4; 5.3.5	75
18.3	Work Areas	V1M2 Secs. 5.3.3; 5.3.4; 5.3.5	5.3.3; 5.3.4; 5.3.5	75
18.4	Floor Plan			76
18.5	Building Security	V1M2 Sec. 5.3.4	5.3.4	76
19.0	TEST METHODS AND METHOD VALIDATION	V1M2 Sec. 5.4.1	5.4.1	65
19.1	Overview	V1M2 Sec. 5.4.1	5.4.1; 5.4.5.1	76
19.2	Standard Operating Procedures (Sops)	V1M2 Secs. 4.2.8.5; 4.3.3.1; 5.4.2	4.3.3.1; 5.4.2	76
19.3	Laboratory Methods Manual	V1M2 Sec. 4.2.8.5		77

Sec. No.	Title	2009 TNI Standard Reference	ISO/IEC 17025:2005 (E) Reference	Page No.
19.4	Selection Of Methods	V1M2 Secs. 4.13.3; 5.4.1; 5.4.2; 5.4.3. V1M4 Secs. 1.4; 1.5.1; 1.6.1; 1.6.2; 1.6.2.1; 1.6.2.2	5.4.1; 5.4.2; 5.4.3; 5.4.4; 5.4.5.1; 5.4.5.2; 5.4.5.3	77
19.5	Laboratory Developed Methods And Non-Standard Methods	V1M2 Sec. 5.4.2. V1M4 Sec. 1.5.1	5.4.2; 5.4.4; 5.4.5.2; 5.4.5.3	81
19.6	Validation Of Methods	V1M2 Sec. 5.4.2. V1M4 Secs. 1.5.1; 1.5.2; 1.5.2.1; 1.5.2.2; 1.5.3	5.4.2; 5.4.4; 5.4.5.2; 5.4.5.3	81
19.7	Method Detection Limits (mdl) / Limits Of Detection (LOD)	V1M2 Sec. 5.9.3. V1M4 Secs. 1.5.2; 1.5.2.1; 1.5.2.2	5.4.5.3	82
19.8	Instrument Detection Limits (Idl)	V1M2 Sec. 5.9.3		83
19.9	Verification Of Detection And Reporting Limits	V1M2 Sec. 5.9.3. V1M4 Sec. 1.5.2.1		83
19.10	Retention Time Windows	V1M2 Sec. 5.9.3		84
19.11	Evaluation Of Selectivity	V1M2 Sec. 5.9.3. V1M4 Sec. 1.5.4; 1.7.3.6		84
19.12	Estimation Of Uncertainty Of Measurement	V1M2 Sec. 5.1.1; 5.1.2; 5.4.6	5.1.1; 5.1.2; 5.4.6.1; 5.4.6.2; 5.4.6.3	84
19.13	Sample Reanalysis Guidelines	V1M2 Sec 5.9.1	5.9.1	85
19.14	Control Of Data	V1M2 Secs. 5.4.7.1; 5.4.7.2; 5.9.1	5.4.7.1; 5.4.7.2; 5.9.1	85
20.0	EQUIPMENT and CALIBRATIONS	V1M2 Secs. 5.5.4; 5.5.5; 5.5.6	5.5.4; 5.5.5; 5.5.6; 5.6.1	93
20.1	Overview	V1M2 Secs. 5.5.1; 5.5.2; 5.5.3; 5.5.5; 5.5.10	5.5.1; 5.5.2; 5.5.3; 5.5.5; 5.5.10; 5.6.1	93
20.2	Preventive Maintenance	V1M2 Secs. 5.5.1; 5.5.3; 5.5.7; 5.5.9	5.5.1; 5.5.3; 5.5.7; 5.5.9; 5.6.1	93
20.3	Support Equipment	V1M2 Secs. 5.5.10; 5.5.11; 5.5.13.1	5.5.10; 5.5.11; 5.6.2.1.2; 5.6.2.2.1; 5.6.2.2.2	94
20.4	Instrument Calibrations	V1M2 Secs. 5.5.8; 5.5.10; 5.6.3.1. V1M4 Sec. 1.7.1.1; 1.7.2	5.5.8; 5.5.9; 5.5.10; 5.6.1; 5.6.2; 5.6.3.1	96
20.5	Tentatively Identified Compounds (TICS) – GC/MS Analysis			99

Sec. No.	Title	2009 TNI Standard Reference	ISO/IEC 17025:2005 (E) Reference	Page No.
20.6	Gc/Ms Tuning			99
21.0	MEASUREMENT TRACEABILITY			104
21.1	Overview	V1M2 Sec. 5.6.3.1	5.6.2.1.2; 5.6.2.2.2; 5.6.3.1	104
21.2	NIST-Traceable Weights And Thermometers	V1M2 Secs. 5.5.13.1; 5.6.3.1; 5.6.3.2	5.6.3.1; 5.6.3.2	104
21.3	Reference Standards / Materials	V1M2 Secs. 5.6.3.1; 5.6.3.2; 5.6.3.3; 5.6.3.4; 5.6.4.1; 5.6.4.2; 5.9.1; 5.9.3	5.6.3.1; 5.6.3.2; 5.6.3.3; 5.6.3.4; 5.9.1	104
21.4	Documentation And Labeling Of Standards, Reagents, And Reference Materials	V1M2 Secs. 5.6.4.2; 5.9.3		105
22.0	SAMPLING			107
22.1	Overview	V1M2 Secs. 5.7.1; 5.7.3	5.7.1; 5.7.3	107
22.2	Sampling Containers			107
22.3	Definition Of Holding Time			108
22.4	Sampling Containers, Preservation Requirements, Holding Times			108
22.5	Sample Aliquots / Subsampling	V1M2 Sec. 5.7.1	5.7.1	108
23.0	HANDLING OF SAMPLES	V1M2 Sec. 5.8.1	5.8.1	108
23.1	Chain Of Custody (COC)	V1M2 Secs. 5.7.2; 5.7.4; 5.8.4; 5.8.7.5; 5.8.8; 5.9.1	5.7.2; 5.8.4; 5.9.1	108
0	Sample **Receipt**	V1M2 Secs. 5.8.1; 5.8.2; 5.8.3; 5.8.5; 5.8.7.3; 5.8.7.4; 5.8.7.5	5.8.2; 5.8.3	110
23.3	Sample Acceptance Policy	V1M2 Secs. 5.8.6; 5.8.7.2		111
23.4	Sample Storage	V1M2 Secs. 5.7.4; 5.8.4	5.8.4	111
23.5	Hazardous Samples And Foreign Soils			112
23.6	Sample Shipping	V1M2 Sec. 5.8.2	5.8.2	112
23.7	Sample Disposal			113
24.0	ASSURING THE QUALITY OF TEST RESULTS			119
24.1	Overview	V1M2 Secs. 5.9.2; 5.9.3	5.9.2	119
24.2	Controls	V1M2 Secs. 5.9.2; 5.9.3	5.9.2	119

Sec. No.	Title	2009 TNI Standard Reference	ISO/IEC 17025:2005 (E) Reference	Page No.
24.3	Negative Controls	V1M2 Secs. 5.9.2; 5.9.3 V1M4 Secs. 1.7.3; 1.7.3.1; 1.7.4.1	5.9.2	119
24.4	Positive Controls	V1M2 Secs 5.9.2; 5.9.3. V1M4 Secs. 1.7.3; 1.7.3.2; 1.7.3.2.1; 1.7.3.2.2; 1.7.3.2.3	5.9.2	120
24.5	Sample Matrix Controls	V1M2 Secs. 5.9.2; 5.9.3. V1M4 Secs. 1.7.3 ; 1.7.3.3; 1.7.3.3.1; 1.7.3.3.2; 1.7.3.3.3	5.9.2	122
24.6	Acceptance Criteria (Control Limits)	V1M2 Sec. 5.9.3. V1M4 Secs. 1.7.4.2; 1.7.4.3		122
24.7	Additonal Procedures To Assure Quality Control	V1M2 Sec. 5.9.3. V1M4 Sec. 1.7.3.4		125
25.0	REPORTING RESULTS			125
25.1	Overview	-V1M2 Secs. 5.10.1; 5.10.2; 5.10.8	5.10.1; 5.10.2; 5.10.8	125
25.2	Test Reports	V1M2 Secs. 5.10.1; 5.10.2; 5.10.3.1; 5.10.3.2; 5.10.5; 5.10.6; 5.10.7; 5.10.8; 5.10.10; 5.10.11	5.10.1; 5.10.2; 5.10.3.1; 5.10.3.2; 5.10.5; 5.10.6; 5.10.7; 5.10.8	125
25.3	Reporting Level Or Report Type	V1M2 Secs. 5.10.1; 5.10.7; 5.10.8	5.10.1; 5.10.7; 5.10.8	128
25.4	Supplemental Information For Test	V1M2 Secs. 5.10.1; 5.10.3.1; 5.10.5	5.10.1; 5.10.3.1; 5.10.5	128
25.5	Environmental Testing Obtained From Subcontractors	V1M2 Secs. 4.5.5; 5.10.1; 5.10.6	5.10.1; 5.10.6	129
25.6	Client Confidentiality	V1M2 Secs. 4.1.5; 5.10.7	4.1.5; 5.10.7	129
25.7	Format Of Reports	V1M2 Sec. 5.10.8	5.10.8	130
25.8	Amendments To Test Reports	V1M2 Sec. 5.10.9	5.10.1; 5.10.9	130
25.9	Policies On Client Requests For Amendments	V1M2 Secs. 5.9.1; 5.10.9	5.9.1; 5.10.1; 5.10.5; 5.10.9	130

LIST OF TABLES

Table No.	Title	2009 TNI Standard Reference	ISO/IEC 17025:2005 (E) Reference	Page No.
12-1	Example – General Corrective Action Procedures	V1M2 Sec. 4.11.6. V1M4 Sec. 1.7.4.1	4.11.2	55
14-1	Record Index		4.13.1.1	59
14-2	Example: Special Record Retention Requirements			61
15-1	Types Of Internal Audits And Frequency		4.14.1	66
20-1	Example: Instrumentation List		5.5.4; 5.5.5	99
20-2	Example: Software List			100
20-3	Example: Schedule Of Routine Maintenance			101
24-1	Example – Negative Controls			119
24-3	Sample Matrix Control			122

LIST OF FIGURES

Figure No.	Title	2009 TNI Standard Reference	ISO/IEC 17025:2005(E) Reference	Page No.
4-1	Corporate And Laboratory Organization Chart	V1M2 Sec. 4.1.5	4.1.3; 4.1.5; 4.2.6	255
8-1	Example - Subcontracted Sample Form			41
12-1	Example - Non Conformance Memo			54
19-1	Example - Demonstration Of Capability Documentation			91
19-2	Example: Work Flow			92
23-1	Example: Chain Of Custody (COC)			114
23-2	Example: Sample Acceptance Policy	V1M2 Sec. 5.8.6; 5.8.7.1. V1M4 Sec. 1.7.5		115
23-3	Example: Cooler Receipt Form		5.8.3	118

LIST OF APPENDICES

Appendix No.	Title	Page No.
1	Laboratory Floor Plan	132
2	Glossary/Acronyms	133
3	Laboratory Certifications, Accreditations, Validations	141
4	Summary Of Calibration, QC And Corrective Action Procedures	142

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
CA-Q-S-009	Root Cause Analysis
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
TA-QA-0528	Document Control
TA-QA-0529	Complaint Resolution
TA-QA-0530	Management of Change
TA-QA-0506	Archiving Reports and Report File Maintenance
TA-QA-0608	Employee Training Procedures
TA-QA-0500	Standard Operating Procedures
TA-QA-0617	Analyst Demonstration of Capability
TA-QA-0602	Method Detection Limit Studies and Limits of Detection
TA-IP-0226	Multi Incremental Subsampling of Soils and Sediments
TA-QA-0028	Subsampling of Solid Samples
TA-QA-0001	Sample Receipt & Login
TA-QA-0610	Laboratory Corrective Action Procedures
TA-QA-0635	Data Review
TA-QA-0014	Selection and Use of Laboratory Balances
TA-QA-0024	Use, Calibration and Maintenance of Laboratory Thermometers
TA-QA-0016	Volumetric Verification
TA-QA-0610	Laboratory Corrective Action Procedures
TA-QA-0619	Receipt, Storage and Verification of Standards
TA-QA-0600	Quality Control Charting and Establishing

SECTION 3. INTRODUCTION, SCOPE AND APPLICABILITY

3.1 Introduction and Compliance References

TestAmerica Seattle'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.
- 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 drinking water, effluent water, groundwater, hazardous waste, sludge, sediments and soils. The Quality Assurance Program contains specific procedures and methods to test samples of differing matrices for chemical and physical 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 the Laboratory Information Management System (LIMS)**.** 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 **annually** by senior laboratory management to assure that it reflects current practices and meets the requirements of the laboratory's clients and regulators as well as the CQMP. Occasionally, the manual may need changes in order to meet new or changing regulations and operations. The QA Manager will review the changes in the normal course of business and incorporate changes into revised sections of the document. All updates will be reviewed by the senior laboratory management staff. The laboratory updates and approves such changes according to our Document Control procedures (refer to SOP No. TA-QA-0528).

SECTION 4. MANAGEMENT REQUIREMENTS

4.1 Overview

TestAmerica Seattle 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 Executive 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 Seattle 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 Seattle laboratory.

4.2.2 Laboratory Director

TestAmerica Seattle's Laboratory Director is responsible for the overall quality, safety, financial, technical, human resource and service performance of the whole laboratory and reports to their respective GM. The Laboratory Director provides the resources necessary to implement and maintain an effective and comprehensive Quality Assurance and Data Integrity Program.

Specific responsibilities include, but are not limited to:

- Provides one or more technical managers for the appropriate fields of testing. The name(s) of the Technical Director will be included in the national database. If the Technical Director is absent for a period of time exceeding 15 consecutive calendar days, the Laboratory Director must designate another full time staff member meeting the qualifications of the Technical Director to temporarily perform this function. If the absence exceeds 65 consecutive calendar days, the primary accrediting authority must be notified in writing.
- 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 that 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.
- Reviews and approves the laboratory specific QAM, policies, SOPs prior to their implementation and ensures all approved procedures are implemented and adhered to.
- Pursues and maintains appropriate laboratory certification and contract approvals. Supports ISO 17025 requirements. Organizes bid activities for prospective new projects and clients.
- Ensures client specific reporting and quality control requirements are met.
- Captains the management team, consisting of the QA Manager, the Technical Director(s), and the Operations Manager as direct reports.
- Annually assesses the effectiveness of the QMP and QAM within the laboratory.

4.2.3 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 the QA officers to accomplish specific responsibilities, which include, but are not limited to:

- Compliance with ISO 17025
- Compliance with DoD ELAP
- 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 monitors 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.
- Evaluation of the thoroughness and effectiveness of training.

4.2.4 Technical Managers or Designees

The Technical Managers report directly to the Laboratory Director. He/she is 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:

- Compliance with ISO 17025
- Compliance with DoD ELAP

- 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.
- 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.
- Coordinates audit responses with the QA Manager.

4.2.5 LIMS Administration/IT Support

LIMS Administration and IT support are managed at the corporate level. In general, the duties of the corporate LIMS administration team consist of:

- Establishes and maintains the laboratory information system (LIMS) for tracking all samples in the laboratory.
- Updates and enhances LIMS.

- Develops expertise in the requirements described in Good Automated Laboratory Practices (GALP) EPA 2185, 1995 Edition, in order to ensure compliance.
- Programs and tests software modifications/changes.
- Coordinates testing to ensure that all LIMS software accurately performs its intended functions. Testing is performed and documented after installation or when modifications/ changes are made.
- 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.
- Develops and verifies security practices to assure the integrity of LIMS data. Identifies threats, potential threats, and future threats.
- Maintains awareness of any environmental conditions of the facility housing the LIMS that may compromise LIMS raw data and informs management.
- LIMS database back-up once daily.

4.2.6 Hazardous Waste Coordinator

The Hazardous Waste Coordinator reports directly to the Laboratory Director. The duties consist of:

- Managing laboratory generated hazardous waste in accordance with appropriate regulations.
- 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

4.2.8 Department Managers

The Department Manager duties at TestAmerica Seattle are performed by the Technical Managers. The responsibilities of the department managers are listed below:

- Ensure that analysts in their department adhere to applicable SOPs and the QA Manual. They perform frequent SOP and QA Manual review to determine if analysts are in compliance and if new, modified, and optimized measures are feasible and should be added to these documents.
- With regard to analysts, participates in the selection, training (as documented in Section 8.1), development of performance objectives and standards of performance, appraisal (measurement of objectives), scheduling, counseling, discipline, and motivation of analysts and documents these activities in accordance with systems developed by the QA and Personnel Departments. They evaluate staffing sufficiency and overtime needs. Training consists of familiarization with SOP, QC, Safety, and computer systems.

- Encourage the development of analysts to become cross-trained in various methods and/or operate multiple instruments efficiently while performing maintenance and documentation, self-supervise, and function as a department team.
- Provide guidance to analysts in resolving problems encountered daily during sample prep/analysis in conjunction with the QA Manager. Each is responsible for 100% of the data review and documentation, non-conformance and CPAR issues, the timely and accurate completion of performance evaluation samples and MDLs, for his/her department.
- Works with the Project Managers and Analysts/Technicians to ensure that the requirements of project are met in a timely manner.
- Ensure all logbooks are maintained, current, and properly labeled or archived.
- Responsible for meeting quality requirements and reporting all non-conformance conditions to the QA Manager and/or Laboratory Director.
- Ensure that preventive maintenance is performed and documented 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.
- Conduct efficiency and cost control evaluations on an ongoing basis to determine optimization of labor, supplies, overtime, first-run yield, capacity (designed vs. demonstrated), second- and third-generation production techniques/instruments, and long-term needs for budgetary planning.
- Develop, implement, and enhance calibration programs.
- Provide written responses and facilitates implementing corrective actions for external and internal audit issues.

4.2.9 Laboratory Analysts

Laboratory analysts are responsible for conducting analysis and performing all tasks assigned to them by the department manager, group leader or supervisor. The responsibilities of the analysts are listed below:

- Perform analyses by adhering to analytical and quality control protocols prescribed by current SOPs, this QA Manual, and project-specific plans honestly, accurately, timely, safely, and in the most cost-effective manner.
- Document standard and sample preparation, instrument calibration and maintenance, data calculations, sample matrix effects, and any observed non-conformance on worklists, benchsheets, lab notebooks and/or the Non-Conformance Database.
- Report all non-conformance situations, instrument problems, matrix problems and QC failures, which might affect the reliability of the data, to their supervisor, the Technical Director, and/or the QA Manager or member of QA staff.
- Ensures sample and data integrity by adhering to internal chain-of-custody procedures.

- Perform 100% review of the data generated prior to entering and submitting for secondary level review.
- Suggest method improvements to their supervisor, the Technical Director, and the QA Manager. These improvements, if approved, will be incorporated. Ideas for the optimum performance of their assigned area, for example, through the proper cleaning and maintenance of the assigned instruments and equipment, are encouraged.
- 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.

4.2.10 Safety Officer

The Safety Officer reports to the Laboratory Director and ensures that systems are maintained for the safe operation of the laboratory. The Safety Officer is responsible to:

- 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.
- Manages facility maintenance.

4.2.11 Sample Receiving Staff

The Sample Receiving Technicians report to the Sample Receiving Supervisor; the Supervisor reports to the Operations Manager. The responsibilities are outlined below:

- Ensures implementation of proper sample receipt procedures, including maintenance of chain-of-custody.
- Reports nonconformances associated with condition-upon-receipt of samples.
- Logs incoming samples into the LIMS.
- Ensures that all samples are stored in the proper environment.
- Ensure the verification of data entry from login.
- Responsible for meeting quality requirements including documenting preservation.
- Responsible for ensuring the timely and correct shipment of sample containers, including proper preservatives and instructions, to clients
- Assists Environmental Health and Safety staff with sample disposal.

4.2.12 Project Manager Supervisor

The Project Manager Supervisor reports to the Client Services 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.
- Has signature authority for laboratory reports.
- Assesses and assures customer satisfaction.
- Provides feedback to management on changing customer needs.
- Works with the Department Managers and/or Analysts/Technicians to ensure the requirements of projects are met in a timely manner.
- Organizes bid activities for prospective new projects and clients.

4.2.13 Project Managers

The Project Managers report to the Customer Service Manager and serve as the interface between the laboratory and the clients. With the overall goal of total client satisfaction, the functions of this position are outlined below:

- Responsible to ensure that clients receive the proper sampling supplies.
- Accountable for response to client inquiries concerning sample status.
- Responsible for assistance to clients regarding the resolution of problems concerning COC.
- Ensuring that client specifications, when known, are met by communicating project and quality assurance requirements to the laboratory. Prepares a Quality Assurance Summary (QAS) or equivalent summary form as needed.
- 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.
- Approves customer requested variances to methods and to standard laboratory protocols.
- Responsible for discussing with client any project-related problems, resolving service issues, and coordinating technical details with the laboratory staff.
- Reports client inquiries involving data quality issues or data acceptability to the facility, CSM, Laboratory Director, QA Manager, and to the appropriate 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. Reviews project data packages for completeness and compliance to client needs and have signature authority for final reports.
- Inform clients of data package-related problems and resolve service issues.
- Coordinate requests for sample containers and other services (data packages).
- Prepares re-issue requests for project data.
- Organizes bid activities for prospective new projects and clients.
- Has signature authority for laboratory reports.

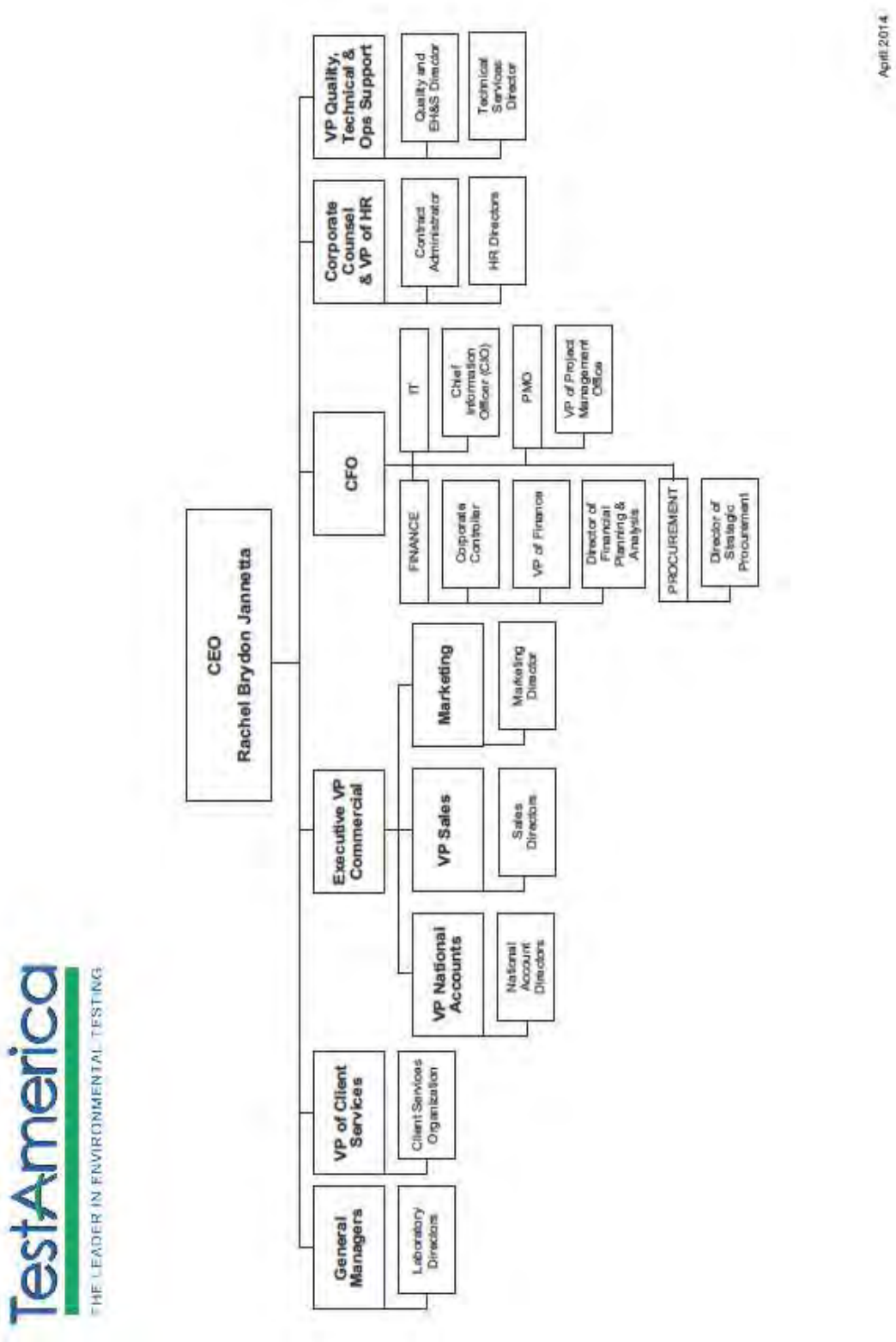
4.3 Deputies

The following table defines who assumes the responsibilities of key personnel in their absence:

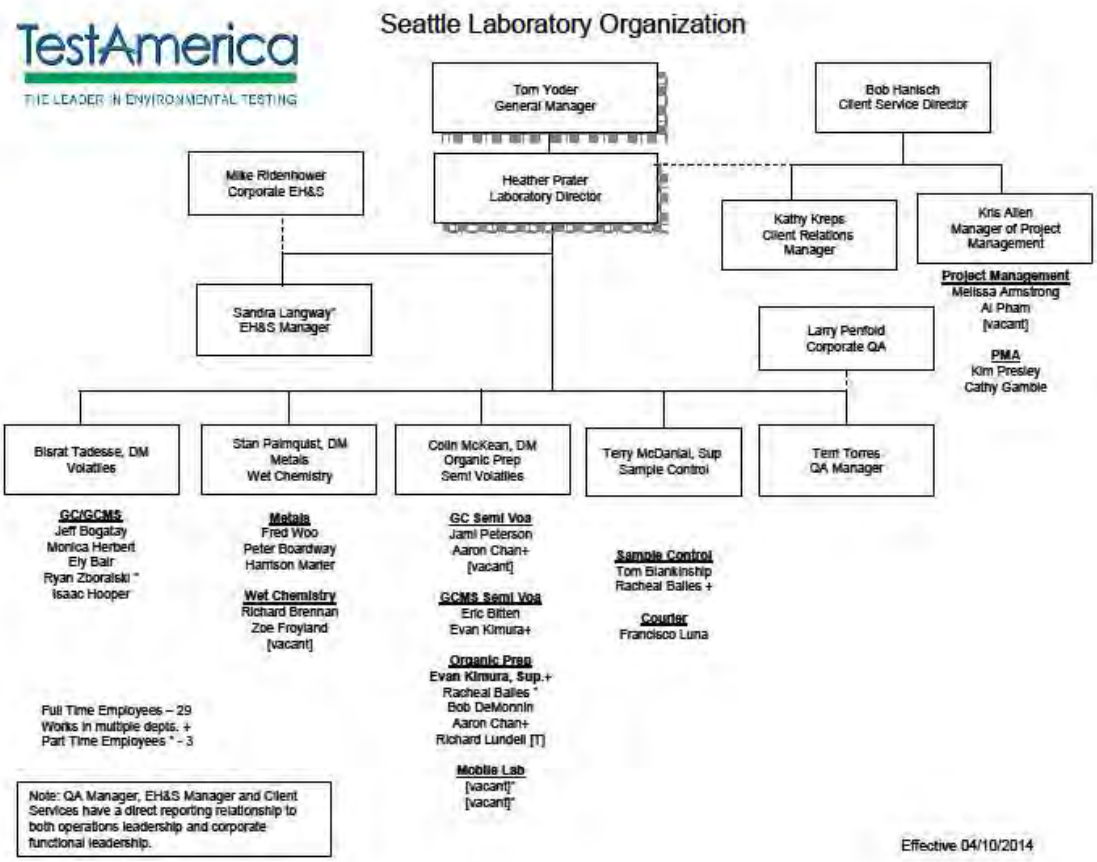
Key Personnel	Deputy
Laboratory Director	QA Manager
QA Manager	Laboratory Director
Inorganic Technical Manager	Laboratory Director

Key Personnel	Deputy
Semivolatile Technical Manager	Laboratory Director
Volatile Technical Manager	Laboratory Director
EHS Coordinator	Laboratory Director
Project Manager Supervisor	Laboratory Director

Figure 4-1. Corporate and Laboratory Organization Charts



TestAmerica Seattle
Organization Chart



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 in the Laboratory Information Management System 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 SOP TA-QA-0600 Control Charting and Establishing Method Warning and Action Limits.

5.6 Statistical Quality Control

Statistically-derived precision and accuracy limits are required by selected methods (such as SW-846) and programs [such as the Ohio Voluntary Action Plan (VAP)]. The laboratory routinely utilizes statistically-derived limits to evaluate method performance and determine when corrective action is appropriate. The analysts are instructed to use the current limits in the laboratory (dated and approved by the 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 laboratory via the 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

As the QC limits are calculated, QC charts are generated showing warning and control limits for the purpose of evaluating trends. The QA Manager evaluates these to determine if adjustments need to be made or for corrective actions to methods. All findings are documented and kept on file. (Refer to SOP TA-QA-0600 Control Charting and Establishing Method Warning and Action Limits.)

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 procedures are defined in SOPs TA-QA-0528, (Document Control) and TA-QA-0506 (Document Archiving).

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 nonconformance memos/corrective action reports. 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 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 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 No. TA-QA-0528 (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 QAM folder for the applicable revision.

For changes to SOPs, refer to SOP No. TA-QA-0500, Standard Operating Procedures. 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 TA-QA-0528 (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. TA-QA-0528 (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
- Customer Services Manager
- Laboratory Operations Manager
- Laboratory and/or Corporate Technical Managers / Directors
- Corporate Information Technology Managers
- Account Executives
- Laboratory and/or Corporate Quality
- Laboratory Environmental Health and Safety Managers
- The Laboratory Director reviews the formal laboratory quote and makes final acceptance for their facility.

The Sales Director, Legal Contracts Director or Account Executive then submits the final proposal to the client.

In the event that one of the above personnel is not available to review the contract, his or her back-up will fulfill the review requirements.

The Legal & Contracts Director maintains copies of all signed contracts**.** The Laboratory Director also maintains copies of all signed contracts on site.

7.3 Documentation

Appropriate records are maintained for every contract or work request. All stages of the contract review process are documented and include records of any significant changes. The reviewed contracts or QAPPs, along with records identifying issues or changes (at a minimum the Additional Project Notes from the quote) will be scanned and stored on the Public server in the QA folder.

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 and all actions agreed upon between the PM and the client are officially documented with a follow-up email.

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 communicated to the laboratory during production or status meetings. These changes are also updated in 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 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).

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.

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 develop 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-NELAC accredited work where required.

Project Managers (PMs), Project Manager Supervior, 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 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;
- 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, 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 or CSM responsible for the project must advise and obtain client consent to the subcontract as appropriate, and provide the scope of work to ensure that the proper requirements are made a part of the subcontract and are made known to the subcontractor.

Prior to sending samples to the subcontracted laboratory, the PM confirms their certification status to determine if it's current and scope-inclusive. The information is documented on a Subcontracted Sample Form (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-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**_____
- 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 completes the Material Request Sheet when requesting reagents, standards, or supplies that are not stocked in on-site consignment system that contains items approved for laboratory use.

The analyst must provide the master item number (from the master item list that has been approved by the Technical Manager), 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 Manager prior to placing the order. The authorized requisitioning agent places the order.

9.3.2 Receiving

It is the responsibility of the receiving clerk 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 QA office**.**

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. 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 megohm-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 Manager 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 stored as scanned images in the LIMS Reagent module. 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 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 Technical 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 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 the 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.

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 Manager 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 laboratory SOP TA-QA-0529 (Client Complaint Resolution).

10.2 External Complaints

An employee that receives a complaint initiates the complaint resolution process by first documenting the complaint according to SOP TA-QA-0529.

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 supervisor for resolution. The supervisor may elect to discuss it with the Technical 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 laboratory's 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 Technical Manager 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-NELAC 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, a 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, 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), Corporate Quality Director 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 and Operations Manager.

The Laboratory Director and/or Operations Manager 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, Operations Manager, Technical 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 Memoranda (NCM) and Corrective Action Reports (CAR) (refer to SOP No. TA-QA-601 and 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 Memorandum (NCM) - is used to document the following types of corrective actions:

- Deviations from an established procedure or SOP
- QC outside of limits (non-matrix related)
- 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 NCMs.
- Issues found while reviewing NCMs that warrant further investigation.
- Internal and external audit findings.
- Failed or unacceptable PT results.
- Corrective actions that cross multiple departments in the laboratory.
- Systematic reporting / calculation errors
- Client complaints
- Data recall investigations
- Identified poor process or method performance trends
- Excessive revised reports
- Health and Safety violations

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. (refer to SOP No. TA-QA-0610 for more detail)

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 Technical 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.

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 arrive at the root cause. Also, 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, thus curtailing issues that could plague the laboratory or operation. (refer to SOP No. CA-Q-S-009 for more detail)

12.3.4 Monitoring of the Corrective Actions

- The Technical 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 Operations Manager and Laboratory Director to ensure final acceptable resolution is achieved and documented appropriately.
- Each NCM and CAR is entered into a database for tracking purposes and a monthly summary of all corrective actions is printed out for review to aid in ensuring that the corrective actions have taken effect.
- The QA Manager reviews monthly NCMs and 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 NCM.

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 and 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 - Non Conformance Memo

01/09/2008 **NonConformance Memo Report** Page 1 of 1

NCM ID: 580-5190 **Date Opened:** 11/30/2007

NCM Type: CCV - %D, High, Sample ND **Date Closed:**

Lab Section: Pesticides and PCBs **Date Verified:**

Narrative

The closing continuing calibration verification (CCV) for Endosulfan II recovered above the upper control limit. The samples associated with this CCV were non-detects for the affected analytes; therefore, the data have been reported.

Affected Items

Description	Project Manager
580-8247-E-1-A	Curbow, Heather
LCS 580-26165/2-A	Curbow, Heather
580-8247-C-2-A	Curbow, Heather
580-8247-E-1-A	Curbow, Heather
LCS 580-26165/2-A	Curbow, Heather
MB 580-26165/1-A	Curbow, Heather
LCSD 580-26165/3-A	Curbow, Heather
580-8247-C-2-A	Curbow, Heather
MB 580-26165/1-A	Curbow, Heather
LCSD 580-26165/3-A	Curbow, Heather

Notifications

User Full Name	Date Received	Date	Notice Level	Verification Type
Mott, Christina M	11/30/2007		Level 1	Review

Job Case Narratives

Job: 580-J8247-1

Job Narrative
580-J8247-1

Comments
No additional comments.

Receipt
All samples were received in good condition within temperature requirements.

GC/MS VOA
No analytical or quality issues were noted.

GC/MS Semi VOA
No analytical or quality issues were noted.

GC Semi VOA
Method 8081: The RPD between the results of the two columns for heptachlor and DDT was >40%. This is due to matrix effect; the lower of the two values (presumably free from matrix interference) is reported as primary.

No other analytical or quality issues were noted.

Metals
No analytical or quality issues were noted.

General Chemistry
No analytical or quality issues were noted.

Organic Prep
No analytical or quality issues were noted.

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, Technical Manager(s))	- 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, Technical Manager(s))	- % 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 LIMS.	- 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 LIMS.	- 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, Technical 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.
Internal / External Audits (QA Manager, Technical 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.

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, Technical Managers, 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 Lab SOP TA-QA-0610 Laboratory Corrective Action Procedures.
Client Complaints (Project Managers, Lab Director, 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, Technical Manager(s))	- 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, Technical Manager(s))	- 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 or for DoD projects the method blank should be below ½ the RL. 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 QA 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 procedures, 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 changes covered under this system include: Facility Changes, Major Accreditation Changes, Addition or Deletion to Division's Capabilities or Instrumentation, Key Personnel Changes, Laboratory Information Management System (LIMS) changes. This process is discussed in further detail in SOP TA-QA-0530, Management of Change.

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, in this instance the electronic copy will be considered to be the vital record). Technical records are maintained by the Technical Managers.

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*

	Record Types [1]:	**Retention Time:**
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, Permits	**Indefinitely**
	Employee Handbook	Indefinitely
	Personnel files, Employee Signature & Initials, Administrative Training Records (e.g., Ethics)	Refer to HR Manual
	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 or the Iron Mountain data storage facility that provide 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 archived off-site are stored in a secure location where a record is maintained of any entry into the storage facility. Whether on-site or off-site storage is used, logs are maintained in each storage box to note removal and return of records. Retention of records are maintained on-site at the laboratory for at least 6 months after their generation and moved

offsite for the remainder of the required storage time. Records are maintained for a minimum of five years unless otherwise specified by a client or regulatory requirement.

For raw data and project records, record retention shall be calculated from the date the project report is issued. For other records, such as Controlled Documents, QA, or Administrative Records, the retention time is calculated from the date the record is formally retired. Records related to the programs listed in Table 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)
Alaska	10 years
Louisiana – All	10 years
Navy Facilities Engineering Service Center (NFESC)	10 years
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. Additional information can also be reference in SOP TA-QA-0506 Archiving Data and Reports.

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 COC is stored with the invoice and the work order

sheet generated by the LIMS in a job folder. The chain of custody would indicate the name of the sampler. If any sampling notes are provided with a work order, they are kept with this package.

- 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 as per SOP TA-QA-0506 Archiving Reports and Report File Maintenance). Instrument data is stored sequentially by instrument. A given day's analyses are maintained in the order of the analysis. Run logs are maintained for each instrument or method; a copy of each day's run log or instrument sequence is stored with the data to aid in re-constructing an analytical sequence. Where an analysis is performed without an instrument, bound logbooks or bench sheets are used to record and file data. Standard and reagent information is recorded in logbooks or entered into the LIMS for each method as required.

- Changes to hardcopy records shall follow the procedures outlined in Section 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. The procedure for this verification can be found in SOP TA-QA-0506 Archiving Reports and Report File Maintenance.

- 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 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 or in the LIMS.
- Instrumentation identification and instrument operating conditions/parameters. Operating conditions/parameters are typically recorded in instrument maintenance logs where available.
- analysis type**;**
- all manual calculations and manual integrations**;**
- analyst's or operator's initials/signature**;**
- sample preparation including cleanup, separation protocols, incubation periods or subculture, ID codes, volumes, weights, instrument printouts, meter readings, calculations, reagents**;**
- test results**;**
- standard and reagent origin, receipt, preparation, and use**;**
- calibration criteria, frequency and acceptance criteria**;**
- data and statistical calculations, review, confirmation, interpretation, assessment and reporting conventions**;**
- quality control protocols and assessment**;**
- electronic data security, software documentation and verification, software and hardware audits, backups, and records of any changes to automated data entries; and
- Method performance criteria including expected quality control requirements. These are indicated both in the LIMS and on specific analytical report formats**.**

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 maintained in the LIMS – no logbooks are used to record that data**.** 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: **100% 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 NELAC 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, NELAC quality systems, client and state requirements, and the effectiveness of the internal controls of the analytical process, including but not limited to data review, quality controls, preventive action and corrective action. The completeness of earlier corrective actions is assessed 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 Method Audits

Method 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. Method audits will include all methods annually.

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 Technical Manager or qualified designee annually. 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, and Soil.

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 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, Technical Managers, Operation Manager, their Quality Director as well as the General Manager. All aspects of the QA system are reviewed to evaluate the suitability of policies and procedures. During the course of the year, the Laboratory Director, General Manager or Corporate QA may request that additional information be added to the report.

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, Technical Managers, Operation Manager, 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 is 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 Support, 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 Managers 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
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

Specialty	Education	Experience
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 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 violations). 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 Employee Training SOP (TA-QA-0608).

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 *fixed* laboratory is a 20,000 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 mobile laboratory is a 23 ft van or client provided work space designed to accommodate an efficient workflow and to provide a safe and comfortable work environment for employees.

The *fixed and mobile* laboratories *are* 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 *at fixed laboratory*), 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. *The fixed* laboratory HVAC and deionized water systems are designed to minimize potential trace contaminants.

The *fixed* 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 *fixed* 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 temperature levels in the laboratory.

When any of the method or regulatory required environmental conditions change to a point where they may adversely affect test results, analytical testing will be discontinued until the environmental conditions are returned to the required levels.

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

The fixed laboratory floor plan can be found in Appendix 1.

18.5 Building Security

Building/Van 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 the laboratory's SOP TA-QA-0500 Standard Operating Procedures.
- 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
- 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
- Plumb, Jr., R.H. 1981, Procedures for Handling and Chemical Analysis of Sediment and Water Samples, Technical Report EPA/C E-81-1. US Army Engineering Waterways Experiment Station, Vicksburg, MS.
- Recommended Protocols for Measuring Conventional Sediment Variables in Puget Sound, April 2003.
- Recommended Guidelines for Measuring Organic Compounds in Puget Sound Waters, Sediment and Tissue Samples, April 1997.
- Recommended Guidelines for Measuring Metals in Puget Sound Waters, Sediment and Tissue Samples, April 1997.

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 (Analyst DOC, Lab SOP # TA-QA-0617) 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 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 or the laboratory's SOP No. TA-QA-0602 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 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 TA-QA-0602 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. 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 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 uncertainties at approximately the 99% confidence level with a coverage factor of k = 3. As an example, for a reported result of 1.0 mg/l with an LCS recovery range of 50 to 150%, the estimated uncertainty in the result would be 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 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 Area Technical Manager or 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 SOP TA-IT-0523 Data Integrity and Security. The laboratory is currently running the TALS system 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 Sequel 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 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 Technical Director and 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 outlined in several SOPs and SOP TA-QA-0635 Procedure for Peer Review of Analytical Data and Project Management Review of Reports to ensure that reported

data are free from calculation and transcription errors and that QC parameters have been reviewed and evaluated before data is reported. The laboratory also utilizes corporate SOP CA-Q-S-002 Acceptable Manual Integration Practices to ensure the authenticity of the data. 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 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.

19.14.4.7 A visual summary of the flow of samples and information through the laboratory, as well as data review and validation, is presented in Figure 19-2.

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,

Document Uncontrolled When Printed

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

TestAmerica Seattle
Analyst Demonstration of Capability-Certification Statement

Initial: _____
Continuing: _____

Date of Preparation: _____ Date of Analysis: _____

Analyst: _____ Analyst: _____

Matrix: Water Soil Sediment Other (Specify):_____

Reference method:_____

Reference SOP:_____

Parameters:_____

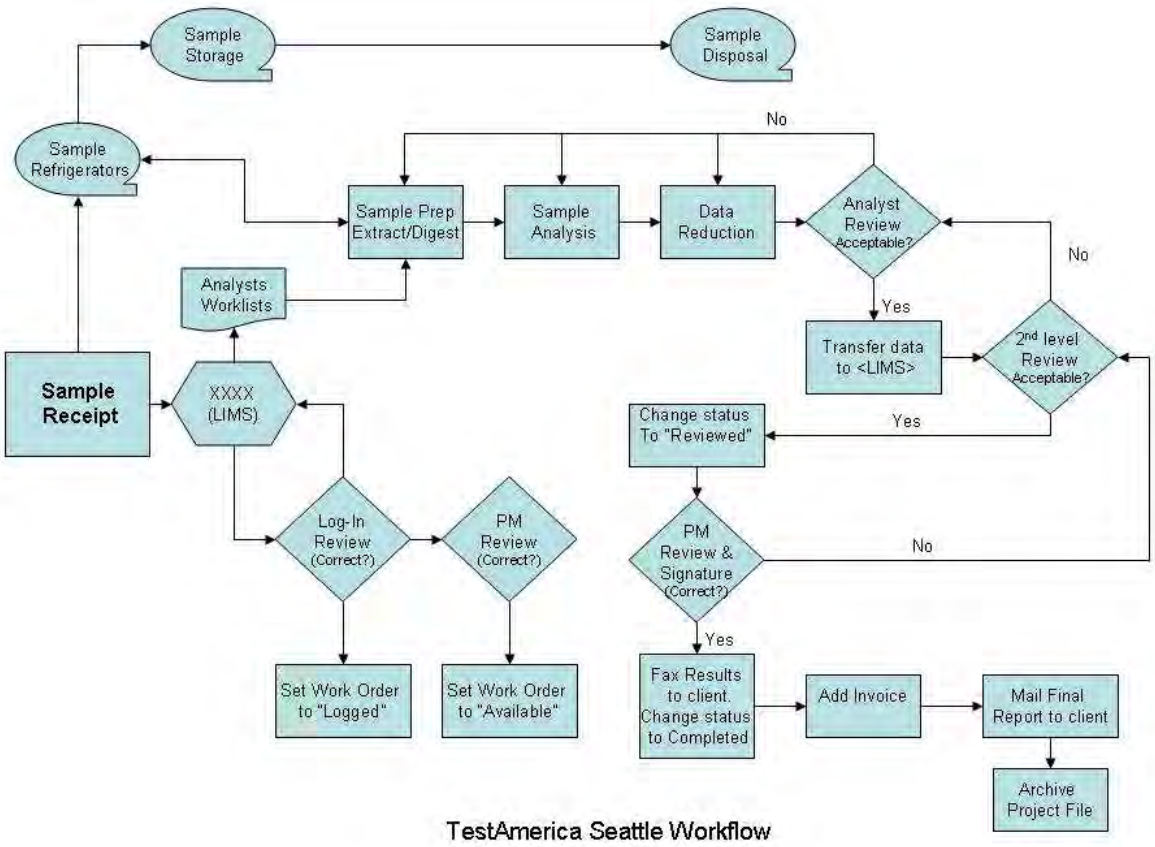
We, the undersigned, CERTIFY that:

- ❑ The analyst identified above, using the cited test method with the specification in the cited SOP, which is in use at this facility for the analyses of samples under the Nation Environmental Laboratory Accreditation Program, have met Demonstration of Capability.
- ❑ The test method was performed by the analyst identified on this certification following the TestAmerica Seattle SOP.
- ❑ A copy of the laboratory specific SOP is available for all personnel on-site.
- ❑ The data associated with the demonstration of capability are true, accurate, complete, and self-explanatory (*).
- ❑ All raw data including a copy of this certification form) necessary to reconstruct and validate these analyses have been retained at the facility, and that the associated information is well organized and available for review by authorized assessors.

Name	Analyst, Signature	Date
Name	Technical Director, Signature	Date
Name	QA Manager, Signature	Date

* True: consistent with supporting data
Accurate: Based on good laboratory practices with sound scientific principles/practices
Complete: includes the results of all supporting performance testing
Self-explanatory: Data properly labeled and stored so that the results are clean and require no additional explanation.

Figure 19-2. Example: Work Flow



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. A list of software is provided in Table 20-2.

Equipment is only operated by authorized and trained personnel. Manufacturer 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 20-3 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 are also outlined in analytical SOPs or instrument manuals. (Note: for some equipment, the log used to monitor performance is also the maintenance log. Multiple pieces of equipment may share the same log as long as it is clear as to which instrument is associated with an entry.)

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, 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 in the QA office. Refer to SOP TA-QA-0014 Selecting and Using Balances for details.

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 at temperatures bracketing the range of use. 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 SOP TA-QA-0024 Use, Calibration, and Maintenance of Laboratory Thermometers.

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. As a DOD-approved lab, sample storage is monitored 7 days a week.

Ovens, water baths and incubators are monitored on days of use.

All of this equipment has a unique identification number, and is assigned a unique thermometer for monitoring.

Sample storage refrigerator temperatures are kept between > 0ºC and ≤ 6 ºC.

Specific temperature settings/ranges for other refrigerators, ovens, water baths, 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 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 SOP TA-QA-0016 Volumetric Verification.

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.

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). 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.

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).

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 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 and reported 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. Alternatively, a reporting limit standard may be analyzed to demonstrate that the laboratory can still support non-detects at their reporting limit**.**

Samples reported by either of the conditions identified above will be appropriately flagged.

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

GC	GC/MS	Ion Trap GC/MS/MS	ICP	ICPMS	CVAA	AutoAnalyzer	IC	TOC
13	15	2	2	2	2	2	2	2

Table 20-2. Example: Software List

Lab Section	Software Title and Version
All (Data Processing and Reporting)	LIMS - TALS (TestAmerica) Ver 1.0.1.330 or higher
ECD, Fuels 8270 and VOA (Data Processing)	CHROM (TestAmerica) Ver 1.2 or higher
Data Acquistion	
ECD	Environmental Chemstation G1701DA Version D.00.01.27
	Envronmental Chemstation MSD Chemstation D.02.00.275
FUELS	Environmental Chemstation G1701DA Version D.00.01.27
	EnviroQuant ChemStation G1701BA Version B.01.00.
8270	Environmental ChemStation MSD ChemStation D.01.00 Build 75 26-Aug-2003.
	Environmental Chemstation G1701DA Version D.00.01.27 08-Nov-2002
	Environmental ChemStation MSD ChemStation E.01.00.237
	EnviroQuant ChemStation G1702BA Version B.01.00.
	Varian Saturn Software Data System
	Finnigan Magnum
	Finnigan GCQ
VOA	Environmental ChemStation MSD ChemStation D.01.00.275.
	EnviroQuant Chemstation G1701BA Version B.01.00.
	Environmental ChemStation MSD ChemStation E.02.00.493.
	Environmental ChemStation MSD ChemStation D.02.00.275.
	Environmental ChemStation MSD ChemStation D.03.00.552.
	Environmental ChemStation G1701DA Version D.00.01.27.
	EnviroQuant ChemStation G1701AA Version A.03.00.
	Environmental ChemStation G1701CA Version C.00.00 21-Dec-1999
Metals	ELAN Version 3.0 Hotfix 3 (Build 3, 0, 6, 48d)
	Agilent G1834B ICP-MS ChemStation B.03.03
	Perkin Elmer WinLab32 for ICP Version 4.0.0.0305
	Thermo iTeVA – Analyst Version 2.2.0.51
	Perkin Elmer WinLab32 for ICP Version 4.0.0.0305
	WinHg Runner 1.4 CT Rev 0.286
Wet Chem	
Lachat QuikChem	Omnion Program version 3.0.220.04
Astoria Pacific	EnviroQuant Chemstation G1701BA Version B.01.00
Dionex DX-500	Dionex Peaknet Run PeakNet 5.11
Dionex ICS 2000	Chromeleon Client Version 6.60 Build1428
Mitsubishi TOX-100	TOX-100 Versions 2.17 Mitsubishi Chemical Corp
OI Analytical	OI Analytical WinTOC for Solids 3.0
Teledyne Tekmar	TOCTalk Ver 4.2.105.
LECO C632	*LECO C632 Carbon Determination Ver 1.60*

Table 20-3. Example: Schedule of Routine Maintenance

Instrument	Procedure	Frequency
Leeman Mercury Analyzer	Check tubing for wear Fill rinse tank with 10% HCl Change drying tube Fill reductant bottle with 10% Stannous Chloride	Daily Daily As required 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 Weekly Weekly Daily Weekly Weekly Monthly Monthly Monthly Monthly
Ion chromatograph	Check seals for leakage Replace seals/valves/lamps Replace suppressor Replace column Clean source/analyzer	Daily As required As required As required As required
UV-Vis Spectrophotometer	Clean ambient flow cell Precision check/alignment of flow cell Wavelength verification check	As required As required Semi-annually
TOC Analyzer	Check gas flow Check fluid level (IC reservoirs) Replace “O” rings Check needle Replace scrubbers (halogen and CO_2) Replace catalyst	Daily Daily As required Daily Yearly As required
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

Instrument	Procedure	Frequency
Hewlett Packard 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
Gas Chromatograph	Compare standard response to previous day or since last initial calibration Check carrier gas flow rate in column Check temp. of detector, inlet, column oven Septum replacement Check system for gas leaks with SNOOP Check for loose/frayed wires and insulation ½"Bake injector/column Change/remove sections of guard column Replace connectors/liners Change/replace column(s)	Daily Daily via use of known compound retention Daily As required W/cylinder change as required Monthly As Required As Required As Required As Required
Electron Capture Detector (ECD)	Detector wipe test (Ni-63) Detector cleaning	Semi-annually As required
Flame Ionization Detector (FID)	Detector cleaning	As required
Balances	Class "S" traceable weight check Clean pan and check if level Field service	Daily, when used Daily At least Annually
Conductivity Meter	0.01M KCl calibration Conductivity cell cleaning	Daily As required
Turbidimeter	Check light bulb	Daily, when used
Deionized/Distilled Water	Conductivity Point Sources Daily conductivity check Check deionizer light Monitor for VOA's System cleaning Replace cartridge & large mixed bed resins	Water Quality SOP UQA-035 Daily Daily As required As required
Drying Ovens	Temperature monitoring Temperature adjustments	Daily As required
Refrigerators/ Freezers	Temperature monitoring Temperature adjustment Defrosting/cleaning	Daily As required As required
Vacuum Pumps/ Air Compressor	Drained Belts checked Lubricated	Weekly Monthly Semi-annually
pH/Specific Ion Meter	Calibration/check slope Clean electrode	Daily As required

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
Zero Headspace Extractors	Verify rotation speed Check for leakage Vendor repair	As required Annually As required
TCLP Extractors	Verify rotation speed	Quarterly

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, 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), or another accreditation organization that is a signatory to a MRA (Mutual Recognition Arrangement) of one or more of the following cooperations – ILAC (International Laboratory Accreditation Cooperation) or APLAC (Asia –Pacific Laboratory Accreditation Cooperation). A calibration 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 reference standards, to the extent available, are purchased from vendors that are accredited to ISO Guide 34 and ISO/IEC 17025. All reference standards from commercial vendors shall be accompanied with a certificate that includes at least the following information:*

- *Manufacturer*
- *Analytes or parameters calibrated*
- *Identification or lot number*
- *Calibration method*
- *Concentration with associated uncertainties*
- *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. 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 has 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 LIMS. 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. These records are maintained in the LIMS. For detailed information on documentation and labeling, please refer to SOP TA-QA-0619, Preparation, Storage, and Verification of Standards.

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 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 (from LIMS)
- 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 in the LIMS and MSD Sheets**.**

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 laboratory SOP TA-QA-0619, Preparation, Storage, and Verification of Standards.

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 SOP TA-QA-0028, Subsampling of Solid Samples.

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 the 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.

23.2 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 on an NCM or the login Checklist in LIMS 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):

Example: 580 - 9608 - A - 1

Location ID | Login ID | Container Occurrence | Sample Number

In the example above, the Location ID code identifies the lab (580 = TestAmerica Seattle), the Login ID or Job Number (unique to a particular client/job occurrence) is 9608 and 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: 580 - 9608 - A - 1 - **A** ← **Secondary Container Occurrence**

Example: 580-9608-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);
- 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 TA-QA-0001 (Sample Receiving and Login).

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. Aqueous samples for metals testing are typically stored at ambient temperature**.** 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. The refrigerator blanks are logged into the LIMS and treated as normal samples with the data stored and archived in the LIMS.

Analysts and technicians retrieve the sample container allocated to their analysis from the designated storage location and place them on carts, analyze the sample, and return the remaining sample or empty container to the storage location 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 designated storage locations for two to four weeks after analysis, which meets or exceeds most sample holding times. After two to four weeks the samples are moved to dry room temperature, sample archive area where they are stored for an additional four weeks before they are disposed of. This eight week holding period allows samples to be checked if a discrepancy or question arises. Special arrangements may be made to store samples for longer periods of time. This extended holding period allows additional metal analyses to be performed on the archived sample and assists clients in dealing with legal matters or regulatory issues**.**

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 isolated areas designated for hazardous waste or foreign soil samples only. 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. Foreign soil samples are sent out for incineration by a USDA-approved waste disposal facility.

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: TA-EHS-0036). All procedures in the laboratory Environmental, Health and Safety Manual are followed during disposal. Samples are normally maintained in the laboratory no longer than ninety days from receipt unless otherwise requested. Unused portions of samples found or suspected to be hazardous according to state or federal guidelines may be returned to the client upon completion of the analytical work.

If a sample is part of a known litigation, the affected legal authority, sample data user, and/or submitter of the sample must participate in the decision about the sample's disposal. All documentation and correspondence concerning the disposal decision process must be kept on file. Pertinent information includes the date of disposal, nature of disposal (such as sample depletion, hazardous waste facility disposal or return to client), names of individuals who conducted the arrangements and physically completed the task. The laboratory will remove or deface sample labels prior to disposal unless this is accomplished through the disposal method (e.g., samples are incinerated). A Waste Disposal Record should be completed.

Figure 23-1. Example: Chain of Custody (COC)

Seattle
5755 8th Street East
Tacoma, WA 98424
253-922-2310

Chain of Custody Record

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

TestAmerica Laboratories, Inc.

Client Contact:	Project Manager:	Site Contact:	Date:	COC No:
Your Company Name here	Tel/Fax:	Lab Contact:	Carrier:	_____ of _____ COCs
Address:	Analysis Turnaround Time			Job No:
City/State/Zip:	Calendar (C) or Work Days (W) _____			
(xxx) xxx-xxxx Phone:	TAT if different from Below _____			
(xxx) xxx-xxxx FAX:	☐ 2 weeks			SDG No:
Project Name:	☐ 1 week			
Site:	☐ 2 days			
P O #	☐ 1 day			

Sample Identification	Sample Date	Sample Time	Sample Type	Matrix	# of Cont.	Filtered Sample																Sample Specific Notes:

Preservation Used: 1= Ice, 2= HCl; 3= H2SO4; 4=HNO3; 5=NaOH; 6= Other _____

Possible Hazard Identification
☐ *Non-Hazard* ☐ *Flammable* ☐ *Skin Irritant* *Poison B* ☐ *Unknown* ☐

Sample Disposal (A fee may be assessed if samples are retained longer than 1 month)
☐ *Return To Client* ☐ *Disposal By Lab* ☐ *Archive For* _____ *Months*

Special Instructions/QC Requirements & Comments:

Relinquished by:	Company:	Date/Time:	Received by:	Company:	Date/Time:
Relinquished by:	Company:	Date/Time:	Received by:	Company:	Date/Time:
Relinquished by:	Company:	Date/Time:	Received by:	Company:	Date/Time:

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.
   - 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. See Lab Sampling Guide.

4) Samples must be preserved according to the requirements of the requested analytical method (See Sampling Guide**.**

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 $\leq$ 10 ^{o}C), the samples must arrive within $\pm$ 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 (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 (Monday unless a holiday). Samples will remain refrigerated and sealed until the time of analysis. The actual times of all "field" sample analyses are noted on the "Short Hold Time Detail Report" 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 and do not seal very well and are prone to intrusion from the water from melted ice.
- Water samples would be best if wrapped with bubble-wrap or paper (newspaper, or paper towels work) and then placed in plastic zip-lock bags.
- Fill extra cooler space with bubble wrap.

Figure 23-3. Example: Cooler Receipt Form

Login Sample Receipt Checklist

Client: Client X

Job Number: 580-43036-1

Login Number: 43036
List Number: 1
Creator: Blankinship, Tom X

List Source: TestAmerica Seattle

Question	Answer	Comment
Radioactivity wasn't checked or is </= background as measured by a survey meter.	True	
The cooler's custody seal, if present, is intact.	True	
Sample custody seals, if present, are intact.	True	
The cooler or samples do not appear to have been compromised or tampered with.	True	
Samples were received on ice.	True	
Cooler Temperature is acceptable.	True	
Cooler Temperature is recorded.	True	
COC is present.	True	
COC is filled out in ink and legible.	True	
COC is filled out with all pertinent information.	True	
Is the Field Sampler's name present on COC?	True	
There are no discrepancies between the containers received and the COC.	True	
Samples are received within Holding Time.	True	
Sample containers have legible labels.	True	
Containers are not broken or leaking.	True	
Sample collection date/times are provided.	True	
Appropriate sample containers are used.	True	
Sample bottles are completely filled.	True	
Sample Preservation Verified.	True	
There is sufficient vol. for all requested analyses, incl. any requested MS/MSDs	True	
Containers requiring zero headspace have no headspace or bubble is <6mm (1/4").	N/A	
Multiphasic samples are not present.	True	
Samples do not require splitting or compositing.	True	
Residual Chlorine Checked.	N/A	

TestAmerica Seattle

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. Quality control samples are to be treated in the exact same manner as the associated field samples being tested. 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;
	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 185%.

- 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. See SOP TA-QA-0600 Quality Control Charting and Establishing Warning and Action Limits.

One example: The QA department generates a Quality Control Limit Summary that contains tables that summarize the precision and accuracy acceptability limits for analyses performed at TestAmerica Seattle. This summary includes an effective date, is updated each time new limits are generated and is located in the LIMS**.** Unless otherwise noted, limits within these tables are laboratory generated. The analysts are instructed to use the current limits in the laboratory (dated and approved by the Technical Director and QA Manager) and entered into the Laboratory Information Management System (LIMS). The Quality Assurance department maintains an archive of all limits used within the laboratory.

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. If agredd to by the client, 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.

Or, for NELAC and Department Of Defense (DOD) work, there are an allowable number of Marginal Exceedances (ME):

<11 analytes	0 marginal exceedances are allowed.
11 – 30 Analytes	1 marginal exceedance is allowed
31-50 Analytes	2 marginal exceedances are allowed
51-70 Analytes	3 marginal exceedances are allowed
71-90 Analytes	4 marginal exceedances are allowed
> 90 Analytes	5 marginal exceedances are allowed

- Marginal exceedances are recovery exceedances between 3 SD and 4 SD from the mean recovery limit (TNI).

- Marginal exceedances must be random. If the same analyte exceeds the LCS control limit repeatedly, it is an indication of a systematic problem. The source of the error must be located and corrective action taken. The laboratory has a system to monitor marginal exceedances to ensure that they are random.

Though marginal exceedences may be allowed, the data must still be qualified to indicate it is outside of the normal limits.

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 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.

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. job 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).

- Any COCs involved with Subcontracting are included.
- In most cases, the applicable COC is paginated as an integral part of the report.
- Any additional addenda to the report are also paginated as an integral part of the report (eg. Sample Receiving Checklist).

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 (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 statement that the report shall not be reproduced except in full, without prior express written approval by the laboratory coordinator.

25.2.19 A signature and title of the person(s) accepting responsibility for the content of the report and date of issue. Signatories are appointed by the Lab Director.

25.2.20 When NELAC 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.

25.2.21 Where applicable, a narrative to the report that explains the issue(s) and corrective action(s) taken in the event that a specific accreditation or certification requirement was not met.

25.2.22 When soil samples are analyzed, a specific identification as to whether soils are reported on a “wet weight” or “dry weight” basis.

25.2.23 Appropriate laboratory certification number for the state of origin of the sample, if applicable.

25.2.24 If only part of the report is provided to the client (client requests some results before all of it is complete), it must be clearly indicated on the report (e.g., preliminary). A complete report must be sent once all of the work has been completed.

25.2.25 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.

25.2.26 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 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 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.
- Level III contains all the information supplied in Level II, but presented on the CLP-like summary forms, and relevant calibration information. A Level II report is not included, unless specifically requested. No raw data is provided.
- Level IV is the same as Level III with the addition of all raw supporting data.

In addition to the various levels of QC packaging, the laboratory also provides reports in 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 Seattle offers a variety of EDD formats including Environmental Restoration Information Management System (ERPIMS), Staged Electronic Data Deliverable (SEDD) Environmental Quality Information System (EQuIS), Electronic Deliverable Format (EDF), Excel and custom 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 report is issued solely for the use of the person or company to whom it is addressed. Any use, copying or disclosure other than by the intended recipient is unauthorized. If you have received this report in error, please notify the sender immediately at 253-922-2310 and destroy this report immediately.

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 job number followed by the appropriate revision number. 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 "Revision" with the appropriate number 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

Key Areas *

1. Admin/HR
2. QA Offices
3. Project Management
4. Sample Control
5. Metals & Mercury Lab
6. Wet / General Chem
7. Metals Prep
8. Organics Prep
9. GC-MS SVOA
10. GC SVOA
11. GC & GC-MS VOA
12. Sample Disposal
13. GeoTech, Storage, Warehouse
14. Conference Room

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 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 ~~is~~ 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
LOQV - Limit of Quantitation Check Standard
MDL – Method Detection Limit
MDLV – MDL Verification Check Standard
MRL – Method Reporting Limit
MS – Matrix Spike
MSD – Matrix Spike Duplicate
MSDS - Material Safety Data Sheet
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 Seattle 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:

Organization	Lab ID Number
DoD ELAP	L2236
ISO 17025	L2236
Alaska	UST-022
California (NELAP)	01115CA
Montana	(UST – no number)
Oregon (NELAP)	WA100007
Washington	C553
USDA Soil Permit	P330-11-00222
USFWS Tissue Import Permit	LE192332-0

The certificates and parameter lists (which may differ) for each organization are available, upon request, from a laboratory representative or may be found on the corporate web site, the laboratory's public server, the final report review table, and in the following offices: QA, marketing, and project management.

Appendix 4: Summary of Calibration, QC and Corrective Action Procedures for GC Organics

Method	QC Check	Frequency	Acceptance Criteria[3]	Corrective Action[4]
SW8081A SW8082	Minimum five-point initial calibration for all target analytes[2]	Initial calibration prior to sample analysis. Perform instrument re-calibration once per year minimum.	Linear regression correlation coefficient $r \geq 0.990$, $r^2 \geq 0.990$. RSD of CF ≤ 20% **For DoD:** Linear regression correlation coefficient $r \geq 0.995$, $r^2 \geq 0.990$. RSD of CF ≤ 20%	Correct problem then repeat initial calibration
	Initial calibration verification (ICV) must be from a 2nd source	Once immediately following initial calibration	All target analytes within 15% of expected value. **For DoD:** All target analytes within 20% of expected value.	Correct problem then repeat initial calibration
	Continuing calibration verification (CCV)	Before sample analysis, after every 10 samples, and at the end of the analysis sequence	All analytes within 15% of expected value and within the RT Window. **For DoD:** All target analytes within 20% of expected value.	Correct problem then repeat initial CCV (re-calibrate if necessary) and re-analyze all samples since last successful CCV.
	Breakdown check (Endrin and DDT)[1]	Before sample analysis	Degradation ≤15% for either Endrin or DDT.	Inlet/column maintenance; repeat breakdown check and re-analyze all samples since last successful breakdown check.
	Method blank	One per analytical prep batch, not to exceed 20 samples in a batch.	No analytes detected ≥ ½ RL or MDL, whichever is greater[5]	Correct problem then re-prep[6] and analyze method blank and all samples processed with the contaminated blank
	LCS for all analytes, must be from a 2nd source.	One per prep batch, not to exceed 20 samples in a batch.	See Control Limits	Re-prep[6] and analyze the LCS and all samples in the affected analytical batch
	Surrogate(s)	Every sample, spike, standard, and method blank	See Control Limits	Check system, re-inject, re-extract[6]
	MS/MSD, must be from a 2nd source. Rotate Aroclors each quarter.	One per batch per matrix, if insufficient sample for MS/MSD, then a LCS/LCSD will be analyzed.	See Control Limits	None (LCS is used to determine if data is acceptable).
	Second-column confirmation	100% for all positive results	Same as for initial or primary column analysis	Same as for initial or primary column analysis. If the relative % difference of results between the 2 columns is greater than 40%, a comment should be placed in LIMS.
	Retention time window calculated for each analyte (see section 9 for how to calculate RTWs).	System set-up, with each new column or major instrument maintenance. Update the mid-RTW at the start of the run or daily.	Each analyte of the LCS, MS/MSD and CCV must be within the calculated RTW.	Correct the problem and re-process or re-analyze samples. If questions, see the supervisor or Technical Manager.
	MDL verification	Minimum yearly **For DOD:** Minimum quarterly	Detectable	Re-evaluate MDL standard used and MDL; see Technical Manager.
	LOQ verification	**For DOD:** Minimum quarterly	Detectable	Re-evaluate LOQ standard used and LOQ; see Technical Manager.

1 --8081A only

Document Uncontrolled When Printed

2 - Method 8082, a five-point calibration is only analyzed for Aroclors 1016 and 1260.
3 - This is a summary of the acceptance criteria, refer to the method SOP for specific or more information.
4 - All abnormalities must be noted in an NCM.
5 - Report all target compounds identified in the method blank above the MDL.
6 - If unable to re-extract the samples because of insufficient sample volume or holding time has expired, then place a comment on the benchsheet and in LIMS.

Company Confidential & Proprietary

Appendix 4: Summary of Calibration, QC and Corrective Action Procedures for GC Organics

Method	QC Check	Frequency	Acceptance Criteria[3]	Corrective Action[4]
EPA608	Minimum three-point (preferably five) initial calibration for all target analytes	Initial calibration prior to sample analysis. Perform instrument re-calibration once per year minimum.	**RSD of CF ≤ 10%** Linear regression - correlation coefficient r ≥ 0.995, r^2 ≥ 0.990.	Correct problem then repeat initial calibration
	Initial calibration verification (ICV) must be from a 2nd source.	Immediately following initial calibration	All analytes within 15% of expected value	Correct problem then repeat initial calibration
	Continuing calibration verification (CCV)	Before sample analysis, and at the end of the analysis sequence	All analytes within **15% of expected value** and within the RTW. 608 (not 608.2[5]) must be within 15% of the true value.	Correct problem then repeat initial CCV (re-calibrate if necessary) and re-analyze all samples since last successful CCV.
	Breakdown check (Endrin and DDT)[1]	Before sample analysis	Degradation ≤15% for either Endrin or DDT.	Inlet/column maintenance; repeat breakdown check and re-analyze all samples since last successful breakdown check.
	Method blank	One per analytical prep batch, **not to exceed 10 samples in a batch**.	No analytes detected ≥ ½ RL or MDL, whichever is greater[6]	Correct problem then re-prep[7] and analyze method blank and all samples processed with the contaminated blank
	LCS (QC check standard) must be from a 2nd source.	One per prep batch, **not to exceed 10 samples in a batch.**	See Control Limits	Re-prep[7] and analyze the LCS and all samples in the affected analytical batch
	Surrogate(s)	Every sample, spiked sample, standard, and method blank	See Control Limits	Check system, re-inject, re-extract[7]
	MS must be from a 2nd source.	One per batch per matrix, 10%, if insufficient sample for MS, then an additional LCS will be analyzed.	See Control Limits	All target compounds should be reported, and any compounds that are outside criteria must be within criteria in the LCS.
	Second-column confirmation	100% for all positive results	Same as for initial or primary column analysis	Same as for initial or primary column analysis. If the relative % difference of results between the 2 columns is greater than 40%, a comment should be placed in LIMS.
	Retention time window calculated for each analyte (see section 9 for how to calculate RTWs).	System set-up, with each new column or major instrument maintenance. Update the mid-RTW at the start of the run or as needed.	Each analyte of the LCS, MS/MSD and CCV must be within the calculated RTW.	Correct the problem and re-process or re-analyze samples. If questions, see the supervisor or Technical Manager.
	MDL verification	Minimum yearly	Detectable	Re-evaluate MDL standard used and MDL; see Technical Manager.

3 - This is a summary of the acceptance criteria, refer to the method SOP for specific or more information.
4 - All abnormalities must be noted in an NCM.
5 – 608.2 compounds are Methoxychlor and Permethrin.
6 - Report all target compounds identified in the method blank above the MDL.
7 - If unable to re-extract the samples because of insufficient sample volume or holding time has expired, then place a comment on the benchsheet and in LIMS.

Appendix 4: Summary of Calibration, QC and Corrective Action Procedures for GC/MS Organics

Method	QC Check	Frequency	Acceptance Criteria[2]	Corrective Action[3]
SW8260B SW8270C SW8151mod	Check of mass spectral ion intensities[1], i.e., Tune	Prior to initial calibration or Continuing calibration verification, every 12 hours	Refer to criteria listed in the method SOP for Tune criteria, including DDT, Benzidine and Pentachlorophenol requirements for 8270.	Retune the instrument and verify (instrument maintenance may be needed).
SW8260B SW8270C	Minimum five-point initial calibration for all target analytes	Initial calibration prior to sample analysis. Perform instrument re-calibration once per year minimum.	VOCs: SPCCs average RF ≥ 0.30 or 0.1 depending on the compound and %RSD for RFs for CCCs ≤ 30% and all other target analytes %RSD for RF ≤ 15%.	Correct problem then repeat initial calibration
			SVOCs: SPCCs average RF ≥ 0.050 and %RSD for RFs for CCCs ≤ 30% and all other target analytes %RSD for RF ≤ 15%.	Correct problem then repeat initial calibration
			option (if %RSD is > 15%)–linear regression $r^2 \geq 0.99$, r ≥ 0.995.	If the calibration is not considered linear by either %RSD or linear regression, then correct the problem and re-calibrate.
	Initial calibration verification (ICV) must be from a 2nd source.	Immediately following five-point initial calibration	All CCCs within 20% of expected value. **For DoD:** All analytes within 20% of expected value. See limits in SOP for poor-performing and other non-standard HSL compounds.	Correct problem then repeat initial calibration
	Relative Retention time window	Each sample	Relative retention time (RRT) of the analyte within 0.06 RRT units of the RRT of the internal standard	Correct problem then reprocess or re-analyze all samples analyzed since the last retention time check
	Continuing calibration verification (CCV)	Daily, before sample analysis and every 12 hours of analysis time	VOCs: SPCCs average RF ≥ 0.30 or 0.1 depending on the compound; **For DoD:** SPCCs average RF ≥ 0.050; CCCs: ≤20% difference (when using RFs) or drift (when using least squares regression). See limits in SOP for poor-performing and other non-standard HSL compounds.	Correct problem then repeat initial calibration and re-analyze all samples since last successful CCV.
			SVOCs: SPCCs average RF ≥ 0.050; and	

Method	QC Check	Frequency	Acceptance Criteria[2]	Corrective Action[3]
	Continuing calibration check		CCCs: ≤20% difference (when using RFs) or drift (when using least squares regression) for standard list analytes, ≤35% for non-standard analytes, and ≤55% for poor performing analytes. **For DoD:** SPCCs average RF ≥ 0.050; CCCs: ≤20% difference (when using RFs) or drift (when using least squares regression). See limits in SOP for poor-performing and other non-standard HSL compounds.	
	Method blank	One per analytical prep batch	No analytes detected ≥ ½ RL or MDL, whichever is greater[4]	Correct problem then re-prep[5] and analyze method blank and all samples processed with the contaminated blank
	Internal Standards	Every sample/standard and blank	Retention time ±30 seconds from retention time of the mid-point std. in the CCV/ICAL (sample/standard). EICP area within -50% to +100% of ICAL mid-point std for the CCV and –50% to +100% of the prior CCV for the samples. **For DoD:** EICP area within -50% to +100% of ICAL mid-point std for the CCV and samples.	Inspect mass spectrometer and GC for malfunctions; mandatory re-analysis of samples analyzed while system was malfunctioning (dilution of the sample may be required, see the supervisor or the Technical Manager for advice).
	LCS for all analytes must be from a 2nd source.	One per prep batch, not to exceed the 20 samples in a batch.	See Control Limits Manual	Correct problem then re-prep[5] and analyze the LCS and all samples in the affected analytical batch
	MS/MSD must be from a 2nd source.	One per batch per matrix, if insufficient sample for MS/MSD, then a LCS/LCSD will be analyzed.	See Control Limits Manual	None (the LCS is used to evaluate to determine if the batch is acceptable).
	Surrogate(s)	Every sample, spike, standard, and blank	See Control Limits Manual	Check system, re-analyze, re-prep[5]
	MDL verification	Minimum yearly **For DOD:** Minimum quarterly	Detectable	Re-evaluate MDL standard used and MDL; see Technical Manager.
	LOQ verification	**For DOD:** Minimum quarterly	Detectable	Re-evaluate LOQ standard used and LOQ; see Technical Manager.
SW8260B	pH check	All 8260 water samples.	pH ≤2.	If the pH is > 2, then comment the data, in the PIPE database, and LIMS.
	Residual chlorine check (North Carolina samples only)	Each sample.	Residual chlorine should be negative.	If the residual chlorine is positive, then comment in the data and LIMS.

Document Uncontrolled When Printed

1 - SW8260B requires BFB; SW8270C requires DFTPP
2 - This is a summary of the acceptance criteria, refer to the method SOP for specific or more information.
3 - All abnormalities must be noted in an NCM.
4 - Report all target compounds identified in the method blank above the MDL.
5 - If unable to re-prep samples because of insufficient sample volume or the holding time has expired, then note in an NCM.

Company Confidential & Proprietary

Appendix 4: Summary of Calibration, QC and Corrective Action Procedures for GC/MS Organics

Method	QC Check	Frequency	Acceptance Criteria[2]	Corrective Action[3]
EPA624 EPA625	Check of mass spectral ion intensities[1] (i.e. Tune)	Prior to initial calibration or Continuing calibration verification every 12 hours.	Refer to criteria listed in the method SOP for Tune requirements including DDT, Benzidine and Pentachlorophenol criteria for 625.	Retune instrument and verify instrument maintenance may be needed.
	Minimum three-point initial calibration for all target analytes.	Initial calibration prior to sample analysis. Perform instrument re-calibration once per year minimum.	%RSD < 35%, if %RSD is > 35% then linear regression is used (for linear regression $r^2 \geq 0.99$), $r \geq 0.995$.	If the calibration is not considered linear by either %RSD or linear regression, then correct problem then repeat initial calibration
	Initial calibration verification (ICV), 20 ug/L, must be from a 2nd source. May be the same as the LCS.	Immediately following initial calibration	See Control Limits Manual.	Correct problem then repeat initial calibration
	Relative Retention time window	Each sample	Retention time (RT) of the analyte within 30 seconds of the RT (± 0.25 min. RTW is used) of the target.	Correct problem then reprocess or re-analyze all samples analyzed since the last retention time check
EPA625	Continuing calibration verification (CCV)	Daily, before sample analysis and every 12 hours of analysis time.	All calibration analytes within 20% of expected value	Correct problem then repeat initial calibration and re-analyze all samples since last successful CCV.
EPA624			See limits in SOP	
EPA624 EPA625	Method blank	One per prep batch (not to exceed 20 samples per batch).	No analytes detected ≥ ½ RL or MDL, whichever is greater[4]	Correct problem then re-prep[5] and analyze method blank and all samples processed with the contaminated blank
	LCS for all analytes, 20 ug/L, must be from a 2nd source. May be the same as the ICV.	One per prep batch (not to exceed 20 samples per batch) or daily.	See Control Limits Manual	Correct problem then re-prep[5] and analyze the LCS and all samples in the affected analytical batch
	MS must be from a 2nd source.	One per batch of 20 per matrix, if insufficient sample for MS, then a-duplicate LCS will be analyzed.	See Control Limits Manual	All target compounds should be reported, and any compound that is outside criteria must be within criteria in the LCS.
	Surrogate(s)	Every sample, spiked sample, standard, and method blank	See Control Limits Manual	Correct problem then re-prep[5] and analyze sample
EPA624 EPA625	Internal Standards	Every sample/standard	Retention time ±30 seconds from retention time of the mid-point std. in the CCV/ICAL (sample/standard). EICP area within -50% to +100% of ICAL mid-point std for the CCV and –50% to +100% of the prior CCV for the samples.	Inspect mass spectrometer and GC for malfunctions; mandatory re-analysis of samples analyzed while system was malfunctioning (dilution of the sample may be required, see the supervisor or the Technical Manager for advice).
EPA624	pH check	All 624 samples after analysis	pH should be ≤ 2.	If the pH is > 2, then comment the data, in the PIPE database, and LIMS.

Method	QC Check	Frequency	Acceptance Criteria[2]	Corrective Action[3]
EPA624	Residual chlorine check (North Carolina samples only)	All samples after analysis	Residual chlorine should be negative.	If the residual chlorine is positive, then comment the data and LIMS.
	MDL verification	Minimum yearly	Detectable	Re-evaluate MDL standard used and MDL; see Technical Manager.

1 - 624 requires BFB; 625 requires DFTPP
2 - This is summary of the acceptance criteria, refer to the method SOP for specific or more information.
3 - All abnormalities must be noted in an NCM.
4 - Report all target compounds identified in the method blank above the MDL.
5 - If unable to re-prep samples because of insufficient sample volume or holding time has expired, then place a comment on the benchsheet and in LIMS.

Appendix 4: Summary of Calibration, QC and Corrective Action Procedures for GC Organics

Method	QC Check	Frequency	Acceptance Criteria[1]	Corrective Action[2]
SW8015[5] SW8021	Five-point initial calibration for all target analytes	Initial calibration prior to sample analysis. Perform instrument re-calibration once per year minimum.	RSD of CF ≤ 20% Linear – least squares regression r^2 ≥ 0.99, r ≥ 0.995	Correct problem then repeat initial calibration
	Initial calibration verification (ICV), must be from a 2nd source.	Immediately following five-point initial calibration	All analytes within 15% of expected value	Correct problem then repeat initial calibration
	LCS for all analytes must be from a 2nd source.	One per prep batch, not to exceed 20 samples in a batch.	See Control Limits	Re-prep[4] and analyze the LCS and all samples in the affected analytical batch
	Continuing calibration verification (CCV)	Before sample analysis, after every 10 samples, and at the end of the analysis sequence	All analytes within 15% of expected value and within the RTW. **For DoD:** All analytes within 20% of expected value and within the RTW.	Correct problem then repeat initial CCV (re-calibrate if necessary) and re-analyze all samples since last successful CCV.
	Method blank	One per analytical prep batch, not to exceed 20 samples in a batch.	No analytes detected ≥ ½ RL or MDL, whichever is greater[3]	Correct problem then re-prep[4] and analyze method blank and all samples processed with the contaminated blank
	Surrogate	Every sample, spiked sample, standard, and method blank	See Control Limits	Check system, re-analyze, re-prep[4]
	MS/MSD must be from a 2nd source.	One per batch per matrix, if insufficient sample for MS/MSD, then a LCS/LCSD will be analyzed.	See Control Limits	None (LCS is used to determine if data is acceptable).
	GC/MS confirmation.	At the clients request or analyst judgment.		
	Retention time window calculated for each analyte (see section 9 for how to calculate RTWs).	System set-up, with each new column or major instrument maintenance. Update the mid-RTW as the start of the run or daily.	Each analyte of the LCS, MS/MSD and CCV must be within the calculated RTW.	Correct the problem and re-process or re-analyze samples. For questions, see the supervisor or Technical Manager.
	MDL verification	Minimum yearly **For DoD:** Minimum quarterly	Detectable	Re-evaluate MDL standard used and MDL; see Technical Manager.
	LOQ verification	**For DoD:** Minimum quarterly	Detectable	Re-evaluate LOQ standard used and LOQ; see Technical Manager.
SW8021	pH Check	All water samples after analysis.	pH should be less than 2.	If pH is > 2, then place a comment on the benchsheet and in LIMS.
MT-VPH MT-EPH WA-VPH WA-EPH NWTPH-EPH NWTPH-VPH	Five-point initial calibration for all target analytes	Initial calibration prior to sample analysis. Perform instrument re-calibration once per year minimum.	RSD of CF ≤ 20% Linear – least squares regression r^2 ≥ 0.99, r ≥ 0.995	Correct problem then repeat initial calibration
	Initial calibration verification (ICV) or Petroleum Performance Check Standard	Immediately following five-point initial calibration	Must be within 70-130% recovery.	Correct problem then repeat initial calibration
	Retention time window calculated for each analyte (see section 9 for how to calculate RTWs).	System set-up, with each new column or major instrument maintenance. Update the mid-RTW as the start of the run or daily.	Each analyte of the LCS, MS/MSD and CCV must be within the calculated RTW.	Correct the problem and re-process or re-analyze samples. For questions, see the supervisor or Technical Manager.

Method	QC Check	Frequency	Acceptance Criteria[1]	Corrective Action[2]
	Continuing calibration verification (CCV)	Before sample analysis, after every 10 samples, and at the end of the analysis sequence	All analytes within 20% of expected value and within the RTW.	Correct problem then repeat initial CCV (re-calibrate if necessary) and re-analyze all samples since last successful CCV.
	Method blank	One per analytical prep batch, not to exceed 20 samples in a batch.	No analytes detected ≥ RL	Correct problem then re-prep[4] and analyze method blank and all samples processed with the contaminated blank
	LCS for all analytes must be from a 2nd source.	One per prep batch, not to exceed 20 samples in a batch.	Must be within 70-130% recovery.	Re-prep[4] and analyze the LCS and all samples in the affected analytical batch
	MS/MSD must be from a 2nd source.	One per batch per matrix, if insufficient sample for MS/MSD, then a LCS/LCSD will be analyzed.	Must be within 50-150% recovery. **For WA-VPH and WA-EPH:** Must be within 70-130% recovery.	None (LCS is used to determine if data is acceptable).
	Duplicate	Each batch, less than 20	±25%	None
	Surrogate	Every sample, spiked sample, standard, and method blank	Low end aliphatic compounds must be within 30-150% recovery and high end aliphatic compounds must be within 50-150% recovery. **For WA-VPH and WA-EPH:** Must be within 60-140% recovery	Check system, re-analyze, re-prep[4]
	MDL and MDL verification	Minimum yearly	Detectable	Re-evaluate MDL standard used and MDL; see Technical Manager.
SW8011 EPA504.1	Five-point initial calibration for all target analytes (calibration standards should be prepped as the samples). Three point minimum for 504.1.	Initial calibration prior to sample analysis. Perform instrument re-calibration once per year minimum.	RSD of CF of each analyte ≤ 20% **RSD of CF < 10% for Method 8011** Linear – $r^2 \geq 0.990$, $r \geq 0.995$	Correct problem then repeat initial calibration
	Initial calibration verification (ICV) must be from a 2nd source.	Immediately following initial calibration	All analytes within 20% of expected value	Correct problem then repeat initial calibration
	Continuing calibration verification (CCV)	Before sample analysis, after every 10 samples, and at the end of the analysis sequence	All analytes within 10% of expected value and within the RTW.	Repeat once. If subsequent CCV fails, correct problem, re-calibrate and re-analyze all samples since last successful CCV.
	Continuing calibration blank (CCB)	After each CCV	No analytes ≥ ½ RL	Correct problem and re-analyze all samples since last successful CCB, unless contamination is <10X the conc present in the sample
	Method blank	One per analytical prep batch, not to exceed 10 samples in a batch.	No analytes ≥ ½ RL	Correct problem then re-prep[3] and analyze method blank and all samples processed with the contaminated blank
	LCS for all analytes must be from a 2nd source.	One per prep batch, not to exceed 10 samples in a batch.	All analytes within 30% of expected value.	Re-prep[3] and analyze the LCS and all samples in the affected analytical batch

Method	QC Check	Frequency	Acceptance Criteria[1]	Corrective Action[2]
	Surrogate	Every sample, spike, standard, and method blank	See Control Limits	Check system, re-inject, re-extract[3]
	MS/MSD must be from a 2nd source.	One per batch per matrix, if insufficient sample for MS/MSD, then a LCS/LCSD will be analyzed.	See Control Limits	(LCS is used to determine if data is acceptable). Add comments in LIMS.
	Second-column confirmation	100% for all positive results	Same as for initial or primary column analysis	Same as for initial or primary column analysis. If the relative % difference of results between the 2 columns is greater than 40%, a comment should be placed in LIMS.
	Retention time window calculated for each analyte (see section 20.1 for how to calculate RTW's).	System set-up, with each new column or major instrument maintenance. Update the mid-RTW as the start of the run or daily.	Each analyte of the LCS, MS/MSD and CCV must be within the calculated RTW.	Correct the problem and re-process or re-analyze samples. For questions, see the supervisor or Technical Manager.
	MDL / MDL verification	Minimum yearly **For DOD:** Minimum quarterly	MDL<Spike<10X MDL Detected	Re-evaluate MDL standard used and MDL; see Technical Manager.
	LOQ verification	**For DOD:** Minimum quarterly	Detectable	Re-evaluate LOQ standard used and LOQ; see Technical Manager.

1 - This is a summary of the acceptance criteria, refer to the method SOP for specific or more information.
2 - All abnormalities must be noted on the data and in an NCM.
3 - Report all target compounds identified in the method blank above the MDL.
4 - If unable to re-prep the samples because of insufficient sample volume or holding time has expired, then note in an NCM.
5 - For GRO and DRO, see state specific SOP/Method for acceptance criteria. If there is not a specific method for that state, then follow the acceptance criteria in this table.

Appendix 4: Summary of Calibration, QC and Corrective Action Procedures for Method SW6010

Method	QC Check	Frequency	Acceptance Criteria	Corrective Action
SW6010B	Initial calibration (minimum 1 standard and a blank)	Daily initial calibration prior to sample analysis.	N/A	N/A
	Second-source calibration verification (ICV)	Daily after initial calibration	All analytes within 10% of expected value	Correct problem then repeat initial calibration
	RL Standard (CRI)	Daily after initial calibration	All others: within 50% of expected value. **For DoD:** within 20% of expected value.	Re-analyzed RL Standard, correct problem no samples may be analyzed without a valid RL Standard.
	Calibration blank (CB)	After every continuing calibration verification	Must be <RL. **For DoD:** Must be < LOD	Correct problem then analyze calibration blank and previous 10 samples
	Continuing calibration verification (CCV)	Before sample analysis, after every 10 samples, and at the end of the analysis sequence	All analytes within 10% of expected value and RSD of replicate integrations <5%	Repeat calibration and re-analyze all samples since last successful calibration
	Method blank	One per prep batch	No analytes detected ≥ ½ RL or MDL, whichever is greater[1] **For DoD:** Must be < ½ RL	Correct problem then re-prep and analyze method blank and all samples processed with the contaminated blank
	Interference check solution (ICS)	At the beginning of an analytical run	ICS-A: Absolute value of concentration for all non-spiked analytes < LOD (unless they are a verified trace impurity form one of the spike analytes) ICS-AB: Within 20% of expected value	Terminate analysis; correct problem; re-analyze ICS; re-analyze all affected samples
	LCS	One per prep batch	See Control Limits	Correct problem then re-prep and analyze the LCS and all samples in the affected analytical batch
	MS/MSD	One per batch per matrix	See Control Limits	None
	Dilution test	Each new sample matrix	1:5 dilution must agree within 10% of the original determination	Perform post digestion spike addition
	Post digestion spike addition	When dilution test fails	Recovery within 25% of expected results	None
	MDL verification	Minimum yearly **For DOD:** Minimum quarterly	Detectable	Re-evaluate MDL standard used and MDL; see Technical Manager.
	LOQ verification	**For DOD:** Minimum quarterly	Detectable	Re-evaluate LOQ standard used and LOQ; see Technical Manager.

1 – Report all targets identified in the method blank above the MDL.

Appendix 4: Summary of Calibration, QC and Corrective Action Procedures for Method SW6020

Method	QC Check	Frequency	Acceptance Criteria	Corrective Action
SW6020	Initial calibration (minimum 1 standard and a blank)	Daily initial calibration prior to sample analysis.	If more than one calibration standard used, r ≥ 0.995	Correct problem then repeat initial calibration
	Second-source calibration verification (ICV)	Daily after initial calibration	All analytes within 10% of expected value	Correct problem then repeat initial calibration
	RL Standard (CRI)	Daily after initial calibration	All others: within 50% of expected value. **For DoD:** within 20% of expected value.	Re-analyzed RL Standard, correct problem no samples may be analyzed without a valid RL Standard.
	Calibration blank (CB)	After every continuing calibration verification	Must be <RL. **For DoD:** Must be < LOD	Correct problem then analyze calibration blank and previous 10 samples
	Continuing calibration verification (CCV)	Before sample analysis, after every 10 samples, and at the end of the analysis sequence	All analytes within 10% of expected value and RSD of replicate integrations <5%	Repeat calibration and re-analyze all samples since last successful calibration
	Linear Dynamic Range	Every 6 months	Within 10% of expected value	N/A
	Method blank	One per prep batch	No analytes detected ≥ RL or MDL, whichever is greater[1] **For DoD:** Must be < ½ RL	Correct problem then re-prep and analyze method blank and all samples processed with the contaminated blank
	Interference check solution (ICS)	At the beginning of an analytical run	ICS-A: Absolute value of concentration for all non-spiked analytes < LOD (unless they are a verified trace impurity form one of the spike analytes) ICS-AB: Within 20% of expected value	Terminate analysis; correct problem; re-analyze ICS; re-analyze all affected samples
	Internal standards (IS)	Every sample	IS intensity within 30-120% of intensity of the IS in the initial calibration	Perform corrective action as described in 6020.
	RL standard	Daily, after one point initial calibration	Within 20% of expected value	Correct problem then re-analyze samples.
	LCS	One per prep batch	See Control Limits	Correct problem then re-prep and analyze the LCS and all samples in the affected analytical batch
	MS/MSD	One per batch per matrix	See Control Limits	None
	Dilution test	Each new sample matrix	1:5 dilution must agree within 10% of the original determination	Perform post digestion spike addition
	Post digestion spike addition	When dilution test fails	Recovery within 25% of expected results	None.
	MDL verification	Minimum yearly **For DOD:** Minimum quarterly	Detectable	Re-evaluate MDL standard used and MDL; see Technical Manager.
	LOQ verification	**For DOD:** Minimum quarterly	Detectable	Re-evaluate LOQ standard used and LOQ; see Technical Manager.

1 – Report all targets identified in the method blank above the MDL.

Appendix 4: Summary of Calibration, QC and Corrective Action Procedures for Method SW7196

Method	QC Check	Frequency	Acceptance Criteria	Corrective Action
SW7196A	Initial calibration (minimum three standards and a blank)	Initial calibration prior to sample analysis.	$r^2 \geq 0.99$, $r \geq 0.995$ for linear regression	Correct problem then repeat initial calibration
	Second-source calibration verification (ICV)	Immediately following initial calibration	All analytes within 10% of expected value	Correct problem then repeat initial calibration
	Continuing calibration verification (CCV)	Beginning and after every 10 samples and at the end of the analysis sequence	All analytes within 20% of expected value	Correct problem then repeat initial calibration and re-analyze all samples since last successful calibration
	Verification check to ensure lack of reducing condition and/or interference	Once for every sample matrix analyzed	Spike recovery between 85-115%	If check indicates interference, dilute and re-analyze sample persistent interference indicates the need to use and alternate method
	Method blank	One per prep batch	No analytes detected ≥ ½ RL or MDL, whichever is greater[1]	Correct problem then re-prep and analyze method blank and all samples processed with the contaminated blank
	MS/MSD	One per 20 samples per matrix	See Control Limits Manual	none
	LCS	One per batch	See Control Limits Manual	Re-prep, re-analyze all affected samples.
	MDL verification	Minimum yearly	Detectable	Re-evaluate MDL standard used and MDL; see Technical Manager.

1 - Report all targets identified in the method blank above the MDL.

Appendix 4: Summary of Calibration, QC and Corrective Action Procedures for Method SW7470/SW7471

Method	QC Check	Frequency	Acceptance Criteria	Corrective Action
SW7470A SW7471A	Initial calibration (minimum 5 standards and a blank)	Daily initial calibration prior to sample analysis. Perform instrument re-calibration once per year minimum.	$r^2 \geq 0.99$, $r \geq 0.995$ for linear regression	Correct problem then repeat initial calibration. If calibration fails again, re-digest the entire digestion batch.
	Second-source calibration verification (ICV)	Immediately following initial daily calibration	Analytes within 10% of expected value	Correct problem then repeat initial calibration. If calibration fails again, re-digest the entire digestion batch.
	Calibration blank	Once per initial daily calibration	No analytes detected ≥ MDL	Correct problem then re-digest and re-analyze calibration and entire digestion batch
	Continuing calibration verification (CCV)	Before sample analysis, after every 10 samples, and at the end of the analysis sequence	Analytes within 20% of expected value	Correct problem then repeat all QC and samples since last successful calibration. If the CCV fails again upon reanalysis, reprep the entire digestion batch.
	Method blank	One per prep batch	No analytes detected ≥ RL **For DoD:** Must be < ½ RL	Correct problem then re-prep and analyze method blank, all samples, and QC processed with the contaminated blank
	LCS	One per prep batch	See Control Limits Manual	Correct problem then re-prep and analyze the LCS, all samples, and QC in the affected analytical batch
	MS/MSD	One per batch per matrix	See Control Limits Manual	None
	MDL verification	Minimum yearly **For DOD:** Minimum quarterly	Detectable	Re-evaluate MDL standard used and MDL; see Technical Manager.
	LOQ verification	**For DOD:** Minimum quarterly	Detectable	Re-evaluate LOQ standard used and LOQ; see Technical Manager.

1 - Report all targets identified in the method blank above the MDL.

Appendix 4: Summary of Calibration, QC and Corrective Action Procedures for Method SW9010/SW9012/SW9014

Method	QC Check	Frequency	Acceptance Criteria	Corrective Action
SW9010 SW9012A SW9014	Initial calibration (six standards and a calibration blank)	Initial daily calibration prior to sample analysis. Perform instrument re-calibration once per year minimum.	$r^2 \geq 0.99$, $r \geq 0.995$ for linear regression	Correct problem then repeat initial calibration
	Distilled standards (one high and one low)	Once per calibration	Analyte within 10% of true value. **For DoD:** Analyte within 15% of true value.	Correct problem then repeat distilled standards
	Second-source calibration verification (ICV)	Immediately following initial daily calibration	Analyte within 15% of expected value.	Correct problem then repeat initial calibration
	Continuing calibration verification (CCV)	Beginning and after every 10 samples and at the end of the analysis sequence	Analyte within 15% of expected value	Correct problem then repeat initial Continuing calibration verification and re-analyze all samples since last successful Continuing calibration verification
	Calibration blank	Once per initial daily calibration	No analytes detected ≥ RL **For DoD:** Must be < ½ RL	Correct problem then re-analyze calibration blank and all samples associated with blank
	Method blank	One per prep batch	No analyte detected ≥ RL or MDL, whichever is greater[1] **For DoD:** Must be < ½ RL	Correct problem then re-prep and analyze method blank and all samples processed with the contaminated blank
	LCS	One per batch per matrix	See Control Limits in LIMS	Re-prep, re-run affected samples
	MS/MSD	One per batch per matrix	See Control Limits in LIMS	None
	MDL verification	Minimum quarterly **For DOD:** Minimum quarterly	Detectable	Re-evaluate MDL standard used and MDL; see Technical Manager.
	LOQ verification	**For DOD:** Minimum quarterly	Detectable	Re-evaluate LOQ standard used and LOQ; see Technical Manager.

Appendix 4: Summary of Calibration, QC and Corrective Action Procedures for Mercury

Method	QC Check	Frequency	Acceptance Criteria	Corrective Action
EPA245.1	Initial calibration (minimum 5 standards and a blank)	Daily initial calibration prior to sample analysis. Perform instrument re-calibration once per year minimum.	$r^2 \geq 0.99$, $r \geq 0.995$ for linear regression	Correct problem then repeat initial calibration
	Second-source calibration verification (ICV)	Immediately following five-point initial calibration	Analyte within 5% of expected value	Correct problem then repeat initial calibration
	Calibration blank	Once per initial daily calibration	No analytes detected ≥ MDL	Correct problem then re-analyze calibration blank and all samples associated with blank
	Continuing calibration verification (CCV)	Before sample analysis, after every 10 samples, and at the end of the analysis sequence	Analyte within 10% of true value	Correct problem then repeat calibration and re-analyze all samples and QC since last successful calibration
	LCS	One per prep batch	All analytes within 15% of expected value	Correct problem then re-prep and analyze the LCS, all samples, and QC in the affected analytical batch
	Matrix Spike/Matrix Spike Duplicate	One per batch or 10 samples	All analytes within 25% of expected value	None
	Method Blank	One per batch	No analytes > ½ RL	Reprep
	MDL verification	Minimum yearly	Detectable	Re-evaluate MDL standard used and MDL; see Technical Manager.

1 - Report all targets identified in the method blank above the MDL.

Appendix 4: Summary of Calibration, QC and Corrective Action Procedures for ICP Metals

Method	QC Check	Frequency	Acceptance Criteria	Corrective Action
EPA200.7	Initial calibration (minimum 1 standard and a blank)	Daily initial calibration prior to sample analysis.	N/A	N/A
	Second-source calibration verification (ICV/QCS)	Each calibration	Value of all analytes within 5% of expected value	Correct problem then repeat initial calibration
	Linear Dynamic Range	Once annually	All analytes within 10% of expected value	Calibration range lowered to meet LDR results
	Calibration blank	After every Continuing calibration verification	No analytes detected (< IDL or MDL, whichever is greater)	Correct problem then analyze calibration blank and previous 10 samples
	Continuing calibration verification (CCV/IPC)	Before sample analysis, after every 10 samples, and at the end of the analysis sequence	All analytes within 10%	Repeat calibration and re-analyze all samples since last successful calibration
	Method blank	One per prep batch	No analytes detected, < 10% of sample conc OR < 2.2 x MDL[1]	Correct problem then re-prep and analyze method blank and all samples processed with the contaminated blank
	Interference check solution (ICSA/ACSAB)	At the beginning of an analytical run, daily	Monitor for interferences.	Terminate analysis; correct problem; re-analyze ICS; re-analyze all affected samples
	LCS/LFB	One per prep batch	All analytes within 15% of expected value	Correct problem then re-prep and analyze the LCS and all samples in the affected analytical batch
	Dilution test	Each new sample matrix	1:5 dilution must agree within 10% of the original determination for concentrations > 50 X IDL	Perform post digestion spike addition
	Post digestion spike addition	When dilution test fails	Recovery within 15% of expected results	Correct problem then re-analyze post digestion spike addition
	Matrix Spike/Matrix Spike Duplicate	One per batch of 10 samples	All analytes within 30% of expected value	None
	MDL verification	Minimum yearly	Detectable	Re-evaluate MDL standard used and MDL; see Technical Manager.

1 - Report all targets identified in the method blank above the MDL.

Appendix 4: Summary of Calibration, QC and Corrective Action Procedures for ICP-MS Metals

Method	QC Check	Frequency	Acceptance Criteria	Corrective Action
EPA200.8	Initial calibration (minimum 1 standard and a blank)	Daily initial calibration prior to sample analysis.	N/A	N/A
	Second-source calibration verification (ICV/QCS)	Each calibration	Value of all analytes within 10% of expected value	Correct problem then repeat initial calibration
	Calibration blank (ICB)	After every Continuing calibration verification	No analytes detected (< RL)	Correct problem then analyze calibration blank and previous 10 samples
	Continuing calibration verification (CCV)	Before sample analysis, after every 10 samples, and at the end of the analysis sequence	All analytes within 10%	Repeat calibration and re-analyze all samples since last successful calibration
	Method Blank/Laboratory Reagent Blank	One per prep batch	No analytes detected, < 10% of sample conc. OR < 2.2xMDL[1]	Correct problem then re-prep and analyze method blank and all samples processed with the contaminated blank
	Interference check solution (ICSA/ICSAB)	At the beginning of an analytical run, daily	Monitor for possible interferences.	Terminate analysis; correct problem; re-analyze ICS; re-analyze all affected samples
	LCS/LFB	One per prep batch	All analytes within 15% of expected value	Correct problem then re-prep and analyze the LCS and all samples in the affected analytical batch
	Internal Standard	Every sample	IS intensity within 60-125% of intensity of the IS in the initial calibration	Perform corrective action as described in 200.8.
	Matrix Spike/Matrix Spike Duplicate (MS/MSD)	One per batch of 10 samples	All analytes within 30% of expected value	None
	MDL verification	Minimum yearly	Detectable	Re-evaluate MDL standard used and MDL; see Technical Manager.

1 - Report all targets identified in the method blank above the MDL.

Document Uncontrolled When Printed

Appendix 4: Summary of Calibration, QC and Corrective Action Procedures for Gravimetric Analyses

Method	QC Check	Frequency	Acceptance Criteria	Corrective Action
EPA160.1 (TDS) SM2540 C (TDS) EPA160.2 (TSS) SM2540D (TSS) EPA160.3 (TS) SM2540B (TS) EPA160.4 (TVS)	Verification standard– single standard (if available)	Each batch	±10%	Repeat
	Method blank	Each batch	No analytes detected ≥ RL	Repeat, rerun
	Duplicate	Each batch, less than 20	±20%	None

Appendix 4: Summary of Calibration, QC and Corrective Action Procedures for Titrimetric Analyses

Method	QC Check	Frequency	Acceptance Criteria	Corrective Action
EPA310.1: Alkalinity. SM2320: HCO_3^-, CO_3^{-2}.	Verification standard– single standard (if available)	Each batch	±10%	Repeat, check
	Method blank	Each batch	No analyte detected ≥ RL.	Repeat batch
	Duplicate	Each batch	±20%	None

Appendix 4: Summary of Calibration, QC and Corrective Action Procedures for Electrometric Analyses

Method	QC Check	Frequency	Acceptance Criteria	Corrective Action
EPA405.1: BOD[1], CBOD[1]. EPA120.1: Cond[1]. SM2510B: Cond[1]. EPA150.1: pH. SW9040B, 9045C:pH. EPA180.1: Turbidity.	Calibration Curve – minimum of 5 standards	Initial Calibration. Perform re-calibration once per year minimum	±10%, $r^2 \geq 0.99$, $r \geq 0.995$.	Recalibrate
	Independent calibration verification (second source) (ICV)	Immediately after initial calibration	±10%	Recalibrate
	Continuing calibration verification (CCV)	Beginning, every 10 samples, and end of batch	±10%	Rerun
	Method blank	Each batch	No analyte detected ≥ ½ report limit or MDL, whichever is greater[2]	Reprep
	LCS	Each batch	±10%	Rerun batch
	MS/MSD	Each batch	± 20%	None
	Duplicate	When spike not available	±20%	None
	MDL verification	Minimum yearly	Detectable	Re-evaluate MDL standard used and MDL; see Technical Manager.

1 - Calibration curve does not apply.

2 - Report all targets identified in the method blank above the MDL.

Appendix 4: Summary of Calibration, QC and Corrective Action Procedures for Spectrophotometric Analyses

Method	QC Check	Frequency	Acceptance Criteria	Corrective Action
EPA350.1: NH_3. EPA410.4: COD. EPA335.4: CN. EPA353.2 NO_2/NO_3 EPA365.1 SM4500-P E T. Phos. EPA415.1: TOC. SM4500-CN^-I: WAD CN EPA9060: TOC. SM4500-Cr D Hexchrome SM4500-N_{org} C	Calibration curve – minimum 5 point	Initial. Perform re-calibration once per year minimum.	RSD <10%, $r^2 \geq 0.99$, $r \geq 0.995$	Recalibrate
	Independent calibration verification – mid-level, second-source required (ICV)	Immediately following initial calibration.	See Control Limits in LIMS	Recalibrate
	Continuing calibration verification (CCV)	Beginning, every 10 samples, and at end of sequence	See Control Limits in LIMS	Correct, recalibrate
	Method blank	Each use	No analyte detected ≥ ½ report limit or MDL, whichever is greater[1]	Reprep, rerun
	MS/MSD	Each batch, less than 20	See Control Limits in LIMS	None
	LCS	Each batch	See Control Limits in LIMS	Rerun
	MDL verification	Minimum yearly	Detectable	Re-evaluate MDL standard used and MDL; see Technical Manager.

Appendix 4: Summary of Calibration, QC and Corrective Action Procedures for Ion Chromatographic Analyses

Method	QC Check	Frequency	Acceptance Criteria	Corrective Action
EPA300: SW9056A: Bromide Chloride Fluoride Nitrate Nitrite Sulfate.	Calibration Curve – Minimum 5-point calibration	Initial calibration. Perform instrument re-calibration once per year minimum.	RSD ± 10%, $r^2 \geq 0.99$, $r \geq 0.995$.	Recalibrate
	Calibration verification (ICV), second source	Immediately following initial calibration	±10%	Recalibrate
	Continuing calibration verification (CCV)	Each use, beginning, every 10 samples, end of batch	± 10%	Rerun affected samples
	Method blank	Each batch	No analyte detected ≥ ½ report limit or MDL, whichever is greater[3]	Rerun batch
	LCS	Each batch	±10%	Rerun batch
	MS/MSD[1]	Each batch	±20%	None, use LCS
	Duplicate[2]	Each batch	±30%	None
	MDL verification	Minimum yearly **For DOD:** Minimum quarterly	Detectable	Re-evaluate MDL standard used and MDL; see Technical Manager.
	LOQ verification	**For DOD:** Minimum quarterly	Detectable	Re-evaluate LOQ standard used and LOQ; see Technical Manager.

1 - Only applies to EPA300, SW9056.
2 - Oil only.
3 - Report all targets identified in the method blank above the MDL.

Appendix 4: Summary of Calibration, QC and Corrective Action Procedures for Physical Analyses

Method	QC Check	Frequency	Acceptance Criteria	Corrective Action
SW1020A: Flash Point. EPA160.5: Settleable Solids.	Method blank	Each batch (if applicable to the method)	No analyte detected ≥ ½ report limit or MDL, whichever is greater[1]	Repeat, rerun
	Two standards for Flash Point One standard for BTU 1 Known for Settleable Solids	Each batch	See Control Limits in LIMS	Rerun batch
	Duplicate	Each batch	See Control Limits in LIMS	None

1 - Report all targets identified in the method blank above the MDL

Document Uncontrolled When Printed

Appendix 4: Summary of Calibration, QC and Corrective Action Procedures for Oil & Grease Analyses

Method	QC Check	Frequency	Acceptance Criteria	Corrective Action
EPA1664A	Verification standard	Single standard	±10% PAR standard	Rerun
	Method blank	Each batch	No analyte detected ≥ report limit	Repeat batch
	LCS	Each batch	See Control Limits	Repeat batch
	Duplicate	Each batch	See Control Limits	
	MS/MSD	Each batch	See Control Limits	None, use LCS
	LOQ verification	**For DOD:** Minimum quarterly	Detectable	Re-evaluate LOQ standard used and LOQ; see Technical Manager.

Title: Semivolatile Petroleum Products Method for Soil and Water [Method NWTPH-Dx mod]

Approvals

Signatures on File

Colin McKean Date Semivolatile Organic Department Manager	Sandra Langway Date Health & Safety Manager / Coordinator
Terri Torres Date Quality Assurance Manager	Heather Prater Date Laboratory Director

Copyright Information:
This documentation has been prepared by TestAmerica Laboratories, Inc. and its affiliates ("TestAmerica"), solely for their own use and the use of their customers in evaluating their qualifications and capabilities in connection with a particular project. The user of this document agrees by its acceptance to return it to TestAmerica upon request and not to reproduce, copy, lend, or otherwise disclose its contents, directly or indirectly, and not to use it for any other purpose other than that for which it was specifically provided. The user also agrees that where consultants or other outside parties are involved in the evaluation process, access to these documents shall not be given to said parties unless those parties also specifically agree to these conditions.

THIS DOCUMENT CONTAINS VALUABLE CONFIDENTIAL AND PROPRIETARY INFORMATION. DISCLOSURE, USE OR REPRODUCTION OF THESE MATERIALS WITHOUT THE WRITTEN AUTHORIZATION OF TESTAMERICA IS STRICTLY PROHIBITED. THIS UNPUBLISHED WORK BY TESTAMERICA IS PROTECTED BY STATE AND FEDERAL LAW OF THE UNITED STATES. IF PUBLICATION OF THIS WORK SHOULD OCCUR THE FOLLOWING NOTICE SHALL APPLY:

©COPYRIGHT 2012 TESTAMERICA LABORATORIES, INC. ALL RIGHTS RESERVED.

Facility Distribution No. 001 **Distributed To:** Lab Intranet

Document Uncontrolled When Printed

1.0 Scope and Application

1.1 Analytes, Matrix(s), and Reporting Limits

1.1.1 This SOP delineates the procedure for the identification and quantitation of semivolatile petroleum products using the WA DOE NWTPH-Dx method. This method is applicable to both soils and waters. Waters may be collected in either 1-liter or 125-mL sample jars.

1.1.2 This SOP does not include the procedures for extracting soil and water samples. Refer to the following SOPs for sample extraction procedures.

TA-OP-0301	Liquid-Liquid Extraction by Separatory Funnel, SW846 3510C
TA-OP-0323	Continuous Liquid-Liquid Extraction, SW846 3520C
TA-OP-03*02*	Sonication Procedure, SW846 3550B
TA-OP-0367	*Microwave Extraction 3546*

1.1.3 Reporting Limits

1.1.3.1 Soil: 25 mg/kg (diesel) and 50 mg/kg (motor oil)

1.1.3.2 Water: 0.125 mg/L (diesel) and 0.25 mg/L (motor oil)

1.2 On occasion clients may request modifications to this SOP. These modifications are handled following the procedures outlined in Section 12.2.1 in the Quality Assurance Manual.

2.0 Summary of Method

This method is used to identify, by pattern matching ("fingerprinting"), and quantitate semivolatile petroleum products. These products include kerosene, jet fuels, diesel oils, fuel oils, lubricating oils, hydraulic fluids, mineral oils and insulating oils such as transformer oils. Soil samples are weighed, dried, surrogate is added and extracted with methylene chloride. Water samples (1-liter or 100-mL volume) are acidified, surrogate is added and extracted with methylene chloride. Extracts are then concentrated and an aliquot of sample is analyzed by GC-FID. The hydrocarbons are quantitated against diesel (nC10-nC24) and motor oil (>nC24-nC36) standards (default) used for calibration and identified by pattern matching to the calibration standard or appropriate library spectra.

3.0 Definitions

3.1 Diesel Range Organics (DRO): The sum of compounds producing chromatographic peaks, both resolved and unresolved, eluting between n-decane (C_{10}) and tetracosane (C_{24}).

3.2 Motor Oil (MO): The sum of the compounds producing chromatographic peaks, both resolved and unresolved, eluting between tetracosane (C_{24}) and n-hexatriacontane (C_{36}).

3.3 Jet Propellant-4 (JP-4): The range is determined by injecting a standard purchased from a vendor and choosing the retention times from the initial low point of the chromatographic peaks to the end of the resolved and unresolved peaks. The hydrocarbon range for this fuel is typically from Toluene to C_{12}; however, LIMS defines this range as n-octane through n-octadecane.

Document Uncontrolled When Printed

3.4 Jet Propellant-8 (JP-8): The range is determined by the same method as used for JP-4. The hydrocarbon range for this fuel is typically from Toluene to C_{12}; however, LIMS defines this range as n-octane through n-hexadecane.

3.5 Mineral Oil (Transformer Oil): The sum of the compounds producing chromatographic peaks, both resolved and unresolved, eluting between dodecane (C_{12}) and tetratriacontane (C_{34}).

3.6 Bunker C (Fuel Oil 6): The sum of the compounds producing chromatographic peaks, both resolved and unresolved, eluting between n-decane (C_{10}) and n-hexatriacontane (C_{36}); however, LIMS defines this range as n-dodecane (C_{12}) through n-octatriacontane (C_{38}).

4.0 Interferences

4.1 Solvents, reagents, glassware, and other equipment coming in contact with the extract may yield interferences.

4.2 Non-petroleum hydrocarbons (non-polar) will also be extracted using this procedure. Hydrocarbons eluting in the ranges described above for fuel hydrocarbons will be detected and reported as false positives. All semi-volatile results must be reported; atypical results should be qualified appropriately.

4.3 Phthalate esters are found in many materials commonly found in the laboratory. In particular, plastics should be avoided because phthalates are routinely used as plasticizers and are easily extracted from the plastic materials.

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

5.1.1 The gas chromatograph contains zones that have elevated temperatures. The analyst needs to be aware of the locations of those zones, and must cool them to room temperature prior to working on them.

5.1.2 There are areas of high voltage in both the gas chromatograph. Depending on the type of work involved, either turn the power to the instrument off, or disconnect it from its source of power.

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
Methylene Chloride	Carcinogen Irritant	25 ppm-TWA 125 ppm-STEL	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.
Sulfuric Acid	Corrosive Oxidizer Dehydrator Poison Carcinogen	1 Mg/M3-TWA	Inhalation produces damaging effects on the mucous membranes and upper respiratory tract. Symptoms may include irritation of the nose and throat, and labored breathing. Symptoms of redness, pain, and severe burn can occur. Contact can cause blurred vision, redness, pain and severe tissue burns. Can cause blindness.
Acetone	Flammable	1000 ppm-TWA	Inhalation of vapors irritates the respiratory tract. May cause coughing, dizziness, dullness, and headache.
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

- Gas Chromatograph, Hewlett Packard 6890 or equivalent with autosampler equipped with a capillary split/splitless injector and an FID.
- Chromatographic column types:

6.1.1 Phenomenex ZB-1: 30 meters x 0.25 mm ID x 0.10 um film thickness cut in half to give two (2) 15 meter columns for dual column capable instruments.

6.1.2 Phenomenex ZB-1: 15 meters x 0.25 mm ID x 0.10 um film thickness.

These are the primary types of columns currently in use. Equivalent versions/types from a different vendor may be substituted. Note that elution chromatography may vary based on the actual column in place.

6.1.3 Analytical Balance, accurate to at least 0.0001g

6.2 Software

6.2.1 Data acquisition system: Agilent's ChemStation, is used for data acquisition and storage on machine-readable media. Since no processing is done by ChemStation and since there are no audit trail functions associated with data acquisition, the audit trail feature for ChemStation may be either enabled or disabled. The other component, Chrom, is used for data processing such as the measurement of peak area or peak height. By design, the audit trail feature for Chrom is always enabled.

6.2.2 Data processing: Chrom version 1.2 or higher

6.2.3 TestAmerica LIMS (TALS), current version

6.3 Supplies

- Volumetric Flasks, 10 mL, 25 mL, 50 mL, 100 mL, 250 mL, Class A, ground glass stoppered
- Scintillation vials with Teflon-lined screw caps
- Glass standard vials with screw caps and Teflon-lined septum
- Autosampler vials, 1.5 mL, crimp top or equivalent
- 20 mL scintillation vials, with Teflon-lined lids or equivalent
- Centrifuge
- Pasteur pipette, disposable

7.0 **Reagents and Standards**

7.1 Document reagents/standards and reagent/standard preparation in TALS using the reagent module as described in SOP TA-QA-0619.

7.2 Methylene Chloride (CH_2Cl_2), analytical reagent grade or equivalent.

7.3 Sodium sulfate, anhydrous powder, reagent grade or equivalent.

Note: Sodium sulfate must be muffled at 400 ℃ for 4 hours prior to use to avoid phthalate contamination.

7.4 Sulfuric acid, concentrated, trace metals grade or equivalent.

7.5 Petroleum product standards, Accustandard FUEL-SET, 20 mg/mL in CH_2Cl_2 or equivalent.

7.6 Check the Balance logbook to determine if the daily calibration check has been completed. If it has not, the analyst must perform this check according to SOP TA-QA-0014.

7.7 Surrogate Stock Standard. Approximately 0.2 g of o-Terphenyl (99%, Aldrich) and 0.2g n-triacontane-d62 (both weighed to the nearest 0.0001 g), and 8 mL of 4-Bromofluorobenzene (EXT-4-BFB_S_0000X from O2Si, Catalog number 020135-08) are diluted to a final volume of 100 mL with an 80:20 mix of DCM:acetone, providing a stock spiking solution of approximately 2,000 mg/L.

Note: n-Triacontane tends to precipitate out of acetone; therefore, the vial of surrogate should be checked for this, and mixed on the vortex until all crystals are back in solution.

7.8 Reference/Stock Standards. Prepare individual petroleum product reference/stock standards; kerosene, JP-5, transformer oil, and bunker crude fuel oil.

7.8.1 Add 5 to 10 drops or the pure petroleum product to a tared 10 mL volumetric flask. Record the weight to the nearest 0.0001g and bring to volume in methylene chloride, stopper and mix by inverting several times. Calculate the concentration of the standard using the equation below.

7.8.1.1 Stock Conc., ug/ml = $\frac{\text{(final wt, mg)-(tare wt, mg)}}{\text{10 mL}} \times \frac{\text{1,000 ug}}{\text{mg}}$

7.8.1.2 These standards are also used to ensure the proper identification of petroleum products by chromatographic pattern matching.

7.8.2 The use of commercially available standards is an acceptable alternative to the above procedure.

7.9 Calibration Working Standards. Using the stock standards, prepare calibration working standards for the identified petroleum product(s) to be quantitated. Serially dilute the reference/stock standard(s) to prepare calibration curve(s). Calibration standards must, at

Document Uncontrolled When Printed

a minimum, provide a minimum 5 (five) point calibration curve, include a sufficiently low standard to provide necessary detection limits, and define a linear working range of the instrument. A mid-range calibration standard should also be prepared for calibration curve verification. The mid-range calibration standard concentration is varied within the instrument calibration range.

7.9.1 In order to be acceptable, each calibration curve must have a*n average %RSD value ≤ 15%, or a* linear correlation coefficient (r) of at least 0.990 and none of the standards may vary from their true value by more than ± 15%. #2 diesel oil and motor oil are the default petroleum product for reporting purposes.

7.10 Retention Time Window Standard. The retention time standard is prepared by appropriately diluting the EPH aliphatic stock standard (EPH AL calstk_0000X), and TPH surrogates (TPH_SURR_0000X) to a final concentration of 20 ug/mL each, with a final volume of 25 mL. The standard contains nC8-nC40 plus surrogates, sans nC39.

7.10.1 Establishing Retention Time Windows. The retention time window for each hydrocarbon range is established using the lower limit of the first eluting compound and the upper limit of the last eluting compound. The upper and lower limits are established by adding or subtracting 3σ from the absolute retention time of the appropriate compound. Alternatively a default standard deviation of 0.01 may be used for a retention time window of 0.03 minutes (EPA Method 8000C section 11.6)

7.11 Diesel and Motor Oil Spiking Solution. A 50,000 ug/mL #2 diesel fuel and motor oil composite standard is prepared and ordered as a custom standard from Restek, part number CS-13305, in a 5:1 DCM:acetone solvent mixture. This standard is logged into the LIMS system as “TPH Spike_RZ_0000X”.

7.12 Other spiking solutions may be prepared using different petroleum products by appropriately diluting a stock standard to a 5,000 ug/mL working solution. #2 diesel and motor oil will be the default spiked products if sample contaminant is unknown.

7.13 Managers/supervisors or a designee are expected to check their areas on a monthly basis for expired standards and dispose of them according to SOP TA-EHS-0036.

8.0 Sample Collection, Preservation, Shipment and Storage

8.1 Water samples may be collected in 1-liter or 125 mL amber glass bottles. Soil samples are typically collected in 4-oz. or 8-oz. glass jars. All sample containers must have Teflon-lined caps.

8.2 All samples shall be stored at 0-6℃ after collecti on. Water samples should be preserved with 1+1 HCl to a pH of ≤ 2.

8.3 Holding time, from the date of sampling to extraction, is 14 days for soil and 14 days for water. Holding time from extraction to analysis is 40 days.

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time	Reference
Water	Glass	1000 mLs	1:1 HCl, pH < 2; Cool 0-6°C	14 Days, Extraction 40 Days, Analysis	40 CFR Part 136.3
Soil	Glass	10 grams	Cool 0-6°C	14 Days, Extraction 40 Days, Analysis	N/A

Document Uncontrolled When Printed

9.0 Quality Control

9.1 The minimum quality controls (QC), acceptance criteria, and corrective actions are described in this section. When processing samples in the laboratory, use the LIMS QC program code and special instructions to determine specific QC requirements that apply.

9.1.1 The laboratory's standard QC requirements, the process of establishing control limits, and the use of control charts are described more completely in SOP TA-QA-0620, Quality Control Program.

9.1.2 Project-specific requirements can override the requirements presented in this section when there is a written agreement between the laboratory and the client, and the source of those requirements should be described in the project documents. Project-specific requirements are communicated to the analyst via special instructions in the LIMS.

9.1.3 Any QC result that fails to meet control criteria must be documented in a Nonconformance Memo (NCM). The NCM is approved by the supervisor and then automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group also receives NCMs by e-mail for tracking and trending purposes. The NCM process is described in more detail in SOP TA-QA-0610. This is in addition to the corrective actions described in the following sections.

9.2 Quality Control Batch

The batch is a set of up to 10 samples of the same matrix processed together using the same reagents and standards. Each quality control batch must contain a method blank (MB), a laboratory control sample (LCS), matrix spike (MS), and duplicate (DUP) pair. Matrix spike/matrix spike duplicate pairs are only performed for Tier 4 projects or by client request. For more details see SOP TA-QA-0620.

9.3 Method Blank (MB)

One method blank is analyzed with every preparation batch or every 20 samples, whichever is more frequent. The method blank consists of either 1 liter of organic-free water (for batches of aqueous samples) or 10 grams of Ottawa sand (for batches of soil samples). The method blank is processed exactly as samples in the batch, and is used to assess whether the laboratory processes have contaminated the samples in the batch.

Acceptance Criteria: Surrogate recoveries must fall within acceptance criteria and the results for the method blank must be less than or equal to the reporting limit concentration or less than 5% of the lowest concentration found in the associated samples. DOD and BP LaMP require MB to be ≤ ½ RL.

Corrective Action: If the method blank acceptance criteria are not met, identify and correct the source of contamination, and re-prepare and reanalyze the associated samples.

Document Uncontrolled When Printed

9.4 Laboratory Control Sample (LCS)

One LCS is analyzed with every preparation batch or every 20 samples, whichever is more frequent. The LCS (and LCSD as appropriate) consists of either 1 liter of organic-free water (for batches of aqueous samples) or 10 grams of Ottawa sand (for batches of soil samples), to which 100 µL of spike solution is added. See Table III for spike levels. The LCS is processed exactly as samples in the batch and is used to assess the accuracy of the analytical system. In the case where insufficient volume is provided for the extraction of an MS/MSD or duplicate sample, an LCSD will also be prepared.

Acceptance Criteria: The percent recovery of the analytes of interest must fall within the established control limits. For all other methods, the control limits are set at ± 3 standard deviations around the calculated mean of the historical LCS recovery data, unless project-specific control limits apply. Current control limits are stored in the laboratory LIMS. See SOP TA-QA-0620 for further details.

Corrective Action: If LCS acceptance limits are not met, the LCS should be reanalyzed once to confirm that the original analysis is reliable. If the results are still outside control limits, the associated samples must be re-extracted and reanalyzed. If the LCS recovery is <u>above</u> the upper control limit, and the associated samples are all <u>below</u> reportable concentrations, the deviation may be described in an NCM, if this is acceptable to the client or allowed by the specific program or project.

9.5 Matrix Spike and Matrix Spike Duplicate (MS/MSD)

When specifically requested, one matrix spike (MS) and one matrix spike duplicate (MSD) are prepared by spiking replicate portions of the selected field sample with the same spiking standard that is used for the LCS. Field blanks and equipment rinses may not be used to prepare the MS and MSD. The MS and MSD are processed exactly as samples in the batch, and are used to assess the effects of sample matrix on the accuracy and precision of the analytical system.

Acceptance Criteria: The percent recovery of the analytes of interest must fall within the established control limits. The control limits are set at ± 3 standard deviations around the calculated mean of the historical MS recovery data, unless project-specific control limits apply. Current control limits are stored in the laboratory LIMS. See Policy QA-0620 for further details.

The relative percent difference (RPD) between the MS and MSD must be less than the established control limit, which is based on 3 standard deviations of the mean of the historical data. RPD control limits are maintained in the laboratory LIMS.

Corrective Action: If the analyte recovery in the MS and/or the RPD between the MS and MSD fails acceptance criteria, but all other QC criteria are met, the MS/MSD failure may be attributed to matrix effects and the associated sample results may be reported as qualified. However, some programs (e.g., USACE) require reanalysis to confirm that presumed matrix effects are reproducible.

Document Uncontrolled When Printed

9.6 Duplicate Sample Analysis

A duplicate pair is required with each analytical batch. The RPD between samples should be ≤35%.

Corrective Action: If the RPD fails the acceptance criterion and the fuel pattern is inconsistent between chromatograms, the sample should be re-extracted and reanalyzed. If only the RPD value exceeds the acceptance criterion, the failure may be attributed to matrix effects or sample inhomogeneity and the associated sample results may be reported as qualified.

9.7 Surrogate Spikes

The o-terphenyl surrogate has chemistry similar to the analytes of interest, but *is* not expected to be found in environmental samples. 100-µL of the surrogate spike solution is added to each field and QC sample in the batch prior to sample extraction and all instrument blanks. See Table III for spike levels. Surrogate results are used to assess the performance of the analytical system for each field and QC sample and instrument blank.

Acceptance Criteria: The percent recovery of the surrogates must fall within 50-150% recovery.

Corrective Action: If surrogate recoveries are outside the established limits, verify calculations, dilutions, and standard solutions. Also verify that the instrument performance is acceptable. High recoveries may be due to co-eluting matrix interference and the chromatogram should be examined for evidence of this. Low recoveries may be due to adsorption by the sample matrix (e.g., clay particles, peat, or organic material in the sample). Recalculate the results and/or reanalyze the extract if the checks reveal a problem.

If the surrogate recovery is outside the established limits due to well-documented matrix effects, the results must be flagged and an explanation included in the report narrative. As with matrix spike failures, some programs (e.g., USACE) may require additional analyses to confirm suspected matrix interferences. The decision to reanalyze or flag the data should be made in consultation with the client. It is only necessary to re-prepare / reanalyze a sample once to demonstrate that a matrix effect is reproducible.

NOTE: For LaMP samples, if the surrogate percent recovery fails, the recovery must be confirmed by re-extraction and reanalysis with the following exceptions:

- The lab has unequivocally demonstrated a sample matrix effect and informed the LaMP client representative.
- The recovery exceeds control limits and all target analytes in the sample are non-detect.

Document Uncontrolled When Printed

9.8 RT Reference Standard

The retention time window is established by injecting a mixture of n-alkanes from Toluene to n-hexatriacontane (C_{36}) three times over a 72 hour period. The mean and standard deviation for the three retention times are calculated. The width of the RT window is set at ±3 times the standard deviations of the mean RT. If the resulting RT window is less than 0.03 minutes, then a window of 0.03 minutes is used.

Acceptance Criteria: Toluene must be resolved from the solvent peak.

Corrective Action: If the acceptance criteria are not met, check instrument conditions and calibration materials, correct as necessary and repeat analysis of the reference standard before proceeding with the analysis of samples.

9.9 **<u>Instrument QC</u>**

9.10 A PIBLK (an instrument blank with surrogate added) needs to be run after each CCV, unless the CCV is followed by a Method Blank. If the PIBLK is analyzed, it needs to be evaluated. The acceptance criterion for the PIBLK is the same as the method blank.

9.11 Initial Calibration (ICAL)

9.11.1 A new calibration curve must be generated initially, after major changes to the system, or when continuing calibration criteria cannot be met. Major changes include installation of new columns and changing FID jets.

9.11.2 The ICAL is performed using the concentration levels described in Table II. A total of four separate initial calibration curves (ICALs) is required to calibrate for all the mixtures. An ICAL must always be analyzed for the diesel fuel as these standards contain the surrogate compounds. ICALs for the other mixtures are analyzed as needed, depending upon the requested parameters. Samples may be calculated as one or more mixtures, dependent upon the project requirements. The lowest calibration concentration is equal to the laboratory reporting limit (RL) concentration. The highest standard defines the highest sample extract concentration that may be reported without dilution. It is not acceptable to remove points from a calibration curve for the purpose of meeting criteria.

9.11.3 The external standardization method is used. Tabulate the area response for each calibration level against the concentration injected. The ratio of the response to the concentration injected, defined as the calibration factor (CF), is calculated for the standard at each concentration as follows:

$$CF_i = \frac{A_{fuel}}{C_{fuel}}$$

Where:

CF_i	=	Calibration factor for the ith calibration level.
A_{fuel}	=	Total area of the fuel calibration standard peak.
C_{fuel}	=	Concentration of fuel calibration standard, mg/mL

9.11.4 If the percent relative standard deviation (%RSD) for the average (mean) of the calculated calibration factors is <u>less</u> than *15*%, the average calibration factor can be used for sample quantitation.

Document Uncontrolled When Printed

$$\text{Average Response Factor} = \overline{CF} = \frac{\sum_{i=1}^{n} CF_i}{n}$$

Where:

CF_i = Calibration factor for the ith calibration level.
n = The number of calibration levels.

9.11.5 If %RSD for the mean calibration factor is <u>greater</u> than *15*%, a linear least-squares regression may be used to fit the calibration data. The linear fit calculates the slope and intercept of a straight line that relates the concentration of each calibration standard to a chromatographic peak area, as follows:

$$A_s = mC_s + b$$

Where:

A_s = Area of the chromatographic peak for the target fuel.
m = Slope of the line as determined by the least-squares regression.
C_s = Concentration of the target fuel in the calibration standard, mg/mL.
b = Intercept of the line as determined by the least-squares regression.

9.11.6 The correlation coefficient of the fitted line must be ≥ 0.990. Note that some programs (e.g., AFCEE and USACE) require that the correlation coefficient is ≥ 0.995, unless approval is given in the project QAPP to use 0.990.

9.11.7 If the ICAL %RSD or correlation coefficient linearity criteria are not met, sample analysis cannot be performed using the calibration. Confirm that the instrument is performing properly, adjust as needed, and confirm that the standards are made correctly. After correcting the problem(s), prepare and reanalyze a new set of calibration standards.

9.11.8 See Corporate SOP CA-Q-S-005 for information on acceptable initial calibration models and associated algorithms.

9.12 Second-Source Initial Calibration Verification (ICV)

A second-source initial calibration verification (ICV) standard is prepared and analyzed immediately after each ICAL. This standard can also be used as the continuing calibration verification (CCV) standard. The response for this standard must be within ± 15% of the response predicted from the ICAL. The percent difference between the measured ICV calibration factor (or the measured concentration of the ICV standard) and the ICAL calibration factor (or the known concentration of the ICV standard) is calculated as follows:

$$\text{Percent Difference} = \frac{\text{R1} - \text{R2}}{\text{R1}} \times 100\%$$

Where:

R1 = Average calibration factor from the calibration curve or the ICV known value.

Document Uncontrolled When Printed

R2 = Calculated calibration factor for the ICV analysis or the measured ICV value.

If the percent difference for the second-source verification falls outside of ± 15%, then sample analysis cannot be performed. Reanalyze the second-source verification standard to confirm the original result. If the second result fails, then re-prepare the verification standard, and/or re-prepare and rerun the ICAL.

9.13 Continuing Calibration Verification (CCV)

9.13.1 A CCV standard is analyzed at the beginning of the analytical sequence, every 12 hours of operation, or every 10 samples (whichever is more frequent), and at the end of the analytical sequence. The response for this standard must be within ± 15% of the response predicted from the ICAL.

In the event of calibration verification failure, corrective action must be taken prior to sample analysis. If routine corrective action procedures fail to produce a second consecutive (immediate) calibration verification within acceptance criteria, then either the lab has to demonstrate acceptable performance after corrective action with two consecutive calibration verifications (using fresh calibration solutions, at low and high concentrations) or, alternatively, a new initial calibration must be established according to Section 10.2. If one of these calibration verification injections fails, a new initial calibration curve must be processed. If a verification standard is not acceptable, all samples analyzed after the last acceptable verification standard must be reanalyzed. Any samples associated with failed closing calibration verifications where the response for an analyte in the calibration verification standard is above the acceptance limit and the analyte was not detected in any of the samples analyzed since the previous passing verification, do not need to be reanalyzed as the verification standard has demonstrated that the analyte would have been detected were it present (see Note below for information relative to DOD samples). Re-analysis is required for all other situations. If for some reason (i.e., lack of sample) re-analysis can't take place, a NCM needs to be initiated and the sample and QC results associated with the failing CCV need to qualified in the final report. Sample results associated with a CCV failure that are uploaded into the LIMS need to be qualified at the analyte level as appropriate. Additional information related to the CCV failure or corrective actions taken should be summarized in a NCM.

NOTE: For DOD samples, a high biased CCV with non-detects in the samples is only acceptable to report if approval is granted by the client. Otherwise, samples associated with a high failing CCV need to be re-analyzed.

9.14 Any extra QC that is analyzed in a batch or sequence must be evaluated using the same criteria as the corresponding QC above.

10.0 Procedure

One-time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using an NCM. The NCM is approved by the supervisor and then automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group also receives NCMs by e-mail for tracking and trending purposes. The NCM process is described in more detail in SOP TA-QA-0610. The NCM shall be filed in the project file and addressed in the case narrative.

Document Uncontrolled When Printed

10.1.1 **Note:** It has been noted that some petroleum products, i.e. heavy oils such as #6 fuel oil or bunker crude may experience a concentration loss of between 10 and 20 percent when subjected to this cleanup technique. This loss appears to be primarily associated with the removal of petroleum compounds which contain sulfur. To account for this loss when analyzing samples that have subjected to the cleanup procedure in preparation for heavy fuel oil determination, the analyst must use standards that have undergone the cleanup technique to calibrate the GC.

10.2 **Calibration**

10.2.1 *Refer to Table 2 for on-instrument calibration levels.*

10.3 The gas chromatograph is set up as follows:

Injector: 300℃

Detector: 350℃

Oven ramping profile for 6890 GC systems with 15 meter column(s): Initial column temperature is set to ~~45~~*30*-60℃, and held for 0.5 minutes, ramped to 340℃ at 30℃/min and held for 2-3 minutes. Flow is set to constant flow at 1.5-3.5 mL/min. A post run is initialized at 340℃ at a flow rate of 5.0 mL/min, and held for 2-4 minutes to clean out the system of contaminants.

Oven ramping profile for 5890 GC systems with 15 meter column(s): The oven temperature program parameters for these systems are similar to that of the 6890 systems, however, the final holding time is increased to 4-6 minutes due to the instrument software not having a post run capability.

Note: the oven ramping profile will vary from instrument to instrument, as each does not perform exactly like one another. In addition, actual column lengths and types vary as well.

Note: each instrument's run method parameters are printed out and stored in each appropriate instrument maintenance logbook.

10.3.1 The FID is allowed to stabilize at manufacturers recommended makeup and carrier gas flows prior to analysis.

10.3.2 Standard and surrogate solutions are allowed to come to room temperature prior to use.

10.4 **Sample Analysis**

10.5 Prior to analysis of any samples or QC samples, the analyst must prepare and analyze a mid-range calibration check standard (CCV) to insure that the instrument is functioning correctly and that the calibration is still valid.

10.5.1 CCVs need to be followed by a blank (method blank or instrument blank). CCVs cannot be preceded by a blank, unless a blank is analyzed before each sample in the bracket.

10.5.2 Instrument response should be monitored daily by recording the absolute response of an individual peak. The surrogate o-Terphenyl is the peak chosen for these instruments. The absolute response for this peak in this standard needs to be documented in the maintenance log book on a daily basis.

Document Uncontrolled When Printed

10.6 The analyst shall use #2 diesel and motor oil as the default product for reporting purposes when no petroleum products were identified in any initial screening or when type(s) of petroleum products are unknown prior to analysis.

10.7 A portion of sample extract stored in an appropriately sized vial is transferred to 1.5 mL autosampler vials.

10.8 Extracts are analyzed by injection of 1 uL on the GC by an autosampler. **Note:** The use of GC/MS or GC/AED may be substituted for GC/FID as long as all other method parameters are met.

10.9 Samples which are expected to contain elevated levels of contamination may be followed by a solvent rinse blank to avoid possibility of carryover.

10.10 A mid-range calibration standard is analyzed every 12 hours of operation, or every 10 samples (whichever is more frequent), and at the end of the analytical sequence.

10.11 If NWTPH-HCID has not been previously performed on the samples and/or the type of petroleum present is unknown, the analyst may prescreen the samples to determine the petroleum product.

10.12 The observed petroleum product shall be determined by pattern matching with standard(s) analyzed the same day. Chromatograms used for this “fingerprinting” should be normalized to approximately 90% of full scale for the largest component of the particular petroleum product observed.

10.13 When reporting results, the analyst should adhere to the following:

10.13.1 If detection is due to a typical fuel pattern other than diesel or motor oil, the analyst must apply the “FUEL” qualifier to the appropriate range(s). The analyst must then follow-up with a “Pattern Recognition” NCM of the appropriate type, explaining the fuel pattern observed in each sample.

10.13.2 If detection is not due to a typical fuel pattern, such as a single peak, the analyst must apply the “NOFUEL” qualifier to the appropriate range(s). The analyst must then follow-up with an “Other – Observation” type NCM, explaining the particular detection.

10.14 For those surrogate compounds that elute within retention time ranges used for petroleum product integration, the analyst must subtract the area of the surrogate from the total area to yield a corrected area of the petroleum product.

10.15 At the discretion of the analyst, the range of components included in the integration may be adjusted in order to minimize the potential contribution of a co-eluting fractions arising from the presence of multiple petroleum products. Any change in the integration range must be reflected in the integration of the calibration standards.

10.16 For the default petroleum products, #2 diesel and motor oil, the calibration standard area of the components from decane (nC_{10}) and tetracosane (nC_{24}) (#2 diesel) and from tetracosane (nC_{24}) to hexatriacontane (nC_{36}) (motor oil) are integrated to the baseline as a group. The data system will automatically remove any surrogate areas that elute within the retention time windows. The samples are integrated in the same manner and the areas compared.

10.17 An example instrument analysis sequence is shown in Attachment 1.

10.18 Upon completion of the analytical sequence:

10.18.1 Review chromatograms via Chrom/Peak Review software and determine

Document Uncontrolled When Printed

whether manual data manipulations are necessary.

10.18.2 All manual integrations must be justified and documented. See Corporate SOP CA-Q-S-002 for requirements for manual integration.

10.18.3 Manual integrations, if necessary, are performed in the Chrom/Peak Review software module, utilizing the appropriate integration type and reason. Before and after chromatograms with appropriate user information are automatically generated in Chrom and is uploaded into LIMS with each sample.

10.18.4 Confirm that run logs have printed on them the instrument ID, the analyst and the method used. If this is not printed on the run logs, this must be entered by hand prior to completing the package.

10.19 Open the analysis batch in the TALS/LIMS Analyst Desktop II module, and allow the system to perform the sample calculations.

10.19.1 Perform a level 1 data review and document the review on the data review checklist (GC Data Review Checklist).

10.19.2 Submit the data folder containing the preparation batch data sheets, worklist information, and review checklist to the peer reviewer for the level 2 review. The data review process is explained in SOP TA-QA-0635.

10.19.3 Update the instrument sequence logbook.

10.20 GC Maintenance

10.20.1 Leak Checking. Leak checking after column installation is recommended. In order to avoid contamination when leak testing fittings and connections, direct a small jet of gas which can be detected by the detector, (for example methane for an FID) at the point to be tested, then use the detector at the maximum sensitivity to detect leakage of gas into the system. Response is rapid at points downstream of the column. Response time will be delayed by the elution time of the gas in the column. A quicker leak test can be performed by placing a drop of isopropanol on the point to be tested. If bubbles form, a leak is indicated. Alternatively, an electronic leak detector may be used.

10.20.2 Column Installation. Columns are replaced every six months or as needed. When a column will not hold its calibration for any length of time, replacement is needed. Poor peak shape and excessive baseline rise not attributed to sample contamination are other indication that the column may need replacement.

10.20.2.1 Remove the old column from the GC oven by loosening the injector and detector nuts with the appropriate wrench.

10.20.2.2 The *injector* end of the new column is installed first. Slide the column nut over the column end.

10.20.2.3 Install the appropriate ferrule onto the column. For the HP 6890/*7890* GCs, the tapered end is placed towards the end of the column.

10.20.2.4 Cut 1 to 2 cm from the end of the column.

10.20.2.5 Uncoil approximately 20 cm of column.

10.20.2.6 Move the column nut and ferrule within 12-15 cm of the end of the column.

10.20.2.7 Cut 1 to 2 cm. off of the end of the retention gap. Insert the column end

Document Uncontrolled When Printed

through the column nut, and then place a graphite ferrule onto the column and column nut. Using an old adaptable FID fitting, pre-crush the graphite ferrule to hold the column in place. For HP 6890/7890 GCs, measure *3* to *4* mm from the end of the column to the beginning of the ferrule.

10.20.2.8 Insert the retention gap into the injector while holding the column in place so it does not slide out of the ferrule. Tighten the column nut to finger tight. Make sure that the mark is visible and in the correct place for HP GCs. Tighten the column nut with a wrench approximately 1/4 turn or until the column cannot be pulled out of the injector.

10.20.2.9 For the detector end, insert the column end through the column nut, and slide a graphite ferrule over the column. Clip approximately 1 – 2 cm off the column end.

10.20.2.10 Partially insert the column into the lower end of the detector. For HP 6890/7890 GCs, the column is inserted 68 mm. CAUTION: The column can break or chip if it is forced into the detector!! Thread the column nut and ferrule until finger tight. For HP 6890/7890 GCs, pull the column back out of the detector approximately 1 mm. Tighten the ferrule and column nut with a wrench approximately 1/3 to 1/2 turn or until the column cannot be removed by pulling on it.

10.20.2.11 Install a ferrule onto the retention gap. For HP 6890/7890 GCs, the tapered end is placed towards the injector.

10.20.2.12 The gas flows are set to manufacturer or method recommended levels and are checked prior to each initial calibration:

Column flow: Approximately 1.5 -11 mL/min.
Make-up gas at the detector (Nitrogen): Column flow + X mL/min. = to 20 mL/min.
Hydrogen: 30 mL/min.
Air: 300 mL/min.
Total Flow: 360 mL/min.

10.20.2.13 Condition the column by heating the oven to 5-10 ℃ above its maximum operating temperature for approximately two hours, or until the baseline drops to its normal operating level. After the column is conditioned, the oven temperature is set at standby. At this point, the instrument is ready to be calibrated.

10.20.3 The injection port septa are replaced at least every 100-150 injections, or as needed under normal operating conditions.

10.20.4 The injection port be cleaned and the liner replaced monthly, or as needed. The frequency is determined by the number of samples analyzed and the amount of contamination introduced into the system. The loss of detector response, particularly in early eluting peaks is an indicator that the liner or retention gap needs replacement. To replace the injection port:

10.20.4.1 Cool the column and injection port to ambient temperature to avoid burns.

10.20.4.2 Unscrew the septum nut from the top of the injector. Remove the septum. Unscrew the nut below the septum plate.

Document Uncontrolled When Printed

10.20.4.3 Using tweezers or needle nose pliers, remove the liner and the viton O-ring. If the O-ring is not damaged, it may be used again. Insert a clean liner into the O-ring with the tapered end up. Insert the liner into the injection port. Tighten the lower nut onto the injection port.

10.20.4.4 Replace the septum with a new septum. Tighten the septum nut until finger tight. Puncture the septum with a syringe. This will also indicate if the septum nut is tight enough.

10.20.5 The oven is heated to maximum operating temperature for approximately one hour to remove any residue or contamination introduced into the system during maintenance.

10.21 FID Maintenance

10.21.1 Maintenance of the FID involves cleaning deposits from internal parts, including the flame tip, and ferrule replacement. These maintenance procedures are performed every six months, or more frequently if there is degradation in the FID performance. "Spiking" signals and carbon build-up are indications that the detector needs cleaning.

10.21.2 Refer to the GC Operator's Manual for proper disassembly and reassembly of the FID.

10.21.3 When removing the FID from the GC, inspect the O-ring at the base of the detector and replace as necessary.

10.21.4 Flame Tip and Internal Parts Cleaning.

10.21.4.1 Using Emery cloth, clean deposits from the bore of the collecting tube, the insulator, and the metal part of the flame tip.

10.21.4.2 If the flame tip is plugged, clear it by inserting a wire through the flame tip orifice.

10.21.4.3 Sonicate the detector parts in a methanol bath for ten to fifteen minutes.

10.21.4.4 Flush all components with methanol and dry in an oven at 100 ℃ for at least 15 minutes.

10.21.4.5 Reinstall the detector.

10.21.5 Ferrule Replacement.

10.21.5.1 If a leak develops around the base of the flame tip assembly, replace the ferrule.

10.21.5.2 Note: A leak is evident by detector noise, instability, and loss of sensitivity.

10.21.6 Autosampler Maintenance

10.21.6.1 Check the wash solvent levels daily. Fill if needed. Empty the waste bottles if necessary.

10.21.6.2 The syringe may need replacement. Follow the procedure outlined in the HP 7673 Automatic Sampler Manual (pp 3-30 through 3-32).

10.21.6.3 Periodically clean the surface of the tray arm, gripper, gripper jaws, and the tray quadrants.

Company Confidential & Proprietary

Document Uncontrolled When Printed

10.22 Spare Parts

10.22.1 Gas Chromatograph.

10.22.1.1 Septa, Merlin Microseal septum and column nut set

10.22.1.2 Injection Port Liners: HP 5890/6890 GCs: 4 mm ID straight

10.22.1.3 Column nut

10.22.1.4 Ferrules: 1/4 in. graphite *0.4* mm ID graphite

10.22.2 Autosampler

10.22.2.1 Syringes: HP7673: HP 10 uL 7673 Std. Plunger, or equivalent

10.22.2.2 Solvent and waste vials and septa

10.22.3 FID

10.22.3.1 FID jet

10.22.3.2 Column insulator

10.22.3.3 Collector tube

All maintenance and repairs need to be documented in the instrument's maintenance logbook. The logbook must include the instrument name, serial number for each major component (e.g., GC, autosampler, column) and the date of start-up. When an instrument is not capable of analyzing samples, it needs to be tagged "Out of Service". Logbook entries must include a description of the problem and what actions were taken to address the problem. After an instrument has undergone maintenance or repairs, the system is evaluated using a CCV or ICAL. If the evaluation is successful, the analyst documents in the logbook that the "System returned to control as indicated by a passing CCV" (or ICAL, MB, etc as may be the case).

If a column was replaced during maintenance procedures the specific make, model and serial numbers of the column installed needs to be entered in the instrument's maintenance logbook.

11.0 Calculations / Data Reduction

11.1 Accuracy

ICV / CCV, LCS % Recovery $= \frac{\text{observed concentration}}{\text{known concentration}} \times 100$

MS % Recovery $= \frac{\text{(spiked sample) - (unspiked sample)}}{\text{spiked concentration}} \times 100$

11.2 Precision (RPD)

Matrix Duplicate (MD) $= \frac{|\text{orig. sample value - dup. sample value}|}{[(\text{orig. sample value + dup. sample value})/2]} \times 100$

Document Uncontrolled When Printed

11.3 Concentration

$$\text{sample conc., mg/L} = \frac{(\text{area count} * \text{response factor}^{1}) * \text{extract volume}}{(\text{volume inj., uL}) * \text{sample volume, mL}}$$

$$\text{sample conc., mg/kg} = \frac{(\text{area count} * \text{response factor}^{1}) * \text{extract volume}}{(\text{volume inj., uL}) * (\text{sample weight, g}) * (\%\text{solids})}$$

1 = ng injected/area count

NOTE: All dry weight corrections are made in LIMS at the time the final report is prepared.

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 (see SOP TA-QA-0602). 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.

Instrumentation software must have each target limit set to the lowest MDL. CHROM (LOD)

12.2 Demonstration of Capabilities

Analyst initial and continuing Demonstrations of Capability (DOC) are performed before any client samples are analyzed and are updated annually. See SOP TA-QA-0617 for details.

12.3 Training Requirements

See SOP TA-QA-0608 for detailed training requirements.

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

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 SOP TA-EHS-0036.

14.1 Waste Streams Produced by the Method

14.1.1 Acidic extracted sample and QC wastewater. After the extraction has been completed the spent water is neutralized and then collected into the organics extraction water conical reservoir. The collected wastewater is then purged with

air to remove any remaining methylene chloride. When the concentration levels are at or below local discharge limits, the wastewater can be discarded down the drain.

14.1.2 Solvent/Methylene Chloride waste. Any waste methylene chloride/solvent is collected in beakers and then poured into a 4-liter amber bottle labeled "Hazardous Waste" located in the hood. After the extraction has been completed, the $MeCl_2$ collected in the 4 L bottles is emptied into the $MeCl_2$ satellite waste barrel located next to the neutralization tank in lab hood #17. The funnel lid on the drum must be closed after each use. At or before the satellite waste reaches 55 gallons, the barrel is transferred to the waste disposal room from where it is sent out for recycling or fuel blending.

14.1.3 Vialed extract waste. Sample extracts that have been placed in vials for analysis are discarded into satellite waste buckets labeled "Hazardous Waste" located underneath the bench top. Once the buckets are full, the GC vials are bulked into the non-PCB GC vial waste barrel located in the waste room and sent out for incineration.

14.1.4 Extract waste. Unused sample extracts are held for at least 40 days, in case further testing is deemed necessary. After at least 40 days has passed, these extracts are transported to the waste room in racks of 100 were they are bulked into a flammable loose pack waste stream and sent out for incineration.

14.1.5 Expired primary and working standards. Expired standards are stored in a canister labeled "Hazardous Waste" at or near the point of generation. At or before the satellite waste reaches 55 gallons, it is removed to the waste warehouse where it is bulked into the non-PCB GC vial waste barrel and sent out for incineration.

15.0 References / Cross-References

15.1 Analytical Methods for Petroleum Hydrocarbons, WA DOE, Toxics Cleanup Program and the Ecology Environmental Laboratory, Publication No. ECY 97-602, June 1997.

15.2 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition, Methods 3510C, 3550B, 3540C, 3580A, 8000C, and 8015B.

16.0 Method Modifications:

Item	Method	Modification
1	NWTPH-Dx	For petroleum products eluting after #2 diesel, e.g. motor oils, hydraulic fluids, and heavy fuel oils, the reporting limit is approximately 50 mg/kg for soil as opposed to 100 mg/kg
2	NWTPH-Dx	Final sample extract volume is 5.0 mL instead of 10 mL for waters analysis.
3	NWTPH-Dx	Silica gel SPE tubes are used instead of silica gel 100/200 mesh.
4	NWTPH-Dx	A full liter of water or 100 mL of water is extracted instead of 400 mL.
5	NWTPH-Dx	Both #2 diesel and motor oil are used as default petroleum products when product is unknown or not identified.
6	NWTPH-Dx	Soil samples may be prepared using a validated modification of method 3550B.

Document Uncontrolled When Printed

17.0 Tables and Attachments

Table I: Aliphatic Hydrocarbon Standard
Table II: Calibration Levels
Table III: Special Hydrocarbon Ranges
Table IV: Spike Levels for Quality Control
Table V: Recommended GC Conditions

Attachment 1: Example Instrument Sequence
Attachment 2: n-Alkane Retention Time Standard Chromatogram

18.0 Revision History

- Revision 13, dated 16 May 2013
  - Updated preparation SOP's in section 1.1.2.
  - Removed Isopropanol reagent from table in section 5.2.
  - Added average response factor criteria for calibrations in section 7.9.1.
  - Updated average response factor %RSD criteria in sections 9.11.4 and 9.11.5.
  - Added section 10.2.1 to refer to Table 2 for on-instrument calibration levels.
  - Updated Table 2 to include new calibration levels to accommodate LVI analysis.

- Revision 12, dated 6 August 2012
  - Updated preparation of stock surrogate standard in section 7.7.
  - Updated GC model number types and re-arranged column installation procedures outlined in sections 10.20.2.3 through 10.20.2.10.
  - Updated detector gas type in section 10.20.2.12.
  - Updated waste streams, section 14.1

- Revision 11, dated 31 May 2011
  - Added references to applicable extraction procedures in section 1.1.2
  - Added RLs in section 1.1.3.
  - Updated carbon ranges for several fuels types discussed in section 3.
  - Incorporated ROMDs 00019 and 00026 in section 6.1
  - Added software descriptions in Section 6.2
  - Added more detail on the preparation of the sodium sulfate in section 7.3.
  - Revised surrogate composition (7.7), RT standard (7.10) and Diesel/Motor Oil Spike (7.11).
  - Incorporated ROMD 00025 in section 9.4.
  - Incorporated ROMD 00022 in section 9.11.8.
  - Incorporated ROMD 00024 in section 9.13
  - Incorporated ROMD 00020 in section 10.3.
  - Incorporated ROMD 00033 in section 10.5.2.
  - Added Item #6 to Method Mods in section 16.
  - Updated information in Tables II and V.

- Revision 10, dated 26 March 2010
  - Added documentation of reagent/standards and reagent/standard preparation Section 7.1.
  - Added removal of expired standards Section 7.13.
  - Added BP requirement for surrogates, Section 9.7.
  - Added criteria for additional QC, Section 9.13
  - Added daily balance check to Section 10.2
  - Added maintenance documentation requirements to the end of section 10.22

Document Uncontrolled When Printed

- Revision 9, dated 26 April 2008
    - Integration for TestAmerica and STL operations.
    - This SOP is the combination of SOPs 0339.9 and 0387.6.

Table I: Aliphatic Hydrocarbon Standard

Compound Boiling Points		
n-Alkane	**Name**	**B.P.$_{760}$ (℃)**
C_{10}	Decane	174
C_{24}	Tetracosane	391
C_{32}	Dotriacontane	468
C_{36}	Hexatriacontane	498

This Table can be used to get the estimated boiling point ranges of the hydrocarbons reported in a given sample.

Table II: Calibration Levels

Standard	***Level 1***	**Level 2**	**Level 3**	**Level 4**	**Level 5**	**Level 6**	**Level 7**	***Level 8***
DRO	*10****	20	50	100	500**	1000	5000	*10000*
Jet Fuels 4 and 8	20	50	100	500**	1000	5000	*n/a*	*n/a*
Motor Oil / RRO	*10**	20	50	100	500**	1000	5000	*10000*
o-Terphenyl (surrogate)	*0.4*	0.8	2.0	4.0	20**	40	200	*400*

***** This level is frequently excluded as a calibration point as the RL for Motor Oil/RRO is 50.
****** Level used for CCV.
**** Level is required in calibration for analyzing LVI samples.*
(All concentrations are in mg/L.)

Table III: Special Hydrocarbon Ranges

Type	**Carbon Ranges**
Gasoline Range	Start of N-C8 through end of N-C12
Kerosene Range	Start of N-C8 through end of N-C20
Mineral Spirits Range	Start of N-C8 through end of N-C12
Mineral (Transformer) Oil Range	Start of N-C12 through end of N-C24
Hydraulic Oil Range	Start of N-C19 through end of N-C36
Heavy Fuel Oil Range	Start of N-C12 through end of N-C38

Document Uncontrolled When Printed

Table IV: Spike Levels for Quality Control

Laboratory Control Samples (LCS) and Matrix Spike/ Spike Duplicate		
Analyte	**Spike Concentration**	
	Water (mg/L)	**Soil (mg/kg)**
Diesel Range Organics	5.0	500
Jet Fuel 8	5.0	500
Jet Fuel 4	5.0	500
Residual Range Organics (or Motor Oil)	5.0	500

Surrogate Control Samples		
Analyte	**Spike Concentration**	
	Water (mg/L)	**Low Soil (mg/kg)**
o-Terphenyl	0.2	20

Table V: Recommended GC Conditions

Hydrogen/Nitrogen Flow Rate	2.5 mL/min
Initial Column Temperature	45 ºC for 0.5 minutes
Temperature Ramp	30 ºC / minute
Final Column Temperature	330 ºC
Injector Temperature	300ºC
FID Temperature	350ºC

Document Uncontrolled When Printed

Attachment 1: Example Instrument Sequence

Page 1

Sequence Log

Directory : e:\Data\042508_a

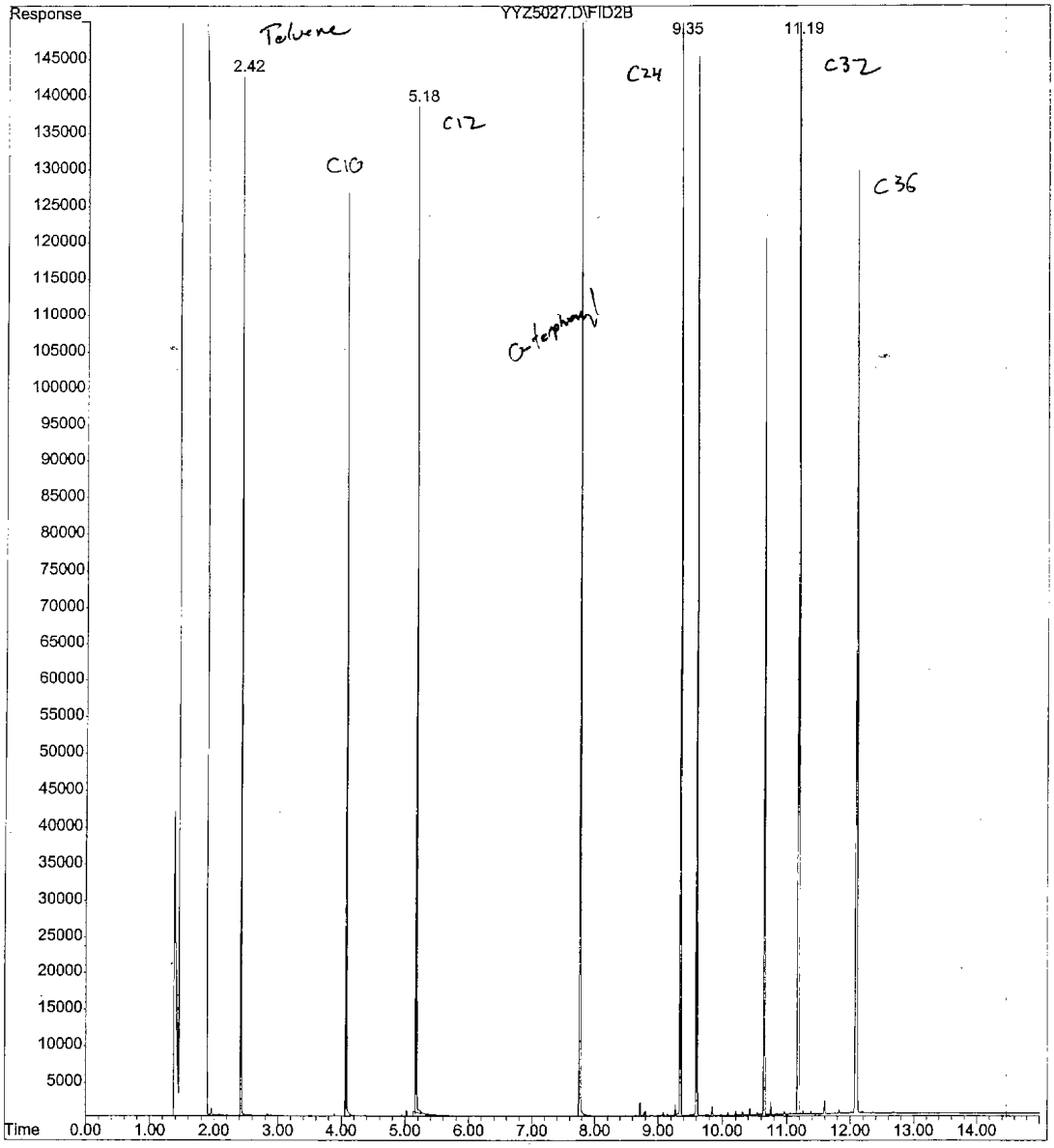
#	Filename	Sample Name	Date/Time
1	fa34544.d	rinse	04/25/08 07:12
2	fa34545.d	RS 176241 8015/NWDX n-alkane	04/25/08 07:31
3	fa34546.d	ccv 254376 1000 NWDX	04/25/08 07:51
4	fa34547.d	MB 580-30606/1-A	04/25/08 09:14
5	fa34548.d	LCS 580-30606/2-A	04/25/08 09:33
6	fa34549.d	580-8586-A-3-C	04/25/08 09:59
7	fa34550.d	580-8586-A-4-C	04/25/08 10:24
8	fa34551.d	580-9702-A-1-F	04/25/08 10:50
9	fa34552.d	580-9702-A-2-B	04/25/08 11:15
10	fa34553.d	580-9702-A-2-C DU	04/25/08 11:40
11	fa34554.d	580-9702-A-3-B	04/25/08 12:06
12	fa34555.d	ccv 254376 1000 NWDX	04/25/08 12:31
13	fa34556.d	MB 580-30606/1-B	04/25/08 13:02
14	fa34557.d	LCS 580-30606/2-B	04/25/08 13:22
15	fa34558.d	580-8586-A-3-D	04/25/08 13:47
16	fa34559.d	580-8586-A-4-D	04/25/08 14:12
17	fa34560.d	ccv 254376 1000 NWDX	04/25/08 14:38

Seq 013
MM

Document Uncontrolled When Printed

Attachment 2: n-Alkane Retention Time Standard Chromatogram

```
File        : F:\DATA\051208_A\YYZ5027.D
Operator    : TMR
Acquired    : 12 May 2008  10:51     using AcqMethod RACQ.M
Instrument  :   SEA014
Sample Name: 1166-95-4 n-alkane rt std
Misc Info   : BT=S014051208
Vial Number: 2
```



Title: Determination of EDB and DBCP in Aqueous and Solid Matrices [Methods SW 8011 and EPA 504.1]

Approvals

Signatures on Files

Colin McKean Date Semivolatile Organic Department Manager	Sandra Langway Date Health & Safety Manager / Coordinator
Terri Torres Date Quality Assurance Manager	Heather Prater Date Laboratory Director

Copyright Information:
This documentation has been prepared by TestAmerica Laboratories, Inc. and its affiliates ("TestAmerica"), solely for their own use and the use of their customers in evaluating their qualifications and capabilities in connection with a particular project. The user of this document agrees by its acceptance to return it to TestAmerica upon request and not to reproduce, copy, lend, or otherwise disclose its contents, directly or indirectly, and not to use it for any other purpose other than that for which it was specifically provided. The user also agrees that where consultants or other outside parties are involved in the evaluation process, access to these documents shall not be given to said parties unless those parties also specifically agree to these conditions.

THIS DOCUMENT CONTAINS VALUABLE CONFIDENTIAL AND PROPRIETARY INFORMATION. DISCLOSURE, USE OR REPRODUCTION OF THESE MATERIALS WITHOUT THE WRITTEN AUTHORIZATION OF TESTAMERICA IS STRICTLY PROHIBITED. THIS UNPUBLISHED WORK BY TESTAMERICA IS PROTECTED BY STATE AND FEDERAL LAW OF THE UNITED STATES. IF PUBLICATION OF THIS WORK SHOULD OCCUR THE FOLLOWING NOTICE SHALL APPLY:

©COPYRIGHT 2012 TESTAMERICA LABORATORIES, INC. ALL RIGHTS RESERVED.

Facility Distribution No. 001 **Distributed To:** Lab Intranet

1.0 Scope and Application

1.1 Analytes, Matrix(s), and Reporting Limits

This SOP provides guidance in the extraction and determination of 1,2-dibromoethane (EDB, CAS # 106-93-4) and 1,2-dibromo-3-chloropropane (DBCP, CAS # 96-12-8) in aqueous matrices utilizing a micro-extraction technique. The determination is made using a gas chromatograph equipped with an electron capture detector. A dual-column and dual detector analysis system is used to confirm compound identification.

The reporting limit for each compound is 0.01 ug/L for water.

On occasion clients may request modifications to this SOP. These modifications are handled following the procedures outlined in the Quality Assurance Manual.

2.0 Summary of Method

A known volume of sample is transferred to a 40 ml VOA vial. Aqueous samples are salted and extracted with hexane. The hexane extract is removed from the sample and analyzed by GC/ECD. Sample introduction is via an auto-sampler system in conjunction with the gas chromatograph. Identification of target analytes is accomplished by retention time comparison to a known standard. Quantitation is based upon target analyte response relative to a calibration curve established by a five-point (or greater) calibration curve. This method is restricted to use by, or under the supervision of, analysts experienced in the use of GC and organic analyses.

3.0 Definitions

The quality control terms used in this procedure are consistent with SW-846 terminology. Definitions are provided in the glossary of the TestAmerica Seattle Quality Assurance Manual (QAM).

4.0 Interferences

4.1 Impurities in the hexane extraction solvent account for a majority of interferences. Whenever interferences are noted in the method or instrument blank, the analyst should reanalyze the hexane to verify purity.

4.2 EDB at low concentrations may be masked by high concentrations of Dibromochloromethane (DBCM).

4.3 Additional calibration levels may be added to extend the calibration range. However, due to the nature of the ECD, the acceptance criteria may not be achieved. If this is the case, calibration levels may be sub-divided into various ranges (i.e., low, mid, high range) and the data file processed against the appropriate curve.

4.4 Sufficient headspace must be allowed in the 40 ml VOA vial to allow for analyte partitioning. Inadequate headspace will produce low recovery of analyte during the extraction process.

4.5 Emulsions may form in the final extract of samples containing petroleum contamination. Extracts exhibiting such emulsion should be centrifuged prior to analysis to aid in the separation of the hexane and contaminate.

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

5.1.1 Unknown samples may contain high concentrations of volatile toxic compounds. Sample containers should be opened in a hood and handled with gloves to prevent exposure.

5.1.2 The gas chromatograph contains zones that have elevated temperatures. The analyst needs to be aware of the locations of those zones, and must cool them to room temperature prior to performing maintenance.

5.1.3 There are areas of high voltage in the gas chromatograph. Depending on the type of work involved, either turn the power to the instrument off, or disconnect it from its source of power.

5.1.4 *The standards used in this method contain known carcinogens, neurotoxins and reproductive hazards. Use appropriate safety precautions and personnel protective equipment when working with the standards.*

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	Hazards	Exposure Limit (1)	Signs and symptoms of exposure
Methanol (MeOH)	Flammable Poison Irritant	200 ppm-TWA	A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness and dizziness. Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes.
Hexane	Flammable Irritant	500 ppm-TWA	Inhalation of vapors irritates the respiratory tract. Overexposure may cause lightheadedness, nausea, headache, and blurred vision. Vapors may cause irritation to the skin and eyes.
EDB	Toxic		Suspected carcinogen
DBCP	Toxic		Suspected carcinogen
DBP			Causes irritation to the eyes and skin. Harmful if inhaled. Causes respiratory tract irritation.
1 – Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

6.1 Instrumentation

- Gas chromatograph - HP5890/6890 or equivalent, equipped with dual ECDs and autosampler.
- Data system: One component of the system, Agilent's ChemStation, is used for data acquisition and storage on machine-readable media. Since no processing is done by Chemstation and since there are no audit trail functions associated with data acquisition, the audit trail feature for Chemstation may be either enabled or disabled. The other component, Chrom, is used for data processing such as the measurement of peak area or peak height. By design, the audit trail feature for Chrom is always enabled.
- Columns:
  - Primary Column: *Zebron Multi residue 1, 30 m, 0.25mm ID, 0.25um* film or equivalent
  - Secondary Column: *Zebron Multi residue 2, 30 m, 0.25mm ID, 0.25um* film or equivalent
  - Note: Other columns may be used. These were the columns in place at the time the SOP was prepared.
- Top loading balance, capable of weighing to 0.01 g

6.2 Supplies

- Clean 40ml VOA vials
- Autosampler vials with seals
- Syringes – 10, 25, 50, 100, 500, 1000 uL, accurate to ± 3%
- Graduated cylinder – Class A, *50* ml
- 1 to 10 mL variable Auto-pipette, calibrated in accordance with SOP TA-QA-0016

7.0 Reagents and Standards

7.1 Document reagent/standards and reagent/standard preparation in TALS using the reagent module as described in SOP TA-QA-0619.

7.2 DBP (Dibromopropane) Surrogate Primary stock solution at a concentration of 10,000 ug/ml – Ultra cat # PPS-400-1.

7.3 DBP primary intermediate working solution

7.3.1 Add approximately 8 ml of methanol to a 10ml volumetric flask.

7.3.2 Add 200 ul of the primary stock solution (section 7.2) to the flask.

7.3.3 Bring to volume with methanol, stopper, and mix

7.3.4 Transfer to an appropriately labeled container and store at < 0°C.

7.3.5 Final concentration of this solution will be 200 ug/ml

7.3.6 Expires 4 weeks from the date made or the manufacturer's expiration date, which ever comes first.

7.4 DBP Surrogate secondary stock solution at a concentration of 100 ug/ml –O2SI Cat # 012340-02

7.5 DBP secondary intermediate working solution.

7.5.1 Add approximately 5ml of methanol to a 10 ml volumetric flask.

7.5.2 Add 2000 ul of the primary stock solution (section 7.4) to the flask

7.5.3 Bring to volume with methanol, stopper, and mix.

7.5.4 Transfer to an appropriately labeled container and store at < 0°C.

7.5.5 Final concentration of this solution will be 200 ug/ml

7.5.6 Expires 4 weeks from the date made or the manufacturer's expiration date, which ever comes first.

7.6 EDB/DBCP/ Stock Solution at a concentration of 200 ug/mL – Restek cat# 30239

7.7 EDB/DBCP//DBP Working Solution (ICAL stock solution)

7.7.1 Add approximately 48 ml of methanol to a 50 ml volumetric flask.

7.7.2 Add 50 uL of the 200 ug/ml stock solution and intermediate EDB solutions(Section 7.1and 7.2) to the flask.

7.7.3 Bring to volume with methanol, stopper, and mix.

7.7.4 Transfer to an appropriately labeled container and store at < 0°C.

7.7.5 Final concentration of this solution will be 200 ug/L.

7.7.6 Expires 4 weeks from the date made or the manufacturer's ampule expiration date – whichever is soonest.

7.8 EDB/DBCP/ Second Source solution at a concentration of 200 ug/mL – Ultra Scientific cat# DWM-514-1.

7.9 EDB/DBCP/ Second Source working spike solution

7.9.1 Add approximately 48 ml of methanol to a 50 ml volumetric flask.

7.9.2 Add 50uL of the 200 ug/ml stock solution (Section 7.8) to the flask.

7.9.3 Bring to volume with methanol, stopper, and mix.

7.9.4 Transfer to an appropriately labeled container and store at < 0°C.

7.9.5 Final concentration of this solution will be 200 ug/L.

7.9.6 Expires 4 weeks from the date made or the manufacturer's ampoule expiration date – whichever is soonest.

7.10 DBP Second Source working surrogate solution

7.10.1 Add approximately 48 ml of methanol to a 50 ml volumetric flask.

7.10.2 Add 50uL of the 200 ug/ml DBP intermediate surrogate solution (Section 7.8) to the flask.

7.10.3 Bring to volume with methanol, stopper, and mix.

7.10.4 Transfer to an appropriately labeled container and store at < 0°C.

7.10.5 Final concentration of this solution will be 200 ug/L.

7.10.6 Expires 4 weeks from the date made or the manufacturer's ampoule expiration date – whichever is soonest.

Document Uncontrolled When Printed

7.11 Hexane, pesticide grade or higher

7.12 Methanol, pesticide grade or higher

7.13 De-ionized water

7.14 Sodium chloride, laboratory grade

7.15 EDB/DBCP Second Source solution at a concentration of 2000 ug/mL – Ultra Scientific cat# HCM-812.

7.16 EDB/DBCP Second Source working solution

7.16.1 Add approximately 48 ml of methanol to a 50 ml volumetric flask.

7.16.2 Add 5 uL of the 2000 ug/ml stock solution (Section 7.8) to the flask.

7.16.3 Bring to volume with methanol, stopper, and mix.

7.16.4 Transfer to an appropriately labeled container and store at < 0°C.

7.16.5 Final concentration of this solution will be at 0.2 ug/ml.

7.16.6 Expires 4 weeks from the date made or the manufacturer's ampoule expiration date – whichever is soonest.

7.17 Managers/supervisors or a designee are expected to check their areas on a monthly basis for expired standards/reagents and dispose of them according to SOP TA-EHS-0036.

8.0 Sample Collection, Preservation, Shipment and Storage

8.1 Aqueous samples should be collected in duplicate 40mL VOA vials with Teflon lined lids and minimal headspace. Samples should be shipped and stored under refrigeration between temperatures of 0°to 6℃.

8.2 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	Extraction Holding Time	Analysis Holding Time	Reference
Waters	40mL VOA vial	40-mL	Cool 0-6°C; Sodium thiosulfate Minimal headspace	14 Days	14 Days from collection or 7 days from extraction, which ever comes first.	40 CFR Part 136.3

Document Uncontrolled When Printed

9.0 Quality Control

9.1 The minimum quality controls (QC), acceptance criteria, and corrective actions are described in this section. When processing samples in the laboratory, use the LIMS QC program code and special instructions to determine specific QC requirements that apply.

9.1.1 The laboratory's standard QC requirements, the process of establishing control limits, and the use of control charts are described more completely in TestAmerica Seattle SOP TA-QA-0620, Quality Control Program.

9.1.2 Project-specific requirements can override the requirements presented in this section when there is a written agreement between the laboratory and the client, and the source of those requirements should be described in the project documents. Project-specific requirements are communicated to the analyst via special instructions in the LIMS.

9.1.3 DoD projects must meet the requirements outlined in the DoD QSM version 4.2.

9.1.4 Any QC result that fails to meet control criteria must be documented in a Nonconformance Memo (NCM). The NCM is approved by the supervisor and then automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group also receives NCMs by e-mail for tracking and trending purposes. The NCM process is described in more detail in SOP TA-QA-0610. This is in addition to the corrective actions described in the following sections.

9.2 Batch Definition

9.3 Batches are defined at the sample preparation stage. The batch is a set of up to 20 samples of the same matrix, plus required QC samples, processed using the same procedures and reagents within the same time period. Batches should be kept together through the whole analytical process as far as possible, but it is not mandatory to analyze prepared extracts on the same instrument or in the same sequence. The method blank must be run on each instrument that is used to analyze samples from the same preparation batch. See QC SOP TA-QA-0620 for further details.

9.4 Method Blank (MB)

At least one method blank must be processed with each preparation batch. The method blank for batches of aqueous samples consists of a 40mL VOA vial of reagent water which is free of any of the analyte(s) of interest. The method blank is processed and analyzed just as if it were a field sample.

Acceptance Criteria: The result for the method blank must be less than the reporting limit for the analyte(s) of interest or less than 10% of the analyte concentration found in the associated samples, whichever is higher. Note that some programs (e.g., DoD QSM, BP LaMP) require that the maximum blank concentration must be less than one-half of the reporting limit or less than 10% of the lowest sample concentration.

Corrective Action: If target analytes in the blank exceed the acceptance limits, the source of the contamination must be investigated. All samples associated with an unacceptable method blank must be re-prepared and reanalyzed. If the analyte was <u>not</u> detected in the samples, then the data may be reported with qualifiers (check project requirements to be sure this is

Document Uncontrolled When Printed

allowed) and it must be addressed in the project narrative. In other circumstances, the entire batch must be re-prepared and reanalyzed.

Note: DoD requirements state samples are to be re-extracted and/or reanalyzed. Reporting data with qualifiers is only appropriate where samples cannot be reanalyzed.

9.5 Laboratory Control Sample (LCS)

At least one LCS must be processed with each preparation batch. For EPA method 504.1, the LCS must be prepared at a frequency of 1/10. For aqueous sample batches, the LCS consists of reagent water, to which the analyte(s) of interest are added at a known concentration. The LCS is carried through the entire analytical procedure just as if it were a sample. When insufficient sample volume exists for the preparation of an MS/MSD an LCSD must be prepared.

Acceptance Criteria: The recovery results for the LCS must fall within the established control limits. Control limits are set at ± 3 standard deviations around the historical mean. Where required, project-specific limits may be used in place of historical limits. Current control limits are maintained in the LIMS.

Corrective Action: If LCS recoveries are outside of the established control limits, the system is out of control and corrective action must occur. If recoveries are above the upper control limit and the analyte(s) of interest is not detected in samples, the data may be reported with qualifiers (check project requirements to be sure this is allowed) and it must be addressed in the project narrative. In other circumstances, the entire batch must be re-prepared and reanalyzed.

Note: DoD requirements state samples are to be re-extracted and/or reanalyzed. Reporting data with qualifiers is only appropriate where samples cannot be reanalyzed.

9.6 Matrix Spike/Matrix Spike Duplicate (MS/MSD)

One MS/MSD pair must be processed with each preparation batch when sufficient volume is available. A matrix spike (MS) is a field sample to which known concentrations of target analytes have been added. It is prepared in a manner similar to the LCS, but uses a real sample matrix in place of the blank matrix. A matrix spike duplicate (MSD) is a second aliquot of the same sample (spiked exactly as the MS) that is prepared and analyzed along with the sample and matrix spike. Some programs allow spikes to be reported for project-related samples only. Samples identified as field blanks cannot be used for the MS/MSD analysis.

Acceptance Criteria: The recovery results for the MS and MSD must fall within the established control limits, which are set at ± 3 standard deviations around the historical mean. The relative percent difference (RPD) between the MS and MSD must be less than the established RPD limit, which is set at 3 standard deviations above the historical mean. Current control limits are maintained in the LIMS.

Corrective Action: If analyte recovery or RPD falls outside the acceptance range, but the associated LCS recovery is in control, and all other QC criteria (e.g., continuing calibration verification) are met, then there is no evidence of analytical problems, and qualified results may be reported. The situation must be described in an NCM and in the final report case narrative. In other circumstances, the batch must be re-prepared and reanalyzed.

9.7 Low-level QC reference sample

A 0.01 ug/L low-level QC reference sample must be prepared and analyzed weekly at a minimum as long as samples are being prepared.(see Section 10.1.5).

Acceptance Criteria: The recovery must be between 60-140% of the expected value.

Corrective Action: When either analyte fails to recover within the limits, repeat the test for that analyte. Another failure confirms a problem with the measurement system or standards. If this occurs, locate and correct the source of the problem and repeat the test.

9.8 *Check Standard*

A 0.02 ug/L standard must be prepared daily to be run with sample analysis

Acceptance Criteria: The recovery must be within 60-140% of the expected value.

Corrective Action: When either analyte fails to recover within the limits, repeat the test for that analyte. Another failure confirms a problem with the measurement system or standards. If this occurs, locate and correct the source of the problem and repeat the test.

9.9 <u>Instrument QC</u>

9.9.1 Initial Calibration

9.9.1.1 The percent RSD for the initial calibration must be $\leq$ 10%. If the %RSD is >10%, linear or nonlinear modeling may be attempted. The correlation coefficient (r) must be $\geq$ 0.995 for all linear or nonlinear calibration curves (R^2 must be $\geq$ 0.99).

9.9.1.2 Note: Non-linear calibrations should not be employed for methods or instruments previously shown to exhibit linear calibration for the analytes of interest. In other words, you can't employ a quadratic model to compensate for detector saturation or lapses in instrument maintenance.

9.9.1.3 If a linear regression is used, the Y-intercept has to be less than the lowest calibration standard.

9.9.1.4 Assess the calibration using a second source standard and refit technique as described in sections 10.2.3.4.1 and 10.2.3.4.2.

9.9.1.5 All analytes in the second source must be within 80-120% of the expected value. If you're analyzing samples for BP, the BP LaMP requires the second source standard to meet the same acceptance criteria as the CCV which for this method is 90-110%. For the refit, the

absolute value of the percent difference between these two amounts for every calibration level should be less than or equal to 20%.

9.9.1.6 If acceptance criteria are not achieved, corrective action must be taken (see Section 10.3.2.5).

9.9.1.7 Any samples associated with a failed initial calibration must be reanalyzed.

9.9.1.8 The initial calibration curve should pass on both columns.

9.9.2 Calibration Verification

9.9.2.1 Calibration verification must be completed prior to sample analysis.

9.9.2.2 A calibration verification check standard (CCV) must be analyzed at the beginning of the sequence, at intervals of not less than once every twenty samples during the sequence, and at the end of the sequence. Analysis of a CCV after every 10 samples is recommended to minimize the number of samples requiring re-injection when QC limits are exceeded.

9.9.2.2.1 Analysis of a CCV after every 10 injections is required for DoD and BP projects.

9.9.2.3 The concentrations of the respective analyte(s) must not vary more than 10% from those established by the initial calibration and the relative standard deviation of replicate measurements must be less than 10%.

9.9.2.4 In the event of a CCV failure, corrective action must be taken prior to sample analysis. Corrective actions include injection port maintenance and the preparation of fresh standards. If the subsequent calibration verification injection fails, a new initial calibration curve must be processed. (i.e., no more than two consecutive injections of the calibration verification may be processed.)

9.9.2.4.1 If a CCV standard is not acceptable, all samples that are bracketed by the failing CCV must be reanalyzed. If deemed necessary, dilute the affected samples sufficiently (but not excessively) to allow for passing CCVs in the re-analysis.

9.9.2.4.2 If a CCV fails high, any samples that are bracketed by the failing CCV and that are ND do not need to be reanalyzed as the verification standard has demonstrated that the analyte would have been detected were it present (check project requirements to be sure this is allowed). The situation must be described in an NCM. In other circumstances, the batch must be re-prepared and reanalyzed.

9.9.2.4.3 **Note:** DoD requirements state samples are to be re-extracted and/or reanalyzed. Reporting data with qualifiers is only appropriate where samples cannot be reanalyzed.

9.9.3 Any extra QC that is analyzed in a batch or sequence must be evaluated using the same criteria as the corresponding QC above.

9.10 Procedure

One-time procedural variations are allowed only if deemed necessary in the professional judgment of management to accommodate variation in sample matrix, chemistry, sample size, or

other parameters. Any variation in procedure shall be completely documented using an NCM. The NCM is approved by the supervisor and then automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA department also receives NCMs by e-mail for tracking and trending purposes. The NCM process is described in more detail in SOP # TA-QA-0610. The NCM shall be filed in the project file and addressed in the case narrative.

9.11 Aqueous Sample Preparation

NOTE: Check the Balance Logbook to determine if the daily calibration check was completed. If the balance requires a check, verify the calibration as detailed in SOP TA-QA-0014.

9.11.1 All calibration standards, QC and samples must go through this process.

9.11.2 Calibration and continuing calibration standards are prepared from the primary source (Section 7.2) according to the table in section 10.3.2.1.1. A set of continuing calibration standards, consisting of the check standard at 0.2ug/L, and a mid point against the calibration is prepared on each day samples are extracted.

9.11.3 Add 6.0 + 0.2 grams of sodium chloride to a labeled 40mL VOA vial.

9.11.4 Using a 50 mL Class A graduated cylinder, quantitatively measure and transfer 35 ml of sample to the appropriate 40mL VOA vial. Gently swirl to dissolve the salt.

9.11.5 If the sample is designated as a blank spike or blank spike duplicate, add 50 uL of the second source working standard (Section 7.8) to a 40mL VOA vial containing 35 mL of DI water and 6.0 g of baked sodium chloride. Also, on a weekly basis and in a similar manner, prepare a single low-level check from the second source using 1.0 uL of the second source working standard. If the sample is designated as a matrix spike or matrix spike duplicate, add 10 uL of the second source working standard to a 40mL VOA vial containing 35mL of sample and 6.0 g of baked sodium chloride. The final effective concentration of the blank spike and matrix spike is 1.0 ug/L, while the final effective concentration of the low-level check is 0.1 ug/L.

9.11.6 Add 2 ml of hexane using a 2.5ml syringe.

9.11.7 Shake on the mechanical shaker for 5 minutes.

9.11.8 Carefully remove the hexane layer (top) and transfer to a clean, labeled sample extract vial. Avoid transferring any of the aqueous phase.

9.11.9 If emulsions are noted in the final extract, centrifuge as noted in Section 4.5.

9.11.10 Transfer the final extracts to the analytical instrument for analysis. If analysis is not to be performed immediately, store the extracts refrigerated.

9.11.11 Record the identification of all reagents and standards on the bench sheet.

9.12 Calibration

Analysis of standards and sample extracts is completed using a GC/ECD combination. Recommended conditions are:

Columns:	*Zebron Multi-Residue 1, 30m, 0.25mm* ID, 0.25um film or equivalent. *Zebron Multi-Residue 2*, 30m, 0.25mm ID, 0.25um film or equivalent.
Injection Port:	210°C
Injection Volume:	3 uL/Split injection setup
Injector:	Split/Splitless with EPC. Delay = 0.5 min
Flow:	Constant flow – 5 psi @ 50°C
Liner:	4mm, single or double gooseneck
Detectors:	300°C
GC Program:	40°C for 3.0 minutes Ramp to 150°C @ 12°/minute Ramp to 280°C @ 10°/minute and hold for 2 minut es
Total Run Time:	Approx 19.5 minutes

Note: Other columns and operating conditions may be used provided they meet the requirements set forth in this SOP. Operating conditions can also be found in the maintenance log book for each instrument.

9.12.1 Approximate retention times:

	Column 1	Column 2
EDB	4.5	6.5
DBP	5	7.5
DBCP	9	11.5

9.12.2 Initial Calibration

All calibration and continuing calibration standards must be processed through the extraction procedure described in Section 10.1 or 10.2. For an overview of the different calibration models used, and the calculations associated with them see CA-Q-S-005.

9.12.2.1 Working calibration standards

9.12.2.1.1 Seven calibration levels are made with the following additions of standards to 200 ml of de-ionized water. Additional levels may be added but must meet acceptance criteria for inclusion in the curve. The method prohibits the analysis of additional standards for the purpose of choosing a set of results that meet the calibration acceptance criteria. The working stock is from section 7.2. Record the identification of the standards on the raw data.

Standard	Amount	On-Column Conc.	Effective Conc.
Level 1 Working Stock	0.5 uL	0.05ug/L	1.43 ng/L
Level 2 Working Stock	1.0 uL	0.10 ug/L	2.86 ng/L
Level 3 Working Stock	2.0 uL	0.20 ug/L	5.71 ng/L
Level 4 Working Stock	5.0 uL	0.50 ug/L	14.3 ng/L
Level 5 Working Stock	10.0 uL	1.00 ug/L	28.6 ng/L
Level 6 Working Stock	25.0 uL	2.50 ug/L	71.4 ng/L
Level 7 Working Stock	50.0 uL	5.00 ug/L	143 ng/L

Document Uncontrolled When Printed

NOTE: The calibration must be analyzed within one day of extraction.

9.12.2.2 Always analyze an acceptable blank prior to analyzing any standards or samples. Then, starting with the lowest level standard, analyze each calibration level using the conditions described in Section 10.3. Record the identification of the standards on the raw data.

9.12.2.3 Use Chrom to calculate the average response factor (RF) for each analyte using external standard calibration. The %RSD for the RFs must be <10%. Utilize "Average of Response Factors" to establish the calibration curve and measure acceptance criteria. The initial calibration curve should pass on both signals.

NOTE: Generally, it is NOT acceptable to remove points from a calibration. If calibration acceptance criteria are not met, the normal corrective action is to examine conditions such as instrument maintenance and accuracy of calibration standards. Any problems found must be fixed and documented in the run log or maintenance log. Then the calibration standard(s) must be reanalyzed.

9.12.2.3.1 If the %RSD for a target analyte is >10%, attempt to fit the data to a linear calibration model based on least squares regression. This will require at least 5 calibration points and those points cannot include the origin (0,0). The correlation coefficient (r) must be >0.995 or coefficient of determination (r2) must be >0.990, and the curve cannot be forced through the origin.

Note: A least squares calibration model may also be employed based on past experience of instrument response.

9.12.2.3.2 If the %RSD for an analyte (or surrogate) is > 10% and the calibration data doesn't fit a linear model, attempt to fit the data to a non-linear, second order polynomial calibration model. This will require at least 6 calibration points and those points cannot include the origin (0,0). The coefficient of determination (r^2) must be >0.990 and the curve cannot be forced through the origin.

Note: Non-linear calibrations should not be employed for methods or instruments previously shown to exhibit linear calibration for the analytes of interest. In other words, you can't employ a quadratic model to compensate for detector saturation or lapses in instrument maintenance.

9.12.2.4 Additional Inspection of Calibration Model

In addition to the criteria specified in 10.3.2.3 for the statistics (%RSD for average response factors and r and r^2 for linear and non-linear least squares regressions) generated from the construction of the calibration model, the evaluation of a calibration model must also include the analysis of a second source standard (10.3.3.4.1) and an assessment of the representativeness of the data used to produce the model (10.2.3.4.2).

9.12.2.4.1 Initial calibration accuracy must be verified through the use of a mid-range second source standard. The source of this standard should be different than the source from which the calibration standards were obtained. In situations where a single vendor is

Document Uncontrolled When Printed

used to provide the primary and secondary standards, the analyst must obtain written warranties that the two references were not prepared from the same reference material. The laboratory control samples prepared in Section 9.4 meet these criteria. The acceptance criteria for the validation is + 20%. (+ 10% for BP LaMP). Record the identification of the second source on the raw data).

9.12.2.4.2 The calibration data must be refit back to the calibration model, allowing a comparison of the calculated amount of each of the standards against the expected amount of the standard. The absolute value of the percent difference between these two amounts for every calibration level should be less than or equal to 20%.

9.12.2.5 Corrective Actions

Corrective actions are required when the criteria for %RSD, r, r^2 or %D are not met.

9.12.2.5.1 Check the instrument operating conditions. Make adjustments and perform maintenance as needed (a dirty injection liner is often to blame), and then re-analyze calibration standards. Calculate and assess the new %RSD, r, r^2 or %D derived from action.

9.12.2.5.2 Review the results for the affected analytes, paying particular attention to proper identification, peak integration, area counts relative to other calibration standards and calculations associated with calibration or response factors and RSD. Correct any discrepancies or if the problem appears to be associated with a single standard, reanalyze the standard once to rule out problems due to random chance. This re-analysis must occur within 8 hours of the original analysis and before any samples are analyzed. If a standard is re-analyzed or a new standard is analyzed, then all of the results from the original analysis of the standard in question must be discarded. Calculate and assess the new %RSD, r, r^2 or %D derived from action.

9.12.2.5.3 Narrow the calibration range by replacing one or more of the calibration standards with standards that cover a narrower range. Modifying the range in this manner may affect the reporting limit or the linear range. Calculate and assess the new %RSD, r, r^2 or %D derived from action.

9.12.2.5.4 Narrow the calibration range by removing data points from either extreme ends of the range. Rejection of such a point may affect the reporting limit or the linear range. It is prohibited to remove data points from within a calibration range, while still retaining the extreme ends of the calibration range. An entire level may not be rejected [this represents a departure from the previous revision of this SOP that permitted a rejection for a valid analytical reason, because frequently no reasons were provided or the reasons were found to be invalid (e.g., when the deleted level was included, the curve would pass for the

majority of the compounds) and the required documentation was lacking in completeness]. When rejecting an extreme end level, remember that there must be a minimum of five standards remaining for a linear model and six points for a quadratic. Calculate and assess the new %RSD, r, r^2 or %D derived from this action.

9.12.2.5.5 When the deviation of any calibration point reprocessed against the new curve exceeds 20%, note the exceptions in an NCM.

9.12.3 Retention Time Windows

9.12.3.1 Using data obtained from three analyses conducted over a 72-hour period of a continuing calibration verification (or mid-point) standard, record the retention time for each analyte and surrogate to three decimal places (e.g., 0.007).

9.12.3.2 Calculate the mean retention time and standard deviation for each analyte.

9.12.3.3 Assign a retention time window of + 3 standard deviations around the mean for each analyte.

9.12.3.4 If the standard deviation is 0.00, use a default retention time window of 0.05 to 0.07 minutes.

9.12.3.5 New RT windows must be established when a new GC column is installed.

9.12.3.6 Establish the center of the retention time window for each analyte by using the absolute retention time for each analyte from the calibration verification standard at the beginning of the analytical shift. For samples analyzed during the same shift as an initial calibration, use the retention time of the mid-point standard of the initial calibration.

9.12.3.7 Whenever the observed retention time of an analyte in a continuing calibration verification standard is outside of the established retention time window, the analyst should determine the cause and correct the problem before proceeding with the analyses.

9.12.4 Continuing Calibration Verification

9.12.4.1 The calibration curve (concentration or RF) must be verified prior to analysis of samples, at intervals of no more than twenty (20) samples (10 samples is recommended to minimize the number of samples requiring re-injection when QC limits are exceeded), or after every 10 injections when required by specific projects or clients (e.g., DoD BP LaMP), and at the conclusion of analysis.

9.12.4.2 Continuing calibration verification is completed using primary source (7.2). *A check standard and a continuing calibration standard at the mid point of the initial calibration is prepared and extracted with each batch of samples (see Section 10.1.2).* For every 10 (but no more than 20) samples throughout the analytical sequence, analyze a continuing calibration standard (alternating between the 1, 5 and 15 ng/mL concentrations).

9.12.4.3 The percent deviation for the measured concentrations or RRFs must be < *15*%. If acceptance criteria are not achieved, the verification process

must be repeated using fresh calibration solution or, alternatively, a new initial calibration must be established according to Section 10.3.2. If the subsequent calibration verification injection fails, a new initial calibration curve must be processed. (i.e., no more than two consecutive injections of the calibration verification may be processed.)

9.12.4.4 Record the identification of the standards on the raw data.

9.13 Sample Analysis

Samples are analyzed in a set referred to as an analysis sequence. Be sure to demonstrate the absence of system contamination by analyzing a method blank or a solvent blank spiked with surrogate at the beginning of each analytical sequence and then after each CCV.

9.13.1 Transfer a small portion of the sample extract to a labeled auto-sampler vial with conical insert and load the vial in the proper position on the auto-sampler tray. If required, perform dilutions prior to loading. Analyze the sample extracts and all QA samples utilizing the same parameters used for the initial calibration and calibration verification. Samples must be analyzed on the same instrument as the batch QC. The only exception would be reanalysis at a dilution.

If batch QC is reanalyzed to confirm recovery failure or blank contamination and the results indicate an improvement such that the reanalysis would be reported, the samples must be reanalyzed as well. The only exception to this would be for an obvious reason (e.g. a bent autosampler needle).

9.13.2 If the concentration of a target compound in a sample exceeds the concentration (of the compound) in the highest calibration standard, the sample must be diluted.

9.13.2.1 Transfer a known amount of the original extract to a clean vial and add the appropriate amount of hexane to achieve the desired dilution. Record the lot # of the hexane on the injection log.

9.13.2.2 Analyze the sample extracts utilizing the same parameters used for the initial calibration and calibration verification.

9.13.2.3 All detections over the calibration range must be flagged in LIMS.

9.13.2.4 Instrument response should be monitored daily by recording the absolute response of an individual peak. The surrogate DBP is the peak chosen for this method. The absolute response for this peak, in the CCVRT, needs to be documented in the maintenance log book, for each column, on a daily basis.

9.13.2.5 An instrument (solvent) blank should be analyzed after each CCV and any sample that has saturated the detector to check that the blank is free of interference and the system is not contaminated.

9.13.2.6 If a CCV failure occurs after analysis of samples and may potentially be due to matrix effects on the detector, the samples should be diluted sufficiently (but not excessively) to allow for passing CCVs.

9.13.3 Compound Identification

9.13.3.1 The RT for the respective analyte must fall within a window based upon + 3 standard deviations of the mean as established in section 10.3.3.

9.13.3.2 Comparison between results from different columns or detector

9.13.3.3 The result from a designated primary column is normally reported. The result from the secondary column is reported if any of the following is true:

9.13.3.3.1 There is obvious chromatographic interference on the primary column.

9.13.3.3.2 The difference between the result on the primary column and the result on the secondary column is > 40% and chromatographic interference is evident.

9.13.3.3.3 A continuing or bracketing standard fails on the primary column, but is acceptable on the secondary column. However, if the difference between the primary column and secondary column results is > 40% and the primary column calibration verification fails, then the sample must be evaluated for reanalysis.

9.13.3.3.4 For BP, the lower concentration of the two results (from the two quantitative columns) is reported.

9.13.3.4 When sample results are confirmed using two dissimilar columns, the agreement between the quantitative results should be evaluated after the identification has been confirmed. Large differences in the numerical results may be indicative of positive interferences with the higher of the results, which could result from poor separation of target analytes or the presence of a non-target compound. Thus, in order to ensure that the results reported are appropriate for the intended application, the analyst should make a formal comparison.

9.13.3.5 Calculate the RPD between the two results using the formula in section 11.2.

9.13.3.5.1 If one result is significantly higher (e.g. >40%), check the chromatograms to see if an obviously overlapping peak is causing an erroneously high result. If no overlapping peaks are noted, examine the baseline. A rising baseline may cause the mis-integration of the peak for the lower result.

9.13.3.5.2 If no anomalies are noted, review the chromatographic conditions. If there is no evidence of chromatographic problems, then it may be appropriate to report the lower result.

9.13.3.5.3 Results are qualified if the RPD is greater than 40%, but either the lower or higher value is reported based on the primary-column determination.

9.13.4 Quantitation

9.13.4.1 Quantitate the initial results against the calibration curve established in Section 10.3.2.

9.13.4.2 Determine the concentration of the injected sample according to the equation documented in Section 11.3.

9.13.5 Manual Integrations

9.13.5.1 Manual integrations should be employed only in situations where the data system has:

9.13.5.1.1 incorrectly identified a signal, or

9.13.5.1.2 Incorrectly quantitated a signal because of an inaccurately drawn baseline and thereby producing an obvious bias.

9.13.5.2 Manual integrations should not be employed to identify a compound in a calibration curve unless it is needed to address retention time changes.

9.13.5.3 Refer to corporate SOP CA-Q-S-002 for specifics on manual integration.

9.14 Instrument Maintenance

9.14.1 Injection Port Maintenance

9.14.1.1 The injection port liner is changed weekly, or whenever the system performance deteriorates, (i.e., endrin breakdown exceeds acceptance limits). The retention gap column is typically replaced along with the injection port liner.

9.14.1.2 Injector septa are changed whenever the injection port liner is changed, and/or after 100 - 120 injections have occurred.

9.14.1.3 The injection port is heated to 300 ℃ until the detector signal returns to normal operation level (approximately 2.5 mV) whenever a new injection port liner is installed.

9.14.2 Column Maintenance

9.14.2.1 Columns are replaced every six months or as needed. The following symptoms are indicators that a new column is needed:

- Excessive baseline rise.
- Calibration curves are not linear, or fail shortly after analysis.
- Poor peak shape.
- Poor peak separation.

9.14.3 Column Replacement

9.14.3.1 Remove the old column from the GC oven by loosening the injector and detector nuts with the appropriate wrench.

9.14.3.2 The detector end of the new column is installed first. Slide the column nut over the column end.

9.14.3.3 Install the appropriate ferrule onto the column. The tapered end of the ferrule is placed towards the column nut.

9.14.3.4 Cut 1 to 2 cm from the end of the column.

9.14.3.5 Uncoil approximately 20 cm of column.

9.14.3.6 Move the column nut and ferrule within 12-15 cm. of the end of the column.

9.14.3.7 Accurately measure 11.5 cm. from the end of the column and mark the distance. Do not allow the column nut past this mark, as the marking media may contaminate the ferrule and the column. Partially insert the column into the lower end of the detector. The column must be inserted all the way to the mark. Thread the column nut and ferrule until finger tight.

9.14.3.8 Cut approximately 0.5 to 1 meter of used column to be used as a retention gap. If capillary fused silica tubing is available, it may also be used. Slide the column nut onto one end of the retention gap.

9.14.3.9 Install a ferrule onto the retention gap. The tapered end of the ferrule is placed towards the column nut.

9.14.3.10 Cut 1 to 2 cm. off of the end of the retention gap.

9.14.3.11 Accurately measure 5- 7 cm. from the end of the column and mark the distance. Do not allow the column nut past this mark, as the marking media may contaminate the ferrule and the column. Partially insert the column into the lower end of the injector. The column must be inserted all the way to the mark. Thread the column nut and ferrule until finger tight.

9.14.3.12 Cut approximately 1 to 2 cm. from the exposed ends of the column and the retention gap. Join with a presstight connector.

9.14.3.13 Tighten all column nuts 1/4 to 1/2 turn or until column cannot be pulled out of the column nut.

9.14.3.14 The gas flows are set to manufacturer or method recommended levels and are checked prior to each initial calibration:

Column flow: Approximately 5 - 5.5 mL/min.
Make-up gas at the detector (Hydrogen): Column flow + X mL/min. = to 25 mL/min.
Conditions may very between instruments.

Condition the column by heating the oven to 5-10 ℃ above its maximum operating temperature for approximately two hours, or until the baseline drops to its normal operating level. After the column is conditioned, the oven temperature is set at standby. At this point, the instrument is ready to be calibrated.

9.14.4 <u>ECD Maintenance</u>

9.14.4.1 Prior to analysis of initial calibration curve standards, the detector is heated to 380 ℃ for at least one hour. The detect or must be stabilized at to normal operating temperature (352 ℃ – 350 ℃) prior to analysis of any standards.

9.14.4.2 The detector is wipe tested and the wipes are sent in to TestAmerica Richland periodically for leak checks in accordance with SOP TA-QA-0409. If the detector is causing excessive baseline noise, is no longer providing a linear response, or will not produce a linear calibration curve, the detector should be sent in for repair. NOTE: ECDs contain radioactive Ni63, and should **<u>never</u>** be opened or maintained by untrained personnel.

9.14.5 <u>Autosampler Maintenance</u>

9.14.5.1 Fill the rinse reservoir daily, or more often if needed. Use the appropriate solvent for the analysis.

9.14.5.2 Check the injector syringe weekly:

9.14.5.3 Check for smooth plunger movement

9.14.5.4 Ensure that the needle tip is not bent, as this will cause septum pieces to be introduced into the injection port, and result in endrin breakdown.

9.14.6 <u>Spare Parts</u>

9.14.6.1 Septa

Document Uncontrolled When Printed

9.14.6.2 Injection Port Liners

9.14.6.3 Column nut

9.14.6.4 Ferrules: 1/4 in. graphite/vespel, 0.4 mm. ID graphite/vespel

9.14.6.5 Syringes, Hamilton 701 or equivalent

9.14.6.6 Solvent and waste vials and septa

9.14.6.7 Y-Splitter

9.14.6.8 Inlet Seal

All maintenance and repairs need to be documented in the instrument's maintenance logbook. The logbook must include the instrument name, serial number for each major component (e.g., GC, autosampler) and the date of start-up. When an instrument is not capable of analyzing samples, it needs to be tagged "Out of Service". Logbook entries must include a description of the problem and what actions were taken to address the problem. After an instrument has undergone maintenance or repairs, the system is evaluated using a tune, CCV or ICAL. If the evaluation is successful, the analyst documents in the logbook that the "System returned to control as indicated by a passing CCV" (or ICAL or MB, etc as may be the case).

If columns were replaced during maintenance procedures the specific make, model and serial numbers of the columns installed need to be entered in the instruments maintenance logbook

10.0 Calculations / Data Reduction

10.1 Accuracy

ICV / CCV, LCS % Recovery = $\frac{\text{observed concentration}}{\text{known concentration}} \times 100$

MS % Recovery = $\frac{\text{(spiked sample) - (unspiked sample)}}{\text{spiked concentration}} \times 100$

10.2 Precision (RPD)

Matrix Duplicate (MD) = $\frac{|\text{orig. sample value - dup. sample value}|}{[(\text{orig. sample value + dup. sample value})/2]} \times 100$

10.3 Sample Concentration

$$\frac{(C_n)(V_e)(d)}{(V_t)}$$

Where:
C_n = Analyte concentration
V_e = Extract volume
V_t = Total sample volume extracted
d = Dilution Factor (if required)

10.4 Analyte Concentration

$$(C_n) = \frac{A_n}{RF}$$

Document Uncontrolled When Printed

Where:

A_n = Analyte area
RF = Response factor

For linear calibration models:

$$Y = MX + B$$

where: Y = raw response of the analyte
M = calculated slope of the line
B = y-intercept
X = analyte concentration

For quadratic models:

$$R = Ax^2 + bx + c$$

Where: R = raw response of the analyte
X = analyte concentration
A,b,c = calculated amounts on calibration curve plot

10.5 Response Factor

$$RF = \frac{A_n}{C_a}$$

where: An = Analyte area
Ca = Analyte concentration

11.0 Method Performance

11.1 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 (see SOP TA-QA-0602). 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.

11.2 Demonstration of Capabilities

Analyst initial and continuing Demonstrations of Capability (DOC) are performed before any client samples are analyzed and are updated annually. See SOP TA-QA-0617 for details.

11.3 Training Requirements

See SOP TA-QA-0608 for detailed training requirements.

12.0 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".

Document Uncontrolled When Printed

13.0 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 SOP TA-EHS-0036.

13.1 Waste Streams Produced by the Method

13.1.1 Vialed extract waste. Sample extracts that have been placed in vials for analysis are discarded into plastic satellite waste buckets labeled "Hazardous Waste" located underneath the bench top. Once the buckets are full the GC vials are bulked into the non-PCB GC vial waste barrel located in the waste room and sent out for incineration.

13.1.2 Solvent/Methylene Chloride waste. Any waste methylene chloride/hexane is collected in beakers and then poured into a 4-liter amber bottle labeled "Hazardous Waste" located in the hood. After the extraction has been completed the $MeCl_2$ collected in the 4 L bottles is emptied into the $MeCl_2$ satellite waste barrel located next to the neutralization tank in lab hood #17. The funnel lid on the drum must be closed after each use At or before the satellite waste reaches 55 gallons the barrel is transferred to the waste disposal room from where it is sent out for recycling or fuel blending.

13.1.3 Extract waste. Unused sample extracts are held for at least 40 days, in case further testing is deemed necessary. After at least 40 days has passed these extracts are transported to the waste room in racks of 100 were they are bulked into a flammable loose pack waste stream and sent out for incineration.

13.1.4 Any used pipettes or disposable glassware is to be discarded in the broken glassware boxes located next to each vialing station. The box needs to be changed before it becomes too full and cannot be closed with out risk of cuts or pokes.

14.0 References / Cross-References

14.1 US EPA Method 8011, "1,2-Dibromoethane 1,2-Dibromo-3-chloropropane by Microextraction and Gas Chromatography", SW-846, Rev. 0, July 1992.

14.2 US EPA Method 8000B, "Determinative Chromatographic Separations", SW-846, Rev. 2, 1996.

14.3 US EPA Method 8000C, "Determinative Chromatographic Separations", SW-846, Rev. 3, 2003.

14.4 US EPA Method 504.1, "EDB, DBCP and 123TCP in Water by Microextraction and Gas Chromatography, 1993.

15.0 Attachments

None

16.0 Revision History

- Revision 10, dated 7 May 2013
  - Changed column type to Zebron Multi Residue 1 and 2 in sections 6.1 and 10.2
  - Removed 1,2,3-Trichloropropane from scope in section 1.1
  - Removed references to soil extraction.
  - Changed CCV acceptance criteria to 15%, per table in method 504.1
  - Added daily analysis of check standard at 0.2 ug/L

- Removed alternating CCV concentration level criteria.
- Added additional information about carcinogens to section 5.1

- Revision 9, dated 2 April 2012
  - Added soil prep procedures in section 10.2.
  - Incorporated ROMD 00033 in section 10.4.2.4
  - Added maintenance procedures in section 10.5

- Revision 8, dated 23 April 2010
  - Declared primary and secondary columns, sections 6.1 and 10.3.3.3
  - Revised supplier and catalog number of second source standard, section 7.9
  - Added section 9.1.3
  - Added LCS frequency for 5.4.1, Section 9.4
  - Added DOD-specified CCV frequency, sections 9.7.2.2 and 10.2.5.1
  - Added DOD-specific corrective actions, Sections 9.3, 9.4 and 9.7.2.4.2
  - Added 504.1 reference, section 14.4

- Revision 7, dated 28 January, 2010
  - Added container information throughout
  - Updated sample volume throughout
  - Added column information Section 10.2.1
  - Added audit trail information Section 6.1
  - Added documentation of standards/reagents and standard/reagent preparation Section 7.1
  - Added removal of expired standards Section 7.10
  - Added criteria for extra QC, Section 9.7.
  - Added requirement for balance calibration Section 10.1
  - Added column information Section 10.2.1
  - Added BP LaMP requirements to relevant sections

- Revision 6, dated 17 August 2009
  - Integration for TestAmerica Bothell and TestAmerica Tacoma operations.
- Revision 5, dated 4 August 2008
  - Updated Sections 9.4.1 and 10.7 to specify that CCB/IBL are to be fortified with surrogate.
  - Updated Section 10.8.2 to indicate the lower concentration of the two results for BP.

Title: Toxicity Characteristic Leaching Procedure (TCLP, Method 1311) and Synthetic Precipitation Leaching Procedure (SPLP, Method 1312)

Approvals

Signatures on File

Colin McKean	Date	Sandra Langway	Date
Semivolatile Organic Department Manager		Health & Safety Coordinator	
Terri Torres	Date	Heather Prater	Date
Quality Assurance Manager		Laboratory Director	

Copyright Information:
This documentation has been prepared by TestAmerica Laboratories, Inc. and its affiliates ("TestAmerica"), solely for their own use and the use of their customers in evaluating their qualifications and capabilities in connection with a particular project. The user of this document agrees by its acceptance to return it to TestAmerica upon request and not to reproduce, copy, lend, or otherwise disclose its contents, directly or indirectly, and not to use it for any other purpose other than that for which it was specifically provided. The user also agrees that where consultants or other outside parties are involved in the evaluation process, access to these documents shall not be given to said parties unless those parties also specifically agree to these conditions.

THIS DOCUMENT CONTAINS VALUABLE CONFIDENTIAL AND PROPRIETARY INFORMATION. DISCLOSURE, USE OR REPRODUCTION OF THESE MATERIALS WITHOUT THE WRITTEN AUTHORIZATION OF TESTAMERICA IS STRICTLY PROHIBITED. THIS UNPUBLISHED WORK BY TESTAMERICA IS PROTECTED BY STATE AND FEDERAL LAW OF THE UNITED STATES. IF PUBLICATION OF THIS WORK SHOULD OCCUR THE FOLLOWING NOTICE SHALL APPLY:

©COPYRIGHT 2012 TESTAMERICA LABORATORIES, INC. ALL RIGHTS RESERVED.

Facility Distribution No. 001 **Distributed To:** Lab Intranet

1.0 Scope and Application

1.1 Both TCLP and SPLP are designed to determine the mobility of both organic and inorganic analytes present in liquid, solid, and multiphasic wastes.

1.2 If a total analysis of the waste demonstrates that individual analytes are not present in the waste, or that they are present but at such low concentrations that the appropriate regulatory levels could not possibly be exceeded, the TCLP or SPLP need not be run.

1.3 If an analysis of any one of the liquid fractions of the TCLP or SPLP extract indicates that a regulated compound is present at such high concentrations that, even after accounting for dilution from the other fractions of the extract, the concentration would be above the regulatory level for that compound, then the waste is hazardous and it is not necessary to analyze the remaining fractions of the extract.

1.4 If an analysis of extract obtained using a bottle extractor shows that the concentration of any regulated volatile analyte exceeds the regulatory level for that compound, then the waste is hazardous and extraction using the ZHE is not necessary. However, extract from a bottle extractor cannot be used to demonstrate that the concentration of volatile compounds is below the regulatory level.

1.5 On occasion, clients may request slight modifications to this SOP. These modifications are addressed on a case by case basis with the supporting demonstration of sensitivity and accuracy (e.g. MDLs, linearity check or PT sample) verified prior to implementation. Any modifications would be written into project special instructions (e.g. Quality Assurance Project Plans), authorized by the laboratory, and mentioned in the report narrative.

2.0 Method Summary

2.1 For liquid wastes (i.e., those containing less than 0.5% dry solid material), the waste, after filtration through a 0.7 µm glass fiber filter, is defined as the TCLP extract.

2.2 For liquid wastes (i.e., those containing less than 0.5% dry solid material), the waste, after filtration through a 0.6 µm to 0.8 µm glass fiber filter, is defined as the SPLP extract.

2.3 For wastes containing greater than or equal to 0.5% solids, the liquid, if any, is separated from the solid phase and stored for later analysis; the particle size of the solid phase is reduced, if necessary. The solid phase is extracted with an amount of extraction fluid equal to 20 times the weight of the solid phase. The extraction fluid employed is a function of the alkalinity of the solid phase of the waste. A ZHE vessel is used when testing for volatile analytes. Following extraction, the liquid extract is separated from the solid phase by filtration through the appropriate glass fiber filter.

2.4 If compatible (i.e., multiple phases will not form on combination), the initial liquid phase of the waste is added to the liquid extract, and these are analyzed together.

2.5 If incompatible, the liquids are analyzed separately and the results are mathematically combined to yield a volume-weighted average concentration.

3.0 Definitions

3.1 Batch: A group of 20 or less samples prepared and/or processed together within the same shift using the same reagents. Each batch must contain a minimum QC of one method blank.

3.2 Leachate: The TCLP or SPLP solution generated after the solids are tumbled with the appropriate fluid.

3.3 Liquid Phase: The liquid fraction or filtrate of a sample that passes through a 0.6 to 0.8µm fiber filter.

3.4 Final Leachate: The final solution generated from this procedure – either a leachate or a leachate combined with a filtrate.

3.5 Percent Solids: The fraction of a sample (as a percentage of the total sample) from which no liquid may be forced out by an applied pressure.

3.6 Solid phase: The material in the filter holder that did not pass through a 0.6 to 0.8µm fiber filter.

4.0 Interferences and Comments

4.1 Oily samples may present unusual filtration and drying problems. For example, the oily sample may pre-maturely clog the filter used in the percent wet solids determination, causing a high-biased result for percent wet solids. As recommended by EPA (see Figure 3 in Appendix B), oily samples will be assumed to be 100% liquid and analysis for total concentrations of contaminants will be performed. This applies specifically to samples containing viscous non-aqueous liquids that would be difficult to filter. For oily samples requiring metals analysis, a suitable sub-lab will be procured. For oily wastes requiring semi-volatile organic analysis, the sample should be logged into the LIMS as a solid matrix for waste dilution extraction. For oily samples requiring volatile organic analysis, the sample should be logged into the LIMS as an aqueous matrix for analysis.

4.2 Samples containing free organic liquids (i.e., those with separable non-aqueous liquid phases) will be assumed to be 100% liquid and totals analysis will be performed to determine if the sample exceeds TCLP limits.

4.3 Phthalates may be eliminated by proper glassware cleanup and by avoiding plastics. Only glass, Teflon or Type 316 stainless steel tumblers may be used for leachates to be analyzed for organics. Plastic tumblers may be used for leachates to be analyzed for the metals.

4.4 Over exposure of the sample to the environment will result in the loss of volatile components.

4.5 Potential interferences that may be encountered during analysis are discussed in the individual analytical methods.

4.6 The laboratory analyst will perform the method in accordance with this SOP. The analyst will resolve non-conformances in methods and data, either individually or with the assistance of the Department Supervisor or Operations Manager. Deviations from this SOP must be documented. Bench sheets and raw data must capture information related to a deviation. The laboratory analyst or supervisor will report deviations or non-conforming events to the Operations, Project and/or QA Manager via a non-conformance report.

4.7 The Department Supervisor, Operations Manager, and/or QA Manager will assist the laboratory analyst in resolving non-conformances.

4.8 The Department Supervisor will review and approve bench sheets and notebooks.

4.9 The QA Manager shall monitor adherence to this SOP through annual audits and non-conformance reports.

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.

Specific safety concerns or requirements as they related to this procedure include.

5.1 The importation and movement of untreated soil is considered by APHIS to be an extremely high-risk activity, because it can readily provide a pathway for the introduction of a variety of dangerous organisms into the United States. In addition to containing high concentrations of hazardous and or toxic compounds, regulated soils and effluents may contain plant, animal or human disease agents.

5.2 Spills of regulated soil and water residue are to be handled as follows:

Spray generously with bleach, before attempting to clean the spill up to prevent the release of airborne contaminates. The area should be cleaned with bleach again after the spill has been removed. Place all clean up material, spilled sample and PPE into a sealable container for autoclaving.

5.3 The filtration of all TCLP samples and extracts are performed in a hood or well-ventilated area. All excess sample/ extract waste is discarded into TCLP (Toxicity Characteristic Leaching Procedure) labeled waste containers for hazardous waste disposal.

5.4 Unknown samples may contain high concentrations of toxic volatile compounds. Sample containers are to be opened in a hood and handled with gloves to prevent exposure.

5.5 Glassware should be inspected for chips or cracks before use. Chipped/broken glassware that poses a safety hazard must be removed from service and repaired or discarded.

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 standards 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. Electronic copies of MSDS can be located on the EH&S webpage of Oasis.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Acetic Acid	Corrosive Poison Flammable	10 ppm-TWA	Contact with concentrated solution may cause serious damage to the skin and eyes. Inhalation of concentrated vapors may cause serious damage to the lining of the nose, throat, and lungs. Breathing difficulties may occur.
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, severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Nitric Acid	Corrosive Oxidizer Poison	2 ppm-TWA 4 ppm-STEL	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Sodium Hydroxide	Corrosive	2 Mg/M3-Ceiling	Severe irritant. Effects from inhalation of dust or mist vary from mild irritation to serious damage of the upper respiratory tract, depending on severity of exposure. Symptoms may include sneezing, sore throat or runny nose. Contact with skin can cause irritation or severe burns and scarring with greater exposures. Causes irritation of eyes, and with greater exposures it can cause burns that may result in permanent impairment of vision, even blindness.
1 – Always add acid to water to prevent violent reactions			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Documentation and Maintenance

6.1 All maintenance performed on any TCLP/SPLP agitation apparatus, ZHE extraction vessel, or Millipore filtration apparatus must be documented in a TCLP Maintenance logbook.

6.2 ZHE Extraction Vessel Pressure Checks

6.2.1 Each ZHE extraction vessel undergoes a one-hour pressure check before each use to identify any major pressure-related malfunctions **prior** to extraction.

6.2.1.1 Fully assemble and pressurize the device to 50 psi, allow it to stand unattended for 1 hour, and recheck the pressure. If pressure is lost, check all fittings and inspect and replace O-rings, if necessary. Retest the device. If leakage problems cannot be solved, the manufacturer must be contacted**.** Recheck the pressure every hour for four hours when doing the monthly pressure check. Log all results in the ZHE pressure check log.

6.2.2 A 4-hour pressure check on each ZHE extraction vessel must be performed and documented monthly. This is done to check for any slow leaks that pass inspection otherwise.

6.2.3 Refer to Appendix A for a template of the ZHE Pressure Check Log.

6.3 All TCLP/SPLP agitation devices must be checked monthly, under a full load, to insure they rotate at 30±2 rpm. Document their rotation in the TCLP Tumbler Check Log (see Appendix B).

6.4 All ZHE metal filter screens are sent through the sonic bath for 15 minutes (in enough MeOH or DIW to cover all the screens) after each use to shake out any sample that may be clogged in the pores of the filters.

6.5 ZHE O-rings should be inspected prior to each use for tears and contamination, and their replacement documented in the ZHE Pressure Check Logbook.

6.6 All extraction vessel Blanks must be documented in the appropriate TCLP/ZHE Logs. (Appendices D & E)

6.7 The ambient temperature must be recorded on the appropriate log sheet. If the *corrected* temperature range during an extraction falls outside the method-specified range of 23 ± 2°C, initiate a NCM as prescribed in TA-QA-610.

7.0 Equipment and Supplies

7.1 Agitation apparatus – Tumbler #1 and #2-Environmental Express Multi-Place, 10 positions, capable of maintaining a tumbling speed of 30± 2 rpm (or equivalent device).

Extraction Vessels

7.1.1 Millipore ZHE Extraction system, Zero-Headspace Extraction Vessels (ZHE), *uniquely* numbered.

Note: This device is for use only when the waste is being tested for the mobility of **volatile** analytes. The ZHE allows for liquid/solid separation within the device, and effectively precludes headspace. For the ZHE to be acceptable for use, the piston within the ZHE must be able to be moved with approximately 15 psi or less. If it takes more pressure to move the piston, the O-rings in the device must be replaced. If this does not solve the problem, the ZHE is unacceptable for TCLP analyses and the manufacturer must be contacted.

7.1.2 ESS 128oz pre-cleaned 1 gallon disposable poly bottles-one time use only

7.1.3 ESS 128oz pre-cleaned 1 gallon disposable borosilicate glass bottles-one time use only.

Note: When the waste is being evaluated using the nonvolatile extraction, a jar with sufficient capacity to hold the sample and the extraction fluid is needed. Headspace is allowed in this vessel. Plastic bottles, other than polytetrafluoroethylene (PTFE), shall not be used if organics are to be investigated.

7.2 Filtration Devices

7.2.1 Positive pressure filtration device -- Millipore Hazardous Waste Filtration System.

7.2.2 ZHE Filters: TCLP Glass Fiber 0.7µm, 90mm. Whatman cat # 1810-090

7.2.3 TCLP Filters: Borosilicate Acid Washed Glass Fiber Filters, pore size 0.6 to 0.8µm, 142 mm. Whatman acid washed low level cat # 1810-142

Note: Glass fiber filters are fragile and should be handled with care.

7.3 pH Meters: Denver Instruments pH meter (or equivalent), resolution to 0.01 pH units.

7.4.1 Calibration of the Denver Instruments pH meter:

7.4.1.1 Turn-on the unit.

7.4.1.2 Dip electrode ½" to 1" into buffer solution of pH = 4.00

7.4.1.3 Press the standardize button to enter Calibration mode. When the 'S' appears on the screen, remove the probe, rinse, and repeat with buffer solution pH 7.00 and 10.00.

7.4.1.4 After reading buffer 10, the calculated slope will appear on the screen. Document this in the TCLP logbook and verify it is within acceptance criteria before proceeding.

7.4.1.5 Calibration with a second source pH = 7.00 is required also and is recorded in the TCLP logbook and must fall with in 7.00 +/- .05.

7.4.1.6 If the probe does not calibrate properly you must perform maintenance and repeat the process.

7.4.2 pH testing with the Denver Instruments pH meter.

7.4.2.1 Turn on the unit

7.4.2.2 Dip the electrode ½" to 1" into the test solution. Stir once and let the reading stabilize. Record the pH to 0.01 pH unit in TCLP logbook or in the TALS worksheet.

7.4 FT Variable Speed Metering Pump (ZHE pump) for transferring ZHE extraction fluid.

7.5 Top-Loading Balance: capable of reading to 0.1g.

7.6 Beaker, plastic, 200 mL

7.7 500 mL and 2000 mL Graduated cylinders

7.8 Watch glass, appropriate diameter to cover beaker.

7.9 Hot plate.

7.10 Wood spatulas or tongue depressors (or equivalent)

7.11 Magnetic stir bar.

7.12 Teflon Boiling Chips

7.13 Teflon Tape

7.14 Centrifuge, Beckman TJ-6 or Equivalent

7.15 Narrow range pH strips- pH 0-6, pH 5-10, pH 7.5-14

8.0 Reagents and Standards

All reagents and standards used in this procedure must conform to the requirements specified in TA-QA-0619. Preparation of reagents and standards will be documented in the LIM's system and a label will be generated to include, as appropriate: the name and expected concentration of the material; the generated LIM's ID number; the date of preparation; the expiration date; the analyst's initials.

8.1 pH Buffers for meter calibration, pH 4, pH 7 and pH 10.

8.2 Glacial acetic acid (CH_3COOH)

8.3 Deionized Water.

8.4 1N Hydrochloric acid (HCl).

8.4.1 In a 1 liter volumetric flask, add 83 mL of concentrated HCl, then dilute to 1 liter with reagent water. Document in the reagent module in TALS, generate a label and place on the bottle.

8.5 Nitric acid (HNO_3).

8.6 2% Nitric acid solution for rinsing TCLP filter.

8.6.1 In a 1L poly wash bottle add between 500 and 800 mls of DI water. Add 20mls of Nitric acid, top off to 1000mls with DI water, and mix.

8.7 Sodium Hydroxide Pellets (NaOH).

8.8 1N Nitric acid (HNO_3)

8.8.1 In a 1 liter volumetric flask, add 64 mL of concentrated HNO_3, then dilute to 1 liter with reagent water. Document in the reagent module in TALS, generate a label and place on the bottle.

8.9 1N Sodium hydroxide (NaOH).

8.9.1 In a 1 liter volumetric flask, add 250 mLs of reagent water. Dissolve 40g of NaOH pellets into this, adding more reagent water as it slowly dissolves. Dilute to a final volume of 1 liter with reagent water. Document in the reagent module in TALS, generate a label and place on the bottle.

8.10 Sulfuric acid/nitric acid (60/40 weight percent) mixture

8.10.1 The amount specified to be used in the extraction fluid preparation is so minimal that it is useful to create a 100X dilution of the mix to increase the measuring accuracy. Amounts are not specified in the EPA method. Only the final pH of the extraction fluid is specified. Therefore, these amounts may need to be adjusted to ensure proper pH.

8.11 Extraction fluids.

8.11.1 The extraction fluids are prepared in-house by the technician using the procedure described below.

8.11.2 TCLP Fluid Mix #1 (Concentrate)

8.11.2.1 In a carboy, dissolve approximately 1040 g NaOH pellets into 10 L reagent water.

8.11.2.2 Add approximately 2.3 L glacial acetic acid to carboy.

8.11.2.3 Dilute to a volume of 20 liters. When correctly prepared, the pH of this fluid will be 4.93 $\pm$ 0.05.

8.11.3 TCLP Fluid # 1 for TCLP and ZHE

from concentrate

8.11.3.1 Add 1 L TCLP Fluid Mix #1 (Concentrate) to 19 L reagent water in a carboy.

8.11.3.2 When correctly prepared, the pH of this fluid will be 4.93 $\pm$ 0.05.

8.11.3.3 Check the pH to 0.01 pH unit daily before use. Document the checked pH as the initial pH of the MB in the TALS prep batch worksheet.

8.11.3.3.1 If the pH isn't within this range, a new solution must be prepared.

-OR- Not from concentrate

8.11.3.4 Add approximately 115 mL glacial acetic acid to 15 L of reagent water in a carboy.

8.11.3.5 Add approximately 1300 mL of 1N NaOH.

8.11.3.6 Dilute to a volume of 20 liters. When correctly prepared, the pH of this fluid will be 4.93 $\pm$ 0.05.

8.11.3.7 Check the pH to 0.01 pH unit daily before use. Document the checked pH as the initial pH of the MB in the TALS prep batch worksheet.

8.11.3.7.1 If the pH isn't within this range, a new solution must be prepared.

8.11.4 TCLP Fluid # 2

8.11.4.1 Add 5.7 mL glacial acetic acid to 500 ml reagent water.

8.11.4.2 Bring to a final volume of 1 liter. When correctly prepared, the pH of this fluid will be 2.88 ± 0.05.

8.11.4.3 Check the pH to 0.01 pH unit daily before use. Document the checked pH as the initial pH of the MB in the TALS prep batch worksheet.

8.9.3.3.1 If the pH isn't within this range, a new solution must be prepared.

8.11.5 SPLP Fluid #1

8.11.5.1 Add 29.25 uL of the 60/40 sulfuric acid/nitric acid mix (8.8) to 18 liters of reagent water in a carboy. If using the 100X acid dilution then add 2.925 mLs of acid to 18 liters of DI water.

8.11.5.2 Dilute to a volume of 20 liters.

8.11.5.3 When correctly prepared, the final pH of this fluid should be 4.20 ± 0.05.

8.11.5.4 Check the pH to 0.01 pH unit daily before use. Document the checked pH as the initial pH of the MB in the TALS prep batch worksheet.

8.9.5.4.1 If the pH isn't within this range, a new solution must be prepared.

8.11.6 SPLP Fluid #2

8.11.6.1 Add 6.75 uL of the 60/40 sulfuric acid/nitric acid mix (8.8) to 18 liters of reagent water in a carboy. If using the 100X acid dilution then add 0.675 mLs of acid to 18 liters of DI water.

8.11.6.2 Dilute to a volume of 20 liters.

8.11.6.3 When correctly prepared, the final pH of this fluid should be 5.00 ± 0.05.

8.11.6.4 Check the pH to 0.01 pH unit daily before use. Document the checked pH as the initial pH of the MB in the TALS prep batch worksheet.

8.11.6.4.1 If the pH isn't within this range, a new solution must be prepared.

8.11.7 SPLP Fluid #3

8.11.7.1 Dionized water

8.12 Managers/supervisors or a designee are expected to check their areas on a monthly basis for expired standards/reagents and dispose of them according to SOP TA-EHS-0036.

Note: These extraction fluids must be documented in the TALS and monitored frequently for impurities. The pH should be checked prior to use to ensure that these fluids are made up accurately. If impurities are found or the pH is not within the above specifications, the fluid must be discarded and fresh extraction fluid prepared.

9.0 Sample Collection, Preservation, Shipment and Storage

9.1 Preservatives shall not be added to samples before extraction.

9.2 Samples are refrigerated at 4°C unless refrigeration results in irreversible physical change to the waste. If precipitation occurs, the entire sample (including precipitate) is extracted.

9.3 When the waste is to be evaluated for volatile analytes, care must be taken to minimize the loss of volatiles. Samples are stored at 4°C to prevent the loss of volatile analytes. Samples are opened only immediately prior to extraction.

9.4 Hold times and minimum sample amounts are specified in Table 1.

9.5 TCLP and SPLP extracts are prepared for analysis and analyzed as soon as possible following extraction. Extracts or portions of extracts for volatile organic analyte determinations are not allowed to come into contact with the atmosphere any longer than is absolutely necessary (i.e., no headspace) to prevent losses. Extracts or portions of extracts for metallic analyte determinations are acidified with nitric acid to a pH < 2, unless precipitation occurs. The metals prep group will adjust the pH so they can add their spike solutions prior to the pH adjustment. Extracts are preserved for other analytes according to the guidance given in the individual analysis methods.

10.0 Quality Control

10.1 Method Blanks

10.1.1 A minimum of one blank (using the same extraction fluid as used for the samples) must be analyzed for every 20 extractions that have been conducted in an extraction vessel.

10.1.2 The method blank should contain no analyte(s) of interest at concentrations greater that the method reporting limit.

10.1.3 If method blank failure occurs, a Non-Conformance Report must be initiated, and all samples associated with the analytical batch must be reprocessed.

10.2 Samples must undergo TCLP or SPLP extraction within the time periods outlined in Table 01. If sample holding times are exceeded, the values obtained will be considered minimal concentrations. Exceeding the holding time is not acceptable in establishing that a waste does not exceed the regulatory level. Exceeding the holding time will not invalidate characterization if the waste exceeds the regulatory level.

10.3 A matrix spike and matrix spike dup must be performed for each waste type unless the result exceeds the regulatory level and the data are being used solely to demonstrate that the waste

property exceeds the regulatory level. This is performed post TCLP as part of the Organic extraction or Inorganic digestion process.

10.3.1 Matrix spike solutions are to be added after filtration of the TCLP or SPLP extract and before preservation.

10.4 Any extra QC that is analyzed in a batch or sequence must be evaluated using the same criteria as the corresponding QC above.

Note: An LCS/LCSD set is created on the TCLP/SPLP/ZHE prep batch but are only there for use when preparing batches for post TCLP processes. Each department uses a portion of the MB extract to create the LCS and LCSD, if necessary. The information in the TCLP prep batch for the LCS and LCSD should be documented identical to the MB.

11.0 Procedure

One-time procedural variations are allowed only if deemed necessary in the professional judgment of the department manager to accommodate variation in sample matrix, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using an NCM. The NCM is approved by the supervisor and then automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group also receives NCMs by e-mail for tracking and trending purposes. The NCM process is described in more detail in SOP TA-QA-0610. The NCM shall be filed in the project file and addressed in the case narrative.

Any deviations from this procedure identified after the work has been completed must be documented in an NCM, with a cause and corrective action described.

Document the results for each sample preparation procedure in TALS (worksheet)

NOTE: SAMPLE PREPARATION
The TCLP extraction depends on the analysis and matrix of each sample. Before the extraction process can be started, the following preliminary evaluations must take place:

- Determination of the percent solids
- Determination of whether the waste contains insignificant solids and is, therefore, its own extract after filtration.
- Determination of whether the solid portion of the waste requires particle size reduction.
- Determination of which of the two extraction fluids are to be used for the nonvolatile TCLP extraction of the waste.

A **minimum** 100 g aliquot of waste is required to perform these preliminary evaluations. This aliquot may not actually undergo TCLP extraction.

11.1 Preliminary Evaluations for TCLP and SPLP

Check the Balance logbook to determine if the daily calibration check has been completed. If it has not, the analyst must perform this check according to SOP TA-QA-0014.

11.1.1 Percent Solids Determination

Percent solids are defined as that fraction of a waste sample from which no liquid may be forced out by an applied pressure.

11.1.1.1 If the waste will obviously yield no liquid when subjected to pressure filtration at 50 PSI for 2 minutes, it is 100% solid. Proceed to section 11.1.2 to determine whether the solid material requires particle size reduction.

11.1.1.2 If the sample is liquid and is obviously <0.5% solids (for instance, a clear sample with no particles present in the sample), then it is 0% solids. If it contains particulates or is cloudy, then a percent solids determination must be performed as described in 11.1.1.3.

Note: In practice, the analyst has been contacting the Project Manager regarding multiphasic samples. If the client requests that each phase be treated separately, percent solids determination is performed on the individual phases. Otherwise, the phases (usually a settled solid layer, and a liquid layer) are homogenized prior to the determination.

11.1.1.3 If the sample is multiphasic, each layer must be separated and a percent solids determination must be performed on each layer.

The sample, a receiving container, and a filter paper are needed for this evaluation.

11.1.1.3.1 Pre-weigh and record each of the individual weights to the nearest tenth of a gram for the sample, the filter, and the container that will receive the filtrate.

11.1.1.3.2 Homogenize the sample with a wood spatula. Weigh the sample of the waste (100 g minimum), and record the weight to the nearest tenth of a gram.

11.1.1.3.3 Allow slurries to stand to permit the solid phase to settle.

Note: If filtration of the waste at 4°C reduces the amount of expressed liquid over what would be expressed at room temperature then allow the sample to warm up to room temperature in the device before filtering.

11.1.1.3.4 Quantitatively transfer the waste sample into the filtration device (liquid and solid phases). Spread the waste sample evenly over the surface of the filter.

Note: If waste material (>1% of original sample weight) has obviously adhered to the container used to transfer the sample to the filtration apparatus, determine the weight of this residue and subtract it from the sample weight to determine the weight of the waste sample that will be filtered.

11.1.1.3.5 Gradually apply vacuum or gentle pressure of 1-10 psi, until air or pressurizing gas moves through the filter. If this point is not reached under 10 psi, and if no additional liquid has passed through the filter in any 2 minute interval, slowly increase the pressure in 10 psi increments to a maximum of 50 psi. After each incremental increase of 10 psi, if the pressurizing gas has not moved through the filter, and if no additional liquid has passed through the filter in any 2 minute interval, proceed to the next 10 psi increment. When the pressurizing gas begins to move through the filter, or when liquid flow has ceased

at 50 psi (i.e., filtration does not result in any additional filtrate within any 2 minute period), stop the filtration.

Note: Instantaneous application of high pressure can degrade the glass fiber filter and may cause premature plugging.

11.1.1.3.6 The material in the filter holder is defined as the solid phase of the waste, and the filtrate is defined as the liquid phase.

Note: Some wastes, such as oily wastes and some paint wastes, will obviously contain some material that appears to be a liquid. Even after applying vacuum or pressure filtration, as outlined above, this material may not filter. If this is the case, the material within the filtration device is defined as a solid. Do not replace the original filter with a fresh filter under any circumstances. **Use only one filter**.

11.1.1.3.7 Determine the weight of the liquid phase by subtracting the weight of the filtrate container from the total weight of the filtrate-filled container.

11.1.1.3.8 Determine the weight of the solid phase of the waste sample by subtracting the weight of the liquid phase from the weight of the total waste sample.

11.1.1.3.9 Record the weight of the liquid and solid phases to the nearest tenth of a gram on the bench sheet.

11.1.1.3.10 Calculate the percent solids as follows:

$$\% \text{ Solids} = \frac{\text{Weight of solid}}{\text{Total weight of waste}} \times 100$$

11.1.1.3.11 If the percent solids are less than 0.5%, then the filtrate is the sample. Proceed to section 11.2.1.3.

11.1.1.3.12 If the percent solids are greater than or equal to 0.5% and it's noticed that a small amount of the filtrate is entrained in the wetting of the filter, determine the percent dry solids.

11.1.1.3.12.1 Remove the solid phase and filter from the filtration apparatus.

11.1.1.3.12.2 Dry the filter and solid phase at 100 ± 20°C. Weigh the filter/solid phase every hour until two successive weights yield the same value within ± 1%.

Note: Use the properly-vented Percent Solids oven for drying these samples.

11.1.1.3.12.3 Record the final weight.

11.1.1.3.12.4 Calculate the percent dry solids as follows:

$$\% \text{ Dry Solids} = \frac{(\text{Wt. of dry waste + filter}) - \text{tared weight of filter}}{\text{Initial weight of waste}} * 100$$

11.1.1.3.12.5 If the percent dry solids is <0.5%, then the waste phase is 0% solids.

11.1.1.3.12.6 If the percent dry solids is ≥ to 0.5%, then proceed to section 11.1.2, Particle Size Reduction.

11.1.1.3.13 If the percent solids are greater than or equal to 0.5%, then proceed to section 11.1.2 to determine whether the solid material requires particle size reduction.

Note: If organic or volatile compounds are to be analyzed, then you must extract/ filter a fresh portion of the sample.

11.1.2 Particle Size Reduction for TCLP and SPLP

This section determines whether the waste requires particle size reduction. This section also describes how to reduce particle size.

11.1.2.1 Using the solid portion of the waste, evaluate the solid for particle size. Particle size reduction is required unless:

11.1.2.1.1 The solid has a surface area per gram of material equal to or greater than 3.1 cm^2.

11.1.2.1.2 The solid is smaller than 1 cm in its narrowest dimension (i.e., is capable of passing through a 9.5 mm (0.375 inch) standard sieve).

Note: Do not sieve the waste if volatiles are being analyzed because some of the volatiles may be lost.

11.1.2.2 If particle size reduction is required, then:

11.1.2.2.1 Prepare the solid portion of the waste for extraction by crushing, cutting, grinding, mashing, ripping, or tearing the waste to a surface area or particle size as described above. Care is taken to reduce contamination of samples by washing the tools used in particle size reduction with water and gentle soap, and rinsing 3 times with DI water.

11.1.2.2.2 If the solids are prepared for organic volatiles extraction, special precautions must be taken so that heat is not generated from the reducing process. The sample, and the tools to be used, should be refrigerated at 4˚ C prior to particle size reduction.

Note: Surface area criteria are meant for filamentous (e.g., paper, cloth, and similar) waste materials. Actual measurement of surface area is not required, nor is it recommended. For materials that do not obviously meet the criteria, sample specific methods would need to be developed and employed to measure the surface area. Such methodology is currently not available.

11.1.2.3 If particle size reduction is required, but not feasible with the available apparatus and equipment, notify the department supervisor and initiate a non-conformance report.

11.1.3 Extraction fluid determination for TCLP and SPLP

This section determines the appropriate TCLP extraction fluid.

11.1.3.1 If the solid content of the waste is greater than or equal to 0.5% and if the sample will be extracted for nonvolatile constituents, determine the appropriate fluid for the nonvolatile extraction as follows:

Note: TCLP extraction for volatile constituents (ZHE) uses only extraction fluid #1 and is prepared with water proven to be interference free.

Note: For Non-aqueous liquid samples, such as products, that need to go through the tumbling process, a pH test is not necessary.

11.1.3.1.1 Weigh out 5g of the sample into a 200 mL plastic beaker. Reduce the solid (if necessary) to a particle size of approximately 1 cm in diameter or less.

11.1.3.1.2 Add 96.5 mL of deionized water to the beaker, drop in a stir bar, cover with a watch glass, and stir vigorously on a magnetic stirrer for 5 minutes. Check the pH and if it is <5.0, use extraction fluid #1. Record the pH in the worksheet in TALS.

11.1.3.1.3 If the pH is >5.0, add 3.5 mL 1N HCl, stir on medium, cover with a watch glass, heat to 50°C ± 5°C on a hot plate. Hold at 50°C ± 5°C for 10 minutes ± 1 minute.

11.1.3.1.4 Remove the watch glass and let the solution cool to room temperature, (around ½ an hour), test and record the pH in TALS.

If the pH is <5.0, use TCLP Fluid #1. Record the lot number of the fluid in the worksheet in TALS.

11.1.3.1.5 If the pH is >5.0, use TCLP Fluid #2. Record the lot number of the fluid in the worksheet in TALS.

11.1.3.1.6 Record the pH to 0.01 pH unit that resulted from this procedure in the worksheet in TALS.

This section determines the appropriate SPLP extraction fluid

11.1.3.2 If the solid content of the waste is greater than or equal to 0.5% and if the sample will be extracted for nonvolatile constituents, determine the appropriate fluid for the nonvolatile extraction as follows:

Note: SPLP extraction for volatile constituents (ZHE) and cyanide uses only SPLP Fluid #3 (Purged reverse osmosis type III water).

11.1.3.2.1 SPLP Fluid #1 is used to determine the leachability of contaminants from soils originating from east of the Mississippi River, as well as the leachability of wastes and wastewaters.

11.1.3.2.2 SPLP Fluid #2 is used to determine the leachability of contaminants from soils originating from west of the Mississippi River.

List of General Analyses for TCLP and SPLP

Table 1 – TCLP and SPLP Method Specifications and Recommended Sample.

Analysis	Type extraction vessel	Where filtered	Type extract container	QC	Extract preservative	Hold time to TCLP	Hold time to extract	Mass solid phase (minimum # grams)	Vol. Final Extract (mLs)
Metals	1L HDPE Poly	Plunger Filters	500mL HDPE Poly or Evergreen tubes	Blank/BS/ MS/MSD every 20 or per batch	pH<2 HNO3 unless Sx precipitates	180 days. Mercury= 28 days	180 days. Mercury =28 days	100	20X mass of solid
Organics in general	2L Teflon	Millipore	1L Amber glass or 40 mL glass VOA	Refer to specific method	Refer to specific method	Refer to specific method	Refer to specific method	Refer to specific method (Usually 100)	20X mass of solid, or refer to method for liquids
8270 C Semi-volatiles	2L Teflon	Millipore	1L Amber glass	Blank/BS/ MS/MSD every 20 or per batch	None	7 days water. 14 days solid	7 days	100	20X mass of solid
Volatiles	ZHE extraction vessel	ZHE extraction vessel	5 mL gass-tight glass syringe	Blank/ 20	No headspace	14 days	14 days	25	20X mass of solid
8081A 8082B Pesticides/ PCB's	2L Teflon	Millipore	1L Amber glass	Blank/BS/ MS/MSD every 20 or per batch	None	7 days water. 14 days solid	7 days	100	20X mass of solid
8310 PAH	2L Teflon	Millipore	1L Amber glass	Blank/BS/ MS/MSD every 20 or per batch	None	7 days water. 14 days solid	7 days	100	20X mass of solid
8015 TPH-D	2L Teflon	Millipore	1L Amber glass	Blank/BS/ MS/MSD every 20 or per batch	None	7 days water. 14 days solid	7 days	100	20X mass of solid
8151 Herbicide	2L Teflon	Millipore	1L Amber glass	Blank/BS/ MS/MSD every 20 or per batch	None	7 days water. 14 days solid	7 days	100	20X mass of solid

Note: The TCLP and SPLP methods require 100 g minimum sample amount. If less than 100 g is used, a Non-Conformance Memo must be issued. ZHE samples use 25 g, and do not require a NCM unless less than 25 g is available.

11.2 TCLP and SPLP Non-Volatiles Extraction Procedure

NOTE: Document sample information in the TCLP/ SPLP Sample Log (see Appendices C & D).

11.2.1 Procedure by matrix

11.2.1.1 If the sample is determined to be 100% solid and the preparation steps in section 11.1 were completed, then weigh out a minimum of 100 g of homogenized sample using a wood spatula or tongue depressor, making sure that there is an appropriate amount of sample to yield the amount of extract required by the specific analysis. Record the weight in the logbook to the nearest tenth of a gram. The ratio of extraction fluid added to 100% solid sample is 20 mLs to 1 g (20:1). In this non-volatile extraction procedure, the initial 100 g weighed out for the preliminary evaluation in section 11.1 may be used to extract the sample. Place the weighed sample and extraction fluid in the correct extraction vessel listed in Table-01. The extraction vessels are numbered. Document the vessel used in the TCLP Vessel Use Log. Go to Section 11.2.3 for Tumbling.

11.2.1.2 If the sample contains less than 100% solids, liquid/solid separation is required. For the non-volatile extraction procedure, the filtrate and solid portion/filter obtained in the percent solids determination (section 11.1.1) may be used. The liquid phase obtained during the percent solids determination is collected in an appropriate, labeled, pre-weighed container, re-weighed, and set aside. The weight of the filtrate and the solid portion/filter was recorded on the bench sheet to the nearest tenth of a gram (see section 11.1.1.3.9). The other preliminary evaluations identified in sections 11.1.2 and 11.1.3 were completed.

11.2.1.2.1 The solid portion/filter is placed in the correct extractor vessel. The extraction vessels are numbered. Document the vessel used in the TCLP Vessel Use Log. The weight of extraction fluid to be added is calculated according to the equation below:

$$\text{Weight of extraction fluid (g)} = \frac{20 \times \text{percent solids (\%)} \times \text{total weight of waste filtered (g)}}{100\%}$$

For example: 100 g of the sample was weighed out and the percent solids were calculated at 25%.

$$\text{Weight of extraction fluid (g)} = \frac{20 \times 25\text{\% solids} \times 100\text{ g total weight of waste filtered}}{100\%}$$

The weight of extraction fluid to add to the extraction vessel in this case is <u>500 g.</u>

11.2.1.2.2 The solid portion will be tumbled as outlined in Section 11.2.3.

11.2.1.3 If the sample is determined to be <0.5% solids, then the filtrate obtained from the percent solids determination is defined as the TCLP extract. Filter enough of the sample so that the amount of filtered liquid will support all of the analyses required of the TCLP extract.

Note: If the client has not provided sufficient sample (100 g minimum), then the Project Manager must be notified through a Non-Conformance Report. If sufficient sample cannot be obtained, then the data will be qualified. The qualifier will read, "Due to insufficient sample provided for analysis, a lesser aliquot was analyzed than the method-required 100 grams."

Note: If the amount of extract generated by a single TCLP extraction will not be sufficient to perform all of the analyses, more than one extraction may be performed and the extracts from each combined and aliquoted for analysis. Also, if you used a portion of your initial 100 g to perform a fluid determination, remember to take that portion into account during the rest of the procedure.

11.2.2 Method Blanks

11.2.2.1 For each extraction fluid used in a batch, prepare a method blank using the extraction fluid and 100g of clean Teflon boiling chips. Document the method blank in the worksheet in TALS.

11.2.3 Tumbling the TCLP or SPLP Sample

11.2.3.1 Close the extractor bottle **tightly** (Teflon tape can be used to ensure a tight seal).

11.2.3.2 Secure container in the rotary agitation device to keep movement of container to a minimum, and rotate at 30 ± 2 rpm for 18 ± 2 hours.

11.2.3.3 Ambient temperature (i.e., temperature of room in which extraction takes place) shall be maintained at 23 + 2°C during the extraction period. *The min/max thermometer shall be set at the start of every extraction. The initial temperature and final temperature during tumbling is recorded in the TALS worksheet for both the corrected and the uncorrected reading. In addition the minimum and maximum recorded temperature for the extraction shall be recorded in the TALS worksheet for both the corrected and the uncorrected reading.*

11.2.3.4 If the temperature range during an extraction falls outside the method-specified range of 23 ± 2°C, initiate a non-conformance report as described in TA-QA-0610.

Note: As agitation continues, pressure may build up within the extractor bottle for some types of wastes (e.g., limed or calcium carbonate containing waste may evolve gases such as carbon dioxide). To relieve excess pressure, the extractor bottle may be periodically opened (e.g., after 15 minutes, 30 minutes, and 1 hour) and vented into a hood.

11.2.3.5 Following the 18 ± 2 hour extraction, remove containers and allow samples to settle. This will aid in filtering. Go to Section 11.2.4.

11.2.4 Filtering TCLP or SPLP extracts.

11.2.4.1 If the extract is to be analyzed for metals, use the acid-washed filter specified in section 7.3.4 or 7.3.3. Due to random Barium contamination another pre-rinse with 2% Nitric acid must be preformed. Organic extracts are filtered using the positive pressure procedure. Gravity filtering is an option if necessary due to equipment issues but is more time consuming.

11.2.5 Gravity Filtration for metals:

11.2.4.2 Place a glass funnel over a 200ml plastic beaker. Fold a TCLP filter and place in the funnel. Pre-rinse with the 2% Nitric acid solution making sure to saturate the entire filter paper with approximately 20mls of solution. Discard of the rinse waste in the neutralization tank.

11.2.4.3 Make sure to check and document the final pH of the extract from the unfiltered leachate.

11.2.4.4 Fill the funnel with leachate being careful to not fill over the top of the filter paper. Continue until the desired volume for digestion is reached.

Filtration by positive pressure

11.2.4.5 Filter sample by pouring it into filter apparatus containing *a* .6 to .8 micron glass fiber filter and sealing. Gradually apply vacuum or gentle pressure of 1-10 psi, until air or pressurizing gas moves through the filter. If this point is not reached under 10 psi, and if no additional liquid has passed through the filter in any 2-minute interval, slowly increase the pressure in 10-psi increments to a maximum of 50 psi. After each incremental increase of 10 psi, if the pressurizing gas has not moved through the filter, and if no additional liquid has passed through the filter in any 2-minute interval, proceed to the next 10 psi increment. When the pressurizing gas begins to move through the filter, or when liquid flow has ceased at 50 psi (i.e., filtration does not result in any additional filtrate within any 2 minute period), stop the filtration.

11.2.5.1 Collect the filtrate in 500 or 1L amber bottles.

Note: Instantaneous application of high pressure can degrade the glass fiber filter and may cause premature plugging.

Note: For final filtration of the TCLP or SPLP extract, the glass fiber filter may be changed, if necessary, to facilitate filtration.

11.2.6 Handling the TCLP or SPLP Extracts.

11.2.6.1 If the sample contained no initial liquid phase, the filtered liquid material is defined as the TCLP extract.

11.2.6.2 If the sample is multiphasic:

11.2.6.2.1 Check the miscibility of the solid phase filtrate with the liquid phase filtrate(s). Pour ~ 5 mL of one of the filtrates into a small glass container (example: a 12mL vial). With a plastic pipette drop a few mLs of the other filtrate into the container. Observe them combine. If they are compatible, (miscible), multiple phases will not result on combination.

11.2.6.2.2 If they are miscible, then combine the phases into one TCLP or SPLP extract.

11.2.6.2.3 If they are not miscible, then do not combine them. Determine the volume of the individual phases (to ± 0.5%) using a graduated cylinder. Conduct the appropriate analyses, and combine the results mathematically as described below:

$$\text{Final Analyte Concentration} = \frac{(V_1)(C_1) + (V_2)(C_2)}{V_1 + V_2}$$

Where:
V_1 = The volume of the first phase (L)
C_1 = The concentration of the analyte of concern in the first phase (mg/L)
V_2 = The volume of the second phase (L)
C_2 = The concentration of the analyte of concern in the second phase (mg/L)

11.2.6.3 If the sample contained <0.5% solids, then its own filtrate is defined as the TCLP or SPLP extract.

11.2.6.4 Following collection of the TCLP or SPLP extract, determine the pH of the extract using a calibrated pH meter. Record the pH of the extracts to the nearest tenth of a pH unit.

11.2.6.5 Metals aliquots must be acidified with nitric acid to pH<2. If precipitation is observed upon addition of nitric acid to a small aliquot of the extract, then the remaining portion of the extract shall not be acidified and the extract shall be analyzed as soon as possible.

11.2.6.5.1 If the leachate is a metals matrix spike or matrix spike duplicate, the metals matrix spike solution must be added to the leachate prior to nitric acid preservation.

11.2.6.5.2 Upon completion of the preservation step, the trace metals chemist will document the preservation of the extracts in the Metals Digestion Log.

11.2.6.6 All other aliquots must be stored under refrigeration (4°C) until analyzed. The TCLP or SPLP extract shall be prepared and analyzed according to appropriate analytical methods.

11.3 ZHE Volatiles Extraction Procedure

Note: Document sample information in the worksheet in TALS.

11.3.1 Use the ZHE device to obtain TCLP or SPLP extract for analysis of volatile compounds only. Extract resulting from the use of the ZHE shall not be used to evaluate the mobility of non-volatile analytes, e.g. metals, pesticides, herbicides, etc.

11.3.2 The ZHE device has approximately a 500 mL internal capacity. The ZHE can thus accommodate a maximum of 25 grams of solid, due to the need to add an amount of extraction fluid equal to 20 times the weight of the solid phase.

11.3.3 Charge the ZHE with sample only once and do not open the device until the final extract has been collected.

11.3.4 Do not allow the waste, the initial liquid phase, or the extract to be exposed to the atmosphere for any more time than is absolutely necessary. Any manipulation of these materials should be done when cold (4°C) to minimize loss of volatiles.

11.3.5 All ZHE metal parts are numbered to track possible contamination. Only use the numbered parts that agree with the vessel number.

11.3.6 Perform a 1 hour pressure check on each ZHE vessel before every use. Set it up empty as outlined below and pressurize to 50 psi. Document the test in the Pressure Check Log. (Appendix A).

11.3.7 Preparation of the extractor vessel

11.3.7.1 Pre-weigh the filtrate collection container.

11.3.7.2 Apply vacuum grease to the piston O-rings and place the ZHE piston within the ZHE body, engraved number facing down. Adjust the piston within the ZHE body to a height that will minimize the distance the piston will have to move once the ZHE is charged with sample. Secure the gas inlet/outlet flange (bottom flange) onto the ZHE body in accordance with the manufacturer's instructions. Secure the glass fiber filter between the support screens and set aside. Set liquid inlet/outlet flange (top flange) aside.

11.3.8 Calculate the weight of waste to charge ZHE vessel.

11.3.8.1 If the waste is 100% solid, weigh out a subsample (25 g. maximum) of the waste and immediately transfer it to the ZHE vessel and attach the ZHE top. The extraction vessels are numbered. Document the vessel used in the TCLP Vessel Use Log. Record the weight in TALS to the nearest tenth of a gram.

11.3.8.2 If the waste contains < 0.5% dry solids, quickly pour 100-500 mLs of the sample into the ZHE vessel. The extraction vessels are numbered. Document the vessel used in the TCLP Vessel Use Log and in the TALS worksheet.

11.3.8.3 For wastes containing between 0.5% and 5% solids, weigh out a 500g. subsample and record the weight. Since the ZHE vessel cannot be opened during the extraction, multiple vessels may be used to extract the sample and the extracts combined for analysis. The extraction vessels are numbered. Document the vessel used in the TCLP Vessel Use Log.

11.3.8.4 For wastes containing ≥ 5% solids, determine the amount of waste to charge into the ZHE as follows:

$$\text{Weight of waste to charge ZHE (g)} = \frac{25}{\text{percent solids}} * 100$$

11.3.8.5 If particle size reduction was required as determined in section 11.1.2, prepare the waste for extraction by crushing, cutting, or grinding the solid portion of the waste to a surface area or particle size specified in section 11.1.2. Waste and appropriate reduction equipment should be refrigerated, if possible, to 4°C prior to particle size reduction. The means used to effect particle size reduction must not generate heat. Also, exposure of the waste to the atmosphere should be kept to a minimum. Don't sieve.

Note: Quantitatively transfer the entire sample (liquid and solid phases) quickly to the ZHE. Don't allow waste slurries to stand and don't centrifuge. Secure the filter and support screens onto the top flange of the device and secure the top flange to the ZHE body in accordance with the manufacturer's instructions. Tighten all ZHE fittings and place the device in the vertical position (gas inlet/outlet flange on the bottom). Do not attach the extract collection device to the top plate.

Note: If waste material (>1% of original sample weight) has obviously adhered to the container used to transfer the sample to the ZHE, determine the weight of this residue and subtract it from the sample weight determined in Section 11.3.8 to determine the weight of the waste sample that will be filtered.

11.3.9 Headspace Elimination and Liquid/Solid Separation

11.3.9.1 Attach a gas line to the gas inlet/outlet valve (bottom flange) and, with the liquid inlet/outlet valve (top flange) open, begin applying gentle pressure of 1-10 psi (or more if necessary) to force all headspace slowly out of the ZHE device into a hood.

11.3.9.2 If the waste is liquid, at the first appearance of liquid from the liquid inlet/outlet valve, quickly close the valve and discontinue pressure. If filtration of the waste at 4°C reduces the amount of expressed liquid over what would be expressed at room temperature, then allow the sample to warm up to room temperature in the device before filtering. Proceed to section 11.3.10.

11.3.9.3 If the waste is 100% solid, slowly increase the pressure to a maximum of 50 psi to force most of the headspace out of the device and proceed to section 11.3.10.

11.3.9.4 Attach the ZHE fluid pump sample tube to the liquid inlet/outlet valve with the other end of the line at the bottom of a 40ml voa vial and open the valve. Begin applying gentle pressure of 1-10 psi to force the liquid phase of the sample into the filtrate collection container. If no additional liquid has passed through the filter in any 2 minute interval, slowly increase the pressure in 10 psi increments to a maximum of 50 psi. After each incremental increase of 10 psi, if no additional liquid has passed through the filter in any 2 minute interval, proceed to the next 10 psi increment. When liquid flow has ceased such that continued pressure filtration at 50 psi does not result in any additional filtrate within a 2 minute period or the syringe is full, stop the filtration. Close the liquid inlet/outlet valve, discontinue pressure to the piston, and disconnect the sample tube. Express any additional liquid phase into a TCLP waste container once the voa vial is full.

Please Note: Instantaneous application of high pressure can degrade the glass fiber filter and may cause premature plugging. An in-line glass fiber filter may be used to filter the material within the ZHE if it is suspected that the glass fiber filter has been ruptured.

11.3.9.5 The material in the ZHE is defined as the solid phase of the waste and the filtrate is defined as the liquid phase.

Please Note: Some wastes, such as oily wastes and some paint wastes, will obviously contain some material that appears to be a liquid. Even after applying pressure filtration, this material will not filter. If this is the case, the material within the filtration device is defined as a solid and is carried through the TCLP or SPLP extraction as a solid. If the original waste contained <0.5% dry solids, this filtrate is defined as the TCLP or SPLP extract and is analyzed directly.

11.3.10 Charging the ZHE Device with TCLP or SPLP Extraction Fluid.

11.3.10.1 The equation to determine the amount of extraction fluid to add to the extractor vessel is:

$$\text{Weight of extraction fluid (g)} = \frac{20 \times \text{percent solids (\%)} \times \text{total weight of waste filtered (g)}}{100\%}$$

For example: 25 g of the sample was weighed out and the percent solids were calculated at 25%.

$$\text{Weight of extraction fluid (g)} = \frac{20 \times 25\% \text{ solids} \times 25 \text{ g total weight of waste filtered}}{100\%}$$

The weight of extraction fluid to add to the extraction vessel in this case is 125 g.

11.3.10.2 TCLP Fluid #1 or SPLP Fluid #3 is used exclusively for the extraction of volatiles using the ZHE. Add the appropriate amount of appropriate fluid to the solid material within the ZHE according to the following steps:

11.3.10.2.1 Pre-flush the extraction fluid lines connected to the ZHE pump with fresh extraction fluid to eliminate any air pockets from the line. Fill the graduated cylinder with the calculated weight of extraction fluid you will add. With the ZHE in the vertical position, attach the extraction fluid line from the pump directly into the liquid inlet/outlet valve.

11.3.10.2.2 Open the top flange and pump all the fluid to the extraction vessel.

11.3.10.2.3 After the extraction fluid has been added, immediately close the liquid inlet/outlet valve. Check the ZHE to ensure that all valves are in their closed positions. Manually rotate the device in an end-over-end fashion 2 or 3 times. Reposition ZHE in the vertical position with the liquid inlet/outlet valve on top.

10.3.10.2.4 Pressurize the ZHE to 40+PSI.

11.3.11 Tumbling the ZHE Samples

11.3.11.1 Method Blank

11.3.11.1.1 One Method Blank must be run through each extraction vessel every 20 times it is used. Vessel usage is tracked in the TCLP Vessel Use Log (see Appendix C). Using this log, select an appropriate vessel for the method blank. Document the method blank in the worksheet in TALS.

11.3.11.2 Secure container in the rotary agitation device to keep movement of container to a minimum, and rotate at 30 ± 2 rpm for 18 ± 2 hours.

11.3.11.3 Ambient temperature (i.e., temperature of room in which extraction takes place) should be maintained at 23 ± 2°C during the extraction period. The initial temperature and final temperature during tumbling is recorded in the TALS worksheet for both the corrected and the uncorrected reading. The range paper with the entire nights reading should be dated and placed in the three ring binder for future reference. If the temperature range during an extraction falls outside the method-specified range of 23 ± 2°C, initiate a non-conformance report as described in B-SOP-QAG-010.

11.3.11.4 Following the 18 ± 2 hour agitation period, check the pressure behind the ZHE piston by reading the pressure meter on the ZHE body. If the pressure has not been maintained (i.e., no gas release observed), the device is leaking. Repair and retest the device or remove it from service. Initiate an NCM, then using a fresh portion of the waste, re-prepare the sample in a properly functioning device.

If the pressure within the device has been maintained, the material in the extractor vessel is once again separated into its component liquid and solid phases. A separate filtrate collection container must be used if combining would create multiple phases, or there is not enough volume left within the filtrate collection container.

11.3.12 Filtering the ZHE Extracts

11.3.12.1 Attach the sample tube line to the liquid inlet/outlet valve with the other end of the tube placed at the bottom of a 40ml voa vial and open the valve. Begin applying gentle pressure of 1-10 psi to force the TCLP liquid phase of the sample into the filtrate collection container. If no additional liquid has passed through the filter in any 2 minute interval, slowly increase the pressure in 10 psi increments to a maximum of 50 psi. After each incremental increase of 10 psi, if no additional liquid has passed through the filter in any 2 minute interval, proceed to the next 10 psi increment. When liquid flow has ceased such that continued pressure filtration at 50 psi does not result in any additional filtrate within a 2 minute period or the voa vial is full, stop the filtration. Close the liquid inlet/outlet valve, discontinue pressure to the piston, and disconnect the sample tube line. Express any additional liquid phase into a TCLP waste container once the VOA vial is full.

Note: Instantaneous application of high pressure can degrade the glass fiber filter and may cause premature plugging. An in-line glass fiber filter may be used to filter the material within the ZHE if it is suspected that the glass fiber filter has been ruptured.

11.3.13 Handling the ZHE Extracts

11.3.13.1 If the sample contained no initial liquid phase, the filtered liquid material is defined as the TCLP or SPLP extract.

11.3.13.2 If the sample is multiphasic:

11.3.13.2.1 Check the miscibility of the solid phase filtrate with the liquid phase filtrate(s) by combining via transfer pipette small portions of each component in a small glass container. If they are compatible, (miscible), multiple phases will not result on combination.

11.3.13.2.2 If they are miscible, then combine the phases into one voa vial as one ZHE extract.

11.3.13.2.3 If they are not miscible, then do not combine them. Determine the volume of the individual phases (to $\pm$ 0.5%) while contained in their respective syringes. Conduct the appropriate analyses, and combine the results mathematically as described below:

$$\text{Final Analyte Concentration} = \frac{(V_1)(C_1) + (V_2)(C_2)}{V_1 + V_2}$$

Where:
V_1 = The volume of the first phase (L)
C_1 = The concentration of the analyte of concern in the first phase (mg/L)
V_2 = The volume of the second phase (L)
C_2 = The concentration of the analyte of concern in the second phase (mg/L)

11.3.13.3 If the sample contained <0.5% solids, then its own filtrate is defined as the ZHE extract.

11.3.13.4 The liquid phase may now be either analyzed or stored at 4°C under minimal headspace conditions until time of analysis.

12.0 Calculations / Data Reduction

12.1 Percent Solids Determination

$$\% \text{ Solids} = \frac{\text{Weight of solid}}{\text{Total weight of waste}} \times 100$$

Document Uncontrolled When Printed

12.2 Percent Dry Solids Determination

$$\% \text{ Dry Solids} = \frac{(\text{Wt. of dry waste + filter}) - \text{tared weight of filter}}{\text{Initial weight of waste}} \times 100$$

12.3 The equation to determine the amount of extraction fluid to add to the extractor vessel is:

$$\text{Weight of extraction fluid (g)} = \frac{20 \times \text{percent solids (\%)} \times \text{total weight of waste filtered (g)}}{100\%}$$

12.4 Final Analyte Concentration from multiple phases

$$\text{Final Analyte Concentration} = \frac{(V_1)(C_1) + (V_2)(C_2)}{V_1 + V_2}$$

Where:
V_1 = The volume of the first phase (L)
C_1 = The concentration of the analyte of concern in the first phase (mg/L)
V_2 = The volume of the second phase (L)
C_2 = The concentration of the analyte of concern in the second phase (mg/L)

12.5 ZHE waste amount for solids

$$\text{Weight of waste to charge ZHE (g)} = \frac{25}{\text{percent solids}} \times 100$$

13.0 Method Performance

13.1 A Method Detection Limit (MDL) study must be performed annually according to the current SOP for MDL completion (TA-QA-0602).

13.2 An IDC must be completed according to the procedures specified in TA-QA-0617.

14.0 Pollution Control Waste

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., processing one set of MDLs on all applicable instruments, examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. *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"*.

15.0 Waste Management

All waste will be disposed of in accordance with Federal, State and Local regulations. Employees will abide by this method and the policies in the Corporate Safety Manual for "Waste Management and Pollution Prevention". Excess reagents, samples and method process wastes are disposed of in an accepted manner. *Waste description rules and land disposal restrictions are followed.* The lab's waste disposal procedures are incorporated by reference to TA-EHS-0036.

15.1 Waste Streams Produced by the Method.

The following waste streams are produced when this method is carried out:

15.1.1 Extraction Fluid. The remaining extraction fluid is decanted into the acid waste drum located in the waste warehouse. This waste stream is sent out for waste water treatment. The remaining solid wastes is removed from the container and placed in the toxic solid debris waste stream. This waste stream is sent out for incineration. The empty container is then placed in the dumpster and sent to the sanitary landfill.

16.0 References

16.1 TestAmerica Seattle Quality Assurance Manual (QAM), current version.

16.2 TestAmerica Environmental Health and Safety Manual, current version.

16.3 US EPA Method 1311, "Toxicity Characteristic Leaching Procedure (TCLP)", SW-846, Rev. 0, July 1992.

16.4 US EPA Method 1312, "Synthetic Precipitation Leaching Procedure (TCLP)", SW-846, Rev. 0, September 1994

16.5 TCLP Flowchart, Genium Publishing Corp., 1997.

17.0 Attachments

17.1 Appendix A: ZHE Pressure Check Log

17.2 Appendix B: TCLP Tumbler Log

17.3 Appendix C: ZHE Vessel Use Log

17.4 Appendix D: pH Meter Log

17.5 Appendix E: TCLP Flowchart

17.6 Appendix F: 1311 TCLP/ZHE Summary

18.0 Revision History

SOP Rev No. and Date:	Revised Section No.:	Revised Text	Comments
Revision 12, Dated 5/7/12	7.3	Updated filter brands	
	Throughout	Removed reference to using EE tube filtration process	
	8.0	Added rinse solution for filters and reagent documentation to be done in TALS	
	10.0	Made reference to LCS/LCSD batching and added requirement for MSD.	
	11.0	Added gravity filtration process and pre-rinse step for filter paper for metals	
	17	Added Appendix F	
Revision 11, Dated 4/25/11	Throughout	Changed documentation requirements from TCLP logbook to TALS worksheet	
	11.2.3, 11.3.11.3	Incorporated ROMD 00021	
	17	Deleted Attachments C (TCLP Log) and D (TCLP Vessel Use Log) because of documentation and vessel change	
Revision 10, Dated 3/10/10	8.0	Added documentation of standards/reagents and standard/reagent preparation	
	8.10	Added removal of expired standards	
	10.4	Addressed the need to evaluate extra QC when present	
	11.1	Added daily balance check	

19.0 Discrepancies to the Method

None.

Appendix A: Example of a ZHE Pressure Check

TestAmerica-Seattle, WA

ZHE PRESSURE CHECK LOG

Date	Time Started	Vessel #	O-ring	PSI 0 hrs	PSI 1 hr	PSI 2 hrs	PSI 3 hrs	PSI 4 hrs	Pass/Fail	Action	Analyst
			OK / Replaced								
			OK / Replaced								
			OK / Replaced								
			OK / Replaced								
			OK / Replaced								
			OK / Replaced								
			OK / Replaced								
			OK / Replaced								
			OK / Replaced								
			OK / Replaced								
			OK / Replaced								
			OK / Replaced								
			OK / Replaced								
			OK / Replaced								
			OK / Replaced								
			OK / Replaced								
			OK / Replaced								
			OK / Replaced								
			OK / Replaced								
			OK / Replaced								
			OK / Replaced								
			OK / Replaced								
			OK / Replaced								
			OK / Replaced								
			OK / Replaced								

Reviewer's Initials & Date: _____

B-NBK-EXT-132

Page 120 of 120

Log

Appendix B: Example of a TCLP Tumbler Log

Tumbler Check should be performed on a monthly basis, while fully loaded

	Tumbler #1		
Date	RPM	Action	Analyst

	Tumbler #2		
Date	RPM	Action	Analyst

	Tumbler #3		
Date	RPM	Action	Analyst

Appendix C: Example of ZHE Vessel Use Log

	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20
Date:																				
Batch:																				
ZHE#	S/B	S/B	S/B	S/B	S/B	S/B	S/B	S/B	S/B	S/B	S/B	S/B	S/B	S/B	S/B	S/B	S/B	S/B	S/B	S/B
1																				
2																				
3																				
4																				
5																				
6																				
7																				
8																				

S=Sample B=Blank

Appendix D: Example of pH Meter Calibration Log

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

pH 3 - Calibration Logbook

Analyst_____				Date_____	
Calibration					
pH 4 Std.	Lot:	Slope =		Acceptance Criteria 92.0-105.0	
pH 7 Std.	Lot:	Slope =			
pH 10 Std.	Lot:	Slope =			
Second Source Check Standard pH 7					
2nd pH 7 Std.	Lot:	Reading =		= 7.0 ± 0.05 pH units	Yes / No

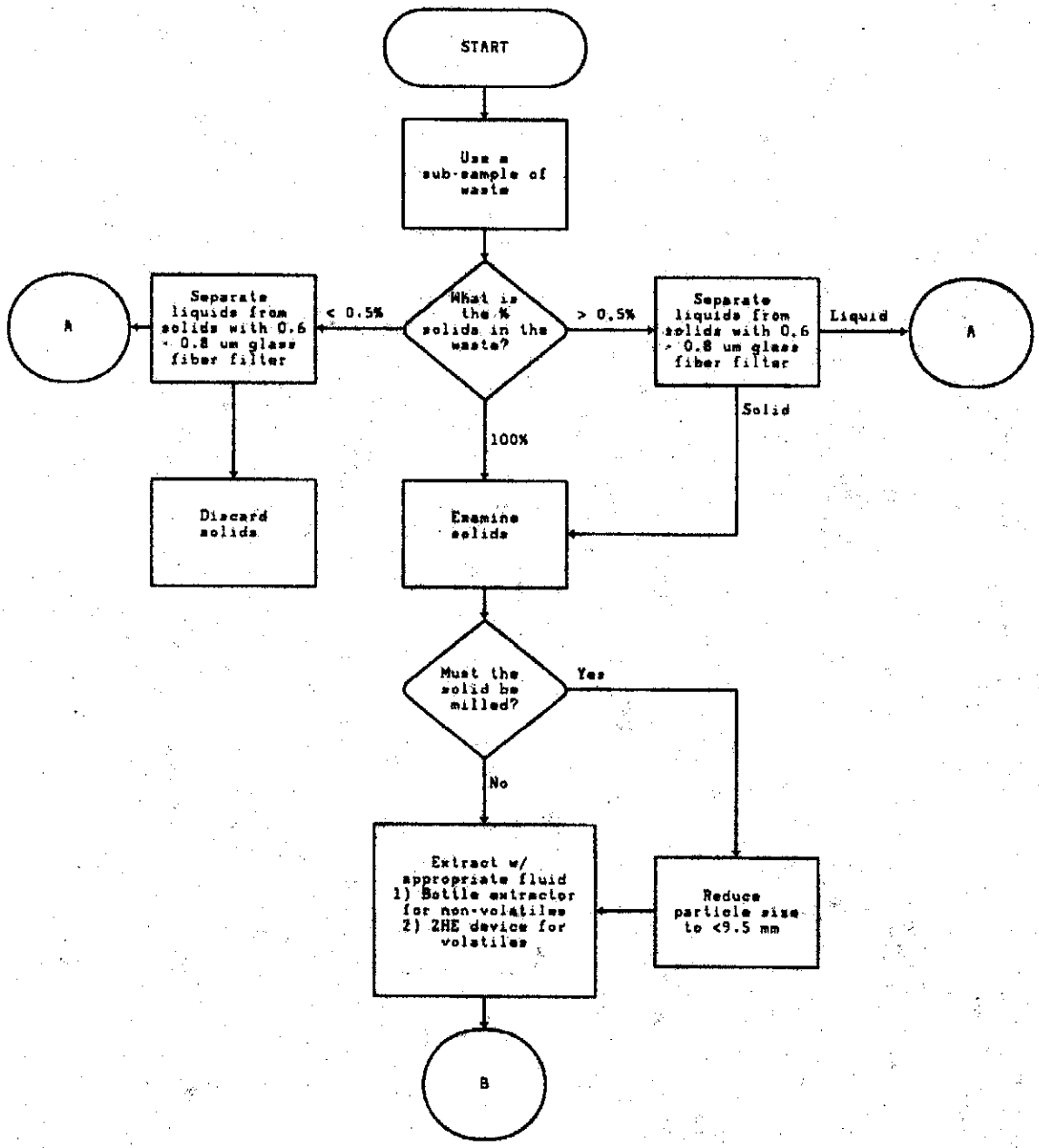
Analyst_____				Date_____	
Calibration					
pH 4 Std.	Lot:	Slope =		Acceptance Criteria 92.0-105.0	
pH 7 Std.	Lot:	Slope =			
pH 10 Std.	Lot:	Slope =			
Second Source Check Standard pH 7					
2nd pH 7 Std.	Lot:	Reading =		= 7.0 ± 0.05 pH units	Yes / No

Analyst_____				Date_____	
Calibration					
pH 4 Std.	Lot:	Slope =		Acceptance Criteria 92.0-105.0	
pH 7 Std.	Lot:	Slope =			
pH 10 Std.	Lot:	Slope =			
Second Source Check Standard pH 7					
2nd pH 7 Std.	Lot:	Reading =		= 7.0 ± 0.05 pH units	Yes / No

Analyst_____				Date_____	
Calibration					
pH 4 Std.	Lot:	Slope =		Acceptance Criteria 92.0-105.0	
pH 7 Std.	Lot:	Slope =			
pH 10 Std.	Lot:	Slope =			
Second Source Check Standard pH 7					
2nd pH 7 Std.	Lot:	Reading =		= 7.0 ± 0.05 pH units	Yes / No

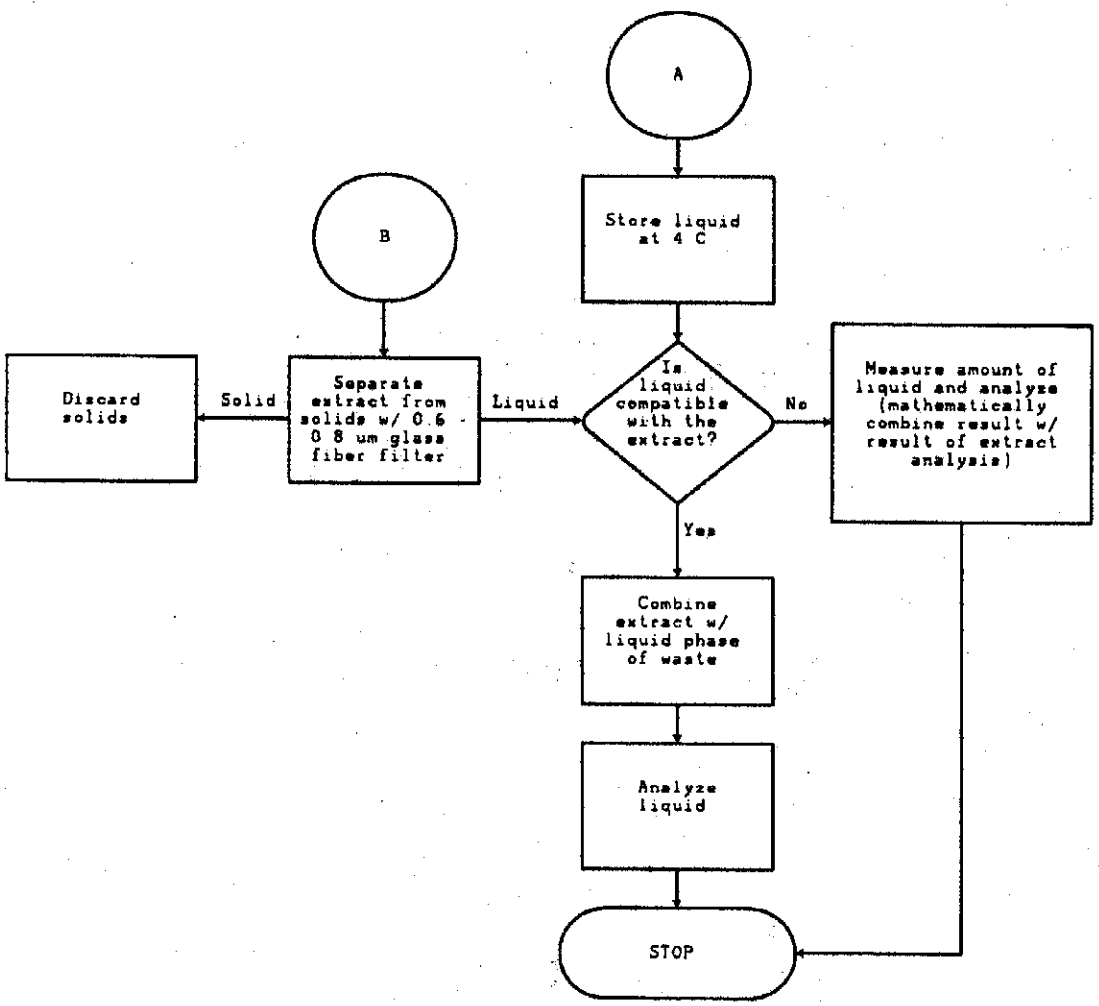
Document Uncontrolled When Printed

Appendix E: TCLP Flowchart



Document Uncontrolled When Printed

Appendix E: TCLP Flowchart (continued)



Company Confidential & Proprietary

Appendix F: 1311 TCLP/ZHE Summary

Quarterly checks:	Pipettor calibration
Monthly checks:	RPM check for tumbler while full, 30 ± 2 RPM required 4 hour pressure check for all working ZHE bodies
Daily Checks:	Daily temperature control unit maintenance: Drain and dump water waste pH probe calibration and fluid pH verification, 4.88 to 4.98

1311 Metals, 8270, 8081, 8151

If the sample is not obviously 100% water or 100% solids:

Percent solids determination:
First weight the sample container and entire contents of the jar.
This weight is the **Total Initial Volume.**
Set up the pressure filter device. Transfer the entire contents of the sample jar into the filtration device.
Weigh the empty jar, this is the **Container weight**.
Total Initial Volume – Container weight = Total sample weight
Weigh an **Empty container**, glass or plastic.
Pressurize the filtration device and capture the filtrate in this container.
Weigh the **Full container** when complete.
Full container – Empty container = Total aqueous weight.
Total soil weight = Total sample weight – Total aqueous weight

% solids = (Total soil weight / Total sample weight) x 100
Check SOP for complete details on re-combining portions

Water samples are filtered only in plastic 200ml beakers for metals and amber bottles for Organics

Soil samples are first pH tested (already determined 100% solid)
Determine if particle size reduction is necessary, material should be broken, cut, torn apart into 1cm³ portions before continuing.

5grams – add DI water up to 101.5 grams
Stir and take pH
If pH is ≤ 5 you're done skip to fluid addition of fluid #1
If pH is >5 then put on the hot plate
Add 3.5 mls of 1N HCL
Heat to 50 degrees +/- 5 degrees keep in range for 10 minutes
Let cool approx 10 minutes
Take the pH
If pH ≤ 5 use TCLP fluid #1
If pH > 5 use TCLP fluid #2
100 grams of soil/solid to 2000mls of fluid
Samples are ready to be tumbled for 18 ± 2 hours

Quality Control:
MB per batch of 20 samples
MB for each fluid type per batch
Corrected Temperature out of range of 21-25 degrees – **NCM**
100grams not available – **NCM** and maintain a 20:1 fluid:sample ratio.
Any required checks not completed – **NCM** and immediately complete check

Appendix F: 1311 TCLP/ZHE Summary (continued)

Filtration Process

Metals –
For MB and sample to be used as Matrix QC filter full 200mls
For the rest of the samples at least 100mls
Gravity filter using a glass funnel and TCLP filter. Pre-rinse the filter using ~ 20mls of the 2% Nitric acid solution. Discard waste before filtering the sample.
Take the final leachate pH on a portion of **un**-filtered leachate
Deliver to metals

8270, 8081, and 8151
Use the positive pressure filtration device with a TCLP filter in place and filter ~ 1L into an amber bottle.
Store in walk-in on designated shelf

1311 ZHE for Volatile Analysis

If the sample is not obviously 100% water or 100% solids:

The determination is the same as for non-volatile analysis except the filtration device is the ZHE body itself.

Water samples are filtered only through a ZHE body complete with TCLP Filter.

Soil samples determined to be 100% solid
Determine if particle size reduction is necessary, material should be broken, cut, torn apart into 1cm³ portions before continuing.

25 grams of soil is placed in the ZHE body
Pressurize the ZHE and remove any headspace by opening the top valve allowing the piston to move up
Add 500mls of TCLP fluid #1 to a class A graduated cylinder
Attach the tubing from the pump to the top of the ZHE body and place the other end of the tubing in the graduated cylinder
Pump the fluid into the ZHE body
Re-pressurize to over 40psi if necessary
Samples are ready to be tumbled for 18 ± 2 hours

Quality Control:
Same as Non-volatile portion
If pressure is lost overnight – **NCM** and possible re-preparation required

Filtration Process

Attach the filtration tubing to the top of the ZHE body
Place the other end of the tube at the bottom of a 40ml voa vial
Fill the voa vial to the top and cap with no head space
Deliver to volatile's analyst or store in voas refrigerator

Appendix F: 1311 TCLP/ZHE Summary (continued)

Fluid Preparation:

TCLP Fluid #1 Concentrate:

In a 20L carboy add 10 L of DI water
Weigh 1040 grams of Sodium Hydroxide pellets
Slowly add the pellets to the carboy adding only approximately 1/4th of the total volume at one time
Cap the carboy and roll on the counter to mix
Let cool and add another 1/4th volume of the pellets and repeat the mixing and cooling step two more times
When all the sodium hydroxide is dissolved and cooled to near room temperature you can proceed
Slowly add 2.3 liters of glacial acetic acid
Fill to the 20L mark on the carboy and mix well
Let cool-mix again and read the pH
Should fall in the range of 4.88 to 4.98

TCLP Fluid #1:

Dilute 1 Liter of concentrate to 20L with DI water in a carboy
Mix well and verify pH between 4.88 and 4.98

TCLP Fluid #2:

Add 22.8 mls of glacial acetic acid to 4 Liters of DI water
Mix and verify pH between 2.83 and 2.93

For more details including preparation for SPLP the SOP must be referenced. This summary is not intended to replace the SOP and any details not referenced with in this summary should be sought out in the SOP.

Title: Mercury Analysis by CVAA [Methods 245.1, 7470A, 7471A]

Approvals

Signatures on File

Stan Palmquist — Date Metals Department Manager	Sandra Langway — Date Health & Safety Manager / Coordinator
Terri Torres — Date Quality Assurance Manager	Heather Prater — Date Laboratory Director

Copyright Information:
This documentation has been prepared by TestAmerica Laboratories, Inc. and its affiliates ("TestAmerica"), solely for their own use and the use of their customers in evaluating their qualifications and capabilities in connection with a particular project. The user of this document agrees by its acceptance to return it to TestAmerica upon request and not to reproduce, copy, lend, or otherwise disclose its contents, directly or indirectly, and not to use it for any other purpose other than that for which it was specifically provided. The user also agrees that where consultants or other outside parties are involved in the evaluation process, access to these documents shall not be given to said parties unless those parties also specifically agree to these conditions.

THIS DOCUMENT CONTAINS VALUABLE CONFIDENTIAL AND PROPRIETARY INFORMATION. DISCLOSURE, USE OR REPRODUCTION OF THESE MATERIALS WITHOUT THE WRITTEN AUTHORIZATION OF TESTAMERICA IS STRICTLY PROHIBITED. THIS UNPUBLISHED WORK BY TESTAMERICA IS PROTECTED BY STATE AND FEDERAL LAW OF THE UNITED STATES. IF PUBLICATION OF THIS WORK SHOULD OCCUR THE FOLLOWING NOTICE SHALL APPLY:

©COPYRIGHT 2012 TESTAMERICA LABORATORIES, INC. ALL RIGHTS RESERVED.

Facility Distribution No. 001 **Distributed To:** Lab Intranet

Document Uncontrolled When Printed

1.0 Scope and Application

1.1 Analytes, Matrix(s), and Reporting Limits

This procedure describes the preparation and analysis of mercury (Hg, CAS # 7439-97-6) by Cold Vapor Atomic Absorption Spectroscopy (CVAA) using SW-846 Method 7470A and MCAWW Method 245.1. Method 7470A is applicable to the preparation and analysis of mercury in ground water, aqueous samples, wastes, wipes, TCLP, EP and other leachates/extracts. Method 245.1 is applicable to the determination of mercury in drinking, surface and saline waters, and domestic and industrial wastes.

Standard Aqueous RL	0.2 µg/L
Brine RL	60 µg/L
TCLP RL	2.0 µg/L
Soil RL	20 µg/kg

On occasion clients may request modifications to this SOP. These modifications are handled following the procedures outlined in Section 12.2.1 in the Quality Assurance Manual.

2.0 Summary of Method

This method describes the determination of mercury in solution by CVAA. Prepared samples are treated so that mercury is reduced to the elemental state and aerated from solution in a closed system. The mercury vapor passes through a cell positioned in the light path of an atomic absorption spectrophotometer at 253.7 nm. Intensity of absorption (attenuation of light signal) is directly related to the concentration of mercury by comparison with standards.

3.0 Definitions

3.1 Dissolved metals = those metals that will pass through a 0.45 um membrane filter.

3.2 Suspended metals = those elements which are retained by a 0.45 um membrane filter.

3.3 Total metals = the concentration determined of an unfiltered sample after digestion or; the sum of the dissolved plus suspended concentrations.

3.4 Total recoverable metals = the concentration determined of an unfiltered sample after treatment with hot, dilute mineral acid.

4.0 Interferences

4.1 Potassium permanganate, which is used to breakdown organic mercury compounds, is added as a reagent to eliminate interference from sulfide.

4.2 Copper also has been reported to interfere; however, copper concentrations as high as 10 mg/L had no effect on the recovery of mercury from spiked samples.

4.3 High chloride concentration requires additional permanganate (free chlorine must be absent during oxidation steps). Excess of hydroxylamine sulfate reagent eliminates chlorine interference.

4.4 Interference from certain volatile organic materials that absorb at the wavelength used for the method may also occur. If suspected, a preliminary run without stannous chloride can determine if this type of interference is present. While the possibility of absorption from

Document Uncontrolled When Printed

certain organic substances present in the sample does exist, this problem is not routinely encountered. This is mentioned only to caution the analyst of the possibility. If this condition is found to exist, the mercury concentration in the sample can be determined by subtracting the result of the sample run without the reducing reagent (stannous chloride) from that obtained with the reducing reagent.

4.5 Samples containing high concentrations of oxidizable organic materials, as evidenced by high COD levels, may not be completely oxidized by this procedure. When this occurs, the recovery of mercury will be low. The problem can be eliminated by reducing the volume of original sample used.

4.6 The most common interference is laboratory contamination, which may arise from impure reagents, dirty glassware, improper sample transfers, dirty work areas, etc. Be aware of potential sources of contamination and take appropriate measures to minimize or avoid them.

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

5.1.1 Samples that contain high concentrations of carbonates or organic material or samples that are at elevated pH can react violently when acids are added.

5.1.2 Potassium permanganate is a strong oxidizing agent. It is incompatible and must be stored separately from hydroxylamine hydrochloride and stannous chloride, the reducing agents used in this procedure, and from acids.

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
Mercury (100 µg/ml in Reagent)	Oxidizer Corrosive Poison	0.1 Mg/M3 Ceiling (Mercury Compounds)	Extremely toxic. Causes irritation to the respiratory tract. Causes irritation. Symptoms include redness and pain. May cause burns. May cause sensitization. Can be absorbed through the skin with symptoms to parallel ingestion. May affect the central nervous system. Causes irritation and burns to eyes. Symptoms include redness, pain, and blurred vision; may cause serious and permanent eye damage.
Sulfuric Acid	Corrosive Oxidizer Dehydrator Poison	1 Mg/M3-TWA	Inhalation produces damaging effects on the mucous membranes and upper respiratory tract. Symptoms may include irritation of the nose and throat, and labored breathing. Symptoms of redness, pain, and severe burn can occur. Contact can cause blurred vision, redness, pain and severe tissue burns. Can cause blindness.
Nitric Acid	Corrosive Oxidizer Poison	2 ppm-TWA 4 ppm-STEL	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Hydrochloric Acid	Corrosive Poison	5 PPM-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Potassium Permanganate	Oxidizer	5 Mg/M3 for Mn Compounds	Causes irritation to the respiratory tract. Symptoms may include coughing, shortness of breath. Dry crystals and concentrated solutions are caustic causing redness, pain, severe burns, brown stains in the contact area and possible hardening of outer skin layer. Diluted solutions are only mildly irritating to the skin. Eye contact with crystals (dusts) and concentrated solutions causes severe irritation, redness, and blurred vision and can cause severe damage, possibly permanent.
Potassium Persulfate	Oxidizer	None	Causes irritation to the respiratory tract. Symptoms may include coughing, shortness of breath. Causes irritation to skin and eyes. Symptoms include redness, itching, and pain. May cause dermatitis, burns, and moderate skin necrosis.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

Document Uncontrolled When Printed

6.0 Equipment and Supplies

6.1 Digestion Tubes (CPI) , certified for cleanliness and volume

If the digestion tubes come with certifications of cleanliness and certifications of volume, the following procedures do not have to be done for each lot of tubes received:

6.1.1 Ten tubes are used to process ten Method Blanks and the digestates are analyzed by ICP-MS. The lot number of the digestion tubes is recorded on the batch sheet and the results of the analyses are place in a three ring binder with the batch sheets. The lot of digestion tubes is considered acceptable if all analytes are less than ½ the ICP-MS RLs.

6.1.2 Volumetric verification is performed on same ten digestion tubes. Each digestion tube is tared on a balance and DI water is filled to the 50 ml mark. The weight of the water is recorded and should equal 50 grams ±0.5 grams to be considered acceptable. This procedure is repeated for 10 replicates on each tube. Verification is documented in the same digestion batch as the Method Blanks in Section 6.

6.2 Instrumentation

- Leeman Labs Hydra AA Automated Mercury Analyzer
- Computer controlled data system for Hydra AA
- Analytical balance, 0.1 mg accuracy
- Hot Bloc digestion block, capable of maintaining constant temperature at 90 - 95℃

6.3 Computer hardware and software

- *Computer with a minimum 1GB memory, Pentium 4 processor, 80 G hard drive or equivalent or as recommended by instrument manufacturer.*
- *Data acquisition/processing system: Envoy 1.8 or higher*
- *LIMS system: TALS version 1.0 or higher*

6.4 Supplies

- Graduated cylinder, 100 mL.
- Volumetric flasks, 100 mL and 1000 mL.
- Disposable polyethylene tubes, 16 x 125 mm.
- Poly dispensing bottles
- Inert utensils

7.0 Reagents and Standards

7.1 Document reagents/standards and reagent/standard preparation in TALS using the reagent module as described in SOP TA-QA-0619.

7.2 ASTM Type II reagent water.

7.3 Argon gas, high purity grade

7.4 Sulfuric acid, concentrated, trace reagent grade.

7.5 Nitric acid, concentrated, trace reagent grade of low mercury content.

7.6 Nitric acid, 50%

7.6.1 Preparation: Dilute concentrated nitric by adding to equal volume of Type II water.

7.7 Hydrochloric acid, concentrated, trace reagent grade.

Document Uncontrolled When Printed

7.8 Stannous chloride, Certified ACS.

7.9 Sodium chloride, Certified ACS.

7.10 Hydroxylamine hydrochloride, Certified ACS.

7.11 Potassium permanganate, Certified ACS.

7.12 Potassium persulfate, Certified ACS.

7.13 Aqua Regia

7.13.1 Preparation: 0.6mL of Nitric acid, 1.8mL of hydrochloric acid, and 2.5mL of deionized water are added directly to each standard and sample before digestion (solid samples only).

7.14 2:1 Sulfuric:Nitric (mixed acid).

7.14.1 Preparation: 700mL trace grade nitric acid mixed into 1400mL trace grade sulfuric acid.

7.15 Stannous chloride ($SnCl_2$) reagent.

7.15.1 Preparation: Add 100mL of trace grade Hydrochloric acid, 100g stannous chloride to 1000mL of warm Type II water.

7.16 Sodium chloride-hydroxylamine hydrochloride solution.

7.16.1 Preparation: Dissolve 500g of sodium chloride and 500g of hydroxylamine hydrochloride type II water and dilute to 3600mL.

7.17 Potassium permanganate, mercury-free, 5% solution (w/v).

7.17.1 Preparation: Dissolve 180 g of potassium permanganate type II water and dilute to 3600mL.

7.17.2 5% Potassium Permanganate solution may also be purchased.

7.18 Potassium persulfate, 5% solution (w/v).

7.18.1 Preparation: Dissolve 180 g of potassium persulfate type II water and dilute to 3600mL.

7.19 Calibration and laboratory control sample/matrix spiking mercury stock standard, 100 µg/mL (AccuStandard or equivalent).

7.20 Working standard preparation: 50mL Type II water mixed with 0.15mL nitric acid, 0.1mL mercury stock standard; dilute to 100mL.

7.20.1 Preparation of calibration standards (0.2, 0.5, 2.0, 5.0, and 10.0µg/l) - All mercury calibration standards are prepped each day and included in the first LIMS prep (digestion) batch of the day.

7.20.1.1 Liquid matrix calibration standards for methods 245.1 and 7470A:

7.20.1.1.1 Perform dilutions of mercury working standard with Type II water into the digestion vessels. The final concentration for each calibration level is listed in the following table:

Document Uncontrolled When Printed

Calibration Level	Volume of Calibration Working Solution (100 µg/L)	Final Volume (mL)	Final Hg Concentration (µg/L)
Blank	**0.0**	**50.0**	**0.0**
1	**0.1**	**50.0**	**0.2**
2	**0.25**	**50.0**	**0.5**
3	**1.0**	**50.0**	**2.0**
4	**2.5**	**50.0**	**5.0**
5	**5.0**	**50.0**	**10.0**

7.20.1.1.2 Add 2.5mL of Sulfuric Acid and 1.25mL of Nitric acid to each digestion vessel.

7.20.1.1.3 Add 7.5mL of potassium permanganate solution to each digestion vessel, and allow to stand for at least 15 minutes.

7.20.1.1.4 Add 4mL of potassium persulfate to each digestion vessel and heat for 2 hours in the Digestion Bloc maintained at 90 - 95℃.

7.20.1.1.5 Cool and add 3mL of sodium chloride-hydroxylamine Hydrochloride to reduce the excess permanganate.

7.20.1.1.6 Bring the cooled solution to a final volume of 50mL with DI water.

7.20.1.2 Solid matrix calibration standards for method 7471A:

7.20.2 Perform dilutions of mercury working standard with Type II water into the digestion vessels. The final concentration for each calibration level is listed in the following table: All mercury calibration standards are prepped each day and included in the first LIMS prep (digestion) batch of the day.

Calibration Level	Volume of Calibration Working Solution (100 µg/L)	Final Volume (mL)	Final Hg Concentration (µg/L)
Blank	**0.0**	**50.0**	**0.0**
1	**0.1**	**50.0**	**0.2**
2	**0.25**	**50.0**	**0.5**
3	**1.0**	**50.0**	**2.0**
4	**2.5**	**50.0**	**5.0**
5	**5.0**	**50.0**	**10.0**

7.20.2.1 Add enough Type II water to each vessel to make a total volume of 10mL. Add 0.6 mL of nitric acid, and 1.8 mL of hydrochloric acid to each standard. Heat for 2 minutes in the Digestion Bloc maintained at 90 - 95℃.

7.20.2.2 Cool and add 25mL of Type II water and 7.5mL of potassium permanganate solution to each bottle.

7.20.2.3 Mix thoroughly and place in the Digestion Bloc maintained at 90 - 95°C for 30 minutes.

7.20.2.4 Cool and add 3mL of sodium chloride-hydroxylamine hydrochloride to reduce the excess permanganate.

7.20.2.5 Bring the cooled solution to a final volume of 50ml with DI water.

7.21 Second Source Check Standard (stock), 100 µg/mL (ELEMENTS or equivalent).

7.22 Second Source Check Standard (working), 100 µg/L, 50mL Type II water mixed with 0.15mL nitric acid, 0.1mL mercury stock standard; dilute to 100mL.

7.23 ICV Solution

7.23.1 Preparation:

- ICV (4 µg/L): 2.0mL of 100 µg/L Second Source Check standard diluted to 50mL with Type II water.

7.24 LCS and MS/MSD Solution / CCV Solution

7.24.1 Preparation:

- LCS and MS/MSD (2 µg/L): 1.0mL of 100 µg/L Calibration Solution diluted to 50mL with Type II water.
- CCV (5 µg/L): 2.5mL of 100 µg/L Calibration Solution diluted to 50mL with Type II water.

7.25 Managers/supervisors or a designee are expected to check their areas on a monthly basis for expired standards/reagents and dispose of them according to SOP TA-EHS-0036.

8.0 Sample Collection, Preservation, Shipment and Storage

8.1 Sample holding time for mercury is 28 days from time of collection to the time of analysis.

8.2 Aqueous samples are preserved with nitric acid to a pH of <2 and may be stored in either plastic or glass. Refrigeration is not required. Preservation must be verified prior to analysis.

8.3 Soil samples do not require preservation, but are to be stored at ≤ 6°C until the time of analysis.

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time [1]	Reference
Waters	HDPE	50mL	HNO_3, pH < 2;	28 Days	40 CFR Part 136.3
Soils	Glass	3 grams	≤ 6 C	28 Days	N/A

[1] Inclusive of digestion and analysis.

9.0 Quality Control

This section describes routine quality control practices, which are also summarized in Attachment 1. Preparation of QC materials is described in Section 7. Initial calibrations and calibration verifications are discussed in Section 10. Initial performance studies are described in Section 12. Current control limits are stored in the laboratory LIMS system.

9.1 The process of establishing control limits, and the use of control charts are described more completely in TA-QA-0620, Quality Control Program.

9.2 QC result that fails to meet control criteria must be documented in a Nonconformance

Document Uncontrolled When Printed

Memo (NCM). *Failing QC that is automatically flagged by TALS does not need a NCM as long as it is a routine failure.* The NCM is approved by the supervisor and then automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA department also receives NCMs by e-mail for tracking and trending purposes. The NCM process is described in more detail in SOP # TA-QA-0610. This is in addition to the corrective actions described in the following sections.

9.3 Project-specific requirements can override the requirements presented in this section when there is a written agreement between the laboratory and the client, and the source of those requirements should be described in the project documents.

9.4 Preparation Batch

A group of up to 20 samples that are of the same matrix and are processed together using the same procedures and reagents. The preparation batch must contain a method blank, an LCS, and a matrix spike/matrix spike duplicate pair (MS/MSD). As discussed in the following sections, special program or project requirements can include additional requirements. Always refer to special project instructions for details before proceeding with the analysis.

9.5 Method Blank (MB)

The MB consists of an empty vessel or <1-mm glass beads (for DoD projects) containing all reagents specific to the method that is carried through the entire analytical procedure, including preparation and analysis. At least one method blank (MB) must be processed with each preparation batch.

Acceptance Criteria: Some programs (e.g., DoD) require control of method blanks to have a concentration less than or equal to one-half of the RL. *Some programs (LaMP) and method 7470A require no detections in the method blank more than 10% of the low limit calibration check solution. This can not be obtained in most cases. TestAmerica Seattle will only evaluate the method blank to 1/2 the RL or Project DQOs and when specific DQOs are not provided by the client the RL will be defined as the DQO.*

Corrective Action: All samples associated with an unacceptable method blank must be re-prepared and reanalyzed. If mercury was not detected in the samples, the data may be reported with qualifiers (check project requirements to be sure this is allowed) and it must be addressed in the project narrative.

9.6 Laboratory Control Sample (LCS)

The preparation of the LCS is described in Section 7.22. At least one aqueous LCS must be processed with each preparation batch. The LCS must be carried through the entire analytical procedure. If there is not sufficient sample volume for a matrix spike duplicate or sample duplicate, then precision information for the batch will need to be derived by processing a LCSD.

Acceptance Criteria: For Methods 7470/7471, the maximum control limits for LCS recoveries are 80-120%. In-house control limits based on three standard deviations of the mean of historical results are used as long as they are at least as tight as 80-120% (see SOP TA-QA-0620 for further details on establishing control limits).

Document Uncontrolled When Printed

For Method 245.1, the maximum control limits for LCS recoveries are 85-115%.

Corrective Action: If LCS recoveries are outside established control limits, the system is out of control and corrective action must occur. If recoveries are above control limits and mercury is not detected in samples, the data may be reported with qualifiers (check project requirements to be sure this is allowed - BP LaMP makes no allowance for out high/ND scenarios) and it must be addressed in the project narrative. In other circumstances, the entire batch must be re-prepared and reanalyzed.

9.7 Matrix Spike/Matrix Spike Duplicate (MS/MSD)

One MS/MSD pair must be processed for each preparation batch. Some programs may require the use of sample duplicates in place of or in addition to MS/MSDs. In addition, some programs will allow spikes to be reported only for project-related samples. Samples identified as field blanks cannot be used for MS/MSD analysis. If there is not sufficient sample volume for a matrix spike duplicate or sample duplicate, then precision information for the batch will need to be derived by processing a LCSD.

Acceptance Criteria: Control limits are statistically determined based on three standard deviations of the mean of the laboratory's historical data. The MS/MSD recoveries must fall within 80-120%; the relative percent difference (RPD) between the MS and MSD cannot exceed 20%.

Corrective Action: If analyte recovery or RPD fails acceptance criteria, the LCS recovery must be in control for the data to be reported. If there is no evidence of analytical problems and all other QC criteria are met, then qualified results may be reported and the situation must be described in the final report case narrative. In other circumstances, the batch must be re-prepared and reanalyzed.

If the native analyte concentration in the MS/MSD exceeds 4 times the spike level for that analyte, the recovery data are reported as NC (i.e., not calculated). If the reporting software does not have the ability to report NC, then the actual recovery must be reported and narrated as follows: "Results outside of limits do not necessarily reflect poor method performance in the matrix due to high analyte concentrations in the sample relative to the spike level."

9.8 **Instrument QC**

9.8.1 Initial Calibration (ICAL)

9.8.1.1 Calibration must be performed daily (every 24 hours) and each time the instrument is set up. The instrument calibration date and time must be included in the raw data.

9.8.1.2 Calibrate using five standards and a blank (see Section 7.20.)

NOTE: It is generally not acceptable to reject calibration points for this method.

9.8.1.3 The calibration curve must have a correlation coefficient of ≥ 0.995 or the instrument shall be stopped and recalibrated prior to running samples.

Sample results cannot be reported from a curve with an unacceptable correlation coefficient.

9.8.2 Initial and Continuing Calibration Blanks

9.8.2.1 An initial calibration blank (ICB) is tested immediately after the daily ICAL standards.

Acceptance Criteria: The absolute value of the blank result must be less than the reporting limit (< Limit of Detection (LOD) for DoD). As noted with the method blank, some programs require that results for blanks must be less than two times the method detection limit (refer to special project requirements). Samples analyzed for BP LaMP must be less than ½ the RL.

Corrective Action: If the blank acceptance limit is exceeded, the analysis should be terminated, the source of contamination identified, and the instrument recalibrated.

9.8.2.2 Continuing calibration blanks (CCBs) are run after every 10 samples and at the end of the run.

Acceptance Criteria: The absolute value of the blank result must be less than the reporting limit (< LOD for DoD). As just noted, some programs require that results for blanks must be less than two times the method detection limit (refer to special project requirements). Samples analysed for BP LaMP must be less than ½ the RL.

Corrective Action: If the blank acceptance limit is exceeded, the analysis should be terminated, the source of contamination identified, and the instrument recalibrated.

9.8.3 RL Calibration Check Standard (for BP and BNSF)

Calibration accuracy at the RL is verified by analyzing a standard prepared within two times the laboratory's standard reporting limit. The preparation of this standard is described in Section 7. For analytical sequences that include BNSF and/or BP LAMP samples, the Calibration Check Standard must be analyzed at the beginning of the analytical sequence, every 8 hours and/or at the end of the analytical sequence.

Acceptance Criteria: The acceptance limits are ± 50% of the expected value.

Corrective Action: If the RL Check standard fails to meet acceptance limits, a single reanalysis may be attempted without modification to the instrument operating conditions. Otherwise, the analysis must be terminated, the problem corrected, the instrument recalibrated, and the calibration re-verified.

Document Uncontrolled When Printed

9.8.4 Initial Calibration Verification (ICV)

The accuracy of the calibration standards is verified by testing a second source standard (ICV).

Acceptance Criteria: For Methods 7470/7471, the ICV recovery must be within 90-110%.

For Method 245.1, the ICV recovery must be 95-105%.

Corrective Action: If the ICV acceptance limit is exceeded, the analysis should be terminated, the accuracy of the calibration standards checked, and the instrument recalibrated.

9.8.5 Continuing Calibration Verification (CCV)

Calibration accuracy is monitored throughout the analytical run through the analysis of a known standard after every 10 samples and at the end of the run.

Acceptance Criteria: For Methods 7470/7471, the CCV recovery must be within 80-120%.

For Method 245.1, the CCV recovery must be 90-110%.

Correction Action: Sample results may be reported only when bracketed by valid CCV pairs. If a CCV fails, the analysis must be terminated, the problem corrected, the instrument recalibrated, the calibration verified, and the affected samples reanalyzed. If the cause of the CCV failure was not directly related to the instrument, the associated samples must be reanalyzed.

9.9 Any extra QC that is analyzed in a batch or sequence must be evaluated using the same criteria as the corresponding QC above.

10.0 Procedure

10.1 Sample Preparation

10.1.1 Liquid sample preparation methods 245.1 and 7470A.

10.1.1.1 Transfer 50mL, or an aliquot diluted to 50mL, to a digestion vessel. TCLP leachates are diluted 1:10 prior to digestion.

10.1.1.2 Add 2.5mL of concentrated sulfuric acid and 1.25mL of concentrated Nitric acid.

10.1.1.3 Add 7.5mL of potassium permanganate solution to each sample bottle

10.1.1.4 Add 4mL of potassium persulfate to each bottle and heat for 2 hours in the Digestion Bloc maintained at 90 - 95℃. Record the corrected and uncorrected temperatures of the digestion within the appropriate field of the batch prep sheet within the LIMS. Monitor the Digestion Bloc temperature during digestion.

10.1.1.4.1 If necessary, add more potassium permanganate until purple color persists for at least 15 minutes

10.1.1.5 Cool and add 3mL of sodium chloride-hydroxylamine hydrochloride to reduce the excess permanganate.

10.1.1.6 Bring the cooled solution to a final volume of 50mL with DI water.

10.1.2 Solid sample preparation method 7471A.

10.1.2.1 Check the Balance Logbook to determine if the daily calibration check was completed. If the balance requires a check, verify the calibration as detailed in SOP TA-QA-0014.

10.1.2.2 Using inert utensils, mix the sample thoroughly to achieve homogeneity (refer to SOP TA-QA-0028 for subsampling procedures).

Soil samples submitted under the BP LaMP must be transferred from their field container to a plastic or Teflon pan and then mixed thoroughly with inert utensils while reducing it to BB-sized clumps. After subsampling for this preparation, return unused portion to the original field container. Document on the container and in the digestion log that sample was "BP LaMP Homogenized.

10.1.2.3 Using an inert utensil, weigh triplicate 0.2 grams portions of sample into a digestion tube.

10.1.2.4 Add 2.5mL of Type II water, 0.6mL of nitric acid, and 1.8mL of hydrochloric acid to each sample, blank, and QC sample. Heat for 2 minutes in the Digestion Bloc maintained at 90 - 95℃. Record the corrected and uncorrected temperatures of the digestion within the appropriate field of the batch prep sheet within the LIMS.

10.1.2.5 Cool and add 25mL Type II water and 7.5mL potassium permanganate solution to each sample vessel. Add more if necessary, until the purple color persists for at least 15 minutes.

10.1.2.6 Mix thoroughly and place in the Digestion Bloc for 30 minutes at 90 - 95℃. Record the corrected and uncorrected temperatures of the digestion within the appropriate field of the batch prep sheet within the LIMS.

10.1.2.6.1 If necessary, add more potassium permanganate until purple color persists for at least 15 minutes

10.1.2.7 Cool and add 3mL of sodium chloride-hydroxylamine hydrochloride to reduce excess permanganate.

10.1.2.8 Bring the cooled solution to a final volume of 50mL with DI water.

10.1.3 *ISM Solid sample preparation method 7471A.*

10.1.3.1 *Check the Balance Logbook to determine if the daily calibration check was completed. If the balance requires a check, verify the calibration as detailed in SOP TA-QA-0014.*

10.1.3.2 *Using inert utensils, mix the sample thoroughly to achieve homogeneity (refer to SOP TA-QA-0028 for subsampling procedures).*

10.1.3.3 *Using an inert utensil, weigh triplicate 1.7 grams portions of sample into a 300 mL glass digestion tube.*

10.1.3.4 *Add 12.5 mL of Type II water, 3.0 mL of nitric acid, and 9 mL of hydrochloric acid to each sample, blank, and QC sample. Heat for 2 minutes in the Digestion Bloc maintained at 90 - 95℃. Record the*

Document Uncontrolled When Printed

corrected and uncorrected temperatures of the digestion within the appropriate field of the batch prep sheet within the LIMS.

10.1.3.5 *Cool and add 125 mL Type II water and 37.5 mL potassium permanganate solution to each sample vessel. Add more if necessary, until the purple color persists for at least 15 minutes.*

10.1.3.6 *Mix thoroughly and place in the Digestion Bloc for 30 minutes at 90 - 95℃. Record the corrected and uncorrected temperatures of the digestion within the appropriate field of the batch prep sheet within the LIMS.*

10.1.3.6.1 *If necessary, add more potassium permanganate until purple color persists for at least 15 minutes*

10.1.3.7 *Cool and add 15 mL of sodium chloride-hydroxylamine hydrochloride to reduce excess permanganate.*

10.1.3.8 *Bring the cooled solution to a final volume of 250 mL with DI water.*

10.2 Instrument Operating Conditions

10.2.1 The current operating conditions of each instrument must be either written, or printed out and attached to the corresponding instrument maintenance logbook.

10.3 Calibration

10.3.1 Initial Calibration Procedures. An instrument blank and five standards (0.2, 0.5, 2.0, 5.0, and 10.0µg/L) are injected in triplicate by the autosampler. The injection order is blank, 0.2µg/L, 0.5µg/L, 2.0µg/L, 5µg/L, and finally the 10µg/L standard. The instrument calculates an average value for each standard and the instrument blank; the average instrument response is used to generate the calibration curve. The %RSD for the triplicate measurements should be less than 20%. For details regarding calibration models and algorithms, refer to corporate SOP CA-Q-S-005.

10.3.2 Continuing Calibration Verification Procedures. After every 10 samples and at the end of the run, a continuing calibration verification (CCV) standard at 5.0µg/L, or other mid-range concentration is injected singly by an auto sampler. The analysis of the CCV is immediately following by the analysis of a continuing calibration blank (CCB).

10.4 Sample Analysis

10.4.1 Samples are poured into disposable culture tubes and placed in an autosampler rack for analysis.

10.4.2 Samples are analyzed using the Leeman system, following manufacturer's instructions. The solution concentrations are determined by the instrument data handling system and reported as raw instrument data.

10.4.3 When a sample concentration is above the upper calibration range the sample must be diluted and reanalyzed in order to bring the concentration within the calibration range.

10.5 Following is a typical analytical sequence:

ICAL
ICV
ICB
CRDL (for BP and BNSF)
Method Blank
LCS and LCSD (in included)
7 injections or 8 if only LCS in included
CCV and CCB
10 injections
CCV and CCB
10 injections
CCV and CCB

10.6 The instrument ID and the analyst initials must be documented on the raw data.

10.7 **Data Reduction and Review**

10.7.1 Upon completion of the analytical sequence, perform a level 1 data review and document the review on the data review checklist.

10.7.2 Submit the data package and review checklist to the peer reviewer for the level 2 review. The data review process is explained in SOP TA-QA-0635.

10.7.3 Update instrument sequence logbook.

10.8 **Instrument Maintenance**

10.8.1 All instrument maintenance must be documented in the instrument maintenance logbook.

- The logbook must include the instrument name, serial number for each major component (e.g., AA, autosampler) and the date of start-up.
- When an instrument is not capable of analyzing samples, it needs to be tagged "Out of Service".
- Routine Maintenance (which includes, but is not limited to daily, weekly, and semiannual maintenance) is completed periodically and does not necessary indicate the instrument is out of control is noted in the logbook with the notation "RM".
- For non-routine maintenance or repairs, logbook entries must include a description of the problem and what actions were taken to address the problem.
  - When non-routine maintenance or repairs are complete, the instruments return to control is noted in the logbook with the notation "RTC".

10.8.2 Daily Use and Maintenance.

10.8.2.1 These procedures are performed daily prior to instrument calibration.

10.8.2.2 Turn off the "overnight macro".

Document Uncontrolled When Printed

10.8.2.3 Check the stability of the diagnostics-aperture (should be ± 200 or better), zero detector, and acquisition 5-volt reference.

10.8.2.4 Check the rinse and $SnCl_2$ levels. The reservoirs should be full.

10.8.2.5 Start the "warmstrt" macro.

10.8.2.6 Run calibration macro ("cal245").

10.8.2.7 The Hg lamp intensities will be monitored each day and the value recorded in the instrument maintenance logbook.

10.8.3 Bi-monthly Maintenance

10.8.3.1 Change tubing as needed if pumping is not consistent.

10.8.3.2 Clean the pump clamps whenever tubing is changed.

10.8.3.3 Oil the autosampler bars.

10.8.4 Quarterly Maintenance

10.8.4.1 If the instrument counts drop below 250,000, clean the optic cell.

10.8.4.2 Clean out the interior of the instrument.

10.8.4.3 Change exhaust lines as needed.

10.8.5 Instrument QC Check

10.8.5.1 Whenever the optic cell is replaced, the "coldstrt" macro is run. This allows the optic cell to warm to a constant temperature, and will also condition new tubing. If the flow is consistent, the instrument is ready for calibration.

10.8.6 Spare Parts

10.8.6.1 Tubing

- Sample tubing.
- Reductant tubing.
- Drain tubing.
- Sample tips.
- Mixing Coil

10.8.6.2 Optics Cell

- Lenses
- Gaskets
- Complete spare optics cell

10.8.6.3 Other Parts

- Liquid gas separator
- Drying tube apparatus.

Document Uncontrolled When Printed

10.9 Trouble Shooting

13 Troubleshooting

13.1 Troubleshooting Tools

13.1.1 Module LED indicators

The LED lamps on the instrument modules are useful for indicating general health of the instrument. The LEDs can be seen on the front of the AA module and the Pump module.

13.1.1.1 AA Module LEDs

As seen from left to right:

Data Acquisition - Pulses for each data acquisition.
Program Mode - On when programming new firmware.
Timer Interrupt (CPU alive) - Pulses indicating the internal CPU is alive.
(Send/Receive) - Communication with PC.
(Send/Receive) - Communication with PC.
Power - On when power is operational.

13.1.1.2 Pump Module LEDs

As seen from left to right:

(Send/Receive) - Communication with PC.
(Send/Receive) - Communication with PC.
Power - On when power is operational.

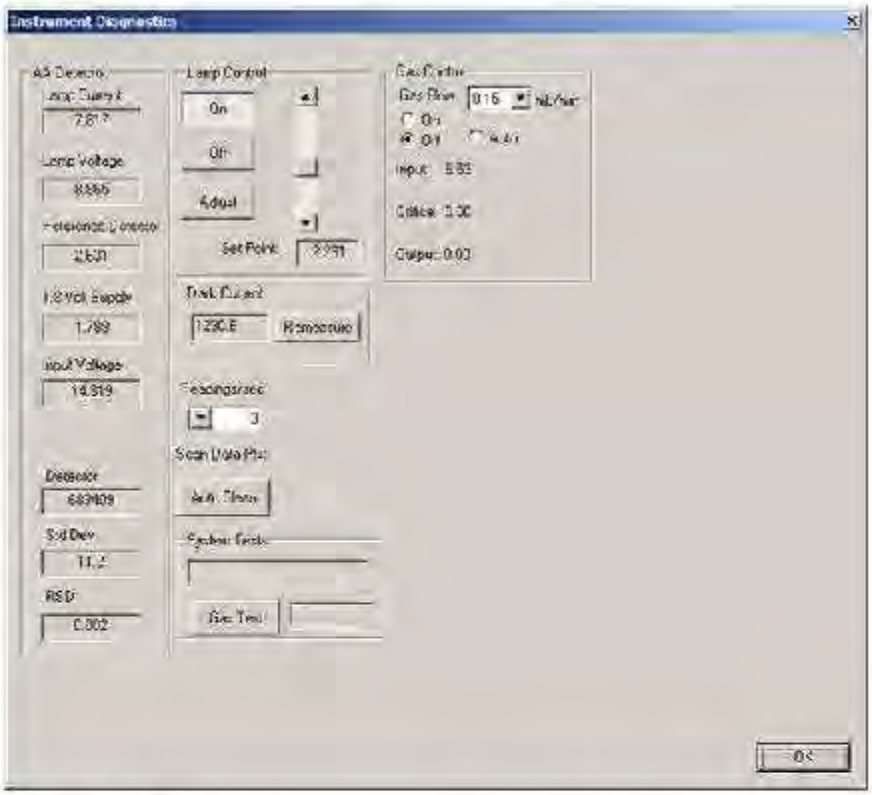
13.1.2 Instrument Diagnostics

Various diagnostic readouts can be viewed using the instrument diagnostic page. To view the instrument diagnostic page use the Tools pulldown menu and select the Instrument Diagnostics.

The Instrument Diagnostic page will open and various parameters will be displayed and updated in real time.

Company Confidential & Proprietary

Document Uncontrolled When Printed



Typical parameter values:

Parameter	Minimum	Maximum
Lamp Voltage	5	12.2
1.8Volt Supply	1.6	2.0
Input Voltage	12	18
Detector (Lamp on)	250000	8000000
Dark Current	10	2500
Orifice pressure (@0.50LPM)	0.59	0.63

13.1.2.1 Future Subject

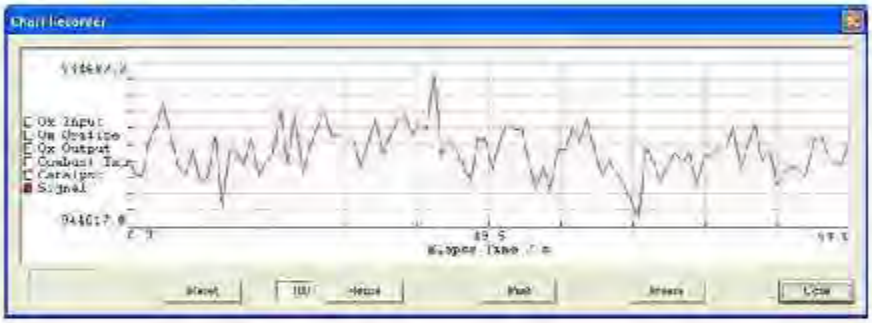
13.1.3 Real-Time Chart Recording

The Real-Time Chart Recorder is a screen that can be opened and viewed while running the instrument. The Chart Recorder can monitor various parameters by plotting values versus time. The chart recording automatically wraps around when it reaches the right most edge. To launch the chart recorder use Tools pulldown and select Chart Recorder.

Company Confidential & Proprietary

Document Uncontrolled When Printed

Select the parameter by clicking it on the left side of the chart recorder. To increase or decrease the time window, enter a new value to the left of the Resize button. Then click the Resize button to accept. Reset will restart the recording on the left side.



13.2 No or Low Signal for Standards

Possible Cause	Corrective Action
Hg Lamp is not turned on	Turn the mercury lamp on.
Stannous Chloride is not being pumped	Check that the pump tubing is not fatigued and that the lever is fully rotated to apply adequate pressure. Verify that the stannous chloride is flowing by temporary removal of the tubing from the reductant bottle and observing air pumping into the tubing.
Stannous Chloride is not potent	Replace the stannous chloride solution if it is old. The solution slowly oxidizes over time. Stannous chloride granules ($SnCl_2 \cdot 2H_2O$)

Company Confidential & Proprietary

Document Uncontrolled When Printed

	should go into solution in 10% HCl very rapidly. If it does not reorder.
Standards are not being pumped	Check that the pump tubing is not fatigued and that the lever is fully rotated to apply adequate pressure.
Standards are old	Standard properly acidified and in glass or Teflon containers should be stable for several days, even at low concentrations. However, if not properly acidified or stored in other types of plastic container, mercury can be lost quickly. Remake standard dilutions from a stock standard of known accuracy. This is especially true in nitric acid. The best stability is in HCl acid.
Carrier gas is not flowing	Check the liquid/gas separator to ensure proper gas flow. Check the exhaust of the CVAAS module by placing tubing into beaker of water and observe bubbling.
A leak in the sample vapor path	Check all tubing connections are tight and that the absorption cell windows are in place.
A blockage in the sample vapor path	Submerge the CVAAS exhaust line in a small beaker of water to confirm that the carrier gas is flowing throughout the system.

13.3 No or Low Signal for Samples (Standards are good)

Possible Cause	Corrective Action
Digestion is incomplete	Stannous chloride reduces Hg^{+2} to Hg^{0}. The oxidation steps in the digestion procedures are designed bring mercury to the +2 state. Samples high in organic content may require additional oxidant or heating during the digestion to complete oxidation
Samples are high in known interferents	If samples are high in interferents such as iodide, the signal response will be low. Diluting the sample will decrease the concentration of the interference and limit its impact.

13.4 Poor Precision for Repeats

Possible Cause	Corrective Action
The Signal has not reached plateau	Increase the uptake time or gas flow.
Integration time is too long	Once the signal has reached plateau it will continue to rise slowly. If repeats are taken

Company Confidential & Proprietary

Document Uncontrolled When Printed

	without returning to rinse, the slight rise is signal over time can result in poorer precision. Drop the integration time to 2 seconds.
Integration time is too short	If repeats are taken with a return to rinse between readings, a longer integration time should result in better precision. Increase the integration time to 5 seconds or more.
More care is required with timing in manual mode	If the sampler is not controlling solution uptake it is critical that the times for rinse and uptake be consistent. Move the probe to the appropriate solution and click OK promptly when directed.
Low light intensity	Check the signal value displayed on the Method/ Instrument Control display. The value should be 250000 or greater. If low make sure the cells are properly held in clamps and clean optics if necessary.

13.5 Lamp Does Not Light

Possible Cause	Corrective Action
Hydra II is not plugged in and powered up.	Check electrical connections and ensure right-most LED is lit on CVAAS module.
Envoy software reports instrument status as IDLE	Make sure all communication Indicators at the bottom right of the screen are green. If not, power down the instrument and exit Envoy software, power up the instrument and restart Envoy program. If problem persists call Customer Support.
Lamp status is Off	View Menu bar/Tools/Instrument Diagnostics. Confirm lamp is on. Click Adjust button and view lamp current & voltage values.
Lamp assembly is not fully inserted in control board	**CAUTION**- Power off instrument before touching Hg lamp. Pull up on the lamp to release contacts, then re-insert lamp noting the increased resistance as the contacts are made.
If all above is correct	Replace lamp assembly PN 122-00189-1. See Hg Lamp Replacement **CAUTION**- Power off instrument before touching Hg lamp. Call Customer Support at 1-800-LEEMANS

Company Confidential & Proprietary

Document Uncontrolled When Printed

11.0 Calculations / Data Reduction

For details regarding calibration models and algorithms, refer to corporate SOP CA-Q-S-005.

11.1 Accuracy

ICV / CCV, LCS % Recovery = $\frac{\text{observed concentration}}{\text{Known concentration}} \times 100$

MS % Recovery = $\frac{\text{(spiked sample) - (unspiked sample)}}{\text{Spiked concentration}} \times 100$

11.2 Precision (RPD)

Matrix Duplicate (MD) = $\frac{|\text{orig. sample value - dup. sample value}|}{[(\text{orig. sample value + dup. sample value})/2]} \times 100$

11.3 Concentration

11.3.1 Liquid samples, report as mg/L.

$$\text{mg/L} = \frac{\text{(instrument reading) * (final dilution)}}{\text{(Initial volume)}}$$

11.3.2 Solid samples (except filters), report as mg/kg:

$$\text{mg/kg} = \frac{\left[\frac{\text{(instrument reading) * (final dilution)}}{\text{(sample weight)}}\right]}{\text{(percent solids)}}$$

11.3.3 Filters, report as µg (or as µg/cm^3)

$$\mu\text{g} = \text{(instrument reading) * (final dilution)}$$

$$\mu\text{g/cm}^3 = \frac{\text{(instrument reading) * (final dilution) * (1000)}}{\text{(air volume in L)}}$$

NOTE: All dry weight corrections are made in LIMS at the time the final report is prepared.

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 (see SOP TA-QA-0602). 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

Analyst initial and continuing Demonstrations of Capability (DOC) are performed before any client samples are analyzed and are updated annually. See SOP TA-QA-0617 for details.

12.3 Training Requirements

See SOP TA-QA-0608 for detailed training requirements.

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

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 SOP TA-EHS-0036. The following waste streams are produced when this method is carried out.

14.1 Waste Streams Produced by the Method

14.1.1 Aqueous Acidic (Metals) - Corrosive. Acidic waste generated by the digestion with a mercury concentration less than 0.2ppm and rinse solutions are poured into the acid neutralization tank, neutralized and then discarded down the drain.

14.1.2 Remaining standard and any sample digestion fluid with a mercury concentration greater than 0.2ppm are disposed of in the high mercury concentration satellite disposal container located in the analysis lab. At or before the satellite waste containers reaches 55 gallons, it is taken to the waste warehouse where it is sent out for retort.

14.1.3 Expired standard and reagents: i.e. Potassium Permanganate or Potassium Persulfate will be lab packed and sent out for incineration.

15.0 References / Cross-References

15.1 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition, Final Update II, Revision I, September 1994, Method 7471A (Mercury).

15.2 Department of Defense Quality Systems Manual for Environmental Laboratories, Final Version 4.1, April 2009.

15.3 EPA-600/4-79-020, Methods for Chemical Analysis of Water and Wastes, March 1983, Methods 245.1 and 245.2.

16.0 Method Modifications:

Item	Method	Modification
1	245.1 and 7470A	This SOP allows for the use of reduced sample volumes to decrease waste generation. Reagent levels are adjusted to maintain the same ratios as stated in the source methods. According to a letter from Robert Booth of EPA EMSL-Cinn to David Payne of EPA Region V, "Reduction in sample size and appropriate corresponding reduction in sample volume is (sic) not considered a significant change in the methodology."
2	7470A and 7471A	Methods 7470A and 7471A state that working mercury standards "should be prepared fresh daily." The laboratory frequently prepares up to three batches of mercury samples, including digested calibration

Document Uncontrolled When Printed

Item	Method	Modification
		standards, each day. The third batch is typically prepared and digested late in the day, and then is analyzed the morning of the next day. The laboratory has developed the following information demonstrating that analysis within 24 hours, but on the second calendar day from preparation produces reliable results and is acceptable to the EPA: • Successful proficiency testing PT results for samples that were prepared and analyzed within 24 hours, but on successive days (e.g., ERA WP-66); • Successful analysis of true NIST mercury standards within every analytical batch; and • A written comment from the EPA MICE Hotline stating that, with the supporting lab data, their opinion was that the laboratory's practice is "within the letter of the method as written
3	7471A	80 mL polyethylene digestion vessels and a Bloc Digester are used during the procedure in lieu of the 300 mL BOD bottles and water bath.
4	SW-846	Chapter 1 of SW846 specifies the use of reagent water with a purity equivalent to ASTM Type II water. This SOP specifies the use of a Millipore DI system or equivalent to produce reagent water. This SOP requires that reagent water must be free of the analytes of interest as demonstrated through the analysis of method blanks
5	7470A	Chapter 1 of SW-846 states that the method blank should not contain any analyte of interest at or above the MDL. This SOP states that the method blank must not contain any analyte of interest at or above the reporting limit.
6	7471A	Method 7471A does not state control criteria within the text of the method. The QC section of 7471A refers the analyst to Section 8.0 of Method 7000A, the generic atomic absorption method, which discusses flame and furnace methods. The ICV criteria stated in Method 7000A is ± 10%. This SOP requires ICV control limits of ± 20% based on the fact that the mercury ICV, unlike the ICV for the flame and furnace analytes, is digested and therefore is equivalent to an LCS. The CLP protocol 245.5 CLP-M recognizes this factor and requires control limits of ± 20%.
7	245.1	Method 245.1 Section 12.8 states that concentrations should be reported as follows: Between 0.1 and 1 µg/g, to the nearest 0.01 µg; between 1 and 10 µg/g, to the nearest 0.1µg; and above 10 µg/g, to the nearest µg. TestAmerica Seattle reports all mercury results under this SOP to two significant figures.
8	*7470A and 7471A*	*Method Blank criteria of no detections in the method blank more than 10% of the low limit calibration check solution. TestAmerica Seattle will only evaluate the method blank to 1/2 the RL or Project DQOs and when specific DQOs are not provided by the client the RL will be defined as the DQO.*

17.0 Attachments

Attachment 1: Summary of Quality Control Requirements

18.0 Revision History

- Revision 22, dated 9 April, 2014

- Added Computer hardware and software, section 6.3
- Updated NCM criteria, section 9.2
- Added the prep procedure for ISM samples section 10.1.3
- Added information on the acceptance of the blank sections 9.5 and 16.0

- Revision 21, dated 29 July 26, 2013
  - Added option of purchasing potassium permanganate solution to section 7.17

- Revision 20, dated 6 August 2012
  - Updated safety intro, section 5.0
  - Updated waste streams, section 14.1

- Revision 19, dated 25 April 2011
  - Updated soil RL in section 1.1
  - Incorporated ROMD 00025 in sections 9.6 and 9.7
  - Incorporated ROMD 00021 in sections 10.1.1.4, 10.1.2.4 and 10.1.2.6.
  - Incorporated ROMD00005 into section 10.1.2.3.
  - Incorporated ROMD 00020 in section 10.2.1
  - Incorporated ROMD 00022 in sections 10.2.1 and 11.0
  - Added new section (10.7) on data reduction and review
  - Incorporated ROMD 00033 in section 10.8.2
  - In section 8.3 added that the required temp is ≤ 6 C
  - In the table under section 8.3 added the preservation temp of ≤ 6 C for mercury soils
  - In section 7.15 change sulfuric acid to hydrochloric acid when making up the stannous chloride reagent
  -
- Revision 18, dated 16 April 2010
  - Added documentation of reagents and reagent preparation (Section 7.1).
  - Added documentation of standards and standard preparation (Section 7.18).
  - Added removal of expired standards Section (7.25).
  - Added Method 245.1 QC acceptance criteria for LCSs in section 9.6 and Attachment 1.
  - Added CRDL standard, section 9.8.3
  - Added Method 245.1 QC acceptance criteria for ICVs and CCVs in sections 9.8.3 and 9.8.4.
  - Added instructions to evaluate extra QC (Section 9.9)
  - Added section 10.1.1.6
  - Added verification of balance calibration check (Section 10.1.2.1)
  - Added BP sample homogenization requirements (Section 10.1.2.2)
  - Updated standard concentrations in section 10.2.1.
  - Added requirement to apply correction factor to temperature readings throughout section 10.1
  - Added maintenance documentation and return to service requirements, Section 10.6.1.
  - Clarified the order of analysis for CCVs and CCBs (Section 10.2.2)
  - Added documentation of analyst and instrument ID (Section 10.5)
  - Updated use of 1:1sulfuric:Nitric acid to concentrated sulfuric acid only(Sections 7.21.1.1.2 and 10.1.1.2)

- Revision 17, dated 5 May 2009

Document Uncontrolled When Printed

  - Updated Summary of Quality Control Requirements table to include requirements from the DoD QSM v. 4.1

- Revision 16, dated 27 March 2008
  - Integration for TestAmerica and STL operations.
  - This revision is a complete rewrite and an expansion of scope.

Document Uncontrolled When Printed

Attachment 1.

Summary of Quality Control Requirements

QC PARAMETER	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
Minimum five-point Initial Calibration	Beginning of every analytical run, every 24 hours, when instrument is modified, or CCV fails.	r ≥ 0.995	Terminate analysis; Correct the problem; Prepare new standards; Recalibrate following system performance.
ICV	Immediately following ICAL	95 -105% for 245.1 90 - 110 % for 7470A/7471A including DoD	Terminate analysis; correct the problem; recalibrate or re-prepare and reanalyze batch.
ICB	Following ICB	Absolute value < RL **For DoD**: < LOD	Terminate analysis; correct the problem; recalibrate or re-prepare and reanalyze batch.
CCV	Every 10 samples and at the end of the run	90 -110% for 245.1 80 - 120 % for 7470A and 7471A including DoD	Terminate analysis; correct the problem; recalibrate and rerun all samples not bracketed by acceptable CCVs or re-prepare and reanalyze batch.
CCB	Immediately following each CCV	Absolute value < RL **For DoD**: < LOD	Terminate analysis; correct the problem; recalibrate and rerun all samples not bracketed by acceptable CCVs or re-prepare and reanalyze batch.
Method Blank	One per sample preparation batch of up to 20 samples.	≤RL; **For DoD**: ½ RL Sample results greater than 10% the blank concentration are acceptable. Samples for which the contaminant is < RL do not require re-digestion.	Re-digest and reanalyze samples. Note exceptions under criteria section.
Laboratory Control Sample (LCS)	One per sample preparation batch of up to 20 samples.	85 - 115% for 245.1. Recovery must be within statistical control limits, not to exceed 80 - 120% for 7470A/7471A.	Terminate analysis; correct the problem; redigest and reanalyze all samples associated with the failed LCS.
Matrix Spike	One per sample preparation batch of up to 20 samples.	Recovery must be within statistical control limits, not to exceed 75-125% (80-120% for DoD). 50-150% for TCLP Leachates	In the absence of client specific requirements, flag the data; no flag required if the sample level is > 4x the spike added.
Matrix Spike Duplicate	See Matrix Spike	Recovery within statistical control limits, not to exceed 75-125 % recovery; RPD ≤ 20% (80-120% for DoD)	See Corrective Action for Matrix Spike.

Summary of Quality Control Requirements

QC PARAMETER	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
Duplicate		≤20% RPD for water ≤35% RPD for solid ≤50% RPD for leachates **For DoD:** ≤20% RPD for all	See Corrective Action for Matrix Spike.

* An RL check standard is analyzed for BP LaMP and BNSF. The acceptance criteria is ±50% of the true value.

Title: Inductively Coupled Plasma-Mass Spectrometry (ICP-MS) [Methods 6020, *6020A*, 200.8]

Approvals

Signatures on File

Stan Palmquist — Date Metals Department Manager	Sandra Langway — Date Health & Safety Manager / Coordinator
Terri Torres — Date Quality Assurance Manager	Heather Prater — Date Laboratory Director

Copyright Information:
This documentation has been prepared by TestAmerica Laboratories, Inc. and its affiliates ("TestAmerica"), solely for their own use and the use of their customers in evaluating their qualifications and capabilities in connection with a particular project. The user of this document agrees by its acceptance to return it to TestAmerica upon request and not to reproduce, copy, lend, or otherwise disclose its contents, directly or indirectly, and not to use it for any other purpose other than that for which it was specifically provided. The user also agrees that where consultants or other outside parties are involved in the evaluation process, access to these documents shall not be given to said parties unless those parties also specifically agree to these conditions.

THIS DOCUMENT CONTAINS VALUABLE CONFIDENTIAL AND PROPRIETARY INFORMATION. DISCLOSURE, USE OR REPRODUCTION OF THESE MATERIALS WITHOUT THE WRITTEN AUTHORIZATION OF TESTAMERICA IS STRICTLY PROHIBITED. THIS UNPUBLISHED WORK BY TESTAMERICA IS PROTECTED BY STATE AND FEDERAL LAW OF THE UNITED STATES. IF PUBLICATION OF THIS WORK SHOULD OCCUR THE FOLLOWING NOTICE SHALL APPLY:

©COPYRIGHT 2012 TESTAMERICA LABORATORIES, INC. ALL RIGHTS RESERVED.

Facility Distribution No. 001 **Distributed To:** Lab Intranet

1.0 Scope and Application

1.1 This procedure describes multi-elemental analysis by inductively coupled plasma-mass spectrometry (ICP/MS) based on EPA Methods 200.8 and 6020.

1.2 Method 200.8 lists twenty-one elements approved for analysis by ICP/MS (Al, Sb, As, Ba, Be, Cd, Cr, Co, Cu, Pb, Mn, Hg, Mo, Ni, Se, Ag, Tl, Th, U, V, and Zn). Method 6020 lists fifteen elements approved for analysis by ICP/MS (Al, Sb, As, Ba, Be, Cd, Cr, Co, Cu, Pb, Mn, Ni, Ag, Tl, and Zn). This procedure has been developed for thirty-five elements (see Table VII), and additional elements may be included provided that the method performance criteria presented in Sections 9 and 10 are met. However, project approval may be required from the controlling agencies for compliance testing beyond the elements included in the promulgated methods. See Table XII for a list of elements and associated reporting limits.

1.3 The procedure is applicable to the analysis of acid digested waters, soils, and wastes. The preliminary acid digestion for aqueous samples is described in SOP TA-IP-0205, and the digestion procedure for soils is given in SOP TA-IP-0220.

1.4 On occasion clients may request modifications to this SOP. These modifications are handled following the procedures outlined in Section 12.2.1 in the Quality Assurance Manual.

2.0 Summary of Method

2.1 Aqueous samples, digestates, or leachates are nebulized into a spray chamber where a stream of argon carries the sample aerosol through the quartz torch and injects it into a R.F. plasma. There the sample is decomposed and desolvated.

2.2 The ions produced are entrained in the plasma gas and by means of a water-cooled, differentially pumped interface, introduced into a high-vacuum chamber that houses a quadrapole mass spectrometer capable of providing a resolution better than or equal to 0.9 amu (see Section 3.1) peak width at 10% of the peak height. For analysis by methods 200.8, the resolution requirement is 1.0 amu at 5% peak height. The ions are sorted according to their mass-to-charge ratio and measured with a channel electron multiplier.

2.3 Interference must be assessed and valid corrections applied, or the data flagged to indicate problems. Interference correction must include compensation for background ions contributed by the plasma gas, reagents, and the constituents of the sample matrix. Recommended elemental equations, which correct for many of these interferences, are listed in Table I. Use of the internal standard technique is required to compensate for suppressions and enhancements caused by sample matrices.

3.0 Definitions

3.1 Atomic Mass Unit (amu) – Obsolete term replaced by “unified atomic mass unit (u)” or “dalton (Da)”, which denotes a small unit of mass that is used to express atomic and molecular masses. It is defined to be 1/12 of the mass of one atom of carbon-12, or $1.66053886 \times 10^{-27}$ kg.

3.2 Dissolved Metals - Those elements which pass through a 0.45-µm membrane filter (sample is acidified after filtration).

3.3 Suspended Metals - Those elements which are retained by a 0.45-µm membrane filter.

3.4 Total Metals - The concentration determined on an unfiltered sample following vigorous acid digestion.

3.5 Total Recoverable Metals - The concentration determined on an unfiltered sample following treatment with hot, dilute mineral acids.

3.6 Instrument Detection Limit (IDL) - See Section 12.1.1.

3.7 Sensitivity - The slope of the analytical curve (i.e., the functional relationship between raw instrument signal and the concentration).

3.8 Tuning Solution - This is a multi-element solution containing analytes which are representative of the entire mass range capable of being scanned by the instrument. It is used to optimize the sensitivity of the instrument and to verify the mass resolution meets method criteria.

3.9 Initial Calibration Verification / Quality Control Standard (ICV/QCS) - A multi-element standard of known concentrations prepared to verify instrument calibration. This solution must be an independent standard prepared near the mid-point of the calibration curve, and at a concentration other than that used for instrument calibration.

3.10 Continuing Calibration Verification (CCV) - A multi-element standard of known concentrations prepared to monitor and verify the instrument daily continuing performance.

3.11 Interference Check Standard (ICS) - A solution containing both interfering and analyte elements of know concentration that is used to verify background and interelement correction factors.

3.12 Laboratory Control Sample / Laboratory Fortified Blank (LCS/LFB) - A multi-element standard of known concentrations that is carried through the entire sample preparation and analysis procedure. This solution is used to verify the accuracy of the sample preparation.

3.13 Reagent Blank - High purity (> 18 megohm-cm) water containing the same acid matrix as the calibration standards that is carried through the entire digestion process.

3.14 Calibration Blank - High purity (> 18 megohm- cm) water acidified with the same acid concentrations present in the standards and samples. Also referred to as the Initial Calibration Blank (ICB) and Continuing Calibration Blank (CCB).

3.15 Method Detection Limit (MDL) - See section 12.1.2.

4.0 Interferences

4.1 Isobaric Interferences

4.1.1 Isobaric interferences in the ICPMS are caused by isotopes of different elements forming ions with the same nominal mass-to-charge ratio (m/z). Most interferences of this type are corrected for by the instrument software.

4.2 Isobaric Molecular and Doubly Charged Ion Interferences

4.2.1 Isobaric molecular interferences are caused by ions consisting of more than one atom or charge. Table III lists isobaric interferences which might possibly affect required analytes. When these interferences cannot be avoided by the use of another isotope with sufficient natural abundance, corrections must be applied and the data flagged to indicate the presence of interferences.

4.2.2 Chloride in samples can produce low recoveries for antimony and silver. If chloride interference is a concern, 1% HCl can be added during digestion, but calibration standards must be adjusted to include 1% HCl also.

4.3 Physical Interferences

4.3.1 Physical interferences are associated with the transport and nebulization process. Internal standards are used to compensate for these types of interferences.

4.3.2 Internal standards should be added at a level to give greater than *100,000* counts of raw signal intensity. The mass of the internal standard should ideally be within 50 amu of the mass of the measured analyte.

4.3.3 Matrix effects are monitored by comparing the internal standard intensity in the sample to the internal standard intensity of the calibration blank. When performing method 6020, the internal standard intensities must be between 30% and 120% of the intensities in the calibration blank. *For method 6020A, the internal standard intensities must be ≥70%.* If they fall outside this window, a five-fold dilution (1:4) is performed on the sample to correct for matrix effects and the sample is reanalyzed. When performing method 200.8, the internal standards must be between 60% and 125% of the calibration blank. If they are outside this window, the calibration blank is reanalyzed to verify internal standard intensities. If the intensities are within limits, the sample is diluted by a factor of 2 (1:1) and is reanalyzed.

4.3.4 Memory effects are dependent on the relative concentration differences between samples and/or standards which are analyzed sequentially. The rinse period between samples must be long enough to eliminate significant memory interference.

4.4 The use of hydrochloric and sulfuric acids should be minimized due to higher incidence of molecular-ion interferences with the presence of these acids. Excessive amounts of nitric acid can also lead to molecular interferences.

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

5.1.1 The ICP plasma emits strong UV light and is harmful to vision. All analysts must avoid looking directly at the plasma. The RF Generator produces strong radio frequency waves, most of which are unshielded. People with pacemakers should not go near the instrument while in operation.

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.

Materials with Serious or Significant Hazard Rating

Material (1)	Hazards	Exposure Limit (2)	Signs and Symptoms of Exposure
Nitric Acid	Corrosive Oxidizer Poison	2 ppm-TWA 4 ppm-STEL	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Hydrochloric Acid	Corrosive Poison	5 ppm-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.

(1) Always add acid to water to prevent violent reactions.
(2) Exposure limit refers to the OSHA regulatory exposure limit.

6.0 Equipment and Supplies

6.1 Instrumentation

- Inductively Coupled Plasma Mass Spectrometer (ICP/MS) capable of providing resolution, less than or equal to 0.9 amu at 10% peak height from 6-253 amu and 1.0 amu at 5% peak height from 6-253 amu with a data system that allows corrections for isobaric interferences and the application of the internal standard technique
- Autosampler with autosampler tubes
- A four-channel peristaltic pump
- Vacuum pump, Recirculating Chiller, Spray Chamber Cooling Power Pack

6.2 Computer hardware and software

- *Computer with a minimum 1GB memory, Pentium 4 processor, 80 G hard drive or equivalent or as recommended by instrument manufacturer.*
- *Data acquisition/processing system: Agilent 7500 ICP-MS ChemStation or equivalent*
- *LIMS system: TALS version 1.0 or higher*

6.3 Supplies

- Calibrated automatic pipettes or Class A glass volumetric pipettes
- Argon gas: High purity grade (99.99%)

7.0 Reagents and Standards

7.1 Document reagent/standards and reagent/standard preparation in TALS using the reagent module as described in SOP TA-QA-0619.

7.2 Standards

7.2.1 Storage and Shelf-Life

7.2.1.1 All standards must be stored in FEP fluorocarbon or previously unused polyethylene or polypropylene bottles. Standards stored at concentrations as received from the vendor and mid-level dilutions must be replaced prior to the expiration date assigned by the vendor. If no expiration date is provided, the stocks and mid-level standards may be stored for up to one year. They must be replaced sooner if verification from an independent source indicates a problem.

7.2.1.2 Working standards, i.e., all standards at concentrations ready to analyze on the ICP/MS (all except tuning mixes, ICSA and ICSAB mixes, which are received at ready-to-use concentrations), are prepared every three months.

7.2.2 The tuning solution is purchased as a custom multi-element mix or as single element solutions. The elements and concentrations of the constituents are shown in Table VIII.

7.2.3 Initial calibration standards are purchased as custom multi-element mixes or as single element solutions. The standards are prepared every three months and diluted to working levels using a combination of 2% nitric acid and *1.5*% hydrochloric acid. The concentrations are given in Table XI.

7.2.4 Initial calibration verification (ICV) standards are obtained from a source different than the source for the calibration standards. The ICV standards are prepared every three months in a solution of 2% nitric acid and *1.5*% hydrochloric acid to the concentrations shown in Table XI.

7.2.5 Continuing calibration verification (CCV) standards are prepared from the same source as the calibration standards. The CCV standards are prepared every three months in a solution of 2% nitric acid and *1.5*% hydrochloric acid. The concentration is different than the ICV, as shown in Table XI.

7.2.6 Reporting limit (RL), *LLICV, and LLCCV* verification standards are prepared every three months from the same stock as the calibration standards using a solution of 2% nitric acid and *1.5*% hydrochloric acid. The concentrations must be less than or equal to the reporting limits.

7.2.7 Linear dynamic range studies are conducted at *10,000* µg/L every six months.

7.2.8 Spiking solutions are CLP Sample Spike solution. Spike concentrations are listed in Table XI.

7.3 Reagents

7.3.1 Reagent Water

ASTM Type I or equivalent for the elements of interest, generated using an ion-exchange water polishing system capable of achieving 18.0 megohm-cm.

7.3.2 Acid Diluent, 2% HNO_3 and *1.5*% HCl

Carefully dilute 220 mL of concentrated HNO_3 and *165* mL of concentrated HCl to 11 L with reagent water. This solution is used to dilute samples, and it is used for calibration blanks.

7.4 Managers/supervisors or a designee are expected to check their areas on a monthly basis for expired standards/reagents and dispose of them according to SOP TA-EHS-0036.

8.0 **Sample Collection, Preservation, Shipment and Storage**

8.1 Aqueous samples and digestates are stored at room temperature.

8.2 Aqueous samples are preserved with nitric acid to a pH of 2, and may be stored in plastic or glass. Preservation must be verified prior to analysis.

8.3 Soil samples do not require preservation, but must be stored at 0-6 ℃ until the time of preparation.

8.4 The analytical holding times for metals are six months from the time of collection.

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time [1]	Reference
Waters	HDPE	50 mLs	HNO_3, pH < 2;	180 Days	40 CFR Part 136.3
Soils with Hg Analysis	Glass	1 gram	≤ 6 C	180 Days	SW-846, Chapter 3 Table 3.1
Soils	Glass	1 gram	None	180 days	SW-846, Chapter 3 Table 3.1

[1] Inclusive of digestion and analysis.

9.0 **Quality Control**

9.1 Quality control requirements are also summarized in TABLE X.

9.2 Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method. Control limits are maintained in the laboratory LIMS system. See QC SOP TA-QA-0620 for definition of QC terms, details about establishing control limits, minimum elements of a preparation batch, and general guidelines for evaluating batch QC.

9.3 QC result that fails to meet control criteria must be documented in a Nonconformance Memo (NCM). *Failing QC that is automatically flagged by TALS does not need a NCM as long as it is a routine failure.* The NCM is approved by the supervisor and then automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate and the NCM can be included in the report narrative. The QA department also receives NCMs by e-mail for tracking and trending purposes. The NCM process is described in more detail in SOP TA-QA-0610.

9.4 Method Blank / Laboratory Reagent Blank (MB/LRB)

For aqueous and soil samples, the method blank consists of reagent water that has been processed in the same manner as the samples. For soil samples analyzed under the DoD QSM, the method blank consists of polyethylene beads that have been processed in the same manner as the samples. One method blank must be processed with each preparation batch. In addition, the method blank should be analyzed at the same dilution as the associated samples.

Acceptance Criteria: Method blank results are acceptable if the concentration for each analyte of interest is less than the applicable reporting limit (RL).

NOTE: Some programs (e.g., DoD) require control of method blanks to have a concentration less than or equal to one-half of the RL. *Some programs (LaMP) and method 6020A require no detections in the method blank more than 10% of the low limit calibration check solution. This can not be obtained in most cases. TestAmeria Seattle will only evaluate the method blank to 1/2 the RL or Project DQOs and when specific DQOs are not provided by the client the RL will be defined as the DQO.*

Corrective Action: If the method blank does not meet the acceptance criteria, the source of contamination should be investigated to determine if the problem can be minimized or eliminated. 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):

- The same analyte was not detected in the associated samples;
- The method blank concentration is less than 1/10 of the measured concentration of any sample in the batch;
- The method blank concentration is less than 1/10 the specified regulatory limit; or
- The analyte is a common laboratory contaminant (copper, iron, lead, calcium, magnesium, potassium, sodium, or zinc) less than 2 times the RL. Note that some programs do not recognize common lab contaminants.

If the above criteria are not met and reanalysis is not possible, then the sample data must be qualified. **This anomaly must be addressed in the project narrative and the client must be notified.**

9.5 Laboratory Control Sample / Laboratory Fortified Blank (LCS/LFB)

The LCS consists of reagent water that is spiked with the analytes of interest as summarized in Table XI. For soil samples analyzed under the DoD QSM, the LCS consists of polyethylene beads that have been spiked with the analytes of interest and processed in the same manner as the samples. One LCS must be processed for each preparation batch. However, if there is not sufficient sample volume for a matrix spike duplicate or sample duplicate, then precision information for the batch will need to be derived by processing a LCSD.

Acceptance Criteria: LCS control limits are based on three standard deviations of past laboratory results. These limits are not to exceed 85-115% recovery for Method 200.8 or 80-120% for Method 6020. The control limits are maintained in the LIMS system.

Corrective Action: If the LCS % recovery falls outside of the control limits for any analyte, that analyte is judged to be out of control. All associated samples must be reprocessed for analysis. One possible exception is a recovery for a given element above the upper control limit with

no detection for the same element in the samples. (No such allowance is permitted for BP LaMP samples).

9.6 Matrix Spike / Matrix Spike Duplicate / Laboratory Fortified Sample Matrix / Laboratory Fortified Matrix Duplicate (MS/MSD/LFM/LFMD)

An MS is prepared by taking a second aliquot of a selected sample and spiking it with the analytes of interest as summarized in Table XI. An MSD is prepared by taking a third aliquot of a selected sample and spiking it with the analytes of interest as summarized in Table XI. The MS and MSD are processed in the same manner as the samples. One MS/MSD pair must be processed for each preparation batch. If there is not sufficient sample volume for a matrix spike duplicate or sample duplicate, then precision information for the batch will need to be derived by processing a LCSD. The spike concentration should be the same level as the LCS. (For BP LaMP samples, a trip blank or field blank should not be used for MS/MSD).

Acceptance Criteria: Control limits are based on three standard deviations of past laboratory results. These limits are not to exceed 80-120% recovery, and 20% relative percent difference (RPD). The control limits are maintained in the LIMS system.

Corrective Action: If MS/MSD results do not meet the acceptance criteria and all other quality control criteria have been met, then matrix interference is suspected. Failed matrix spikes are flagged automatically, and are discussed in the final report case narrative.

9.7 Interference Check Solutions (ICSA/ICSAB) **method 6020 *and 6020A* only**

NOTE: It may not be possible to obtain pure ICSA or ICSB standards

The interference check solution is prepared with known concentrations of interfering elements so a determination may be made as to the magnitude of the interference on analytes of interest as well as a test of any software corrections. The required elements and their concentrations are listed in Table VI. The interference check solutions must be analyzed at the beginning of every analytical run or once every 12 hours (for BP LaMP samples - prior to analytical run, every 8 hours and after analysis), whichever is more frequent. The results of solution "A" and solution "AB" should be monitored for possible interferences. See Table VI for analyte concentrations.

Acceptance Criteria: The results for the interference solution (A portion) must be $\leq$ LOD (unless they are a verified trace impurity form one of the spike analytes).

Corrective Action: If ICSA results exceed the LOD and the suspected trace impurities aren't verified, then the analysis sequence must be terminated. The problem must be investigated and fixed. The ICS and all affected samples must be re-analyzed. If the contamination can be confirmed by another method (e.g., ICPAES), acceptance criteria will be applied at that level and the data accepted.

Acceptance Criteria: The results for the trace elements (B portion) must be within ± 20% of the expected value.

Corrective Action: If the ICSAB results exceed the 20% limit, then the analysis sequence must be terminated. The problem must be investigated and fixed. The ICS and all affected samples must be re-analyzed.

9.8 Internal Standards Evaluation for Samples

9.8.1 Method 6020

The IS recovery in samples must be between 30% and 120% of the intensity of the calibration blank. *For 6020A, IS recovery must be greater or equal to 70%.* If sample IS recoveries fall outside of this criterion, a five-fold (1:4) dilution must be performed, the dilution analyzed, and the same acceptance criteria applied.

9.8.2 Method 200.8

The internal standards in samples must be between 60% and 125% of the intensity in the calibration blank. If the sample intensities fall outside this range, the calibration blank is reanalyzed to confirm the instrument has not drifted out of control. If the criteria are met, the sample is diluted by a factor of 2 (1:1) and reanalyzed.

9.8.3 IS limits and corrective actions for standards and blanks are described in Section 10.

9.9 Serial Dilution **method 6020 *and 6020A* only**

One serial five-fold dilution should be analyzed per batch for each matrix. If the analyte concentration is within the linear range of the instrument and sufficiently high (generally, a factor of *10* times above the *RL or for BNSF and BP LaMP a factor of 5 times above the RL*), the serial dilution must agree to within 10% of the original analysis. If not, an interference effect is suspected, which must be described in an anomaly report and included in the final report narrative. Samples identified as blanks cannot be used for serial dilution.

9.10 Post-Digestion Spike Addition (PDS) **method 6020 *and 6020A* only**

A PDS is performed for each batch. An analytical spike added to a portion of a prepared sample, or its dilution, should be recovered to within 75 - 125% of the known value. If the PDS fails to meet this criterion, matrix interference should be suspected. *For 6020A, PDS recoveries are 80 - 120%.*

9.11 For analytical sequences that include BNSF and/or BP LAMP samples, the RSD between multiple instrument integrations must be <20% if the analyte is greater than the reporting limit. If the RSD is above 20% then the laboratory must reanalyze the sample.

9.12 Any extra QC that is analyzed in a batch or sequence must be evaluated using the same criteria as the corresponding QC above. *Corrective action for a failing PDS is a Serial Dilution Test. This test is run as standard practice in all analytical batches.*

10.0 Procedure

One-time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using an NCM. The NCM is approved by the supervisor and then automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA department also receives NCMs by e-mail for tracking and trending purposes. The NCM process is described in more detail in SOP TA-QA-0610. The NCM shall be filed in the project file and addressed in the case narrative.

Any unauthorized deviations from this procedure must also be documented as a nonconformance, with a cause and corrective action described.

10.1 Sample Preparation

Solid and aqueous samples must be digested prior to analysis by the appropriate method (see SOPs TA-IP-0204 and TA-IP-0205).

10.2 Instrument Start Up

Set up the instrument according to manufacturer's operating instructions. Allow the instrument to become thermally stable for at least 30 minutes before tuning. The current operating conditions of each instrument must be either written, or printed out and attached to the corresponding instrument maintenance logbook.

10.3 Instrument Tuning / Mass Calibration

10.3.1 Tune the instrument with a solution containing elements representing all of the mass regions of interest. The relative standard deviations must be less than 5% after running the tuning solution a minimum of 4 times. For method 200.8, the tuning solution must be analyzed 5 times with a relative standard deviation less than 5%.

10.3.2 Mass calibration and resolution checks using the tuning solution must be completed at the beginning of every day.

10.3.2.1 Mass Calibration Check - The mass calibration results must be within 0.1 amu from the true value. If this criterion is not met, the mass calibration must be adjusted before running samples.

10.3.2.2 Mass Resolution Check - The resolution must be verified to be less than 0.9 amu full width at 10% peak height. Due to a limitation of the instrument software, the resolution requirement for method 200.8 of 1.0 amu full width at 5% peak height cannot be verified automatically. If the mass resolution requirement of 0.9 amu at 10% peak height is met, the 200.8 requirement is also satisfied.

10.4 Initial Calibration

10.4.1 The ICP/MS is calibrated each day of operation using a blank and five standards (see Section 7.2.3). Report the average of at least three integrations. For details regarding calibration models and algorithms, refer to corporate SOP CA-Q-S-005.

10.4.2 The validity of the calibration is determined by the subsequent calibration verifications, which are performed at concentrations as described in the next sections.

10.4.3 The internal standard recoveries for 6020 must fall between 30% and 120% of true values, *6020A must be ≥70%,* and for 200.8 must fall between 60% and 125% of true values.

10.5 Second-Source Initial Calibration Verification (ICV)

An ICV standard (see Section 7.2.4) is analyzed immediately after the initial calibration. This is a standard obtained from a different vendor than the standard used for calibration. This analysis also satisfies the Method 200.8 requirement for a Quality Check Standard (QCS).

Acceptance Criteria: The ICV recovery must be within 90-110%. The ICV can be reanalyzed, but must be successful twice in succession or corrective action must be taken.

Corrective Action: If the ICV results are outside of the acceptance limits, investigate the accuracy of the standards, correct as necessary, and recalibrate.

10.6 Calibration Blank

An initial calibration blank (ICB) is analyzed after the ICV. Continuing calibration blanks (CCBs) are analyzed after each continuing calibration verification.

Acceptance Criteria: Results for the calibration blanks must be less than the RL.

Note: All projects that are under the DoD QSM will evaluate the calibration blanks to ensure the criteria of no analytes being detected above the LOD. Any analyte above the LOD will be qualified B on all associated samples.

BP LAMP requires control of calibration blanks to a concentration less than 1/ 2 RL.

Corrective Action: If the calibration blank exceeds acceptance limits, then the possibility of instrument contamination should be examined, particularly the possibility of carry-over from high level samples. The blank can be reanalyzed, and if successful, analysis can continue. However, samples tested after high-level samples should be retested. If the reanalysis is not successful, then the analysis should be terminated. After the problem is corrected, recalibrate and reanalyze all samples tested since the last acceptable CCB.

10.7 Reporting Limit (RL) Verification Standard, *LLICV, LLCCV*

An independent standard is analyzed after the ICV to monitor the lab's ability to produce reliable results at RL-level concentrations. The RL verification standard (see Section 7.2.6) is analyzed after the daily ICB. (For BP LaMP the RL verification standard is run prior to analysis, every 8 hours and after analysis. The standard must be within 2 times the RL concentration.)

Acceptance Criteria: For project reporting limits at or above two times the MDL, the results should be within 50% of the expected value. Note that the DoD QSM requires control of the low-level calibration check standard to ±20% of the expected value, in which case the RLs will need to be three or more times the MDL concentration. *For 6020A and DoD QSM 5.0, the acceptance limit is ± 20% for LLICV. For 6020A, the acceptance limit is ± 30% for LLCCV. For BP LAMP, the acceptance limit is ± 30% for LLICV and LLCCV.*

Corrective Action: If the RL verification fails to meet acceptance limits, data for the associated samples must be assessed. For example, if the results are high, consider blank contamination, and if the results are low, consider MDL verifications. At a minimum, sample results must be qualified in the final report.

10.8 Continuing Calibration Verification (CCV) Standard

A 50-µg/L CCV standard is analyzed after every set of ten samples and at the end of the analytical sequence.

Acceptance Criteria: The CCV recovery must be within 90-110%. If CCV results are not within these limits, the CCV can be reanalyzed, but it must be successful twice in succession or further corrective action must be taken.

Corrective Action: If the CCV fails acceptance criteria, then the analysis should be terminated. Recalibrate and reanalyze all samples tested since the last acceptable CCB.

Calibration Controls	Sequence	Control Limit
Calibration Standards	5-point (minimum) linearity	r≥ 0.995
Cont. Cal. Verif. (CCV)	Prior to / after every 10 injections	Recovery 90-110 %
Cont. Cal. Blank (CCB)	Following ICV/CCB	<2X MDL

10.9 Sample Analysis

10.9.1 Report the average of at least three integrations for all field and QC samples analyzed.

10.9.1.1 For analytical sequences that include BNSF and/or BP LAMP samples the RSD between multiple instrument integrations must be <20% if the analyte is greater than the reporting limit. If the RSD is above 20% then the laboratory must reanalyze the sample.

10.9.2 Flush the system with the rinse blank for at least 30 seconds between samples and standards during the analytical run.

10.9.3 Masses, which would affect the data quality, must be monitored during the analytical run to determine the potential effects of matrix on a given element.

10.9.4 Dilute and reanalyze samples that are more concentrated than the linear range for an analyte or specific isotope of interest. No analyte may be reported from an analysis of a diluted sample in which the analyte concentration is less than 5 times the IDL. (The sample should be diluted to the approximate midrange of the analytical curve.)For samples analyzed under method 200.8, a 4-mL portion of digestate is diluted to 10 mL prior to analysis.

NOTE: Method 200.8 describes the digestion of 100 mL of sample, a final digestion volume of 50 mL, and Section 11.2.8 of the method adds a subsequent dilution of 20 mL of digestate to 50 mL prior to analysis. The digestion in TestAmerica Seattle's SOP TA-IP-0204 uses 50 mL of sample, a final digestion volume of 25 mL, and then 4 mL of digestate is diluted to 10 mL prior to analysis. The effective dilution factor is 1.25 in both the method and this SOP.

10.9.5 The analytical run sequence should be performed as follows to meet all quality control criteria:

Instrument initialization
Warm-up
Tune instrument
Perform mass calibration
Perform resolution check

Validate tuning criteria
Calibration blank
Calibration standards
ICV
ICB
RL verification standard / *LLICV*
ICSA
ICSAB
CCV
LLCCV (6020A)
CCB
10 Samples (which can include all sample types)
CCV
LLCCV (6020A)
CCB
10 samples
CCV
LLCCV (6020A)
CCB

10.10 Data Reduction and Review

10.10.1 Upon completion of the analytical sequence, review the raw data to determine if dilutions are necessary and then perform a level 1 data review and document the review on the data review checklist.

10.10.2 Submit the data package and review checklist to the peer reviewer for the level 2 review. The data review process is explained in SOP TA-QA-0635. All data is calculated using the formulas in Section 11.

10.10.3 Update instrument sequence logbook.

10.11 Instrument Maintenance

10.11.1 All instrument maintenance must be documented in the instrument maintenance logbook.

- Routine Maintenance (which includes, but is not limited to daily, weekly, and semiannual maintenance) is completed periodically and does not necessary indicate the instrument is out of control. It is noted in the logbook with the notation “RM”. **RM maintenance might include weekly cleaning of cones and changing tubing.**
- The logbook must include the instrument name, serial number for each major component (e.g., AA, autosampler) and the date of start-up.
- When an instrument is not capable of analyzing samples, it needs to be tagged “Out of Service”.
- For non-routine maintenance or repairs, logbook entries must include a description of the problem and what actions were taken to address the problem.
- When non-routine maintenance or repairs are complete, the instruments return to control is noted in the logbook with the notation “RTC”.

10.11.2 Daily Use and Maintenance

- For both the P.E. and Agilent instruments, the daily tune and daily mass calibration will be printed, monitored for changes and included in the instrument sequence logbook.

10.11.3 Weekly Maintenance (or more frequently if needed)

- Change the pump tubing as needed.
- Check pump and pump rollers.

10.11.4 Monthly Maintenance (or more frequently if needed)

- Clean the nebulizer as needed when instrument reading are inconsistent.
- Clean the torch as needed. Sonicate the nebulizer in mild soap for 10 minutes to clean.
- Clean the spray chamber if dirty.
- Check the air filters on the power supply and spectrometer, and clean if dirty.

10.11.5 Annual and Semiannual Maintenance (or more frequently if needed)

- Clean the chiller.
- Change the oil in the rough pump
- Change the Lens as needed
- Change the cell annually or as needed

10.11.6 Spare Parts

10.11.6.1 Instrument supplies

- Purge windows.
- Injector tip.
- Lens
- Cell

10.11.6.2 Plasma Torch Assembly.

- Quartz torch.
- Spray chamber.
- Nebulizer.

10.11.6.3 Tubing

- Sample tubing.
- Drain tubing.
- Internal standard tubing
- Sample capillary tubing and sample probe

10.12 Troubleshooting

10.12.1 *Refer to Appendix A, Troubleshooting Guide.*

11.0 Calculations / Data Reduction

For details regarding calibration models and algorithms, refer to corporate SOP CA-Q-S-005.

11.1 Accuracy

ICV / CCV, LCS % Recovery = $\frac{\text{observed concentration}}{\text{known concentration}}$ x 100

MS % Recovery = $\frac{\text{(spiked sample) - (unspiked sample)}}{\text{spiked concentration}}$ x 100

11.2 Precision (RPD)

Matrix Duplicate (MD) = $\frac{|\text{orig. sample value - dup. sample value}|}{[(\text{orig. sample value + dup. sample value})/2]}$ x 100

11.3 Concentration

The final concentration for an aqueous sample is calculated as follows:

$$\text{Result } (\mu\text{g/L}) = \frac{C \times V1 \times D}{V2}$$

Where:

C	=	Concentration from instrument readout, ppb
D	=	Instrument dilution factor
V1	=	Final volume in liters after sample preparation
V2	=	Initial volume of sample digested in liters

NOTE: Samples prepared for total recoverable analytes by Method 200.8 will have a preparation dilution factor of 1.25.

The concentration determined in digested solid samples when reported on a wet weight basis is as follows:

$$\text{Result } (\mu\text{g/kg}) = \frac{C \times V \times D}{W}$$

Where:

C	=	Concentration from instrument readout, ppb
D	=	Instrument dilution factor
V	=	Final volume in liters after sample preparation
W	=	Weight, in g, of wet sample digested

NOTE: All dry weight corrections are made in LIMS at the time the final report is prepared.

12.0 Method Performance

12.1 Instrument and Method Detection Limit Studies

12.1.1 Instrument Detection Limit (IDL) - IDLs are determined by analyzing seven replicates of low concentration undigested standards on each of three non-consecutive days, calculating the standard deviation for each day's results, and calculating the average of the three standard deviations. The IDL must be performed every three months.

12.1.2 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 (see SOP TC-QSM-0602). 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 or program requirements require a greater frequency.

12.1.3 Linear Dynamic Range (LDR)

Linear range standards (see Section 7.2.7 for preparation) must be analyzed semiannually.

Acceptance Criteria: The highest standard must produce a recovery within 90-110% of the expected value. Then the highest LDR is 90% of the highest successful standard.

Correction Action: Samples producing results above the LDR must be diluted reanalyzed.

12.1.3.1 Linear Range Verification

The LDR should be verified whenever, in the judgment of the analyst, a change in the analytical performance caused by either a change in instrument hardware or operating conditions would dictate the necessity to re-establish them.

Acceptance Criteria: The LDR verification standard must produce a result within 90-110% of the expected value.

Corrective Action: If this limit is not met, then a new LDR study is required.

Some programs (e.g., USACE/Navy) require verification of linear ranges in each analytical run or the samples must be diluted at the concentration of the high standard.

Acceptance Criteria: Results must be within 90-110% of the expected value.

Corrective Action: Samples producing results greater than the concentration of the daily check standard will be diluted and reanalyzed.

12.2 Demonstration of Capabilities

Analyst initial and continuing Demonstrations of Capability (DOC) are performed before any client samples are analyzed and are updated annually. See SOP TC-QSM-0617 for details.

12.3 Training Requirements

See SOP TC-QSM-0608 for detailed training requirements.

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

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 Waste Disposal SOP TA-EHS-0036.

14.1 Waste Streams Produced by the Method

Metals acid waste – Acid waste consisting of sample and rinse solution: Waste in which the only hazardous constituent is its pH is neutralized and sewered. Standards and any other high metal waste are collected in satellite waste stations. The waste is then taken to the waste warehouse where it is bulked and sent out for chemical precipitation with or without treatment.

15.0 References / Cross-References

15.1 Test Methods For Evaluating Solid Waste, EPA SW-846, 3rd Edition, Final Update II, Method 6020: "Inductively Coupled Argon Plasma - Mass Spectrometry", Revision 0, September 1994.

15.2 Test Methods For Evaluating Solid Waste, EPA SW-846, 4th Edition, Draft Update IVA, Method 6020A: "Inductively Coupled Argon Plasma - Mass Spectrometry", Revision 1, January 1998.

15.3 Environmental Monitoring Systems Laboratory, EPA Method 200.8, "Determination of Trace Elements in Waters and Wastes by Inductively Coupled Plasma - Mass Spectrometry", Revision 5.4, EMMC Version.

16.0 Method Modifications:

Item	Method	Modification
1	6020, 6020A, 200.8	Commercially available standards are purchased and verified at the laboratory rather than being prepared from the solid material. These verification records are kept on file.
2	6020, 6020A, 200.8	The results of the calibration blank as well as all other blanks must be less than the reporting limit, not 3 times the instrument IDL.
3	6020, 6020A	The serial dilution results are evaluated when the original result is greater than 100 times the MDL rather than 100 times the concentration in the reagent blank.
4	6020, 6020A	Corrective action for a PDS failure will be limited to generating an NCM indicating the failed analyte and the recovery rather than diluting and reanalyzing the sample.
5	6020, 6020A	Internal standard recoveries are based on the intensities of the internal standards in the most recent calibration blank rather than the intensities of the internal standards in the initial calibration standard.
6	6020, 6020A	The internal standard recovery limits for the ICV/CCV and ICB/CCB results is 30 - 120% for all analyses as stated in method 6020A rather than 80-120% as stated in method 6020.
7	200.8	Resolution criteria of the mass calibration are met if the resolution criteria for method 6020 are satisfied.
8	*6020A*	*Method Blank criteria of no detections in the method blank more than 10% of the low limit calibration check solution. TestAmeria Seattle will only evaluate the method blank to 1/2 the RL or Project DQOs and when specific DQOs are not provided by the client the RL will be defined as the DQO.*

17.0 Tables

Table I: Recommended Elemental Equations

Table II: Contributions of Contaminant Elements when Resolution and Measurement Schemes Vary

Table III: Isobaric Molecular-Ion Interferences Which Could Affect the Analytes

Table IV: Changes in Isobaric Molecular-Ion Interferences with Changing Plasma Conditions

Table V: Recommended Internal Standards

Table VI: Interference Check Sample Components and Concentrations

Table VII: Suggested Mass Choices

Table VIII: Tuning Solution

Table IX: Suggested Tuning and Response Factor Criteria

Table X: Summary of Quality Control Requirements

Table XI: Calibration, Calibration Verification, and Spike Concentrations

Table XII: Reporting Limits

Appendix A: Troubleshooting guide

18.0 Revision History

- Revision 24, dated 4 January 2014
  - Updated section 4.3.3 with 6020A internal standard intensity criteria
  - Added computer hardware and software, section 6.2
  - Updated section 7.2.6 to include LLICV and LLCCV
  - Updated NCM criteria, section 9.3
  - Updated method blank criteria, section 9.4
  - Updated section 9.8 with 6020A internal standard intensity criteria
  - Updated serial dilution criteria for LaMP, section 9.9
  - Updated PDS criteria fro 6020A and added corrective action, section 9.10
  - Updated section 10.4.3 with 6020A internal standard intensity criteria
  - Updated section 10.7 to include LLICV and LLCCV
  - Updated section 10.9.5 to include LLICV and LLCCV
  - Added troubleshooting, section 10.12
  - Updated section 16.0 to add modification to 6020A
  - Added Appendix A, section 17.0

- Revision 23, dated 6 August 2012
  - Update safety intro, section 5.0
  - Added instructions for diluting the method blank by the same factor as associated samples, section 9.4
  - Updated pollution control, section 13.0
  - Updated waste streams, section 14.1

- Revision 22, dated 25 April 2011
  - Under 4.3.2 the IS should be added at a level greater than 100,000 not 1,000,000
  - Preservation temps aligned with SW-846 chapter 3 were added in section 8.4
  - Incorporated ROMD 00025 in sections 9.5 and 9.6
  - Incorporated ROMD 00020 in section 10.2
  - Incorporated ROMD 00022 in sections 10.4.1 and 11.0
  - Added section 10.4.3 about IS limits
  - The reference under 10.7 section 7.1.6 was changed to 7.2.6
  - Made corrections to the table under 10.8
  - Section 10.10 was added about data review
  - Under 10.11.1, an explanation of what RM consists of
  - Added more complete instrument maintenance information in section 10.11
  - Incorporated ROMD 00033 in section 10.11.2
  - Table V was revised
  - Table VI was changed to reflect the dilution of this standard
  - Strontium was added to table VII
  - Suggested Mass Calibration information was updated in Table IX
  - Updated soil RLs in Table X11

- Revision 21, dated 16 April 2010
  - Added documentation of standards/reagents and standard/reagent preparation Section 7.1
  - Updated concentrations for nitric/hydrochloric acid mixture Section 7
  - Added removal of expired standards Section 7.4.
  - Updated Method Blank criteria for BP LaMP, Section 9.4
  - Updated LCS criteria for BP LaMP, Section 9.5

- Updated 9.7 ICS-A/AB criteria for BP LaMP, Section 9.7
- Added criteria for additional QC, Section 9.11.
- Updated Calibration Blank control criteria 10.6
- Updated MRL Standard criteria for BP LaMP, Section 10.7
- Added maintenance documentation and return to service requirements, Section 10.10.1
- Updated TableVI with interference check sample components and concentrations

- Revision 20, dated 13 August 2009
  - Added Table XII.X. Summary of Quality Control Requirements

- Revision 19, dated 28 February 2009
  - Updated RLs in Table XII.

- Revision 18, dated 22 February 2008
  - Integration for TestAmerica and STL operations.

TABLE I: Recommended Elemental Equations

Element	Isobaric Correction	Mathematical Equation
Al	none	(1.0000)(27M)
Sb	none	(1.0000)(121M)
As	ArCl, Se	(1.0000)(75M) - (3.1278)(77M) + (1.0177)(78M)
Ba	none	(1.0000)(135M)
Be	none	(1.0000)(9M)
Cd	MoO, Sn	(1.0000)(114M) - (0.0268)(118M) - (1.0000)(135M)
Ca	none	(1.0000)(44M)
Cr	none	(1.0000)(52M)
Co	none	(1.0000)(59M)
Cu	none	(1.0000)(65M)
Fe	none	(1.0000)(57M)
Pb	none	(1.0000)(208M) + (1.0000)(207M) + (1.0000)(206M)
Mg	none	(1.0000)(25M)
Mn	none	(1.0000)(55M)
Hg	none	(1.0000)(200M)
Ni	none	(1.0000)(60M)
K	none	(1.0000)(39M)
Se	Ar2	(1.0000)(78M) - (1.1869)(76M)
Ag	none	(1.0000)(107M)
Na	none	(1.0000)(23M)
Tl	none	(1.0000)(205M)
V	ClO, Cr	(1.0000)(51M) - (3.1081)(53M) + (0.3524)(52M)
Zn	none	(1.0000)(66M)
6Li	Li (natural)	(1.0000)(6M) - (0.0813)(7M)
Sc	none	(1.0000)(45M)
Y	none	(1.0000)(89M)
Rh	none	(1.0000)(103M)
In	Sn	(1.0000)(115M) - (0.0149)(118M)
Tb	none	(1.0000)(159M)
Ho	none	(1.0000)(165M)
Bi	none	(1.0000)(209M)

Where M = Total ion count rate at the specified mass.

TABLE II: Contributions of Contaminant Elements when Resolution and Measurement Schemes Vary

Concentrations listed are the approximate level (µg/L) measured when the interferent is present at 100 mg/L.

Analyte	Interferent Element	Peak Width at 10% of the Peak Height			
		1.0 amu Integration Width		0.8 amu Integration Width	
		0.9 amu	0.3 amu	0.9 amu	0.3 amu
^{121}Sb	^{120}Sn	820	5	10	1
^{121}Sb	^{122}Te	77	None	1	none
^{75}As	^{74}Se, ^{76}Se	910	4	3	none
^{9}Be	^{10}B	1,200	12	9	1
^{112}Cd	^{113}In	1,700	8	10	none
^{114}Cd	^{115}In	5,000	150	180	18
^{116}Cd	^{115}In	30	None	5	none
^{52}Cr	^{51}V	1.4	1.5	none	none
^{53}Cr	^{54}Fe	650	7	1	none
^{59}Co	^{58}Ni, ^{60}Ni	1,500	6	2	none
^{63}Cu	^{62}Ni, ^{64}Ni	190	1	none	none
^{63}Cu	^{64}Zn	4,000	14	9	none
^{65}Cu	^{64}Ni	1	1	none	none
^{65}Cu	^{64}Zn, ^{66}Zn	4,400	22	15	none
^{208}Pb	^{209}Bi	140	14	57	none
^{55}Mn	^{54}Fe, ^{56}Fe	900	8	4	none
^{58}Ni	^{59}Co	3,000	96	75	7
^{60}Ni	^{59}Co	9	4	10	5
^{62}Ni	^{63}Cu	8,500	690	4,500	16
^{107}Ag	^{106}Pd, ^{108}Pd	2,400	22	80	4
^{107}Ag	^{106}Cd, ^{108}Cd	130	3	5	2
^{109}Ag	^{108}Pd, ^{110}Pd	1,800	12	36	3
^{109}Ag	^{108}Cd, ^{110}Cd	1,600	10	37	3
^{51}V	^{52}Cr	2,100	45	410	1
^{64}Zn	^{65}Cu, ^{63}Cu	7,800	57	410	2
^{66}Zn	^{65}Cu	2	none	3	2

TABLE III: Isobaric Molecular-Ion Interferences Which Could Affect the Analytes

Analyte	Interferences						
	Oxygen	Hydroxyl	Nitrogen	Chlorine	Sulfur	Carbon	Other
^{121}Sb	PdO		AgN			AgC	
^{123}Sb	AgO		AgN	SrCl	ZrS	CdC	
^{75}As	CoO	NiOH	NiN	ArCl	CaS	CuC	
^{138}Ba	SnO	SbOH					
^{137}Ba	SbO	SnOH		MoCl			
^{136}Ba	SnO	SnOH				SnC	
^{135}Ba	SnO	SnOH		MoCl			
^{134}Ba	SnO	SnOH	SnN	MoCl		SnC	
^{132}Ba	SnO, CdO	InOH	SnN	MoCl	MoS	SnC	
^{130}Ba	CdO	CdOH	SnN, CdN	MoCl	MoS	SnC	
^{9}Be							
^{114}Cd	MoO	MoOH	MoN	SeCl	SeS		
^{112}Cd	MoO, ZrO	MoOH	MoN	AsCl, SeCl	SeS	MoC	
^{111}Cd	MoO	MoOH	MoN	GeCl			
^{110}Cd	MoO, ZrO		MoN, ZrN	GeCl, AsCl	SeS	MoC	
^{113}Cd	MoO	MoOH		SeCl, AsCl			
^{116}Cd	MoO						
^{106}Cd	ZrO		MoN, ZrN		GeS	MoC, ZrC	
^{108}Cd	MoO, ZrO	ZrOH	MoN, ZrN	GeCl	SeS, GeS	MoC, ZrC	
^{52}Cr	ArO	ClOH				ArC	
^{53}Cr	ClO	ArOH	KN	NCl, OCl		KC	
^{50}Cr	SO		ArN		SO	ArC	Mo^{++}
^{54}Cr		ClOH	ArN, CaN			CaC	
^{59}Cr	CaO	CaOH	ScN	MgCl	AlS	TiC	Sn^{++}
^{63}Cu	TiO, PO_2	TiOH	TiN	SiCl, MgCl	PS	VC	ArNa
^{65}Cu	TiO	TiOH	VN	SiCl	SS, SO_2H	CrC	
^{208}Pb							
^{206}Pb							
^{207}Pb							
^{204}Pb							
^{55}Mn	KO	ArOH	KN		NaS	CaC	Cd^{++}

TABLE III: (cont.) Isobaric Molecular-Ion Interferences Which Could Affect the Analytes

Analyte	Interferences						
	Oxygen	Hydroxyl	Nitrogen	Chlorine	Sulfur	Carbon	Other
^{202}Hg	WO						
^{200}Hg	WO	WOH	WN				
^{199}Hg	WO	WOH					
^{201}Hg		WOH					
^{198}Hg	WO	TaOH	WN			WC	
^{204}Hg							
^{196}Hg			WN				
^{58}Ni	CaO	KOH	CaN	NaCl	MgS	TiC	Cd^{++}, Sn^{++}
^{60}Ni	CaO	CaOH	TiN	MgCl, NaCl	SiS	TiC	Sn^{++}
^{62}Ni	TiO	ScOH	TiN	AlCl, MgCl	SiS	TiC, CrC	Sn^{++}
^{61}Ni	SeO	CaOH	TiN	MgCl	SiS	TiC	
^{64}Ni	TiO	TiOH	TiN, CrN	SiCl, AlCl	SS	CrC	
^{80}Se	ZnO	CuOH	ZnN	ScCl, CaCl	TiS	ZnC	
^{78}Se	NiO	NiOH	ZnN	CaCl, KCl	TiS	ZnC	
^{82}Se	ZnO	CuOH	ZnN	TiCl, ScCl	TiS, CrS		
^{76}Se	NiO	CoOH	NiN	KCl	CaS	ZnC	
^{77}Se	NiO	CuN	CuN	CaCl, ArCl	ScS	CuC	
^{74}Se	NiO	NiN	NiN	ClCl, KCl	CaS	NiC	
^{107}Ag	ZrO	ZrOH		GeCl	AsS	MoC	
^{109}Ag		MoOH	MoN	GeCl	SeS	MoC	
^{205}Tl							
^{203}Tl		WOH					
^{51}V	ClO	SOH	ClN	ClO, ClN	FS	KC	
^{50}V	SO		ArN			ArC	Mo
^{64}Zn	TiO	TiOH	TiN, CrN	SiCl, AlCl	SS	CrC	
^{66}Zn	TiO	TiOH	CrN	PCl, SiCl	SS	FeC	
^{68}Zn	CrO	VOH	FeN	PCl	ArS	FeC	Ba^{++}
^{67}Zn	VO	TiOH, Cr	CrN	SCl	ClS	MnC	Ba^{++}
^{70}Zn	FeO	CrOH	GeN	ClCl	ArS	NiC	

NOTE: The information provided in this table does not indicate that all of the described interferences need to be tested. However, the table can be consulted for informational purposes if unusual samples are encountered.

Table IV: Changes in Isobaric Molecular-Ion Interferences with Changing Plasma Conditions**

	Molecular Interference	Nebulizer Flow Rate		
		High	Average	Low
Oxides	ScO/Sc	0.00326	0.00055	0.00116
	YO/Y	0.00568	0.00395	0.00353
	TbO/Tb	0.0156	0.00648	0.00614
	ClO, Cl	0.00725	0.00227	0.00233
Hydroxides	ScOH/Sc	0.00040	0.00011	0.00000
	YOH/Y	0.00078	0.00044	0.00048
	TbOH/Tb	0.00034	0.00008	0.00011
	ClOH/Cl	0.00048	0.00031	0.00029
Chlorine	ClO/Cl	0.00725	0.00227	0.00233
	ClOH/Cl	0.00048	0.00031	0.00029
	ArCl/Cl	0.00605	0.00091	0.00477

** Information for this table is being determined by the EPA.

Table V: Recommended Internal Standards

Method 6020	Method 200.8
Li 6	Li 6
Rh	Rh
Sc	Sc
Ge	Ge
Ho	Ho
Lu	Lu
Bi	Bi

Table VI: Interference Check Sample Components and Concentrations
(ICSAB minors are suggested spike levels)

Interference Component	Solution A Concentration (mg/L)	Solution AB Concentration (mg/L)
Al	50	50
Ca	60.0	60.0
Fe	50.0	50.0
Mg	20.0	20.0
Na	50.0	50.0
P	20.0	20.0
K	20.0	20.0
S	20.0	20.0
C	40.0	40.0
Cl	200.0	200.0
Mo	0.4	0.4
Ti	0.4	0.4
As	0.0	0.02
Cd	0.0	0.02
Cr	0.0	0.04
Co	0.0	0.04
Cu	0.0	0.04
Mn	0.0	0.04
Ni	0.0	0.04
Se	0.0	0.02
Ag	0.0	0.01
V	0.0	0.04
Zn	0.0	0.02

Table VII: Suggested Mass Choices

Boldface masses indicate the masses which must have the most impact on data quality and the elemental equations used to collect the data. Suggested masses for method 200.8 are in "quotes."

Mass	Element of Interest
"**27**"	Aluminum
121, "**123**"	Antimony
"75"	Arsenic
138, "137", 136, **135**, 134, 132, 130	Barium
"9"	Beryllium
114, 112, "111", 110, 113, 116, 106	Cadmium
42, 43, **44**, 46, 48	Calcium
"**52**", **53**, **50**, 54	Chromium
"**59**"	Cobalt
"**63**", **65**	Copper
56, **54**, **57**, 58	Iron
"**208**", "**207**", "**206**", 204	Lead
24, **25**, **26**	Magnesium
"**55**"	Manganese
196, 198, 199, **200**, **201**, "**202**", 204	Mercury
58, "**60**", 62, **61**, 64	Nickel
39	Potassium
80, **78**, "**82**", **76**, **77**, 74	Selenium
"**107**", **109**	Silver
23	Sodium
88	Strontium
"**205**", 203	Thallium
"**51**", **50**	Vanadium
64, "**66**", **68**, **67**, 70	Zinc
72	Germanium
139	Lanthanum
118	Tin
35, 37	Chlorine
"98", 96, 92, **97**, 94	Molybdenum

NOTE: It is strongly recommended that elements other than those of interest be monitored to indicate other potential molecular interferences that could affect the data quality.

Table VIII: Tuning Solution

A tuning solution containing elements representing all of the mass regions of interest must be analyzed. Below are two groups of suggested solutions that cover a typical mass calibration range.

Method 6020

Element	Concentration (µg/L)
Solution A	
Mg	10
Rh	10
Pb	10
Solution B	
Li	10
Co	10
In	10
Tl	10

Method 200.8

Element	Concentration (µg/L)
Be	10
Mg	10
Co	10
In	10
Pb	10

Table IX: Suggested Tuning and Response Factor Criteria

Minimum Response from Tuning Solution:

Be	>1,000
Mg	>2,000
Rh	>20,000
Pb	>10,000
Li	>2,000
Co	>20,000
In	>1,000
Tl	>1,000

Suggested Mass Calibration:

Be	9
Mg	24
Rh	103
Pb	208
Li	7
Co	59
In	115
Tl	203

Table X: Summary of Quality Control Requirements

QC Parameter	Frequency*	Acceptance Criteria	Corrective Action
IS	*Every reading.*	*6020 IS, 30-120% rec.* *200.8 IS, 60-125% rec.* *6020A IS ≥ 70% rec.*	*Terminate analysis; correct the problem; recalibrate.*
ICV/QCS	Beginning of every analytical run.	90 - 110% recovery.	Terminate analysis; correct the problem; recalibrate.
ICB/CB	Immediately after each ICV	The result is < RL. **For DoD QSM, < LOD.**	Terminate analysis; correct the problem; recalibrate.
RL/LLICV	Beginning of every analytical run.	6020 & 200.8: 50 - 150% rec. *6020A: 80-120% rec.* *DoD: 80-120% rec.*	Terminate analysis; correct the problem; recalibrate.
CCV	Beginning and end of run and every 10 samples OR every 2 hours, whichever is more frequent. Beginning and end of each lot.	90 - 110% recovery.	Reanalyze once. If acceptable, continue. If unacceptable, terminate analysis; correct the problem recalibrate the instrument, reverify calibration and rerun all samples since the last acceptable CCV.
CCB	Immediately following each CCV.	The result must be < RL. *For DoD QSM, < LOD.*	Reanalyze once. If acceptable, continue. If unacceptable, terminate analysis; correct the problem, recalibrate the instrument, verify calibration and rerun all samples since the last acceptable CCB.
ICSA	Beginning and every 12 hours.	Monitor for possible interferences. **For DoD QSM, < LOD (unless they are a verified trace impurity form one of the spike analytes).**	See Section 9.7

QC Parameter	Frequency*	Acceptance Criteria	Corrective Action
ICSAB	Immediately following each ICSA.	Monitor for possible interferences. **For DoD QSM, ± 20%.**	See Section 9.7
Method Blank/Laboratory Reagent Blank	One per lot of 20 field samples or fewer.	The result must be < RL. Sample results greater than 10x the blank concentration or samples for which the contaminant is < RL, do not require redigestion or reanalysis. **For DoD QSM, < ½ RL.**	Redigest and reanalyze samples. Note exceptions under criteria section. See Section 9.4 for additional requirements.
Laboratory Control Sample (LCS)	One per sample preparation batch of up to 20 samples.	LCS must be within 80 - 120% recovery *or QSM 5.0 control limits.* (85-115% for 200.8) Samples for which the contaminant is < RL and the LCS results are > 120% (115% for 200.8) may not require redigestion or reanalysis (see Section 9.5).	Terminate analysis; Correct the problem; Redigest and reanalyze all samples associated with the LCS.
Matrix Spike (MS)	One per sample preparation batch of up to 20 samples.	75 – 125% recovery or tighter in-house control limits. **For DoD:** Use LCS control limits.	In the absence of client specific requirements, flag the data; no flag required if the sample level is > 4x the spike added.
Matrix Spike Duplicate (MSD)	One per sample preparation batch of up to 20 samples. 10% frequency for some programs (see **Error! Reference source not found.**)	75 – 125 % recovery; RPD ≤ 20% or tighter in-house control limits. **For DoD:** Use LCS control limits.	See Corrective Action for Matrix Spike.
Serial Dilution (6020 Only)	One per batch of 20 field samples or fewer.	90 - 110% recovery	See Section 9.9 for additional requirements.

QC Parameter	Frequency*	Acceptance Criteria	Corrective Action
Post-Digestion Spike (6020 Only)	One per batch of 20 field samples or fewer.	75-125% recovery *80-120% for 6020A*	See Section 9.10.
Matrix Spike/Laboratory Fortified Matrix	One per lot of 20 field samples or fewer.	Must be within laboratory control limits	See Section 9.6 for additional requirements.

Table XI: Calibration, Calibration Verification, and Spike Concentrations

Element	Initial Calibration Levels (µg/L)					ICV (µg/L)	CCV (µg/L)	LCS (µg/L)	MS/MSD (µg/L)
	1	2	3	4	5				
Aluminum	1	10	100	500	1000	400	500	80	80
Antimony	0.1	1	10	50	100	40	50	60	60
Arsenic	0.1	1	10	50	100	40	50	80	80
Barium	0.1	1	10	50	100	40	50	80	80
Beryllium	0.1	1	10	50	100	40	50	2	2
Cadmium	0.1	1	10	50	100	40	50	2	2
Calcium	10	100	1000	5000	10,000	4000	5000	400	400
Chromium	0.1	1	10	50	100	40	50	8	8
Cobalt	0.1	1	10	50	100	40	50	20	20
Copper	0.1	1	10	50	100	40	50	10	10
Iron	10	100	1000	5000	10,000	4000	5000	440	440
Lead	0.1	1	10	50	100	40	50	20	20
Magnesium	10	100	1000	5000	10,000	4000	5000	400	400
Manganese	0.1	1	10	50	100	40	50	20	20
Mercury	0.005	0.05	0.5	2.5	5	2	2.5	1.0	1.0
Molybdenum	0.1	1	10	50	100	40	50	*100*	*100*
Nickel	0.1	1	10	50	100	40	50	20	20
Potassium	1	10	100	500	1000	4000	5000	400	400
Selenium	0.1	1	10	50	100	40	50	*80*	*80*
Silver	0.1	1	10	50	100	40	50	12	12
Strontium	0.1	1	10	50	100	40	50	*40*	*40*
Thallium	0.1	1	10	50	100	40	50	80	80
Tin	0.1	1	10	50	100	40	50	*100*	*100*
Titanium	0.1	1	10	50	100	40	50	*100*	*100*
Uranium	0.1	1	10	50	100	40	50	*40*	*40*
Zinc	0.1	1	10	50	100	40	50	20	20

This procedure has been developed for thirty-five elements (See Table VIII). Additional elements may be included in the calibration solution at the above levels. Levels may be adjusted to meet specific regulatory or client programs.

Table XII: Reporting Limits

Element	Water 6020, 200.8 (µg/L)	Soil 6020 (µg/kg)
Aluminum	*500*	30,000
Antimony	2	200
Arsenic	*5*	500
Barium	6	200
Beryllium	2	200
Cadmium	2	200
Calcium	50,000	100,000
Chromium	2	200
Cobalt	2	200
Copper	5	*400*
Iron	200	20,000
Lead	2	200
Magnesium	*50000*	100,000
Manganese	2	500
Mercury	*0.625*	200
Molybdenum	4	400
Nickel	*15*	*500*
Potassium	50,000	100,000
Selenium	*5*	700
Silver	2	200
Sodium	*50000*	*150000*
Strontium	2	200
Thallium	*5*	*500*
Tin	7	500
Titanium	5	500
Uranium	2	200
Vanadium	*10*	*700*
Zinc	*20*	*2000*

Appendix A – Troubleshooting Guide

Error Messages

The following messages can appear in the ChemStation green message bar when you operate the Agilent 7500. When an error message is displayed, the instrument runs a safety sequence.

Click on an error No., and you will jump to the error message corresponding to the error No.

NOTE:
An * (asterisk) indicates that an error number has multiple messages and contents. Please check the error message and error number to properly identify the problem.

1000	1001	1002	1003	1004	1005	1006 *
1007	1008	1009 *	1010 *	1011	1012 *	1013
1014	1015	1016 *	1017	1018	1019	1020
1021	1022	1023	1024	1025	1026	1027
1090	1099	1100	1101 *	1102 *	1103 *	1104 *
1109 *	1110 *	1111 *	1112 *	1113 *	1114	1115 *
1116 *	1117	1118 *	1119 *	1120 *	1121 *	1122
1123	1124	1125	1126	1127	1128	1129
1130	1131	1132	1133 *	1134	1135	1136
1137	1138	1139 *	1140	1141	1142	1143 *
1144	1145	1146	1147	1148	1149 *	1150
1151 *	1152	1153	1200	1201	1202	1203
1204	1205	1206	1208 *	1209	1210	1211 *
1212	1213	1214	1215 *	1216	1217	1218
1219 *	1220	1221 *	1222	1223	1226	1300
1301	1302	1303	1304	1305	1306	1307
1308	1309	1400 *	1402	1410	1411	1413
1414	1415	1416	1417	1418	1422	1430
1432	1433	1434	1435	1436	1440 *	1441
1442	1443	1445	1446	1449	1450	1451
1452	1453	1454	1455	1518	1519	1520
1521	1522	1523	1524	1525	1526	

Alarm Messages Number List

NOTE:
An * (asterisk) indicates that an error number has multiple messages and contents. Please check the error message and error number to properly identify the problem.

2150 *	2151 *	2152	2153	2400 *	2402
2403	2404	2405	2412	2413	2414
2415	2416	2433	2435	2605	2606
2607	2608	2609	2610	3000	3001
3002	3003	3004			

1000 **Execution Error: Instrument is busy- Between Transition Modes.**
Attempted to execute a command that cannot be executed during mode transition. Transitioning modes.

Problem	***Possible Cause/ Solution***
High Blanks	*Increase rinse time* *Clean or replace tip* *Clean or replace torch* *Clean or replace sample tubing* *Clean or replace nebulizer* *Clean or replace mixing chamber*
Instrument Drift	*RF not cooling properly* *Replace torch (Crack)* *Clean or replace nebulizer (blockage)* *Check room temperature (changing)* *Replace pump tubing* *Room humidity too high* *Clean torch tip (salt buildup)* *Check for argon leaks* *Adjust sample carrier gas* *Replace PA tube*
Erratic Readings, *Flickering Torch or* *High RSD*	*Check for argon leaks* *Adjust sample carrier gas* *Replace tubing (clogged)* *Check drainage(back pressure changing)* *Increase uptake time (too short)* *Increase flush time (too short)* *Clean nebulizer, torch or spray chamber* *Increase sample volume introduced* *Check that autosampler tubes are full* *Sample or dilution of sample not mixed* *Increase integration time (too short)* *Realign torch* *Reduce amount of tubing connectors*

Title: Determination of Volatile Organic Compounds by GC/MS [Methods 8260B, 8260C and 624]

Approvals

Signatures on File			
Bisrat Tadesse	Date	Sandra Langway	Date
Volatiles Department Manager		Health & Safety Manager / Coordinator	
Terri Torres	Date	Heather Prater	Date
Quality Assurance Manager		Laboratory Director	

Copyright Information:
This documentation has been prepared by TestAmerica Laboratories, Inc. and its affiliates ("TestAmerica"), solely for their own use and the use of their customers in evaluating their qualifications and capabilities in connection with a particular project. The user of this document agrees by its acceptance to return it to TestAmerica upon request and not to reproduce, copy, lend, or otherwise disclose its contents, directly or indirectly, and not to use it for any other purpose other than that for which it was specifically provided. The user also agrees that where consultants or other outside parties are involved in the evaluation process, access to these documents shall not be given to said parties unless those parties also specifically agree to these conditions.

THIS DOCUMENT CONTAINS VALUABLE CONFIDENTIAL AND PROPRIETARY INFORMATION. DISCLOSURE, USE OR REPRODUCTION OF THESE MATERIALS WITHOUT THE WRITTEN AUTHORIZATION OF TESTAMERICA IS STRICTLY PROHIBITED. THIS UNPUBLISHED WORK BY TESTAMERICA IS PROTECTED BY STATE AND FEDERAL LAW OF THE UNITED STATES. IF PUBLICATION OF THIS WORK SHOULD OCCUR THE FOLLOWING NOTICE SHALL APPLY:

©COPYRIGHT 2012 TESTAMERICA LABORATORIES, INC. ALL RIGHTS RESERVED.

Facility Distribution No. 001 **Distributed To:** Lab Intranet

1.0 Scope and Application

1.1 This method is applicable to the determination of volatile organic compounds (VOCs) in water, wastewater, soils, sludges, and other solid matrices. Standard analytes are listed in Tables 1, 2, and 3.

1.2 This SOP is applicable to Method 8260B and 8260C, which is appropriate for compliance testing under RCRA regulations. Appendix A presents modifications to the procedures in the main SOP that are necessary for analysis of wastewater by Method 624 (CWA compliance testing). It is important that the differences among these methods are carefully observed.

1.3 This method can be used to quantify most volatile organic compounds that have boiling points below 200 °C and are insoluble or slightly soluble in water. Volatile water-soluble compounds can be included in this analytical technique; however, for more soluble compounds, quantitation limits are approximately ten times higher because of poor purging efficiency.

1.4 The method is based upon a purge-and-trap, gas chromatograph/mass spectrometric (GC/MS) procedure. The approximate working range is 1 to 200 µg/L for 8260B and 8260C waters, 1 to 200 µg/kg for low-level soils, and 8 to 8,000 µg/kg for high-level soils. The working range for Method 624 (5 mL purge) is 1-200 µg/L.

1.5 Reporting limits are listed in Tables 1, 2 and 3.

1.6 Method performance is monitored through the use of surrogate compounds, matrix spike/matrix spike duplicates (MS/MSD), and laboratory control spike samples (LCS).

1.7 On occasion clients may request modifications to this SOP. These modifications are handled following the procedures outlined in Section 12.2.1 in the Quality Assurance Manual.

2.0 Summary of Method

2.1 Volatile compounds are introduced into the gas chromatograph by the purge and trap method. The components are separated via the gas chromatograph and detected using a mass spectrometer, which is used to provide both qualitative and quantitative information.

2.2 Aqueous samples are purged directly. Generally, soils are preserved by extracting the volatile analytes into methanol. If especially low detection limits are required, soil samples may be preserved with sodium bisulfate; sampled directly into pre-tarred VOA vials which contain 5mL reagent free water, a magnetic stirrer bar, and are immediately frozen; or collected in a suitable container to be transferred in total or by aliquot to a VOA vial and purged directly.

2.3 In the purge-and-trap process, an inert gas (generally *Nitrogen*) is bubbled through the solution at ambient temperature and the volatile components are efficiently transferred from the aqueous phase to the vapor phase. The vapor is swept through a sorbent column where the volatile components are trapped. After purging is completed, the sorbent column (trap) is heated and back flushed with inert gas to desorb the components onto a gas chromatographic column. The gas chromatographic column is then heated to elute the components, which are detected with a mass spectrometer.

2.4 Qualitative identifications are confirmed by analyzing standards under the same conditions used for samples and comparing the resultant mass spectra and GC retention times. Each identified component is quantified by relating the MS response for an appropriate selected ion produced by that compound to the MS response for another ion produced by an internal standard.

3.0 Definitions

3.1 Both SW-846 (RCRA) and drinking water (SDWA) terminology are used in this section for cross-reference purposes. Elsewhere in the SOP, the SW-846 terminology is used exclusively.

3.2 Batch

The batch is a set of up to 20 samples of the same matrix processed using the same procedures and reagents within the same time period. Using this method, each 4-bromofluorobenzene (BFB) analysis will normally start a new batch. Batches for high-level soils are defined at the sample preparation stage and may be analyzed on multiple instruments over multiple days, although reasonable effort should be made to keep the samples together.

The Quality Control batch must contain a matrix spike/spike duplicate pair (MS/MSD), a Laboratory Control Sample (LCS), and a method blank. If there is insufficient sample to perform the MS/MSD, a duplicate LCS is used to establish batch precision when requested by the client. Refer to SOP TA-QA-0620 for further details of the batch definition.

3.3 Method Blank (MB) or Laboratory Reagent Blank (LRB)

A method blank consisting of all reagents added to the samples must be analyzed with each batch of samples. The method blank is used to identify any background interference or contamination of the analytical system, which may lead to the reporting of elevated concentration levels or false positive data. Sparged water or water that has been boiled then cooled to ambient temperature is used as the blank medium for water batches and muffled Ottawa Sand for soil batches. Prepared (muffled at 800C for at least 2 hours) batches of Ottawa sand are tracked using the reagent data base in the Laboratory Information Management System (known as TALS) and are at the time of the writing of this SOP named with the following convention: voasand_XXXXX.

3.4 Laboratory Control Sample (LCS) or Laboratory Fortified Blank (LFB)

A blank matrix (reagent water or muffled Ottawa Sand) is spiked with the analytes of interest and is carried through the entire analytical procedure. Analysis of this sample with acceptable recoveries of the spiked materials demonstrates that the laboratory techniques for this method are acceptable.

3.5 Surrogates

Surrogates are organic compounds that are similar to the target analyte(s) in chemical composition and behavior in the analytical process, but that are not normally found in environmental samples. Each sample, blank, LCS, and MS/MSD is spiked with surrogate standards. Surrogate spike recoveries must be evaluated by determining whether the concentration (measured as percent recovery) falls within the required recovery limits.

3.6 Matrix Spike/Matrix Spike Duplicate (MS/MSD) or Laboratory Fortified Sample Matrix (LFM)

A matrix spike is an environmental sample to which known concentrations of target analytes have been added. A matrix spike duplicate is a second aliquot of the same sample, which is prepared and analyzed along with the sample and matrix spike. Matrix spikes and duplicates are used to evaluate accuracy and precision in the actual sample matrix.

3.7 Calibration Check Compound (CCC)

CCCs are a representative group of compounds that are used to evaluate initial calibrations and continuing calibrations. Relative percent difference for the initial calibration and % drift (%D) for the continuing calibration response factors are calculated and compared to the specified method criteria.

3.8 System Performance Check Compounds (SPCC)

SPCCs are compounds that are sensitive to system performance problems and are used to evaluate system performance and sensitivity. A response factor from the continuing calibration is calculated for the SPCC compounds and compared to the specified method criteria.

3.9 Initial Calibration Verification (ICV) or Quality Control Sample (QCS)

The ICV is a second-source calibration verification standard. The QCS is reagent water or an environmental sample that is fortified with target analytes at known concentrations. This too is a second-source standard, i.e., different than the source of calibration standards. In this SOP, the LCS and the MS/MSD spikes are second-source standards, and serve to meet method requirements for the QCS.

3.10 Continuing Calibration Verification (CCV) or Laboratory Performance Check Solution (LPC)

A solution of method analytes, surrogate compounds, and internal standards used to evaluate the performance of the instrument system with respect to a defined set of method criteria.

4.0 Interferences

4.1 Method interferences may be caused by contaminants in solvents, reagents, glassware, and other processing apparatus that lead to discrete artifacts. All of these materials must be routinely demonstrated to be free from interferences under conditions of the analysis by running laboratory method blanks as described in the Quality Control section. The use of ultra high purity gases, boiled and cooled to ambient or sparged purified reagent water, and approved lots of purge-and-trap-grade methanol will greatly reduce introduction of contaminants. In extreme cases, the purging vessels may be pre-purged to isolate the instrument from laboratory air contaminated by solvents used in other parts of the laboratory.

4.2 Samples can be contaminated by diffusion of volatile organics (particularly Methylene chloride and fluorocarbons) into the sample through the septum seal during shipment and storage. A field blank prepared from reagent water and carried through the sampling and handling protocol can serve as a check on such contamination.

4.3 Matrix interferences may be caused by non-target contaminants that are co-extracted from the sample. The extent of matrix interferences will vary considerably from source to source depending upon the nature and diversity of the site being sampled.

4.4 Cross-contamination can occur whenever high-level and low-level samples are analyzed sequentially or in the same purge position on an autosampler. Whenever an unusually concentrated sample is analyzed, it should be followed by one or more blanks to check for cross-contamination. The purge and trap system may require extensive bake-out and cleaning after a high-level sample.

Note: Due to the large number of analytes analyzed for in this method, some with higher boiling points are considered Semi-Volatile analytes. It may be necessary to evaluate for cross-contamination at levels above 10 ug/L for Naphthalene and 1,2,3-Trichlorobenzene; above 20 ug/L for 2-Ethyl-1-hexanol, n-Butylbenzene and Hexachlorobutadiene; above 50 ug/L for tert-Butylbenzene, sec-Butylbenzene, 4-Isopropyltoluene, 1,2,4-Trichlorobenzene and Ethylbenzene; above 100 ug/L for Methacrylonitrile, 1,3,5-Trichlorobenzene, 1,2-Dibromo-3-Chloropropane and Toluene, and all other analytes should be evaluated for potential cross-contamination above a detected concentration of 150 ug/L or more. (These concentrations are based on a carry-over study conducted by the laboratory detecting cross-contamination above ½ the RL). It may, therefore, be necessary to run an instrument rinse after laboratory spiked samples, such as high calibration levels, CCVs, LCS and MS to ensure no cross contamination occurs. **All client samples are also evaluated using the same criteria.**

4.5 Some samples may foam when purged due to surfactants present in the sample. When this kind of sample is encountered, an antifoaming agent (e.g., J.T. Baker's Antifoam B silicone emulsion) can be used.

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

5.1.1 The gas chromatograph and mass spectrometer contain zones that have elevated temperatures. The analyst needs to be aware of the locations of those zones, and must cool them to room temperature prior to working on them.

5.1.2 The mass spectrometer is under deep vacuum. The mass spectrometer must be brought to atmospheric pressure prior to working on the source.

5.1.3 There are areas of high voltage in both the gas chromatograph and the mass spectrometer. Depending on the type of work involved, either turn the power to the instrument off, or disconnect it from its source of power.

5.1.4 Cut resistant gloves or a protective cloth must be used when opening voa vials.

5.1.5 The toxicity or carcinogenicity of each reagent used in this method has not been fully established. Each chemical should be regarded as a potential health hazard and exposure should be as low as reasonably achievable. Cautions are included for known extremely hazardous materials.

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	Hazards	Exposure Limit (1)	Signs and symptoms of exposure

Methanol (MeOH)	Flammable Poison Irritant	200 ppm-TWA	A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness and dizziness. Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes.
1 – Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

6.1 Instrumentation

- Gas Chromatograph: The gas chromatograph (GC) system must be capable of temperature programming.
- Gas Chromatographic Columns: Capillary columns are used. Some typical columns are listed below:

  Column 1: 60 m X 0.32 ID DB-624 with 1.8 µm film thickness.

  Column 2: 75 m X 0.53 ID DB-624 wide bore with 3 µm film thickness.

  Column 3 20 M X 0.18 ID DB-624 micro 1.0um film thickness

  Note: Other columns may be used. *Column 3 was* in place at the time the SOP was prepared. The serial number of the column used is documented in the instrument maintenance logbook.
- Mass Spectrometer: The mass spectrometer must be capable of scanning 35-300 amu every two seconds or less, using 70 volts electron energy in the electron impact mode and capable of producing a mass spectrum that meets the required criteria.
- GC/MS interface: In general glass jet separators are used but any interface (including direct introduction to the mass spectrometer) that achieves all acceptance criteria may be used.
- Purge and Trap Device: The purge and trap device consists of the sample purger, the trap, and the desorber.
- Sample Purger: The recommended purging chamber is designed to accept between 5 mL and 25 mL samples with a water column at least 3 cm deep. The purge gas must pass through the water column as finely divided bubbles, each with a diameter of less than 3 mm at the origin. The purge gas must be introduced no more than 5 mm from the base of the water column. Alternative sample purge devices may be used provided equivalent performance is demonstrated. Low level soils are purged directly from a VOA vial.
- Trap: A variety of traps may be used, depending on the target analytes required. The O.I. #10 (Tenax/Silica gel/Carbon Molecular Sieve) is recommended. Other traps such as the Vocarb 3000 or Vocarb 4000 may be used if the Quality Control criteria are met.
- Desorber: The desorber should be capable of rapidly heating the trap up to 270 °C depending on the trap packing material. Many such devices are commercially available.
- Purge-and-trap Autosampler: An autosampler capable of sampling from a sealed vial, Varian Archon, or equivalent.

6.2 Computer hardware and software

- *Computer with a minimum 1GB memory, Pentium 4 processor, 80 G hard drive or equivalent or as recommended by instrument manufacturer.*
- LIMS system: TALS version 1.0 or higher.
- Data acquisition system: Agilent (Hewlett Packard) ChemStation for Windows 95 (version G1701AA) or equivalent. Agilent's ChemStation, is used for data acquisition and storage on machine-readable media. Since no processing is done by ChemStation and since there are no audit trail functions associated with data acquisition, the audit trail feature for ChemStation may be either enabled or disabled. The other component, Chrom, is used for data processing such as the measurement of peak area or peak height. By design, the audit trail feature for Chrom is always enabled.
- Data processing: Chrom version 1.2 or higher. The computer must have software that allows searching any GC/MS data file for ions of a specified mass and plotting such ion abundances versus time or scan number. This type of plot is defined as an Extracted Ion Current Profile (EICP). Software must also be available that allows integrating the abundances in any EICP between the specified time or scan-number limits. In addition, for the non-target compounds, software must be available that allows for the comparison of sample spectra against reference library spectra. The most recent release of the NIST/EPA mass spectral library should be used as the reference library. The computer system must also be capable of backing up data for long-term off-line storage.

6.3 Supplies

- Microsyringes: 0.5 µL gas tight and larger, 0.006-inch ID needle
- Syringe: 5, 10 or 25 mL glass
- Balance: Top-loading balance capable of weighing 0.1 g. The balance used for sample preparation is calibrated daily by a designated primary analyst (a back-up analyst is also assigned should the primary be unavailable). The analyst must perform this check according to SOP TA-QA-0014. It is also the responsibility of any analyst performing work on the balance to check the Balance logbook to determine if the daily calibration check has been completed, before beginning work.
- Scintillation Vials: 20 mL with screw caps.
- Volumetric flasks: 10 mL to 100 mL, class A with ground-glass or Teflon ® stoppers.
- Spatula: Stainless steel.
- Disposable pipettes: Pasteur.
- pH paper: Wide range (0-14) and narrow range (0-2.5).
- Helium: Ultra high purity, gr. 5, 99.999%.
- Nitrogen: Ultra high purity, from cylinders or gas generators, may be used as an alternative to helium for purge gas.

  Note: The use of Nitrogen as a purge gas is not allowed for analysis of VOA contaminants in drinking waters.
- Compressed air: Used for instrument pneumatics.

7.0 Reagents and Standards

7.1 Document reagent/standards and reagent/standard preparation in TALS using the reagent module as described in SOP TA-QA-0619.

7.2 **Reagents**

7.2.1 Methanol: Purge and Trap Grade, High Purity

7.2.2 Reagent Water: High purity water that meets the requirements for a method blank when analyzed. (See Section 9.3.) Reagent water may be purchased as commercial distilled water and prepared by purging with an inert gas for a minimum of 1 hour or boiling and cooling to ambient temperature prior to use. Other methods of preparing reagent water are acceptable.

7.3 **Standards**

7.3.1 If stock or secondary dilution standards are purchased in sealed ampoules they may be used up to the manufacturer's expiration date.

7.3.1.1 Purchased standards are stored at the manufacturer's specifications (i.e. ambient, freezer, refrigerator). Standards prepared from these purchased standards are stored in the freezer.

7.3.2 Calibration Stock Standard Solutions: Components of stock solutions may be purchased as certified solutions from commercial sources or prepared from pure standard materials as appropriate. These standards are prepared in methanol and stored in Teflon-sealed screw-cap bottles with minimal headspace at $\leq$-10°C. Each week, new aliquots of the permanent gas standards will be dispensed and given a one week expiration date. Note: Permanent gas standards may be prepared on a more frequent basis based on analyst observed signs of degradation. Other stock standards must be replaced at least every 6 months.

7.3.3 Calibration Working standards: A working solution containing the compounds of interest prepared from the stock solution(s) in methanol. These standards are stored in the freezer. Working standards are monitored by comparison to the initial calibration curve. If any of the calibration check compounds drift in response from the initial calibration by more than 20%, then corrective action is necessary. This may include steps such as instrument maintenance, preparing a new calibration verification standard or tuning the instrument. If the corrective actions do not correct the problem (two CCVs in a row fail), then a new initial calibration must be performed.

7.3.4 Aqueous calibration standards are prepared in reagent water using the secondary dilution standards. These aqueous standards must be prepared daily.

Likewise, high level methanolic calibration standards are prepared in reagent water with a matrix matched methanol concentration of one fortieth P & T methanol using the secondary dilution standards and must be prepared daily.

7.3.5 Internal standards (IS) are added to all samples, standards, and blank analyses. Refer to Table 5 for internal standard components.

7.3.6 Surrogate Standards: Refer to Table 6 for surrogate standard components and spiking levels.

7.3.7 Laboratory Control Sample Spiking Solutions: Refer to Tables 7 and 7a for LCS components and spiking levels.

7.3.8 Matrix Spiking Solutions: The matrix spike contains the same components as the LCS. Refer to Tables 7 and 7a.

7.3.9 Tuning Standard: A standard is made up that will deliver 50 ng of 4-Bromofluorbenzene on column upon injection.

7.4 As soon as standard preparations for the day are completed, the standards must be returned to the freezer.

7.5 Managers/supervisors or a designee are expected to check their areas on a monthly basis for expired standards/reagents and dispose of them according to SOP TA-EHS-0036.

8.0 Sample Collection, Preservation, Shipment and Storage

8.1 Water samples are normally preserved at pH < 2 with 1:1 hydrochloric acid. The holding time for acid-preserved samples is 14 days from sample collection. For compliance with Method 624, 8260B and 8260C, unpreserved samples must be tested within 7 days of collection.

8.2 There are two exceptions to the information provided in Section 8.1 above:

8.2.1 2-Chloroethyl vinyl ether is hydrolyzed in the presence of acid. For samples collected for analysis of this compound, a separate vial without acid should be recommended.

8.2.2 Aromatic compounds are susceptible to degradation when biological activity isn't suppressed. Therefore, the hold time is 0 days for aromatic compounds (BTEX), as measured from the time of collection, for an unpreserved sample (Reference SW-846, chapter 4.1.2). Due to the potential for biological activity, results obtained from unpreserved aqueous samples will need to be qualified. Also, document the issue in an NCM.

8.3 Soils

8.3.1 *Approved sampling containers for Method 5035 are EnCores, VOAs (with or without water and stir bar), and VOAs preserved with methanol or with sodium bisulfate*

8.3.2 *The holding time for sodium bisulfate and methanolic preserved samples is 14 days.*

8.3.3 *Soil collected using the EnCore™ sampler must be preserved in the laboratory within 48 hours of sampling. At specific client request, if the data is to be reported as 5030, the hold time for preservation and analysis is 14 days from collection. The holding time for EnCore™ samples varies based on client specifications and can be 48 hours, 7 days, or 14 days, see the table in Section 8.4.*

8.3.4 *The holding time for VOAs (with or without water and stir bar) varies based on client specifications and requirements and can be 48 hours, 7 days, or 14 days, see the table in Section 8.4.*

8.4 Listed below are the holding times and the references that include preservation requirements.

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time [1]	Reference
Waters	Three 40-mL VOA vials	40 mLs	HCl, pH < 2; Cool 0-6°C	14 Days	40 CFR Part 136.3 / 5030
Waters	Three 40-mL VOA vials	40 mLs	Cool 0-6℃	7 Days (0 Days for aromatics if not preserved)	40 CFR Part 136.3 / 5030
Soils	Three Encore Samplers	5 grams	Cool 0-6℃ or -10 to -20℃	48 Hrs for Preservation or 14 Days	5035A
Soils	Three VOA vials	5 grams	Sodium bisulfate Cool 0-6℃	14 Days	5035A
Soils	40 ml VOA vial or 4oz septa top jar	10 grams or 25 grams	Methanol Cool 0-6℃	14 Days	5035A
Soils	Three 40-mL VOA vials	5 grams	With or without DI Water -10 to -20℃	14 Days	5035A
Soils	Glass Jar	10 grams	Cool 0-6°C	5035A:48 Hrs for Preservation 14 Days 5030: 14 Days	5035A or 5030C
Waste	Glass Jar	10 grams	Cool 0-6°C	14 Days	5030C

[1] Inclusive of preparation and analysis.

8.5 Aqueous samples are stored *in three 40 ml glass VOA vials* with Teflon lined septa at 0-6°C, with minimum headspace.

8.6 *Soil* Sample Collection for High-Level Analysis using Field Methanol Preservation

8.6.1 A pre-tared four ounce volatile soil jar with an accompanying VOA vial containing 25ml of a methanol/surrogate solution containing the surrogate TFT is sent out for each sample required when the sampling occurs in the state of Alaska. Otherwise a pre-tarred VOA vial containing 10 mls of methanol is sent out for each sample required. In addition the appropriate amount of trip blanks are also sent out. All bottles sent to the field are labeled with the tare weight and lot number of the methanol/surrogate solution or methanol.

8.6.2 For sampling in the state of Alaska, twenty-five gram field samples are collected by placing an appropriate amount of sample in the four ounce soil jar with *septa lined lid* and then adding one VOA vial containing *the 25 mls of* methanol/surrogate solution. Otherwise, ten gram field samples are

collected by adding an appropriate amount of sample to a 40 mL VOA vial containing 10 mL of methanol.

8.7 *Soil Sample Collection for High-Level Analysis using EnCore™ or TerraCore™ samplers.*

8.7.1 If the sample is collected a sample in an EnCore™ sampler a minimum of one EnCores should be provided to the lab. If the samples are collected using the TerraCore sampler a minimum of one 40-ml VOA vial with 5 grams of soil. Samples must be received prior to 48 hours from sampling in order to be frozen or extracted in methanol. Following shipment back to the laboratory, the soil preserved in methanol.

8.8 *Soil Sample Collection for High-Level Analysis Unpreserved*

8.8.1 *When specifically requested by a client, unpreserved soils packed into glass jars or brass tubes may be accepted, subsampled and methanol preserved in the laboratory. These samples have a hold time of 48 hours from sample collection to sub-sampled and preserved if following Method 5035. Otherwise if following Method 5030 the holding time is 14 days to analysis.*

8.9 *Soil* Sample Collection for Low-Level Procedure

8.9.1 Samples may be collected in 5g EnCore™ sampling device (it is recommended that a minimum of two 5g EnCore™ samples are collected, but three are preferred). Soil samples collected in a 5g EnCore™ sampling device and returned to the laboratory are frozen upon receipt.

8.9.2 *Samples may be collected in vendor purchased pre-tared VOA vials with or without 10 mL of DI Water with a magnetic stir bar. Samples collected in pre-tared VOA vials are stored at -10 to -20℃ in the volatile laboratory soils freezer until sample analysis.*

8.9.3 Samples may be collected in vendor purchased pre-tared VOA vials containing 5 or 10 mL of 20% Sodium Bisulfate solution with a magnetic stir bar. Due to the exhibited potential of positive interferences in the purchased prepared vials (most notably ketones and BTEX compounds). Samples collected in pre-tared Sodium Bisulfate preserved sample vials are stored at 0-6℃ in the volatile laboratory soils refrigerator until sample analysis.

8.9.3.1 Soils containing carbonates may effervesce when added to the sodium bisulfate solution. If this is the case at a specific site, samples should be taken in a 5g EnCore™ sampling device, and stored at ≤–10℃ until analysis.

8.9.3.2 If client specifications require field preservation, samples may be collected in pre-tared VOA vials containing a magnetic stir bar and 5 mL to 10 mL of reagent water. Sample collected in this manner must be received and frozen by the laboratory within 48 hours of sampling. Samples stored in this manner **MUST** be frozen on their sides to minimize possible breakage of the sample container due to expansion of water as it freezes.

8.10 A refrigerator or freezer blank is stored in each refrigerator or freezer with the samples. This is analyzed at minimum every 14 days, but may be analyzed more frequently as needed (see SOP TA-QA-0616). The refrigerator or freezer blank should be run immediately after the method blank.

8.11 Percent Moisture Correction for Soils

A percent moisture correction may be performed on soil samples to adjust the extraction final volume of the sample in order to allow for the miscible solvents effect, as required by the client. Percent moisture must be determined if results will be reported as dry weight and percent moisture correction to be performed; refer to SOP TA-WC-0125 for determination of percent moisture. For all methanolic samples with a % moisture of greater than 10%, the following formula is used to determine the corrected final volume:

Corrected FV = ((g of sample * % Moisture/100) + mL of Methanol) * 40

(Also noted in section 12.9)

9.0 Quality Control

9.1 The minimum quality controls (QC), acceptance criteria, and corrective actions are described in this section. When processing samples in the laboratory, use the LIMS QC program code and special instructions to determine specific QC requirements that apply.

9.1.1 The laboratory's standard QC requirements, the process of establishing control limits, and the use of control charts are described more completely in the TestAmerica Seattle QAM.

9.1.2 Specific QC requirements for Federal programs, e.g., USACE and Navy projects, are described in DoD QSM v5 or the latest promulgated version.

9.1.3 Project-specific requirements can override the requirements presented in this section when there is a written agreement between the laboratory and the client, and the source of those requirements should be described in the project documents. Project-specific requirements are communicated to the analyst via special instructions in the LIMS and may also come in the form of email or written notifications distributed at "project kick off" meetings.

9.1.4 Any QC result that fails to meet control criteria must be documented in a Nonconformance Memo (NCM). The NCM is approved by the supervisor and then automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group also receives NCMs by e-mail for tracking and trending purposes. The NCM process is described in more detail in SOP TA-QA-0610. This is in addition to the corrective actions described in the following sections.

9.2 Batch Definition

Batches are defined at the sample preparation stage. The batch is a set of up to 20 samples of the same matrix, plus required QC samples, processed using the same procedures and reagents within the same time period. Batches should be kept together through the whole analytical process as far as possible, but it is not mandatory to analyze prepared extracts on the same instrument or in the same sequence. The method blank must be run on each instrument and in each analytical batch.

9.3 Method Blanks

For each batch of samples, analyze a method blank. The method blank is analyzed after the calibration standards and before any samples. For low-level volatiles in water, the method blank consists of reagent water. For low-level volatiles in soil, the blank medium is muffled Ottawa sand. For high-level volatiles, the method blank consists of 10 mL of *reagent grade methanol* and ten grams of muffled Ottawa sand. Surrogates are added and the method blank is carried through the entire analytical procedure.

Acceptance Criteria: *For DoD/LaMP projects* the method blank must not contain any analyte of interest at or above one-half the reporting limit *or above 1/10 the measured concentration of the analyte in the associated samples or 1/10 the regulatory limits, whichever is greater. For DoD projects, when written approval is received (method notes will contain "2CLC" or "Std Var App" to indicate approval has been received), the method blank must not contain any common laboratory contaminants above the reporting limit.* Contamination up to the reporting limit is allowed for non DoD/LaMP projects or at or above *1/10* of the measured concentration of that analyte in the associated samples, whichever is higher.

The method blank must have acceptable surrogate recoveries.

Corrective Actions: For DoD projects, if the analyte is a common laboratory contaminant (i.e., *acetone, 2-butanone, carbon disulfide and methylene chloride*), *and written approval has been received*, the data may be reported with qualifiers if the concentration of the analyte is less than the reporting limit. For non-DoD if the analyte is a common laboratory contaminant (i.e., methylene chloride, acetone, 2-butanone, ethyl ether, Acetonitrile and hexane) the data may be reported with qualifiers if the concentration of the analyte is less than five times the reporting limit.

Reanalysis of samples associated with an unacceptable method blank is required when reportable concentrations are determined in the associated samples.

If there is no target analyte greater than the RL (less than one half the RL for DoD clients) in the samples associated with an unacceptable method blank, the data may be reported with qualifiers for non-DoD clients. For DoD clients the data may only be reported if written approval has been received.

If surrogate recoveries in the blank are not acceptable, the data must be evaluated to determine if the method blank has served the purpose of demonstrating that the analysis is free of contamination. If surrogate recoveries are low and there are reportable analytes in the associated samples, re-extraction of the blank and affected samples will normally be required. Consultation with the client should take place.

If reanalysis of the batch is not possible due to limited sample volume or other constraints, the method blank is reported, all affected analytes in the associated samples are flagged as required by the project, and appropriate comments may be made in a narrative to provide further documentation.

9.4 Surrogates

Every sample, blank (including instrument blanks), and QC sample is spiked with surrogates. Surrogate recoveries in samples, blanks, and QC samples must be assessed to ensure that recoveries are within established limits. The compounds included in the surrogate spiking solutions are listed in Table 6.

Acceptance Criteria: Acceptance limits for surrogate recoveries are set at ± 3 standard deviations around the historical mean *or as defined by project or program requirements*. Surrogate recovery limits are updated at a fixed frequency by QA and stored in the LIMS.

Corrective Actions: If any surrogates are outside limits, the following corrective actions must take place (except for dilutions):

- Check all calculations for error.
- Ensure that instrument performance is acceptable.
- Recalculate the data and/or reanalyze if either of the above checks reveal a problem.
- Re-prepare and reanalyze the sample or flag the data as "Estimated Concentration" if neither of the above resolves the problem.

The decision to reanalyze or flag the data should be made in consultation with the client. It is necessary to re-prepare/reanalyze a sample only once to demonstrate that poor surrogate recovery is due to matrix effect, unless the analyst believes that the repeated out of control results are not due to matrix effect.

If the surrogates are out of control for the sample, matrix spike, and matrix spike duplicate, then matrix effect has been demonstrated for that sample and re-preparation/reanalysis is not necessary. If the sample is out of control and the MS and/or MSD is in control, then reanalysis or flagging of the data is required.

Re-analysis is not necessary if obvious matrix effect is shown in the chromatograms (e.g. a large co-eluting peak with the same quantitation ion, or non-target interferences) or were noted in sample prep (e.g. high percent moisture content without moisture correction). A non-conformance memo is generated stating the reason for not re-analyzing the affected sample.

NOTE: For LaMP client samples, if the surrogate percent recovery fails, the recovery must be confirmed by re-extraction and reanalysis with the following exceptions:

- The lab has unequivocally demonstrated a sample matrix effect and informed the LaMP client representative.
- The recovery exceeds control limits and all target analytes in the sample are non-detect.

9.5 Laboratory Control Samples (LCS)

An LCS is analyzed for each batch. The LCS is analyzed after the calibration standard and the method blank, and normally before any samples. The LCS is prepared from a different source than are the calibration standards. The LCS contains all of the analytes of interest (see Table 7a) and must contain the same analytes as the matrix spike.

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 *or based on project/program limits*. 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. Recovery limits are updated at a set frequency by QA and are stored in the LIMS.

If there are a large number of analytes in the LCS, then a specified number of results may 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 exceedances are recognized and allowed by NELAC and DoD. For DoD, Marginal exceedances are not allowed for those analytes determined by a project to be target analytes (i.e. "risk drivers") without project specific approval. 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.

For non-DoD projects, if the LCS recovery is high and there are no detections in the associated samples for the affected analytes the data may be reported with qualifiers. For DoD projects the data may only be reported with qualifiers if approval has been received in writing (method notes will contain "3HR" or "Std Var App" to indicate approval has been received).

Note: For DOD projects, all exceedances of LCS Control Limits, marginal or otherwise, are subject to corrective action. Therefore, all instances of LCS failures including the high bias but not detected in the associated samples scenario, must be investigated. For example and as noted above, the randomness of these failures can be evaluated or the spike solution can be re-verified. When the source of the problem is identified, corrective action is taken.

Corrective Actions:

If any analyte or surrogate is outside established control limits as described above, the system is out of control and corrective action must occur. Corrective action will normally be re-preparation and reanalysis of the batch.

If the batch is not re-extracted and reanalyzed, the reasons for accepting the batch must be clearly presented in the project records (via NCMs and the case narrative) and in the final report. Examples of acceptable reasons for not reanalyzing might be that the matrix spike and matrix spike duplicate are acceptable, and sample surrogate recoveries are good, demonstrating that the problem was confined to the LCS. This type of justification should be reviewed and documented with the client before reporting.

If re-extraction and reanalysis of the batch is not possible due to limited sample volume or other constraints, the LCS is reported, all associated samples are flagged, and appropriate comments are made in a narrative to provide further documentation.

9.6 Matrix Spike and Matrix Spike Duplicate (MS/MSD)

For each QC batch, analyze a matrix spike and matrix spike duplicate. Spiking compounds and levels are given in Tables 7 and 7a. The selection of the spike solution is dependent on client program requirements. The matrix spike/duplicate must be analyzed at the same base dilution as the unspiked sample, even if the matrix spike compounds will be diluted out. Dilutions (beyond the base dilution if necessary) of MS/MSD analyses are not required unless there are specific client instructions to do so. If necessary, this requirement will be passed to the laboratory through the PM by means of the mechanisms described in section 9.1.3 of this SOP.

Acceptance Criteria: The MS/MSD 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. The relative percent difference (RPD) between the MS and the MSD must be less than the established RPD limit, which is based on statistical analysis of historical data. MS/MSD recovery and RPD limits are updated at a regular frequency by QA and are stored in the LIMS.

Corrective Actions: If any individual recovery or RPD falls outside the acceptable range, corrective action must occur. The initial corrective action will be to check the recovery of that analyte in the LCS. Generally, if the recovery of the analyte in the LCS is within limits, then the laboratory operation is in control and analysis may proceed. The reasons for accepting the batch must be documented.

If the recovery for any component is outside QC limits for both the matrix spike/matrix spike duplicate and the LCS, the laboratory is out of control and corrective action must be taken. Corrective action will normally include reanalysis of the batch, except in cases where a high bias is indicated and no target is detected above the reporting limit in any associated sample.

If an MS/MSD is not possible due to limited sample, then a LCS duplicate (LCSD) should be analyzed. The RPD between the LCS and LCSD is compared to the established acceptance limit.

9.7 If batch QC samples or trip blanks are re-analyzed to confirm a recovery or result, and an improvement in results would cause the re-analysis to be reported, then the associated client samples must also be re-analyzed. The only exception to this protocol would be if an obvious analytical problem occurred during the initial analysis (i.e. no internal standard added, bent autosampler needle, etc).

9.8 Any extra QC that is analyzed in a batch or sequence must be evaluated using the same criteria as the corresponding QC above.

10.0 Procedure

10.1 Samples scheduled for EPA 624 will be analyzed separately (different ICAL and sequence) from samples scheduled for EPA 8260B *or 8260C*. Refer to appendix A for Modifications for Method 624.

10.2 One time procedural variations are allowed only if deemed necessary in the professional judgment of management to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation shall be completely documented using a Nonconformance Memo and approved by a Supervisor or group leader and QA Manager. If contractually required, the client shall be notified. The Nonconformance Memo shall be filed in the project file.

10.3 Any unauthorized deviations from this procedure must also be documented as a nonconformance, with a cause and corrective action described.

11.0 Calibration

11.1 Summary

Prior to the analysis of samples and blanks, the GC/MS system must be tuned and calibrated. Tuning is accomplished by analyzing 4-bromofluorobenzene (BFB) to establish that the GC/MS system meets the standard mass spectral abundance criteria. The GC/MS system must be calibrated initially at a minimum of five concentrations to determine the linearity of the response utilizing target calibration standards. The calibration must be verified each twelve-hour time period for each GC/MS system. The use of a separate calibration is required for low level water.

11.2 Recommended Instrument Conditions

11.2.1 General

Electron Energy:	70 volts (nominal)
Mass Range:	35–300 amu
Scan Time:	to give at least 5 scans/peak, ≤ 2 seconds/scan
Injector Temperature:	200 – 250 ˚C
Source Temperature:	According to manufacturer's specifications
Transfer Line:	Temperature: 250 – 300 ˚C
Purge Flow:	40 mL/minute *(± 5* mL/min*)*
Carrier Gas Flow:	1-15 mL/minute, dependent upon column specifications

11.2.2 Gas Chromatograph Temperature Program

The temperature programs vary with the column type and instrumentation used. The GC run program on each instrument should be optimized so that each peak is broad enough to accommodate at least 5 scans across the peak (not counting the scans at the baseline start and end of the peak). The actual individual method parameters used are stored in each individual instrument methods folder on the network and can be referenced there.

11.3 Instrument Tuning

Each GC/MS system must be hardware-tuned to meet the abundance criteria listed below and in Table 8 for a maximum of a 50 ng injection or purging of BFB. Analysis must not begin until these criteria are met. These criteria must be met for each twelve-hour time period. The twelve-hour time period begins at the moment of injection of BFB. Its critical to accurately estimate the number of samples that can be analyzed <u>within</u> the 12-hour window. When a tune isn't analyzed every 12-hours (i.e., samples are analyzed outside of the 12-hour window), the event must be documented in a non-conformance memo and corrective must be taken. Whenever feasible, samples that were analyzed outside of the 12-hour window will be re-analyzed within a new 12-hour window. When reanalysis is not feasible, results for the affected samples can only be reported if it's technically justified (e.g., subsequent tune passes), the data has been qualified, and it's been authorized and accepted by the client. The BFB may be taken from a specified BFB Tune injection or from the CCVIS. In the case of a calibration sequence, a specified BFB Tune must be injected prior to the injection of the first calibration standard. If an acceptable tune is not achieved, the autosampler prepares another tune standard by adding BFB to either the CCVIS or an instrument blank. The autotune process is repeated once. If the subsequent tune attempt fails, one or more of the corrective actions suggested in the TestAmerica, Inc. corporate tune policy, CA-Q-QM-002 are attempted.

Mass	Ion Abundance Criteria
50	15 to 40 % of Mass 95
75	30 to 60 % of Mass 95
95	Base Peak, 100 % Relative Abundance
96	5 to 9 % of Mass 95
173	Less than 2 % of Mass 174
174	*50 to 120* % of Mass 95
175	5 to 9 % of Mass 174
176	Greater than 95 %, but less than 101 % of Mass 174
177	5 to 9 % of Mass 176

11.4 Initial Calibration

11.4.1 A series of five or more initial calibration standards is prepared and analyzed for the target compounds. Nominal calibration levels for a *standard* level water purge, *low level soil purge* and high level methanolic extract purge are 0.2, *0.4,* 1, 2, 5, 10, 20, 50, 100 and 150 µg/L. Low level

waters curves are prepared at 0.02, 0.04, 0.10, 0.20, 0.40, 1, 2, 5, 10, 20, 40, and 100 ug/L. Certain analytes are prepared at higher concentrations due to poor purge performance. Table 4 shows the calibration levels for each analysis. The purge volume *is 10 mL.* Other calibration levels and purge volumes may be used depending on the capabilities of the specific instrument or program requirements. Calibration levels may also vary based on analyst discretion in so far as the minimum number of calibration points are met for the curve type utilized (five for average response factor and first order curves, six for second order curves) and the lowest point on the curve is at or below the current TestAmerica Seattle reporting limit.

11.4.2 The same purge volume must be used for calibration and sample analysis, and the low level standard must be at or below the reporting limit.

11.4.3 It may be necessary to analyze more than one set of calibration standards to encompass all of the analytes required for some tests.

11.4.4 Rejection of Calibration Points

Calibration levels below the reporting limit may be removed provided that the minimum number of calibration points are met for the curve type utilized (five for average response factor and first order curves, six for second order curves), and the lowest standard is at or below the TestAmerica Seattle reporting limit.

High point calibration levels may also be removed in so far as the minimum number of calibration points are met for the curve type utilized (five for average response factor and first order curves, six for second order curves) and the midpoint of the curve (the ICIS) is not the highest point of the calibration range.

Generally, it is NOT acceptable to remove mid-points from a calibration. If calibration acceptance criteria are not met, the normal corrective action is to examine conditions such as instrument maintenance and accuracy of calibration standards. Any problems must be fixed and documented in the maintenance logbook. Then the calibration standards must be reanalyzed. If, however, there is documented evidence of a problem with a calibration point (e.g. misinjection, poorly sealed vial, etc…) then one point might be rejected, but it is recommended to re-calibrate.

Refer to Corporate SOP GA-Q-S-005, Calibration Curves, for further details.

11.4.5 Internal Standards

Internal standard calibration is used. The internal standards are listed in Table 5. Target compounds should reference the nearest internal standard. Each calibration standard is analyzed and the response factor (RF) for each compound is calculated using the area response of the characteristic ions against the concentration for each compound and internal standard. See Corporate SOP CA-Q-S-005, for calculation of response factor and other related algorithms.

11.4.6 Calibration Check Compounds (CCC), 8260B

For 8260B the % RSD of the calibration check compounds (CCC) must be less than or equal to 30% even in cases where a first or second order regression is used for the calibration curve. Refer to Table 10. If the %RSD exceeds 30% for any CCC, the system must be evaluated (e.g. maintenance and accuracy of calibration standards) and the calibration re-run.

11.4.7 System Performance Check Compounds (SPCC), 8260B

The average RF must be calculated for each compound. A system performance check is made prior to using the calibration curve. *For 8260B* the five system performance check compounds (SPCC) are checked for a minimum average response factor. Refer to Table 9 for the SPCC compounds and required minimum response factors. If the minimum response factors are not met for any CCC, the system must be evaluated (e.g. maintenance and accuracy of calibration standards) and the calibration re-run.

11.4.8 *For 8260C the most common target analytes are checked for a minimum response factor for each calibration level. See Table X for the compound list and minimum relative response factor criteria. In addition, meeting the minimum response factor criteria for the lowest calibration standard is critical in establishing and demonstrating the desired sensitivity. Due to the large number of compounds that may be analyzed by this method, some compounds will fail to meet this criteria. For any anlayte non-detect associated with a calibration that fails the minimum response factor criteria there must be a demonstration of adequate sensitivity at the quantitaion limit. This is achieved by the successful*

analysis of a LLCCV (CCV at the reporting limit) in the same analytical batch. The criterion for the LLCCV is detection only but the standard qualitative identification criteria in the method must be met.

11.4.9 If all of the %RSD values in the calibration are ≤ 15% *for 8260B*, then all analytes may use average response factor for calibration. If all of the %RSD values in the calibration are *≤20% for 8260C*, then all analytes may use average response factor for calibration. *For analytes that fail the RSD criteria, use linear or quadratic curve.*

11.4.10 If the software in use is capable of routinely reporting curve coefficients for data validation purposes and the necessary calibration reports can be generated, then the analyst should evaluate analytes with %RSD ≤ 15% (8260B or DoD) or ≤ 20% (8260C) for calibration on a curve. If it appears that substantially better accuracy would be obtained using quantitation from a curve, then the appropriate curve should be used for quantitation. The correlation coefficient (r) must be ≥ 0.990 for SW-846 and must be ≥ 0.995 for DoD clients. For non-linear curves, the coefficient of determination (r^2) must be ≥ 0.990 for both SW-846 and DoD requirements.

11.4.11 If the software in use is capable of routinely reporting curve coefficients for data, then calibration on a curve must be used for all analytes with %RSD > 15% *(8260B) or %RSD > 20% (8260C).* The analyst should consider instrument maintenance to improve the linearity of response. Otherwise, the correlation coefficient (r) must be ≥ 0.990 for SW-846 and must be ≥ 0.995 for DoD clients. For non-linear curves, the coefficient of determination (r^2) must be ≥ 0.990 for both SW-846 and DoD requirements.

11.4.12 *For 8260C no more than 10% of compounds can fail the 20% RSD/0.990 correlation coefficient requirement. Any individual analyte result that fails the 20%RSD/0.990 correlation coefficient requirement must be flagged or narrated as an estimated concentration.*

11.4.13 *For any anlayte non-detect associated with a calibration that fails the 20%RSD/0.990 correlation criteria there must be a demonstration of adequate sensitivity at the quantitaion limit. This is achieved by the successful analysis of a LLCCV (CCV at the reporting limit) in the same analytical batch. The criterion for the LLCCV is detection only but the standard qualitative identification criteria in the method must be met.*

11.4.14 See Corporate SOP CA-Q-S-005 for information on acceptable initial calibration models and associated algorithms.

11.4.15 Initial Calibration Verification (ICV)

Once the initial calibration has been evaluated and determined to be valid, the calibration must be verified with an Initial Calibration Verification (ICV) using a standard prepared from an alternate source. The ICV is generally run at 50 ug/L for *standard* level water*, low-level soil and methanol preserved soil*, and 10 ug/L for low level water curves. As the ICV concentration must not be equal to or greater than the highest calibration level, other ICV levels than those previously listed may be used or multiple levels of ICV may be needed to validate all compounds in the initial calibration curve.

For DoD and LaMP projects each target compound in the ICV must be <20% drift when compared to the initial calibration. The same criteria apply to BTEX and oxygenate compounds in the LaMP program. For *8260B* non-DoD and non-LaMP projects, all compounds must be <40% drift when compared to the initial calibration, except for poorly performing compounds listed in Table 11, which must be <45% drift. *For 8260C non-DoD and non-LaMP projects, all compounds must be <30% drift when compared to the initial calibration, except for poorly performing compounds listed in Table 11, which must be <45% drift.*

For DoD projects analyses may continue if the drift is <30% for identified poor performing analytes with written approval from the client. (Method comment 4PP). See table 11 for the list of indentified poor performers.

For non-DoD and LaMP projects analyses may continue for those analytes that fail the criteria with approval from the client and an understanding that these results would be considered estimates and could be used for screening purposes.

Corrective Action: If the %drift falls outside acceptance criteria, assess the system for possible problems (standard degradation, etc.), re-prepare the verification standard and re-analyze. If the second ICV also fails, corrective action is required (e.g. system maintenance, re-preparing intermediate standards, etc.) and the calibration must be re-prepared and re-analyzed. An

acceptable ICV must be achieved before sample analysis. No samples may be run until calibration has been verified.

11.4.16 If time remains in the 12-hour period initiated by the BFB injection before the initial calibration, samples may be analyzed. Otherwise, proceed to continuing calibration, Section 11.5.

11.5 Continuing Calibration

11.5.1 The initial calibration must be verified every twelve hours. *For DoD projects the calibration must also be verified with a closing continuing calibration standard (CCVC) at the end of each analytical sequence.*

11.5.2 Continuing calibration begins with analysis of BFB as described in Section 11.3. If the system tune is acceptable, the continuing calibration standard(s) are analyzed. The level 6 calibration standard is generally used as the *CCV and CCVC*.

11.5.3 *For 8260B non-DoD projects* the RF data from the standards are compared with the initial multi-point calibration to determine the percent drift of the CCC and target compounds. The % drift limits of CCC, target and surrogate compounds are summarized in Table 12.

11.5.3.1 If not all of the CCCs are required analytes, project specific calibration specifications (which may include the use of the CCCs listed in Table 10) must be agreed with the client.

11.5.3.2 Non CCC target compounds that exceed the specified limits for Drift or Difference should be flagged.

11.5.3.3 For sub lists having less then 10 target analytes, the % drift of difference should be <20% before analysis proceeds.

11.5.4 *For 8260B (both DoD and non-DoD),* the SPCCs are also monitored. The SPCCs must meet the criteria described in Table 9.

11.5.5 *For 8260C non-DoD projects, the percent difference or drift (%D) of target and surrogate compounds must be within ± 20% for 80% of the target compounds.*

11.5.6 For DoD *and LaMP projects*, the percent difference or drift (%D) of target and surrogate compounds must be within ± 20% *for the opening CCV.* The same criteria apply to BTEX, oxygenate and surrogate compounds in the LaMP program.

11.5.7 For DoD projects t*he percent difference or drift (%D) of target and surrogate compounds must be within ± 50% for the closing CCV (CCVC).*

11.5.8 *For non-DoD* projects the retention time of the internal standards in the continuing calibration standard cannot change by more than 30 seconds (0.5 min) when compared to the most recent multi-point calibration. *For DoD projects the retention time of the internal standards in the continuing calibration standard cannot change by more than 10 seconds when compared to the most recent multi-point calibration. The 30 second criteria may only be used for DoD projects if prior written approval is received (method notes will contain "8ISRT" or "Std Var App" to indicate approval has been received).* The internal standard areas must not change by more than a factor of 2 (50 - 200 %) from the mid point standard of the most recent multi-point calibration.

11.5.9 If CCC, SPCC*,* target and/or surrogate compounds do not meet the criteria in Sections 11.5.3 through 11.5.8, the system must be evaluated and corrective action must be taken. The BFB tune and continuing calibration must be acceptable before analysis begins. Extensive corrective action, such as a different type of column, will require a new initial calibration. *For non-DoD and LaMP,* if two CCVs in a row fail, a new initial calibration must be performed. *For DoD two additional consecutive CCVs must be run immediately. If both pass, samples may be reported without reanalysis. If either fails corrective action and re-calibration is required.*

Corrective Actions:

For non-DoD projects if the CCV recoveries of the CCC, target or surrogates compounds exceed their specified limits, and there are no associated sample detections above the RL, the data may be qualified and reported as the system has shown a potentially high bias. *For DoD projects, if the CCV recoveries for target compounds exceed their specified limits, and there are no associated sample detections above the RL, the data may be qualified and reported as the system has shown a potentially high bias only if prior approval has been received in writing (method notes will contain "3HR" or "Std Var App" to indicate approval has been received).* For all other cases, results may not

be reported without a valid CCV. Flagging is only appropriate in cases where the samples cannot be reanalyzed.

11.5.10 Once the above criteria have been met, sample analysis may begin. Initial calibration average RFs (or the calibration curve) will be used for sample quantitation, not the continuing calibration RFs. Analysis may proceed until 12 hours from the injection of the BFB have passed. (A sample desorbed less than or equal to 12 hours after the BFB is acceptable.)

11.6 Sample Preparation

Check the Balance logbook to determine if the daily calibration check has been completed. If it has not, the analyst must perform this check according to SOP TA-QA-0014

11.6.1 Sample extraction for High-Level Analysis using in house extraction.

11.6.1.1 When extracting the sample, extrude (for 5 g EnCore™ or TerraCore™) or weigh (for 10 g in house soil extraction) the sample into a tared 20 mL scintillation vial. Record scintillation vial lot number, the sample weight, the methanol lot number, the preparation date and the analyst who prepared it in the 5035 *preparation batch in TALS*. *Sample weights are calculated in the laboratory by adding the tare weight of the scintillation vial to the "Tare Weight" column and then entering the weight of the scintillation vial plus sample into the "Vial & Sample" column of the preparation batch sheet for the corresponding sample container ID. This can be done by either a direct read from the balance in the volatiles prep area (preferred method), or by manually entering the weight. If the samples were preserved in MeOH at another TALS lab the calculated initial sample weight can be manually entered into the "Initial Amount" column of the preparation batch sheet for the corresponding sample container ID.*

11.6.1.2 For each batch of up to 20 samples a method blank (MB) and a Laboratory Control Sample (LCS) are also extracted. To prepare the method blank (MB); add 10-mL of methanol to 10 g Ottawa sand. To prepare the blank spike (LCS)/blank spike duplicate (LCSD), add 160-uL of each 8260 Working Spike Solution and 10-mL of methanol to 10 g muffled Ottawa sand. (NOTE: The same metal spatulas to weigh soil samples must be used for measuring out the Ottawa sand).

11.6.1.3 If sufficient sample is available, one matrix spike (MS)/matrix spike duplicate (MSD) pair is extracted per extraction batch of up to 20 samples. If the sample set being extracted consists of products, waste oils, or other sample matrixes which based on analyst experience and discretion would not yield acceptable spike results due to matrix effects, a matrix duplicate (MD) and/or MD/MS may be prepared in lieu of and MS/MSD. To prepare the matrix spike (MS)/matrix spike duplicate (MSD), add 160-uL of each 8260 Working Spike Solution and 10-mL of methanol to 10 g pre-weighed soil samples.

11.6.1.4 Add 5 mL (for EnCore™ or TerraCore™) or 10 mL (in house soil extraction) of methanol to all vials immediately after recording sample weight.

11.6.1.5 Vortex the samples for the extraction batch for approximately 10 to 30 seconds to break up any large clumps in the extraction vials. This is especially important for extruded samples as they may be compacted in the EnCore™ or TerraCore™ sampling device and come out as a pellet. If after 30 seconds a pellet still remains, vortex for an additional 30 seconds. If pellet still remains, further vortex mixing is not recommended, proceed to next step. It should be noted that the MB and LCS must be vortex mixed the same amount of time as the longest associated sample.

11.6.1.6 After all samples have been vortex mixed, place all samples for the extraction batch into shaker box and set timer for five minutes. It is recommended that the caps of all vials are checked and tightened before placing in shaker box to prevent leaking. If samples are present which still contain pelletized sample after vortex mixing in step 11.6.1.5, set the timer for 10 to 15 minutes. It is not recommended to shake samples for more than 15 minutes. If a sample still contains pelletized sample after shaking for 15 minutes, vortex the sample for an additional 15 seconds and shake entire batch for additional 5 minutes. Any pelletized sample remaining after second shaking is noted in and NCM in the extraction batch.

11.6.1.7 After samples are shaken, place samples four at a time into centrifuge with inserts for scintillation vials. Spin samples for a sufficient time to create a transparent but not necessarily uncolored layer of methanol extract above the extracted material. The time will vary depending on the nature and particle size of the extracted material. Three to five minutes at 50% speed is usually sufficient. Again it should be noted that the MB and LCS must be centrifuged at the same rate and for the same time as the longest centrifuged associated sample.

11.6.1.8 High-level solid extracts are stored in the scintillation vial used for extraction and are stored at 0-6°C. The extracts are removed from cold storage and are allowed to return to ambient temperature prior to analysis.

11.6.2 Sample extraction for High-Level Analysis field preserved.

11.6.2.1 Each containers tare weight is recorded *in the TALS preparation batch.* Most containers will contain a bar code with the tare weight information that can be scanned for automatic entry into *the tare weight entry field in the preparation batch*. Sample weights are calculated in the laboratory by adding the received weight of the sample jar to the *"Vial & Sample"* column *of the preparation batch sheet for* the corresponding sample container ID. This can be done by either a direct read from the balance in the volatiles prep area (preferred method), or by manually entering the weight. *If the samples received are in 4 oz jars with 25 mls of methanol (AK samples) the calculated initial weight of the sample must be adjusted to correct for the weight of the methanol which is not included in the container tare weight. TALS will perform this calculation, however the analyst must enter a "1" into the "Tare Incl MeOH" column of the preparation batch sheet for the corresponding sample container ID.*

11.6.2.2 For each batch of up to 20 samples a method blank (MB) and a Laboratory Control Sample (LCS) are prepared by the laboratory prior to sample analysis. A MB and an LCS sample consists of 10 g of muffled Ottowa Sand added to a scintillation vial followed by 10-mL of methanol.

11.6.2.3 Add the correct amount of matrix spiking solutions to all LCS samples. An aliquot of 160 uL of each 8260 Working Spike Solution is added to 10 mL extracts. The addition of the spike solutions introduces a slight error, which can be neglected in the calculations. The listed volumes are halved (80 uL) for 5-mL extracts.

11.6.2.4 The MB, LCS and any received samples which appear to contain "clumps" of sample which could be broken up with agitation are vortex mixed for up to 1 minute. If no samples require agitation, only the MB and LCS are vortex mixed for approximately 15 seconds to ensure mixing of the sand and added solutions.

11.6.3 Sample preparation for Low-Level Analysis EnCore™.

11.6.3.1 Prior to analysis, low level soil samples collected in 5g EnCore™ sampling devices are removed from the freezer and allowed to partially thaw in order to facilitate extrusion from the sampling device. *Low level soil samples collected in VOA vials with or without water are removed from the freezer and allowed to thaw.* Samples should not be allowed to remain at room temperature for more than 30 minutes prior to extrusion into a VOA vial or analysis.

11.6.3.2 The weight of the samples is accurately recorded by either direct connection from the analytical balance to the batch record (preferred method) or by typing the balance reading into the batch record.

11.6.3.3 The extruded sample then has a cleaned magnetic stir bar added to it. *The extruded sample or the sample received in a VOA vial without water has ten mls of reagent water added to it. The sample received in a VOA vial with 5 mls of water has 5 mls of reagent water added to it.*

11.7 **Sample Analysis**

11.8 Preliminary Evaluation

11.8.1 Sample screening

Where possible, samples are screened by headspace or GC/MS off-tune analysis to determine the correct aliquot for analysis. Alternatively, an appropriate aliquot can be determined from sample histories. Refer to section 11.15 for Dilutions.

11.9 Sample Analysis Procedure

11.9.1 All analysis conditions for samples must be the same as for the continuing calibration standards (including purge volume, time and flow, desorb time and temperature, column temperatures, multiplier setting etc.).

11.9.2 All samples must be analyzed as part of a batch. The batch is a set of up to 20 samples of the same matrix processed using the same procedures and reagents within the same time period. The batch also must contain a MS/MSD (if sufficient sample volume allows), an LCS, and a method blank.

11.9.2.1 Laboratory generated QC samples (Blank, LCS, MS/MSD) do not count towards the maximum 20 samples in a batch. Field QC samples are included in the batch count.

11.9.2.2 It is not necessary to reanalyze batch QC (except for the method blank) with reanalysis of samples. However, any re-runs must be as part of a valid batch.

11.10 Water Samples

11.10.1 All samples and standard solutions must be at ambient temperature before analysis.

11.10.2 *For low-level analysis* water samples are sub sampled by the autosampler at the appropriate volume (10 mL).

11.10.3 *For standard level analysis 10 mls of each sample or QC is sub sampled by the analyst using a pre-cleaned 10 ml gas-tight syringe. This aliquot may then transferred to a purge vessel.*

11.10.4 *For low-level analysis* an internal standards and surrogates mix is added by the autosampler. Refer to Tables 5 and 6.

11.10.5 *For standard level analysis 2ul of internal standards and surrogate mix is added by the analyst by either direct injection into the purge vessel or directly into the syringe containing the sub-sampled aliquot and then transferring to a purge vessel. The purging vessel needs to be sealed quickly to keep the compounds from releasing into the air. Refer to Tables 5 and 6.*

11.10.6 MS/MSD samples are prepared by injecting each 8260 Working Spike Solution through the VOA vial septa using a bevel tipped syringe *or directly into the syringe containing the sub-sampled aliquot.*

11.10.7 Purge the sample at ambient temperature with a trap temperature of 25°C.

11.10.8 After purging is complete, dry purge and desorb the sample, start the GC temperature program, and begin data acquisition. After desorption, bake the trap for 5-10 minutes to condition it for the next analysis. When the trap is cool, it is ready for the next sample.

11.10.9 Purge Time, dry purge time, desorb time, bake time, and temperature are optimized for the type of trap in use and the analytical system. The same conditions must be used for samples and standards. Current at the time of writing this SOP, purge time for all instruments is 8 to 11 minutes, dry purge is 1 minute or 2 minutes, bake time is 6 to 9 minutes, desorb temperature is 195 to 260°C, and bake temperature is 220 to 270°C.

11.10.10 Immediately after analysis or immediately after opening the sealed VOA vial and obtaining the necessary aliquots for dilutions, the analyst must check the pH of aqueous samples with narrow range pH paper to ensure the pH is < 2. Record the pH of aqueous samples *and the lot number of the pH paper used* on the analytical batch sheets. In those cases where the pH is > 2, initiate a non-conformance report and qualify the data, noting if the sample(s) was analyzed outside of the shortened seven-day hold time or zero-day hold time for aromatics.

11.11 TCLP Leach Samples

11.11.1 Follow the instructions for water samples using a 100 times dilution for the method blank, laboratory control sample, and all samples. An MS/MSD pair must be prepared per batch by the method described in section 11.10.6.

11.12 Methanol Extract samples

11.12.1 Sample collection and initial preparation for medium level soil samples has been discussed in section 8.6, 8.7, 8.8 and 11.6.

11.12.2 *Fill a pre-cleaned 10 ml gas-tight syringe with 9.75 mls of reagent water.*

11.12.3 *Add 250 uL of methanolic extract to the syringe. This aliquot may then transferred to a purge vessel.*

11.12.4 *The final volume of reagent water and methanolic extract used is entered into Chrome which then is uploaded into the analytical batch in TALS.*

11.12.5 As with water samples, *2ul of internal standards and surrogate mix is added by the analyst by either direct injection into the purge vessel or directly into the syringe containing the sub-sampled aliquot and then transferring to a purge vessel. The purging vessel needs to be sealed quickly to keep the compounds from releasing into the air. Refer to Tables 5 and 6.*

11.12.6 Load the sample in the autosampler and proceed to analyze.

11.12.7 MS/MSD samples for in house extracts are prepared at time of extraction and are prepared for analysis as above. For field preserved samples, an in house post spike of the prepared sample is necessary, and is prepared by injecting each 8260 Working Spike Solution through the VOA vial septa using a bevel tipped syringe *or directly into the syringe containing the sub-sampled aliquot.*

11.12.8 Dilutions of methanolic extracts are made by adding proportional amounts of 250 uL to the 10 ml gas-tight syringe with reagent water. Samples of greater than 5x do not require the removal of water from the syringe prior to addition of methanolic extract.

11.12.9 Purge the sample at ambient temperature with a trap temperature of 25°C.

11.12.10 After purging is complete, dry purge and desorb the sample, start the GC temperature program, and begin data acquisition. After desorption, bake the trap for 5-10 minutes to condition it for the next analysis. When the trap is cool, it is ready for the next sample.

11.12.11 Purge Time, dry purge time, desorb time, bake time, and temperature are optimized for the type of trap in use and the analytical system. The same conditions must be used for samples and standards. Current at the time of writing this SOP, purge time for all instruments is 8 to 11 minutes, dry purge is 1 minute or 2 minutes, bake time is 6 to 9 minutes, desorb temperature is 195 to 260°C, and bake temperature is 220 to 270°C.

11.13 Low-Level Solids Analysis using Discrete Autosamplers

11.13.1 Sample collection and initial preparation for low level soil samples has been discussed in section 8.9 and 11.6.

11.13.2 *2ul of internal standards and surrogate mix is added by the analyst by direct injection into the VOA vials containing 10mls of water.* It is recommended that the needle is pushed through the vial septa, and the vial is tilted sufficiently to allow the tip of the needle to enter the water in the vial prior to pushing the plunger and expelling the solution. This facilitates a more accurate dispensing by ensuring that a drop of solution does not remain on the tip of the syringe as it is pulled back out of the vial septa. *Refer to Tables 5 and 6.*

11.13.3 MS/MSD samples for low level soil samples prepared by injecting each 8260 Working Spike Solution through the VOA vial septa using a bevel tipped syringe. Direct purge soil analyses are spiked during the preparation of the extraction batch through the septa after capping (as applicable).

11.13.4 When it is feasible to perform dilutions of low level soils, a smaller amount of sample is weighed and analyzed. When dilutions of low level soil samples are not possible, any analyte that exceeds the calibration range must be E flagged on the appropriate reporting form, NCM filled out, and must be noted in the case narrative. In addition, if sufficient volume was provided, a methanolic extract must be prepared and analyzed.

11.13.5 Purge the sample at ambient temperature with a trap temperature of 25°C.

11.13.6 After purging is complete, dry purge and desorb the sample, start the GC temperature program, and begin data acquisition. After desorption, bake the trap for 5-10 minutes to condition it for the next analysis. When the trap is cool, it is ready for the next sample.

11.13.7 Purge Time, dry purge time, desorb time, bake time, and temperature are optimized for the type of trap in use and the analytical system. The same conditions must be used for samples and standards. Current at the time of writing this SOP, purge time for all instruments is 8 to 11 minutes, dry purge is 1 minute or 2 minutes, bake time is 6 to 9 minutes, desorb temperature is 195 to 260°C, and bake temperature is 220 to 270°C.

11.14 Initial Review and Corrective Actions

11.14.1 Retention Times

For 8260B if the retention time for any internal standard in the continuing calibration changes by more than 30 seconds from the mid-level initial calibration standard, the chromatographic system must be inspected for malfunctions and corrected. Reanalysis of samples analyzed while the system was malfunctioning is required.

For DoD projects of 8260C if the retention time for any internal standard in the continuing calibration changes by more than 10 seconds from the mid-level initial calibration standard, the chromatographic system must be inspected for malfunctions and corrected. Reanalysis of samples analyzed while the system was malfunctioning is required.

11.14.2 Internal Standard Response

If the internal standard response in the daily continuing calibration is more than 200% or less than 50% of the response in the mid-level of the initial calibration standard, the chromatographic system must be inspected for malfunctions and corrected. Reanalysis of samples analyzed while the system was malfunctioning is required.

For non-DOD samples, the internal standard response in a sample is compared to the internal standard response in the daily continuing calibration. For DOD samples, the internal standard response in a sample is compared to the internal standard response of the mid point standard in the initial calibration (not the daily CCV). For this reason, separate analytical sequences need to be employed for DOD samples. Responses from 50% to 200% are acceptable. If a sample fails to meet these internal standard criteria, further investigation is necessary. If the change in sensitivity is a matrix effect confined to an individual sample, reanalysis is not necessary, though may be required to prove matrix effect. If the change in sensitivity is due to instrumental problems, all affected samples must be reanalyzed after the problem is corrected. If the ISTD response falls below 50% and the sample has no target analyte detections above the RL (1/2 the RL for DoD projects), the data may be qualified and reported. This should be done in consultation with the PM and client, an NCM must be written.

11.14.3 Surrogate Standard Recoveries

The surrogate standard recoveries are evaluated to ensure that they are within limits. Corrective action for surrogates out of control will normally be to reanalyze the affected samples. However, if the surrogate standard response is out high and there are no target analytes or tentatively identified compounds, reanalysis may not be necessary. Out of control surrogate standard response may be a matrix effect. It is only necessary to reanalyze a sample once to demonstrate matrix effect (this may be demonstrated by a MS/MSD analysis as well). Reanalysis at a dilution should be considered if appropriate. If a diluted analysis is necessary and surrogate recoveries are in control or less affected, this is sufficient to demonstrate matrix interference.

Re-analysis is not necessary if obvious matrix effect is shown in the chromatograms (e.g. a large co-eluting peak with the same quantitation ion, or non-target interferences) or were noted in sample prep (e.g. high percent moisture content without moisture correction). A non-conformance memo is generated stating the reason for not re-analyzing the affected sample.

11.15 Dilutions

Dilutions should be done just prior to the GC/MS analysis of the sample. Dilutions are made directly using *a 10 ml syringe*. *The syringe* should be filled with reagent water and the appropriate amount of water should be removed *from the syringe*. Sample is then added to the *syringe and the aliquot is immediately transferred to a purge vessel*.

If the response for any compound exceeds the working range of the GC/MS system, a dilution of the sample/extract is prepared and analyzed. A dilution should be prepared to ensure that the majority of compounds being diluted for fall in the middle to upper part of the calibration curve (i.e. from 40-90% of their respective calibration range). All reported dilutions must be within the calibration range of the respective analytes and should be compared to other dilutions to ensure that the diluted data “makes sense” as a check for possible dilution errors. If this cannot be accomplished with a single dilution, multiple sample dilutions are necessary. Dilution levels should be considered carefully and it is recommended that a “complicated” sample (one which has more than three compounds which require dilution) is discussed with another analyst or area supervisor as necessary in order to minimize the number of dilutions required. Samples may be screened to

determine the appropriate dilution for the initial run or historical site data may be used to determine initial dilutions.

11.15.1 Guidance for Dilutions Due to Matrix

If the sample is initially run at a dilution and the baseline rise is less than half the height of the internal standards, or if individual non target peaks are less than twice the height of the internal standards, then the sample should be reanalyzed at a more concentrated dilution. This requirement is approximate and subject to analyst judgment and reasonable client requirements and requests.

11.15.2 Reporting Dilutions

The most concentrated dilution will be reported as the base dilution. Other dilution levels will report only the required diluted compounds and all other compounds and surrogates in the dilution will be set to acceptable in the LIMS system and not reported. Other reporting techniques may be required by specific project requirements or client request and will be transferred to the laboratory by the PM using the mechanisms previously discussed. See Section 9.1.3.

11.15.3 Percent Moisture

Analytical results may be reported as dry or wet weight, as required by the client. Percent moisture must be determined if results will be reported as dry weight. Refer to SOP TA-WC-0125 for determination of percent moisture.

11.16 Instrument Maintenance

11.16.1 Agilent 5973 Inert, 5973 Network, 5975, 5975B, and 6890N

11.16.1.1 All circuit boards and peripheral attachments are dusted and vacuumed of debris and all plumbing and electrical connections inspected and adjusted; any replacement of worn parts is also done at this time.

11.16.1.2 The injection port is cleaned of debris by removing the injector and column nut and forcing clean methanol through the top of the injector into a waste container at the bottom. The inlet liner and gold seal are replaced as needed (e.g. visibly dirty or discolored, active site issues, etc.) The ion source is disassembled, cleaned, and reassembled with new filaments and insulators, if needed.

11.16.2 Column installation is performed when the following conditions are encountered;

- Heavy column bleed that cannot be eliminated by thermal conditioning.
- Loss of early eluting peaks due to column cutting.
- Inability to chromatographically resolve method performance compound peaks.
- Distortion of peak shapes i.e.; broadening, ghost peaks, split peaks that can't be resolved by injection port maintenance or flow control.

11.16.2.1 Turn the GC oven off and let the system cool to room temperature. Remove the column nut, liner, septum, and press tight inlet connector. Dispose of old column appropriately.

11.16.2.2 Cut approximately six inches off of the end of new columns. Install new column using appropriate sized ferrules and nuts.

11.16.2.3 Turn the GC on and set the injector temperature to 230 ℃, oven to the manufacturers recommended isotherm temperature or 10℃ below the manufacturers max temperature if an isotherm is not provided and condition for five minutes.

11.16.2.4 Perform an air water check on the system. When the air water spectrum shows acceptable levels, proceed with the mass calibration procedure. For additional information of column replacement see the manufacturer's operator's manual.

11.16.3 Tekmar 3000 Thermal Purge and Trap Concentrator Unit

11.16.3.1 A new Supelco K-trap (Vocarb 3000) is installed and conditioned by stepping the unit through to the bake setting. A new trap should be conditioned for a minimum of one hour prior to use, but an initial conditioning of overnight is recommended as time allows.

11.16.3.2 Sample lines, internal valves, sparge cells, and sparge cell mounts and fittings are rinsed with purge and trap grade methanol or replaced as necessary.

11.16.3.3 All dust and debris is removed from the circuit boards and tubing replaced where necessary.

11.16.3.4 The purge gas flow rate (40 mL/min ± 5 mL/min) should be measured at the vent and recorded in the maintenance logbook.

11.16.4 Archon 2000 or equivalent type auto sampler.

11.16.4.1 Remove debris and perform a calibration per manufacturer's instructions.

11.16.4.2 All dust and debris is removed from the circuit boards and tubing replaced where necessary.

11.16.4.3 The guide rails are wiped down with a Kim wipe and methanol to remove grease buildup and debris.

11.16.4.4 The syringe is cleaned per manufacturer's instructions to remove debris.

11.16.4.5 The DI reservoir is refilled prior to analysis.

11.16.4.6 The Internal Standard/Surrogate Standard vial is refilled as needed and primed after filling to ensure the instrument is drawing volume.

11.16.5 Major Maintenance

A new initial calibration is necessary following certain maintenance procedures. These maintenance procedures include changing the column, cleaning the source and replacing the multiplier. In addition, a new initial calibration may be necessary if a large amount of routine maintenance occurs at once. This will be determined by evaluating the system using a CCV.

11.16.6 Maintenance Logbook

All maintenance and repairs need to be documented in the instrument's maintenance logbook. The logbook must include the instrument name, serial number for each major component (e.g., GC, autosampler, *column*) and the date of start-up. When an instrument is not capable of analyzing samples, it needs to be tagged "Out of Service". Logbook entries must include a description of the problem and what actions were taken to address the problem. After an instrument has undergone maintenance or repairs, the system is evaluated using a tune, CCV or ICAL. If the evaluation is successful, the analyst documents in the logbook that the "System returned to control as indicated by a passing CCV" (or ICAL, MB, tune, etc as may be the case).

If columns were replaced during maintenance procedures, the specific make, model and serial numbers of the columns installed need to be entered in the instruments maintenance logbook.

11.17 Troubleshooting

11.17.1 QUESTIONS TO ASK YOURSELF

11.17.1.1 MECHANICAL ISSUE?

- Has the problem just started, or has it been getting worse with time?
- Has anything recently been done to the instrument that may have caused the problem to start?
- Have there been any nasty samples run on the instrument recently?

11.17.1.2 ANALYTE ISSUE?

- Are there any other compounds out or trending out?
- Did the CCV and/or LCS show similar trends?
- Is this a one time event? Or did you see similar signs yesterday?
- Is it failing on other mass specs?
- Did anything change from the last time it passed?
- Have there been any nasty samples run on the instrument recently?

11.17.1.3 INTERNAL/SURROGATE ISSUE?

- Have they been stable up until now?
- Is the gas on and are the standards fresh?
- Are the glass vials correctly positioned and tight?
- Do they look like they are trending in any direction or just fluctuating?
- Is the tune stable? Are the ratios close to the ICAL tune?

11.17.2 BEST PRACTISE

11.17.2.1 If you can – replace the most likely piece in the system, label the old one with which instrument it's from, the date out and possibly the problem you are having e.g. R1, 071010 Bromoform. Test if the problem is fixed. If not, move onto the next thing and replace until the problem is fixed. Then work backward and reinstall the old pieces which are still OK. If you no longer need a part that is still good, label it as such. If a piece is bad and not even good for testing with throw it out and tell your department manager what piece and part number.

11.17.3 TYPICAL FIRST THINGS TO LOOK AT AND TRY

11.17.3.1 CHECK YOUR FLOWS (For soil mode, purge on archon)

- Set the Archon to purge for 111mins instead of 11mins then set a vial to purge and "pause" the concentrator. This will give ample time to troubleshoot.
- Use the flow meter to check the flow out the purge vent using a small clear tube as an adapter – you should have about 40mls.
- Take the transfer line off at the entrance to the concentrator and measure the flow – you should also have about 40mls.
- Generally this would suggest you don't have a blockage or a leak, but check the following first
- Find a clear tube that has been blocked at one end, place this on the purge vent while the vial is purging and establish the following:
- Did the bubbles stop straight away <2mins?
- Yes – the leak is in the Archon
- Have the bubbles stopped over a 5 – 7 min period?
    - Yes – Most probably not a leak but could be a blockage (which includes the tube you put on the vent)
    - No – There is a leak somewhere, most likely the concentrator.
- If there is no soil valve attached then remove the transfer line inlet (to concentrator) fitting and replace with a capped fitting, Run the concentrator full leak check.
- If all of the above passes then you do not have a problem with the flows/blockage/leak.
- Remember to set Archon back to 11mins purge and bake off trap before running again.

11.17.3.2 IF YOU HAVE A LEAK

- In the Archon – purge a vial and leak check around the top of the needle, the transfer line and the pencil filter in the back of the Archon.
- In the concentrator – with a vial purging leak check around all the fittings starting with the entry from the transfer line, the 4-port valve, the 6-port valve ad around the water management and trap.
- You can also check the flows coming out of the 4-port, leaving the 6-port, there is an adapter which can be added instead of the trap to measure through the water management, from the trap. If all of these measures around 40mls and still you have less than 37 coming out the purge vent it could be leaking across the valves in the side of the concentrator.
- REMEMBER – any Archon that has been changed to Nitrogen will not be able to be checked with the leak detector.

11.17.3.3 COMMON PLACE TO LOOK IF IT'S A ANALYTE PROBLEM

- **1,1,2,2-Trichloroethane RRF** – Could be any of the lines as often an active site. Start with transfer line, lines in concentrator, water management and trap, soil valve if installed.
- **Bromoform** – Flow is not 40mls, water management, transfer line, small lines in concentrator. This can sometime be a tune issue as well if it is somewhat different from normal.
- **Chloromethane carryover** – Often the trap, replace the trap with the wire at the bottom and condition at 210°C for 30min.
- **Napthalene carryover/low response** – Often water management, change this out and condition with 5 or 6 varying concentrations of an old main or 524 std. e.g. 20ppb, 80ppb, 10ppb, 50ppb etc. Could also be transfer line.
- **2-Cleve** – could be anywhere. Start with transfer line and work way through system, small lines in concentrator, purge needle as last resort. Could also be water management, soil valve if installed.

- **MTBE** – often an active site could be transfer line or trap. Sometimes the instrument just needs recalibrating.
- **Vinyl Chloride** – Usually the first compound to fail low in the standard mix – simply move up to next standard. If you are changing to a new standard every couple of days then your ICAL standards were too fresh and you need to ICAL again.
- **1,1-Dichloroethene** – Usually the first compound to fail low in the Main mix – simply move up to next standard. If you are changing to a new standard every couple of days then your ICAL standards were too fresh and you need to ICAL again.
- **Chloroform** – usually your tune is differing from the ICAL
- **Contamination** – look back at previous runs, if you have high hits for this compound then run 20 – 30 blanks to clean it up. If you still have high concentrations then the sample possibly blew back into the instrument so could be purge valves or the pencil filter in the back of the Archon.
- **Ethylbenzene** – usually your tune is differing from the ICAL.

11.17.3.4 PEAK SHAPE DIFFERENT

- If you have peak tailing on the front end compounds such as Vinyl Chloride, Chloromethane etc then it is often a result of a worn trap.

11.17.3.5 SURROGATES FAILING

- If you have 1,2-DCA failing and all the other surrogates are OK, then the likely cause is the tune. Often the ratios will have moved. Follow the surrogates, if low, then drop ratios etc
- If you have all the surrogates out and it seems to be consistently that way, it could be the following,
    - Is the gas turned on?
    - Is the surrogate vial positioned properly on the archon
    - Your concentration levels in the ICAL maybe incorrect if you have just ICALed.
    - The valve has perhaps worn a little and the nominal value in target needs to be updated, but this can only happen after an ICAL.
    - If the surrogates need to be weighted linear or linear in an ICAL and you see them trending this way for a while its possible the column has gone bad or you may have a standard valve leak.
    - You can check to see if it's the archon causing the problem by manually injecting IS/SS and turn off the IS and SS on the archon
- If two surrogates are high and two are low, sometimes this is because your Archon surrogate is not matching with the surrogate you ICAL'd with. Try swapping out the Archon surrogate and if this does not fix it, re-ICAL the Supp with the new surrogate.

11.17.3.6 INTERNALS FAILING, TRENDING OR BOUNCING

- How fresh is your internals? If they are more than say a month old, they may have gone bad. Is the vial on properly and done up tight (not too tight).
- Is the gas turned on?
- How long has the instrument been using the current filament? More than 4weeks then you may want to change to the other or change the filaments if they are both spent.
- If the internals bouncing seems to correlate with the tune then most likely you want to change filament or clean the source.
- If the internals fall to a value and sit there or raise to a value and sit there you may want to consider ICAL it at that tune setting, instruments have a kind of sweet spot.
- Internals falling off maybe a water buildup and the parameters need looking at on the concentrator. Have a look at other instruments to see how they are set up and try one or two different settings to establish if this helps. Always keep a note of where they were at and what you have changed them too. Best place to record this is on your sequence and in the white folder under daily changes.
- Split ratios can also effect internals, try increasing the ratio to more like 45:1 if not already at this level.

11.17.3.7 CONCENTRATOR STOPS

- Turn it off and on again, it is common for the concentrator to lose the plot every so often.
- Shows a temperature error – check to make sure the trap is covered and the main cover is down properly, any heat escape will cause an error.

11.17.3.8 SEQUENCE STOPS

- You have started on a file that already exists and you have not checked the overwrite file box.
- The computer hard drive is full. You need to back up all but the last months files from C:/HPCHEM/DATA to the G:/RAWDATA/GCMSVOA/INSTRUMENT/ if you do not have access to the G drive, then save to the sample place on the L drive and copy over to the G drive from your office computer. This should be done each month.
- Communication problem with the GC and MS. Turn off the MS and GC and restart the computer. Restart the MS and GC.

11.17.3.9 ARCHON STOPS

- Standard home errors – syringe already home – use the maintenance menu on the archon to step you through cleaning the syringe. Before putting back together smear the inside of the syringe with 'Nose grease'.
- Standard home errors -

12.0 Calculations / Data Reduction

12.1 Qualitative Identification for Full Scan Analysis

12.1.1 An analyte is identified by retention time and by comparison of the sample mass spectrum with the mass spectrum of a standard of the suspected compound (standard reference spectrum). Mass spectra for standard reference may be obtained on the user's GC/MS by analysis of the calibration standards or from the NIST Library (same library as used for routine sample analysis). Two criteria must be satisfied to verify identification: (1) elution of sample component at the same GC retention time as the standard component; and (2) correspondence of the sample component and the standard component characteristic ions.

NOTE: Care must be taken to ensure that spectral distortion due to co-elution is evaluated.

12.1.1.1 The sample component retention time must compare to within ± 0.06 RRT units of the retention time of the standard component. For reference, the standard must be run within the same twelve hour tune as the sample.

12.1.1.2 All ions present in the standard mass spectra at a relative intensity greater than 10% (most abundant ion in the spectrum equals 100%) should be present in the sample spectrum.

12.1.1.3 The relative intensities of ions should agree to within ±30% between the standard and sample spectra. (Example: For an ion with an abundance of 50% in the standard spectra, the corresponding sample abundance must be between 20 and 80%.)

12.1.2 If a compound cannot be verified by all the above criteria, but in the technical judgment of the analyst, the identification is correct, then the analyst shall report that identification and proceed with quantitation.

12.1.3 All data are subject to two levels of technical review, as described in SOP TA-QA-0635.

12.2 Tentatively Identified Compounds (TICs)

12.2.1 If the client requests components not associated with the calibration standards, a search of the NIST library may be made for the purpose of tentative identification. The following guidelines apply:

12.2.1.1 Relative intensities of major ions in the reference spectrum (ions > 10% of the most abundant ion) should be present in the sample spectrum.

12.2.1.2 The relative intensities of the major ions should agree to within 20%. (Example: If an ion shows an abundance of 50% in the standard spectrum, the corresponding sample ion abundance must be between 30% and 70%).

12.2.1.3 Molecular ions present in the reference spectrum should be present in the sample spectrum.

12.2.1.4 Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible background contamination or presence of co-eluting compounds.

12.2.1.5 Ions present in the reference spectrum but not in the sample spectrum should be reviewed for possible subtraction from the spectrum because of background contamination or co-

eluting peaks. (Data system reduction programs can sometimes create these discrepancies.)

12.2.1.6 Computer-generated library search routines should not use normalization routines that would misrepresent the library or unknown spectra when compared to each other. Only after visual inspection of the sample with the nearest library searches should the analyst assign a tentative identification.

12.2.1.7 Once tentative identifications are assigned, these results are uploaded into LIMS with the other data and the TICs are automatically reported to the client from the LIMS.

12.3 Accuracy

ICV / CCV, LCS % Recovery = $\frac{\text{observed concentration}}{\text{known concentration}} \times 100$

MS % Recovery = $\frac{\text{(spiked sample) - (unspiked sample)}}{\text{spiked concentration}} \times 100$

12.4 Precision (RPD)

Matrix Duplicate (MD) = $\frac{|\text{orig. sample value - dup. sample value}|}{[(\text{orig. sample value + dup. sample value})/2]} \times 100$

12.5 Response Factor (RF)

$$RF = \frac{A_x C_{is}}{A_{is} C_x}$$

Where:

A_x = Area of the characteristic ion for the compound to be measured.
A_{is} = Area of the characteristic ion for the specific internal standard.
C_{is} = Concentration of the specific internal standard, ng.
C_x = Concentration of the compound being measured, ng.

12.6 Standard deviation (SD)

$$SD = \sqrt{\frac{\sum_{i=1}^{n}\left(X_i - \overline{X}\right)^2}{n-1}}$$

Where:

X_i = Value of X at i through n.
n = Number of points.
$\overline{X}$ = Average value of Xi.

12.7 Percent relative standard deviation (%RSD)

$$\%RSD = \frac{SD}{\overline{RF}} \times 100\%$$

Where $\overline{RF}$ is the mean of RF values for the calibration.

12.8 **Percent drift between the initial calibration and the continuing calibration:**

$$\%Drift = \frac{C_{expected} - C_{found}}{C_{expected}} \times 100\%$$

Where:

$C_{expected}$ = Known concentration in standard.
C_{found} = Measured concentration using selected quantitation method.

12.9 **Concentration** = mg/kg or L = $\frac{C \times V \times D}{W}$

Where:
C = sample concentration in extract (ppm)
V = Volume of extract (mL)
D = Dilution Factor

W = Weight/Volume of sample aliquot extracted (grams or mLs)

NOTE: All dry weight corrections are made in LIMS at the time the final report is prepared.

Note: For all methanolic samples with a % Moisture of greater than 10%, it is necessary to adjust the extraction final volume of the sample in order to allow for the miscible solvents effect. This is done by the following equation:

Corrected FV = ((g of samples * % moisture/100) + ml of MeOH) * 40

In these situations, an "other observation" NCM must be generated and the correction and above formula must be noted in the case narrative.

12.9.1 **Calculation of Results for Methanol Extracts**

Sample Conc (ug/Kg) = [On Column (ug/L)] x $\frac{\text{Extraction Final Volume (mL)}}{\text{Amount of Soil Sample (g)}}$ x $\frac{\text{Purge vessel Volume (mL)}}{\text{Amt of MeOH Extract (mL)}}$ x $\frac{\text{1L (CF)}}{\text{1000mL}}$ x $\frac{\text{1000g (CF)}}{\text{1Kg}}$

Purge vessel volume is 10 ml and when the extract doesn't require a dilution, 0.250 mL of the methanol extract is used. So, the equation becomes:

Sample Conc (ug/Kg) = [On Column (ug/L)] x $\frac{\text{Extraction Final Volume (mL)}^{1}}{\text{Amount of Soil Sample (g)}^{2}}$ x $\frac{\text{10 (mL)}}{\text{0.250 (mL)}}$ x $\frac{\text{1L (CF)}}{\text{1000mL}}$ x $\frac{\text{1000g (CF)}}{\text{1Kg}}$

[1]Extract Final Volume, miscible solvent corrected (mL) = ((g of samples * % moisture/100) + ml of MeOH) * 40 (used when % Moisture of the soil sample is greater than 10%).

[2]Amount of Soil, dry-weight corrected (g) = sample mass (g) * (100 - % moisture/100)

12.10 Upon completion of the analytical sequence:

12.10.1 Review chromatograms online and determine whether manual data manipulations are necessary.

12.10.2 Manual Integrations

All manual integrations must be justified and documented. See Corporate SOP CA-Q-S-002 for requirements for manual integration.

12.10.3 Manual integrations may be processed using Chrom, which stores the before and after chromatograms and the reason for the change, and attaches the analyst's electronic signature.

12.10.4 Alternatively, the manual integration may be processed manually. In the latter case, print both the both the before and after chromatograms and record the reason for the change and initial and date the after chromatogram. Before and after chromatograms must be of sufficient scale to allow an independent reviewer to evaluate the manual integration.

12.10.5 Confirm that run logs have printed on them the instrument ID, the analyst and the method used. If this is not printed on the run logs, this must be entered by hand prior to completing the package.

12.11 Compile the raw data for all the samples and QC samples in a batch. The analytical batch is defined as containing no more than 20 field samples.

12.11.1 Perform a level 1 data review and document the review on the data review checklist (GCMS Data Review Checklist).

12.11.2 Submit the data package and review checklist to the peer reviewer for the level 2 review. The data review process is explained in SOP TA-QA-0635.

12.12 Method Performance

12.13 Method Detection Limit Study (MDL)/Detection Limit

The method detection limit (MDL) *or* detection limit (DL) 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/DL is determined according to the laboratory's MDL/DL procedure (see SOP TA-QA-0602). MDL/DLs 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/DL studies for analyses performed; these are verified at least quarterly unless method requirements require a greater frequency.

For instruments that run samples which fall under LaMP regulations, a yearly MDL Study must be performed and MDLV starting at 1X the MDL.

12.14 Limit of Detection

The limit of detection (LOD) is determined for each analyte and matrix by spiking a quality system matrix at approximately two to four times the detection limit. This spike concentration establishes the LOD. The LOD is verified quarterly for each method and matrix on each instrument that analyzes said method/matrix. Refer to the laboratory's LOD procedure (see SOP TA-QA-0602)

12.15 Limit of Quantitation

The limit of Quantitation (LOQ) is verified quarterly for each method and matrix on each instrument that analyzed said method/matrix. Refer to the laboratory's LOQ procedure (see SOP TA-QA-0618)

12.16 Demonstration of Capabilities

Analyst initial Demonstrations of Capability (DOC) are performed after completing a read and understand memo for the SOP and before any client samples are analyzed. DOCs are updated annually (continuing DOC). See SOP TA-QA-0617 for details.

12.17 Training Requirements

See SOP TA-QA-0608 for detailed training requirements.

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

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 Waste Disposal SOP TA-EHS-0036.

14.1 Waste Streams Produced by the Method

14.1.1 VOA vials containing acidic water; VOA vials containing extracted acidic water and small amounts of methanol are collected in large plastic satellite waste bins marked "Hazardous Waste." At or before the waste reaches 55 gallons, the contents are transferred to the waste warehouse where the vials are bulked into a 55 gallon waste barrel and sent out for incineration.

14.1.2 VOA vials containing extracted soil samples, which will contain small amounts of methanol and possibly sodium bisulfate. Unused sample extracts are held for at least 40 days, in case further testing is deemed necessary. After at least 40 days have passed these sample extracts are transported to the waste room were they are bulked into the flammable liquid loose pack barrel and sent out for incineration.

14.1.3 Expired Standards. Expired standards are collected in satellite containers marked "Hazardous Waste." At or before the containers reach 55 gallons the containers are taken to the waste warehouse where they are bulked into an expired standards lab pack and sent out for incineration.

15.0 References / Cross-References

15.1 Method 8260B, Volatile Compounds by Gas Chromatography/Mass Spectrometry (GC/MS), Revision 2, December, 1996, SW-846, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, Third Edition and all promulgated updates, EPA Office of Solid Waste, January 2005.

15.2 Method 8260C, Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS), Revision 3, August, 2006, SW-846, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, Fourth Edition, EPA Office of Solid Waste.

15.3 Method 5035A, Closed-System Purge-and-Trap and Extraction for Volatile Organics in Soil and Waste Samples, Revision 1, July, 2002, SW-846, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, Third Edition and all promulgated updates, EPA Office of Solid Waste, January 2005.

15.4 40CFR, Part 136, Appendix A (Method 624).

16.0 Method Modifications:

Item	Method	Modification
1	8260B	The quantitation and qualifier ions for some compounds have been changed from those recommended in SW-846 in order to improve the reliability of qualitative identification.
1	8260C	Storage conditions for standards are listed as <6C or as recommended by manufacturer. TestAmerica Seattle follows 8260B guidelines to store standards at -10C or as recommended by manufacturer.
2	5035A	The aliquot of methanol extract taken for analysis is 125 uL rather than the 100 uL specified in Table 1 of the method.

17.0 Tables and Appendices

Table 1 TestAmerica Primary List Reporting Limits for 8260B and 8260C
Table 2 8260B and 8260C Additional Analyte List Reporting Limits
Table 3 Appendix IX List Reporting Limits
Table 4 Typical Calibration Levels
Table 5 Internal Standards
Table 6 Surrogate Standards
Table 7 Short List LCS and Matrix Spike Standard
Table 7a Full List LCS and Matrix Spike Standard
Table 8 BFB Key Ion Abundance Criteria
Table 9 8260B SPCC Compounds and Minimum Response Factors
Table 10 8260B CCC Compounds
Table 11 Poorly Performing Compounds
Table 12 CCV % Drift Criteria for Non-DOD Projects
Table 13 Summary of QC Requirements
Table 14 8260C Minimum Relative Response Factor Criteria
Appendix A Modifications for Method 624
Table A-1 Method 624 Analytes and Reporting Limits, 10-mL Purge
Attachment I Process Flow for Seattle VOA

18.0 Changes from last revision

- Revision 23, dated 5 January 2014
  - Section 2.3, updated purge gas.
  - Section 6.2, added computer hardware
  - Section 8.3, updated to clearly define different soil sample techniques
  - Section 8.5, updated container
  - Secton 8.6.2, updated container and volume of MeOH

- Section 8.8, added section for unpreserved soils
- Section 8.9.2, added VOA vials for low-level soils.
- Section 9.3, added clarification for DoD and LaMP program requirements
- Sections 9.4 and 9.5, added program requirements
- Section 11.3, updated BFB criteria for mass 174
- Section 11.4 updated to current practice
- Section 11.4.4, added reference to corporate SOP on calibrations
- Sections 11.4.8, 11.4.9, 11.4.11 and 11.4.12, added the criteria for 8260C
- Section 11.4.15, added clarification for DoD and LaMP program requirements
- Sections 11.5.1 and 11.5.2, added DoD QSM 5.0 requirement for closing CCV
- Section 11.5.5, added the criteria for 8260C
- Section 11.5.7, added DoD QSM 5.0 requirement for closing CCV
- Sections 11.5.8 and 11.5.9, added clarification for DoD and LaMP program requirements
- Sections 11.6.1.1, 11.6.2.1, 11.6.3.1 and 11.6.3.3, added more detail for the procedure.
- Sections 11.10, 11.12 and 11.13, updated to new procedures
- Section 11.14.1, added the criteria for 8260C
- Section 11.15, updated to new procedures
- Section 11.17, added section for troubleshooting
- Updated and added Tables and Attachments

- Revision 22, dated 13 March 2013
  - Added section 11.7 to describe the bulk soil preparation procedures and documentation.
  - Added section 15.1 Waste Streams Produced by the Method.

- Revision 21, dated 12 October 2012.
  - Added an elaborated calculation for methanolic extracts 12.10.1.

- Revision 20, dated 20 June 2011
  - Incorporated ROMDs 00019 and 00026 in sections 6.1 and 11.16.6.
  - Added “Note” in section 4.4 addressing cross-contamination from laboratory spiked samples.
  - Addressed shelf life of gas standards in section 7.3.2.
  - Add instructions to return standards to freezer when daily prep is completed, section 7.4
  - Added hold time NCM for unpreserved aromatic compounds in Section 8.2.
  - Added clarification in section 8.5.2 that TCLP samples require a MS/MSD.
  - Added hold time for unpreserved aromatic compounds in Section 8.13.
  - Added clarification on spike solutions in sections 9.5 and 9.6.
  - Incorporated ROMD 00025 in section 9.6.
  - Incorporated ROMD 00020 in section 11.2.2.
  - Added optimization objectives for the GC run programs in section 11.2.2.
  - Incorporated ROMD 00022 in section 11.5.12.
  - Incorporated ROMD 00033 in section 11.6.3
  - Incorporated ROMD 00024 in section 11.6.8.
  - Updated corrective actions for CCV failure in section 11.6.8.
  - Defined CCV % Drift Criteria for non-DOD projects in section 11.6.6 and Table 12
  - Added section 11.11 for TCLP sample preparation/analysis.
  - Added pH check procedures for water samples in section 11.10.9.
  - Revised IS acceptance criteria in section 11.14.2.
  - Expanded list of compounds in Table 7 for compliance with NELAC.

- Revision 19, dated 16 September 2010
  - Updated corrective actions for ICV and CCV failures, sections 11.5 and 11.6.
  - Left italicized text from revision 18 since this revision occurred just days after.

- Revision 18, dated 13 September 2010
  - Added more detail to tuning procedures, section 11.
  - Updated corrective actions for ICV and CCV failures, sections 11.5 and 11.6.
  - Added qualitative identification requirements for SIM analysis, section 12.2

- Revision 17, dated 16 April 2010
  - Removed Chlorobenzen-D5 from Table 5. Added Pentafluorobenzene and 1,4-Dichlorobenzene and updated quantitation ion.

- Added Sections on 8260_SIM. Including MS parameters and recommended ICAL and spike ranges. Updates Scope section 1.2.1. Updated Calibration 11.4.2. Added section 11.2.3.1 SIM parameters
- Added a range of spiking amounts and added 10mL purge spiking amounts to sections 11.9
- Added documentation of standards/reagents and standard/reagent preparation Section 7.1
- Added clarifications about standard storage conditions, section 7.3.1
- Added removal of expired standards Section 7.4.
- Added instructions for the analysis of storage blanks, section 8.11
- Added clarification about re-analyzing batch QC and trip blanks, section 9.7
- Added criteria for additional QC, Section 9.8.
- Added daily balance check to Section 6.2.
- Added LaMP client surrogate criteria, Section 9.4.
- Added requirement for running 624 and 8260 methods on separate sequences Section 10.1.
- Added measurement of gas flow rate, Section11.14.3.4.
- Added maintenance logbook documentation requirements, section 11.14.5
- Added specifications (MDLs) and clarifications (DOCs), section 13
- Updated appendix A to include purge and desorb requirements for method 624.
- Integration for TestAmerica Seattle and TestAmerica Tacoma operations.

- Revision 16, dated 16 September 2009
  - Description of preparation requirements for reagent water have been updated in sections 3.3 and 7.1.2 to reflect current practice of by purging with an inert gas for a minimum of 1 hour prior to use rather than for a period of overnight.

- Revision 15, dated 22 July 2009
  - Method modifications section updated to identify volume of methanol extract used for analysis.
  - References section updated to include Method 5035A.
  - Nomenclature for spiking solutions has been updated.
  - Spiking volumes have been updated.
  - Calibration levels have been updated.
  - Corrected typographical errors.
  - Updated RLs in Tables 1, 2, 3, and A-1.
  - Added Table 12. Summary of QC Requirements

- Revision 14, dated 16 March 2009
  - Method modifications section updated to identify volume of methanol extract used for analysis.
  - References section updated to include Method 5035A.
  - Nomenclature for spiking solutions has been updated.
  - Spiking volumes have been updated.
  - Calibration levels have been updated.
  - Corrected typographical errors.
  - Updated RLs in Tables 1, 2, 3, and A-1.

- Revision 13, dated 22 March 2008
  - Integration for TestAmerica and STL operations.
  - This revision is a complete rewrite and an expansion of scope.
  - This SOP is the combination of SOPs 0312.12, 0327.5, and 0381.6.

Table 1

TestAmerica Primary List Reporting Limits for 8260B and 8260C[2]

Compound	CAS Number	Reporting Limits[1]			
		Standard Level Water (µg/L)	Low Level Water (µg/L)	Low Soil (µg/kg)	Med Soil (µg/kg)
Dichlorodifluoromethane	75-71-8	1.0	0.4	1.0	40
Chloromethane	74-87-3	*1.0*	0.1	1.0	*200*
Bromomethane	74-83-9	5.0	0.1	*5.0*	140
Vinyl chloride	75-01-4	*0.2*	0.02	*0.2*	8
Chloroethane	75-00-3	5.0	0.2	*5.0*	*200*
Trichlorofluoromethane	75-69-4	1.0	0.1	1.0	40
Acetone	67-64-1	*20*	2.0	*20*	*800*
Carbon disulfide	75-15-0	1.0	0.1	1.0	40
Methylene chloride	75-09-2	3.0	0.1	*3.0*	*16*
1,1-Dichloroethene	75-35-4	1.0	0.1	*1.0*	*16*
1,1-Dichloroethane	75-34-3	1.0	0.1	1.0	40
trans-1,2-Dichloroethene	156-60-5	1.0	0.1	1.0	40
Methyl tert-butyl ether (MTBE)	1634-04-4	1.0	0.1	1.0	40
cis-1,2-Dichloroethene	156-59-2	1.0	0.1	1.0	40
Chloroform	67-66-3	1.0	0.1	1.0	40
1,2-Dichloroethane	107-06-2	1.0	0.1	1.0	*16*
Dibromomethane	74-95-3	*2.0*	0.1	*2.0*	*80*
2-Butanone (MEK)	78-93-3	10.0	0.5	*10.0*	400
1,1,1-Trichloroethane	71-55-6	1.0	0.1	1.0	40
Carbon tetrachloride	56-23-5	*0.2*	0.1	*0.2*	*16*
Bromodichloromethane	75-27-4	1.0	0.1	1.0	40
1,2-Dichloropropane	78-87-5	1.0	0.1	1.0	*16*
cis-1,3-Dichloropropene	10061-01-5	1.0	0.1	1.0	*40*
Trichloroethene	79-01-6	1.0	0.1	1.0	*20*
Dibromochloromethane	124-48-1	1.0	0.1	1.0	*16*
1,2-Dibromoethane (EDB)	106-93-4	*0.4*	0.1	*0.4*	*16*
1,2,3-Trichloropropane	96-18-4	*2.0*	0.2	*2.0*	*80*
1,1,2-Trichloroethane	79-00-5	1.0	0.1	1.0	*16*
Benzene	71-43-2	1.0	0.1	1.0	16
trans-1,3-Dichloropropene	10061-02-6	1.0	0.1	1.0	*40*
Bromoform	75-25-2	1.0	0.1	1.0	40
Compound	**CAS Number**	**Reporting Limits[1]**			
		Standard Level Water (µg/L)	**Low Level Water (µg/L)**	**Low Soil (µg/kg)**	**Med Soil (µg/kg)**
4-Methyl-2-pentanone (MIBK)	108-10-1	5.0	0.5	5.0	200
2-Hexanone	591-78-6	5.0	1.0	5.0	200
Tetrachloroethene	127-18-4	*0.4*	0.1	*0.4*	*16*

Table 1

TestAmerica Primary List Reporting Limits for 8260B and 8260C[2]

Toluene	108-88-3	1.0	0.1	*1.0*	40
1,1,2,2-Tetrachloroethane	79-34-5	1.0	0.1	*1.0*	*16*
Chlorobenzene	108-90-7	1.0	0.1	1.0	40
Ethylbenzene	100-41-4	1.0	0.1	1.0	40
Styrene	100-42-5	1.0	0.1	1.0	40
m- and p-Xylenes	136777-61-2	2.0	0.2	2.0	40
o-xylene	95-47-6	1.0	0.1	1.0	40
1,3-Dichlorobenzene	541-73-1	1.0	0.2	1.0	40
1,4-Dichlorobenzene	106-46-7	1.0	0.2	1.0	40
1,2-Dichlorobenzene	95-50-1	1.0	0.2	1.0	40
2,2-Dichloropropane	590-20-7	*2.0*	0.1	*2.0*	*80*
Bromochloromethane	74-97-5	1.0	0.1	1.0	40
1,1-Dichloropropene	563-58-6	*2.0*	0.1	*2.0*	*80*
1,3-Dichloropropane	142-28-9	1.0	0.1	1.0	40
Bromobenzene	108-86-1	1.0	0.1	*1.0*	40
n-Propylbenzene	103-65-1	1.0	0.1	1.0	40
2-Chlorotoluene	95-49-8	1.0	0.1	*1.0*	40
4-Chlorotoluene	106-43-4	1.0	0.2	*1.0*	40
1,3,5-Trimethylbenzene	108-67-8	1.0	0.1	*1.0*	40
tert-Butylbenzene	98-06-6	1.0	0.1	*1.0*	40
1,2,4-Trimethylbenzene	95-63-6	1.0	0.1	*1.0*	40
sec-butylbenzene	135-98-8	1.0	0.1	*1.0*	40
4-Isopropyltoluene	99-87-6	1.0	0.2	*1.0*	40
n-Butylbenzene	104-51-8	1.0	0.1	*1.0*	40
1,2,4-Trichlorobenzene	120-82-1	1.0	0.2	*1.0*	40
Naphthalene	91-20-3	1.0	0.4	*1.0*	40
Hexachlorobutadiene	87-68-3	1.0	0.2	1.0	40
1,2,3-Trichlorobenzene	87-61-6	*2.0*	0.4	2.0	*80*
Compound	**CAS Number**	**Reporting Limits[1]**			
		Standard Level Water (µg/L)	**Low Level Water (µg/L)**	**Low Soil (µg/kg)**	**Med Soil (µg/kg)**
1,1,1,2-Tetrachloroethane	630-20-6	1.0	0.1	1.0	40
1,2-Dibromo-3-chloropropane (DBCP)	96-12-8	2.0	0.2	2.0	200
Isopropylbenzene	98-82-8	1.0	0.1	*1.0*	40

1 Reporting limits listed for soil/sediment are based on wet weight. The reporting limits calculated by the laboratory for soil/sediment, calculated on dry weight basis, will be higher.

Table 2

8260B and 8260C Additional Analyte List Reporting Limits

Compound	CAS Number	Reporting Limits[1]			
		Standard Level Water µg/L	Low Level Water µg/L	Low Soil µg/kg	High Soil µg/kg
Acrolein	107-02-8	5.0	2.0	5.0	200
1,1,2-Trichloro-1,2,2-trifluoroethane	76-13-1	1.0	0.2	1.0	40
Iodomethane	74-88-4	5.0	0.5	*5.0*	200
2-Chloroethyl vinyl ether[2]	110-75-8	10[2]	2.0[2]	10.0	200
Vinyl acetate	108-05-4	*10*	0.5	*10*	*400*
Methyl acetate	79-20-9	*10*	--	*10*	*400*
2-Methyl-2-propanol	75-65-0	25.0	10.0	*25.0*	*800*
tert-Butyl Ethyl Ether	637-92-3	1.0	0.5	*1.0*	40
trans-1,4-Dichloro-2-butene	110-57-6	6.0	1.0	*6.0*	200
Total Xylenes	1330-20-7	*3.0*	0.1	*3.0*	*80*
Acrylonitrile	107-13-1	*2.0*	2.0	*2.0*	*400*
Hexane	110-54-3	1.0	--	*1.0*	40
Tetrahydrofuran	109-99-9	*10*	--	*10*	*400*
Cyclohexane	110-82-7	1.0	--	*1.0*	40
tert-Amyl Methyl Ether	994-05-8	*10*	0.5	10	*400*
Methylcyclohexane	108-87-2	1.0	--	1.0	40
cis-1,4-Dichloro-2-butene	1476-11-5	5.0	--	5.0	200
Hexachloroethane	67-72-1	1.0	0.1	1.0	40
2-Ethyl-1-hexanol	104-76-7	50	--	75	1000
1,3,5-Trichlorobenzene	108-70-3	1.0	0.2	*1.0*	40

1 Reporting limits listed for soil/sediment are based on wet weight. The reporting limits calculated by the laboratory for soil/sediment, calculated on dry weight basis, will be higher.
2 2-Chloroethyl vinyl ether cannot be reliably recovered from acid preserved samples

Table 3

Appendix IX List Reporting Limits

Compound	CAS Number	Reporting Limits[1]			
		Standard Level Water µg/L	Low Level Water µg/L	Low Soil µg/kg	High Soil µg/kg
Acetonitrile	75-05-8	*2.0*	--	*2.0*	400
Isopropyl ether	108-20-3	1.0	--	*1.0*	40
n-Butanol	71-36-3	100	--	100	4000
Methacrylonitrile	126-98-7	5.0	--	5.0	200
Isobutanol	78-83-1	100	--	100	4000
Ethyl ether	60-29-7	5.0	--	5.0	200
Ethyl Acetate	141-78-6	10	--	*10*	200

[1] Reporting limits listed for soil/sediment are based on wet weight. The reporting limits calculated by the laboratory for soil/sediment, calculated on dry weight basis, will be higher.

Table 4

Typical Calibration Levels

Standard level water, Low-level Soil and Methanol preserved soil purge	**Calibration Levels, µg/L**											
	0.2	**0.4**	**1.0**	**2.0**	**5.0**	**10**	**20**	**50**	**100**	**150**		
Low level water purge	**Calibration Levels, µg/L**											
	0.02	**0.04**	**0.10**	**0.20**	**0.40**	**1.0**	**2.0**	**5.0**	**10.0**	**20.0**	**40.0**	**100**

Table 5

Internal Standards

Internal Standard	Standard Concentration (mg/L)	Quantitation Ion
1,4-Dichlorobenzene-d4	250	152
Chlorobenzene-d5	250	117
Fluorobenzene	250	96
Dioxane-d8	5000	96
Tert-butanol-d9	5000	65

Table 6

Surrogate Standards

Surrogate Compounds	Standard Concentration (mg/L)
1,2-Dichloroethane-d4	150
4-Bromofluorobenzene	150
Dibromofluoromethane	150
Toluene-d8	150
Trifluorotoluene	150

NOTES:

1) Recovery and precision limits for the surrogates are generated from historical data and are maintained by the QA department.

Table 7

Full List LCS and Matrix Spike Compounds

Compound	Standard Concentration (mg/L)
Acrolein	250
Vinyl acetate	250
2-Chloroethyl vinyl ether	250
1,4-Dioxane	1250
2-Butanone (MEK)	250
2-Ethyl-1-hexanol	1250
2-Hexanone	250
2-Methyl-2-propanol	250
4-Methyl-2-pentanone (MIBK)	250
Acetone	250
Acetonitrile	500
Acrylonitrile	250
Bromomethane	250
Chloroethane	50
Chloromethane	50
Cis-1,4-Dichloro-2-butene	50
Dichlorodifluoromethane	50
Ethyl acetate	250
Ethyl ether	250
Iodomethane	250
Isopropyl ether	50
Methacrylonitrile	250
Methyl acetate	250
Methyl tert-butyl ether	50
Tert-amyl methyl ether	50
Tert-butyl ethyl ether	50
Tetrahydrofuran	250
Trans-1,4-Dichloro-2-butene	250
Trichlorfluoromethane	50
Vinyl chloride	50
1,1,1,2-Tetrachloroethane	50
1,1,1-Trichloroethane	50

Compound	Standard Concentration (mg/L)
1,1,2,2-Tetrachloroethane	50
1,1,2-Trichloro-1,2,2-trifluoroethane	50
1,1,2-Trichloroethane	50
1,1-Dichloroethane	50
1,1-Dichloroethene	50
1,1-Dichloropropene	50
1,2,3-Trichloropropane	50
1,2,4-Trichlorobenzene	50
1,2,4-Trimethylbenzene	50
1,2-Dibromo-3-chloropropane	50
1,2-Dichlorobenzene	50
1,2-Dichloroethane	50
1,2-Dichloropropane	50
1,3,5-Trichlorobenzene	50
1,3,5-Trimethylbenzene	50
1,3-Dichlorobenzene	50
1,3-Dichloropropane	50
1,4-Dichlorobenzene	50
2,2-Dichloropropane	50
2-Chlorotoluene	50
4-Chlorotoluene	50
4-Isopropyltoluene	50
Benzene	50
Bromobenzene	50
Bromoform	50
Carbon disulfide	50
Carbon tetrachloride	50
Chlorobenzene	50
Chlorobromomethane	50
Chlorodibromomethane	50
Chloroform	50
Cis-1,2-Dichloroethene	50
Cis-1,3-Dichloropropene	50
Cyclohexane	50

Compound	Standard Concentration (mg/L)
Dibromomethane	50
Dichlorobromomethane	50
Ethylbenzene	50
Ethylene Dibromide	50
Hexachlorobutadiene	50
Hexachloroethane	50
Hexane	50
Isobutyl alcohol	5000
Isopropylbenzene	50
Methylcyclohexane	50
Methylene chloride	50
m- & p-Xylene	100
Naphthalene	50
n-Butanol	5000
n-Butylbenzene	50
n-Propylbenzene	50
o-Xylene	50
sec-Butylbenzene	50
Styrene	50
tert-Butylbenzene	50
Tetrachloroethene	50
Toluene	50
trans-1,2-Dichloroethene	50
trans-1,3-Dichloropropene	50
Trichloroethene	50

NOTES:

1) Recovery and precision limits for the LCS, MS, and MSD are generated from historical data and are maintained by the QA department.

Table 8

BFB Key Ion Abundance Criteria

Mass	Ion Abundance Criteria
50	15 to 40 % of Mass 95
75	30 to 60 % of Mass 95
95	Base Peak, 100 % Relative Abundance
96	5 to 9 % of Mass 95
173	Less than 2 % of Mass 174
174	50 to 120 % of Mass 95
175	5 to 9 % of Mass 174
176	Greater than 95 %, but less than 101 % of Mass 174
177	5 to 9 % of Mass 176

Table 9

8260B SPCC Compounds and Minimum Response Factors

Compound	8260B Min. RF
Chloromethane	0.10
1,1-Dichloroethane	0.10
Bromoform	0.10
1,1,2,2-Tetrachloroethane	0.30
Chlorobenzene	0.30

Table 10
8260B CCC Compounds

Compound	Max. %RSD from Initial Calibration	Max. %D for continuing calibration
Vinyl Chloride	≤ 30	≤ 20
1,1-Dichloroethene	≤ 30	≤ 20
Chloroform	≤ 30	≤ 20
1,2-Dichloropropane	≤ 30	≤ 20
Toluene	≤ 30	≤ 20
Ethylbenzene	≤ 30	≤ 20

Table 11

Poorly Performing Compounds

Acetone	Isopropyl alcohol
Acetonitrile	Dichlorofluoromethane
Acrolein	**1,2-Dibromo-3-chloropropane (DBCP)**
Acrylonitrile	1,4-Dioxane
n-Butanol	Ethyl acetate
2-Butanone (MEK)	**2-Hexanone**
Bromomethane	Methacrylonitrile
Bromoform	Methyl acetate
Carbon disulfide	Methyl methacrylate
2-Chloroethyl vinyl ether	**4-Methyl-2-pentanone (MIBK)**
Chloroethane	**Naphthalene**
Chloromethane	2-Nitropropane
Dichlorodifluoromethane	Proprionitrile
cis-1,4-Dichloro-2-butene	Tetrahydrofuran
trans-1,4-Dichloro-2-butene	1,1,2-Trichloro-1,2,2-trifluoroethane
Ethanol	**1,2,3-Trichlorobenzene**
Ethyl methacrylate	**Trichlorofluoromethane**
Iodomethane	Vinyl acetate
Isobutyl alcohol	Tert butyl alcohol

The laboratory's GC/MS group identified this list of compounds based on current and historical performance. The recovery performance was reviewed against full spike recovery data and method performance data, where available, to validate each compound as a "poor performer." This is not a comprehensive list and is subject to change. Each DoD projects' target analyte list should be evaluated for poor performers.

Analytes that are in bold are also represented in Table 1 Reporting Limits for Standard Analytes.

.

Table 12

8260B CCV % Drift Criteria for Non-DOD Projects

Analyte	Cas No	CCV %D
1,1,1,2-Tetrachloroethane	630-20-6	30
1,1,1-Trichloroethane	71-55-6	30
1,1,2,2-Tetrachloroethane	79-34-5	30
1,1,2-Trichloro-1,2,2-trifluoroethane	76-13-1	50
1,1,2-Trichloroethane	79-00-5	30
1,1-Dichloroethane	75-34-3	30
1,1-Dichloroethene	75-35-4	20
1,1-Dichloropropene	563-58-6	30
1,2,3-Trichlorobenzene	87-61-6	40
1,2,3-Trichloropropane	96-18-4	30
1,2,4-Trichlorobenzene	120-82-1	40
1,2,4-Trimethylbenzene	95-63-6	30
1,2-Dibromo-3-Chloropropane	96-12-8	50
1,2-Dichlorobenzene	95-50-1	30
1,2-Dichloroethane	107-06-2	30
1,2-Dichloropropane	78-87-5	20
1,3,5-Trichlorobenzene	108-70-3	30
1,3,5-Trimethylbenzene	108-67-8	30
1,3-Dichlorobenzene	541-73-1	30
1,3-Dichloropropane	142-28-9	30
1,4-Dichlorobenzene	106-46-7	30
2,2-Dichloropropane	594-20-7	40
2-Butanone (MEK)	78-93-3	50
2-Chloroethyl vinyl ether	110-75-8	50
2-Chlorotoluene	95-49-8	30
2-Ethyl-1-Hexanol	104-76-7	50
2-Hexanone	591-78-6	50
2-Methyl-2-propanol	75-65-0	50
4-Chlorotoluene	106-43-4	30
4-Isopropyltoluene	99-87-6	30
4-Methyl-2-pentanone (MIBK)	108-10-1	50
Acetone	67-64-1	50
Acetonitrile	75-05-8	50
Acrolein	107-02-8	50
Acrylonitrile	107-13-1	50
Benzene	71-43-2	30
Bromobenzene	108-86-1	30
Bromoform	75-25-2	40
Bromomethane	74-83-9	50
Carbon disulfide	75-15-0	50
Carbon tetrachloride	56-23-5	30
Chlorobenzene	108-90-7	30

Analyte	Cas No	CCV %D
Chlorobromomethane	74-97-5	40
Chlorodibromomethane	124-48-1	40
Chloroethane	75-00-3	50
Chloroform	67-66-3	20
Chloromethane	74-87-3	50
cis-1,2-Dichloroethene	156-59-2	30
cis-1,3-Dichloropropene	10061-01-5	30
cis-1,4-Dichloro-2-butene	1476-11-5	50
Cyclohexane	110-82-7	30
Dibromomethane	74-95-3	30
Dichlorobromomethane	75-27-4	30
Dichlorodifluoromethane	75-71-8	50
Ethyl acetate	141-78-6	50
Ethyl ether	60-29-7	40
Ethylbenzene	100-41-4	20
Ethylene Dibromide	106-93-4	30
Hexachlorobutadiene	87-68-3	40
Hexachloroethane	67-72-1	40
Hexane	110-54-3	40
Iodomethane	74-88-4	50
Isobutyl alcohol	78-83-1	50
Isopropyl ether	108-20-3	30
Isopropylbenzene	98-82-8	30
Methacrylonitrile	126-98-7	50
Methyl acetate	79-20-9	50
Methyl tert-butyl ether	1634-04-4	30
Methylcyclohexane	108-87-2	30
Methylene Chloride	75-09-2	40
m-Xylene & p-Xylene	179601-23-1	30
Naphthalene	91-20-3	40
n-Butanol	71-36-3	50
n-Butylbenzene	104-51-8	30
N-Propylbenzene	103-65-1	30
o-Xylene	95-47-6	30
sec-Butylbenzene	135-98-8	30
Styrene	100-42-5	30
Tert-amyl methyl ether	994-05-8	40
Tert-butyl ethyl ether	637-92-3	30
tert-Butylbenzene	98-06-6	30
Tetrachloroethene	127-18-4	40
Tetrahydrofuran	109-99-9	50
Toluene	108-88-3	20
trans-1,2-Dichloroethene	156-60-5	30
trans-1,3-Dichloropropene	10061-02-6	30
trans-1,4-Dichloro-2-butene	110-57-6	50
Trichloroethene	79-01-6	30
Trichlorofluoromethane	75-69-4	50

Analyte	Cas No	CCV %D
Vinyl acetate	108-05-4	50
Vinyl chloride	75-01-4	20
Xylenes, Total	1330-20-7	

Table 13

Summary of QC Requirements

QC Parameter	Frequency	Acceptance Criteria	Corrective Action
BFB Tune	Prior to ICAL and at the beginning of each 12-hour period.	See Section 11.3	Retune instrument and verify. Rerun affected samples.
Minimum 5-point Initial Calibration, 6-point for quadratic curves (minimum 3-point Initial Calibration for Method 624)	Initial calibration prior to sample analysis	For Method 624: RSD for RFs: ≤ 35% for all analytes. For Method 8260B: 1. Average Response Factor for SPCCs: ≥ 0.30 for chlorobenzene, and 1,1,2,2-tetrachloroethane; ≥ 0.10 for chloromethane, bromoform, and 1,1-dichloroethane 2. RSD for RFs for CCCs: ≤ 30% For Method 8260C: 1. Average Response Factor for specified compounds: See table 14 2. RSD for all compounds: ≤ 20% **<u>For DOD requirements above and one option below</u>:** <u>Option 1</u>: RSD for each analyte ≤ 15% <u>Option 2</u>: Linear regression r ≥ 0.995 <u>Option 3</u>: Non linear regression r^2 ≥ 0.990 and 6 points must be used.	Terminate analysis; correct the problem; recalibrate. Problem must be corrected. No samples may be run until ICAL has passed.
ICV or QCS	Following initial calibration.	Method control limits of all analytes under method 624 8260B: 60-140% for non-DoD projects and 45-155% for poor performers 8260C: 70-130% for non-	Terminate analysis; correct the problem; recalibrate.

QC Parameter	Frequency	Acceptance Criteria	Corrective Action
		DoD projects and 45-155% for poor performers **For DoD:** 80 - 120% recovery and 70-130% for poor performers with prior written approval	
Relative Retention Times (RRT)	With each sample	RRT of each target analyte within ± 0.06 RRT units.	Correct problem, then rerun ICAL. Laboratory may update RTs based on the CCV to account for minor performance fluctuations or after routine system maintenance (e.g. column clipping).
CCV	Daily before sample analysis and every 12 hours of analysis time.	8260B: 1. Avg RF for SPCCs: ≥ 0.30 for chlorobenzene and 1,1,2,2-tetrachloroethene; ≥ 0.10 for chloromethane, bromoform, and 1,1-dichloroethane; 2. %D/Drift for CCCs ≤ 20%D. 3. %D/Drift for nonCCCs ≤ limits in Table 12. 8260C: 1. Avg RF for specified compounds: See table 14 2. %D/Drift for 80% of compounds ≤ 20%D. **For DoD:** 1. Avg RF: see method 2. Opening CCV: %D/Drift for all target compounds and surrogates ≤ 20%D. 3. Closing CCV: %D/Drift for all target compounds and surrogates ≤ 50%D.	Correct problem, then rerun CCV. If that fails, then repeat ICAL. Reanalyze all sample since the last successful CCV.
Internal Standards (IS) verification	Every field sample, standard, and QC sample	Retention time ± 30 seconds from RT of the midpoint standard in ICAL; EICP area within -50% to +100% of ICAL	Inspect mass spectrometer and GC for malfunctions. Reanalysis of samples while system was malfunctioning is mandatory.

QC Parameter	Frequency	Acceptance Criteria	Corrective Action
		midpoint standard. **For DoD:** Retention time ± 10 seconds from RT of the midpoint standard in ICAL; EICP area within -50% to +100% of ICAL midpoint standard.	
Method Blank	One per batch of 20 field samples or fewer.	The result must be < RL or < *5%* the amount measured in any sample or 1/10 the regulatory limit. **For DoD:** No analytes detected > ½ RL and > 10% the amount measured in any sample or 1/10 the regulatory limit. For common laboratory contaminants no analytes detected > RL.	Re-extract and reanalyze samples. Note exceptions under criteria section. See Section 9.3 for additional requirements.
LCS	One per batch of 20 field samples or fewer.	Must be within laboratory control limits. **For DoD:** Must contain all analytes to be reported. QC acceptance criteria specified by DoD, if available. Otherwise, use in-house control limits.	See Section 9.5 for additional requirements.
Surrogate	All field and QC samples.	Must be within laboratory control limits, unless it fails high and the sample is ND, or matrix interference is confirmed by a reanalysis or MS/MSD performed on the sample, or client specific requirements exist. **For DoD:** QC acceptance criteria specified by DoD, if available. Otherwise, use in-house control limits.	See Section 9.4 for additional requirements.
Matrix Spike/Laboratory Fortified Matrix	One per lot of 20 field samples or fewer.	Must be within laboratory control limits. **For DoD:** Must contain all analytes to be reported and must use LCS control limits. MD/MSD RPD ≤20%	See Section 9.5 for additional requirements.

Document Uncontrolled When Printed

Table 14

8260C Minimum Relative Response Factor Criteria for Initial and Continuing Calibration Verification

Volatile Compounds	Minimum Response Factor (RF)a	Typical Response Factor (RF)b
Dichlorodifluoromethane	0.100	0.327
Chloromethane	0.100	0.537
Vinyl chloride	0.100	0.451
Bromomethane	0.100	0.255
Chloroethane	0.100	0.254
Trichlorofluoromethane	0.100	0.426
1,1-Dichloroethene	0.100	0.313
1,1,2-Trichloro-1,2,2-trifluoroethane	0.100	0.302
Acetone	0.100	0.151
Carbon disulfide	0.100	1.163
Methyl Acetate	0.100	0.302
Methylene chloride	0.100	0.380
trans-1,2-Dichloroethene	0.100	0.351
cis-1,2-Dichloroethene	0.100	0.376
Methyl tert-Butyl Ether	0.100	0.847
1,1-Dichloroethane	0.200	0.655
2-Butanone	0.100	0.216
Chloroform	0.200	0.557
1,1,1-Trichloroethane	0.100	0.442
Cyclohexane	0.100	0.579
Carbon tetrachloride	0.100	0.353
Benzene	0.500	1.368
1,2-Dichloroethane	0.100	0.443
Trichloroethene	0.200	0.338
Methylcyclohexane	0.100	0.501
1,2-Dichloropropane	0.100	0.382
Bromodichloromethane	0.200	0.424
cis-1,3-Dichloropropene	0.200	0.537
trans-1,3-Dichloropropene	0.100	0.515
4-Methyl-2-pentanone	0.100	0.363
Toluene	0.400	1.577
1,1,2-Trichloroethane	0.100	0.518
Tetrachloroethene	0.200	0.606
2-Hexanone	0.100	0.536
Dibromochloromethane	0.100	0.652
1,2-Dibromoethane	0.100	0.634
Chlorobenzene	0.500	1.733
Ethylbenzene	0.100	2.827
meta-/para-Xylene	0.100	1.080
Volatile Compounds	Minimum Response Factor (RF)a	Typical Response Factor (RF)b
ortho-Xylene	0.300	1.073
Styrene	0.300	1.916
Bromoform	0.100	0.413
Isopropylbenzene	0.100	2.271
1,1,2,2-Tetrachloroethane	0.300	0.782
1,3-Dichlorobenzene	0.600	1.408
1,4-Dichlorobenzene	0.500	1.427
1,2-Dichlorobenzene	0.400	1.332

Document Uncontrolled When Printed

1,2-Dibromo-3-chloropropane	0.050	0.129
1,2,4-Trichlorobenzene	0.200	0.806

a The project-specific response factors obtained may be affected by the quantitation ion selected and when using possible alternate ions the actual response factors may be lower than those listed. In addition, lower than the recommended minimum response factors may be acceptable for those compounds that are not considered critical target analytes and the associated data may be used for screening purposes.

b Data provided by EPA Region III laboratory.

Company Confidential & Proprietary

Appendix A - Modifications for Method 624

Requirements for EPA 624

1. Method 624 is required for demonstration of compliance with CWA permits, e.g., NPDES wastewater discharge permits. This method can be applied only to aqueous matrices. The standard analyte list and reporting limits are listed in Table A-1. If compounds are added to the analysis, all of the method criteria must be satisfied for the additional compounds.
2. The tune period for this method is defined as 24 hours, which is the maximum elapsed time before the tune check is performed. Calibration verifications are done at the same 24 hour frequency.
3. The initial calibration curve for this method requires at least three points.
4. Sample concentrations are calculated using the average RRF from the initial calibration curve.
5. CCC evaluation is consistent with the published 624 CCC recovery criteria. Refer to method 624 for ranges.
6. Each target analyte is assigned to the closest eluting internal standard.
7. Initial demonstration of Proficiency
   - The spiking level for the four replicate initial demonstration of proficiency is 20 µg/L.
8. Initial calibration curve requirements:
   - Target compounds must have RSD ≤ 35%.
   - If this requirement can not be met, a regression curve must be constructed for the non-compliant compounds. There is no correlation coefficient requirement for the regression curve.
9. Continuing calibration verification requirements:
   - The laboratory control standard is from a different source than the initial calibration standard. The daily CCAL concentration is 50 ug/L. The LCS concentration is 20 ug/L.
10. Matrix Spike and LCS Requirements
    - The matrix spike and LCS/LCSD are spiked at 20 µg/L, prepared from the same source containing all analytes of interest. A matrix spike duplicate is not necessary for this method.
11. Consistent with the other volatile methods, corrections for recovery are not allowed.
12. Qualitative Identification - The relative intensities of ions should agree to within ±20% between the standard and sample spectra. (Example: For an ion with an abundance of 50% in the standard spectra, the corresponding sample abundance must be between 30 and 70 percent.)
13. Method clarifications, modifications and additions
    - Section 5.2.2 of the source method describes the trap packing materials as Tenax GC, Methyl silicone, silica gel and coconut charcoal. TestAmerica routinely employs the Supelco VOCARB 3000, which consists of Carbopack B and Carboxen 1000 and 1001.
    - Section 5.3.2 of the source method describes a packed analytical column. TestAmerica routinely employs capillary columns when performing this method.
    - The source method provides a suggested list of compounds for internal and surrogate standards. Others are permitted by the method. TestAmerica uses three internal standards, including 1,4-dichlorobenzene-d4, which are not listed in Table 3 of the source method. Toluene-d8 is used as a surrogate compound, which is also not listed in the source method.
    - The lab is preparing internal standards at 10 ug/L and applying the same criteria designed for 30 ug/L in the Method. The lower the concentration is consistent with the greater sensitivity provided by capillary columns as compared to the older packed columns described in the method. It could only be more challenging for the lab to meet the acceptance criteria at 10 ug/L; it provides a higher level of data quality.
    - Method 624 describes a mass scan range of 25 to 260 amu. Table 13 lists all of the ions used for analysis. None of the ions are below 35 amu. Therefore, we scan from 35 to 300 and include all ions needed for analysis.

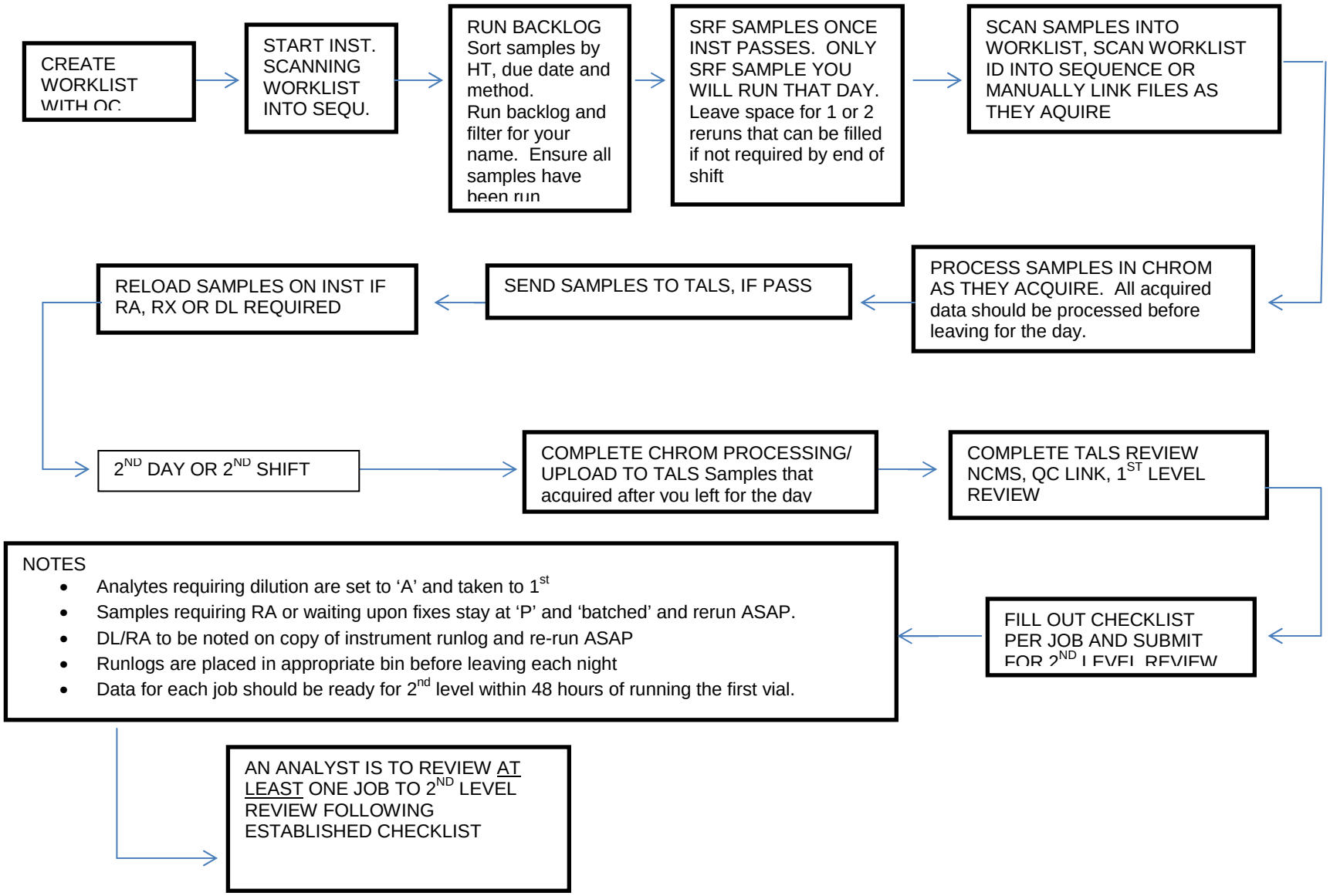
- Method 624 describes dilutions “if response of any m/z” exceeds the response for the highest m/z in the ICAL. As the m/z ratio is always directly proportional to the concentration, evaluation based on dilution (per 11.13) is equivalent.
- Method 624 has criteria for unresolved isomers. The problems of isomeric resolution for the routine analytes listed in this SOP were worked through when the laboratory developed its implementation of the method. For example, we know through experience that meta and para xylenes will not be resolved and it was not necessary to include an evaluation for the xylenes in each analysis. Any development work to add compounds would take this into account.
- Method 624 has requirements for purge time and desorb conditions. Purge time for samples is 11 minutes ± 0.1 minutes at ambient temperature. After the 11-minute purge time, attach the trap to the chromatograph, adjust the purge and trap system to the desorb mode, and begin to temperature program the gas chromatograph. Introduce the trapped materials to the GC column by rapidly heating the trap to 180℃ while backflushing the trap with an inert gas for four minutes.
- The purge gas flow rate (40 mL/min ± 5 mL/min) should be measured at the vent and recorded in the maintenance logbook.
- Method 624 requires the trap to be baked for 10 minutes prior to analysis.

Table A-1

Method 624 Analytes and Reporting Limits, 10-mL Purge

Analytes	µg/L
Acrolein	5
Acrylonitrile	2
Benzene	1
Bromodichloromethane	1
Bromoform	1
Bromomethane	5
Carbon tetrachloride	0.2
Chlorobenzene	1
Chloroethane	5
2-Chloroethyl vinyl ether	10
Chloroform	1
Chloromethane	5
Dibromochloromethane	1
1,2-Dichlorobenzene	1
1,3-Dichlorobenzene	1
1,4-Dichlorobenzene	1
1,1-Dichloroethane	1
1,2-Dichloroethane	1
1,1-Dichloroethene	1
trans-1,2-Dichloroethene	1
1,2-Dichloropropane	1
cis-1,3-Dichloropropene	1
trans-1,3-Dichloropropene	1
Ethylbenzene	1
Methylene chloride	3
1,1,2,2-Tetrachloroethane	1
Tetrachloroethene	0.4
Toluene	1
1,1,1-Trichloroethane	1
1,1,2-Trichloroethane	1
Trichloroethene	1
Trichlorofluoromethane	1
Vinyl chloride	0.2

Attachment 1; PROCESS FLOW FOR SEATTLE VOA



Title: Gasoline Range Organics Analysis [Methods 8015B, AK101, NWTPH-Gx]

Approvals

Signatures on File	
Bisrat Tadesse Date	Sandra Langway Date
Volatiles Department Manager	Health & Safety Manager / Coordinator
Terri Torres Date	Heather Prater Date
Quality Assurance Manager	Laboratory Director

Copyright Information:
This documentation has been prepared by TestAmerica Laboratories Inc. and its affiliates ("TestAmerica"), solely for their own use and the use of their customers in evaluating their qualifications and capabilities in connection with a particular project. The user of this document agrees by its acceptance to return it to TestAmerica upon request and not to reproduce, copy, lend, or otherwise disclose its contents, directly or indirectly, and not to use it for any other purpose other than that for which it was specifically provided. The user also agrees that where consultants or other outside parties are involved in the evaluation process, access to these documents shall not be given to said parties unless those parties also specifically agree to these conditions.

THIS DOCUMENT CONTAINS VALUABLE CONFIDENTIAL AND PROPRIETARY INFORMATION. DISCLOSURE, USE OR REPRODUCTION OF THESE MATERIALS WITHOUT THE WRITTEN AUTHORIZATION OF TESTAMERICA IS STRICTLY PROHIBITED. THIS UNPUBLISHED WORK BY TESTAMERICA IS PROTECTED BY STATE AND FEDERAL LAW OF THE UNITED STATES. IF PUBLICATION OF THIS WORK SHOULD OCCUR THE FOLLOWING NOTICE SHALL APPLY:

©COPYRIGHT 2012 TESTAMERICA LABORATORIES, INC. ALL RIGHTS RESERVED.

Facility Distribution No. 001 **Distributed To:** Lab Intranet

Document Uncontrolled When Printed

1.0 Scope and Application

1.1 Analytes, Matrix(s), and Reporting Limits

This SOP delineates the specific requirements for analyzing TPH as gasoline. This method is applicable to soil analysis via 5035, water analysis via 5030 and providing simultaneous confirmation above the RL-secondary column confirmation is not required. Gasoline may be reported without the associated individual analytes, per client request. Table 1 provides a list of target analytes and associated RLs.

On occasion clients may request modifications to this SOP. These modifications are handled following the procedures outlined in Section 12.2.1 in the Quality Assurance Manual.

2.0 Summary of Method

This method describes the analysis of Gasoline Range Organics in soil and water matrices. Soil samples are extracted in methanol. An aliquot of this extract is diluted in water and the sample is analyzed by purge and trap GC with FID. Water samples are analyzed with no dilution, or diluted with reagent water; the sample is analyzed using purge and trap GC with FID.

3.0 Definitions

3.1 Gasoline Range Organics (GRO): All chromatographic peaks eluting between the following ranges are attributed to GRO:

AK101: The area including n-Hexane to the start of n-Decane

8015B: *The area including* Toluene through *the end of* n-Dodecane, inclusive

Hawaii: *The area including* Hexane through *the end of* n-Dodecane, inclusive

California 8015B: *The area including* 2-methylpentane through *the end of* 1,2,4-trimethylbenzene*, inclusive*

NWTPH-GX: at a minimum *the area including* Toluene through *the end of* 1-Methylnaphthalene inclusive is integrated and plotted against the known concentration of gasoline standard added.

Quantitation is based on a direct comparison of the area within this range to the total area of the calibration standard within this range.

4.0 Interferences

4.1 High levels of heavier petroleum products such as diesel fuel may contain some volatile components that produce a response within the retention time range for gasoline. Other organic compounds, including chlorinated solvents, ketones, and ethers are measurable.

4.2 Samples contaminated with a single compound that is detectable using this method may result in a biased value for the compound. This is caused by the different response factors for gasoline and other various solvents.

4.3 Samples can become contaminated by diffusion of volatile organics during shipment and storage. A trip blank prepared from reagent water or methanol and carried through sampling, storage, and handling is recommended.

4.4 Contamination by carryover can occur whenever high-level and low-level samples are sequentially analyzed. Whenever an unusually concentrated sample is encountered, it

Document Uncontrolled When Printed

should be followed by an analysis of a solvent blank of reagent water to check for cross contamination. For volatile samples containing high concentrations of water-soluble materials, suspended solids, high boiling compounds or organohalides, it may be necessary to wash the syringe or purging device with a detergent solution, rinse with distilled water, and then dry in a 105℃ oven between analyses. The trap and other parts of the system are also subject to contamination; therefore, frequent bake-out and purging of the entire system may be required.

4.5 Any co-eluting compound with a quantitation ion identical with the compounds of interest will affect results.

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

5.1.1 Eye protection that satisfies ANSI Z87.1 (per the Corporate Safety Manual), laboratory coat, and appropriate gloves must be worn while samples, standards, solvents, and reagents are being handled. Latex, nitrile, or vinyl gloves must be worn while handling samples, standards, solvents, and reagents. Cut resistant gloves must be worn when using sharp tools or when washing glassware. Disposable gloves that have been contaminated will be removed and discarded; other gloves will be cleaned immediately.

5.1.2 Purge vessels on purge-and-trap instruments can be pressurized by the time analysis is completed. Vent the pressure prior to removal of these vessels to prevent the contents from spraying out.

5.1.3 GC VOA instruments use an ultraviolet (UV) light source, which must be shielded from view. There should also be a warning label/sticker on each instrument that identifies it as a UV light source.

5.1.4 The gas chromatograph contains zones that have elevated temperatures. The analyst needs to be aware of the locations of those zones, and must cool them to room temperature prior to working on them.

5.1.5 There are areas of high voltage in the gas chromatograph. Depending on the type of work involved, either turn the power to the instrument off, or disconnect it from its source of power.

5.1.6 The toxicity or carcinogenicity of each reagent used in this method has not been fully established. Each chemical should be regarded as a potential health hazard and exposure should be as low as reasonably achievable. Cautions are included for known extremely hazardous materials.

Company Confidential & Proprietary

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	Hazards	Exposure Limit (1)	Signs and symptoms of exposure
Methanol (MeOH)	Flammable Poison Irritant	200 ppm-TWA	A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness and dizziness. Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes.
1 – Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

6.1 Computer hardware and software

- *Computer with a minimum 1GB memory, Pentium 4 processor, 80 G hard drive or equivalent or as recommended by instrument manufacturer.*
- *LIMS system: TALS version 1.0 or higher.*
- *Data acquisition system: Agilent (Hewlett Packard) ChemStation for Windows 95 (version G1701AA) or equivalent. Agilent's ChemStation, is used for data acquisition and storage on machine-readable media. Since no processing is done by ChemStation and since there are no audit trail functions associated with data acquisition, the audit trail feature for ChemStation may be either enabled or disabled. The other component, Chrom, is used for data processing such as the measurement of peak area or peak height. By design, the audit trail feature for Chrom is always enabled.*
- *Data processing: Chrom version 1.2 or higher. The computer must have software that allows searching any GC/MS data file for ions of a specified mass and plotting such ion abundances versus time or scan number. This type of plot is defined as an Extracted Ion Current Profile (EICP). Software must also be available that allows integrating the abundances in any EICP between the specified time or scan-number limits. In addition, for the non-target compounds, software must be available that allows for the comparison of sample spectra against reference library spectra. The most recent release of the NIST/EPA mass spectral library should be used as the reference library. The computer system must also be capable of backing up data for long-term off-line storage.*

6.2 Instrumentation

- Chromatographic column: DB-624, 30 meters x 0.25 mm ID x 1.4 um film thickness (J & W Scientific) or equivalent with DB-Wax, 15 meters x 0.25 mm ID x 0.5 um film thickness (J & W Scientific) post column OR

- Chromatographic columns: split post column DB-624, 30 meters x 0.32 mm ID x 1.4 um film thickness (J & W Scientific) or equivalent to FID, 1.0 meter 0.18 inert column to MS.
- Varian Archon Autosampler or Tekmar Aquatek 70 Autosampler or equivalent
- Purge/Trap Liquid Concentrator, Tekmar 3000 or equivalent
- Gas chromatograph and detector: Hewlett Packard 5890 Series II Gas Chromatograph or equivalent with OI model 4430 Photoionization Detector (PID) or equivalent and OI Flame Ionization Detector (FID) or equivalent
- Tekmar 3100 Purge & Trap Concentrator or equivalent
- Columns: Restek RTX-VRX (75 m x 0.45 mm x 2.5 µm) or equivalent
  Note: Other columns may be used. This was the column in place at the time the SOP was prepared. The serial number of the column used is documented in the instrument maintenance logbook.
- Analytical balance, 0.0001 g accuracy
- Drying oven

6.3 **Supplies**

- Scintillation vials
- Volumetric flasks: 10-mL, 50-mL, and 100-mL
- Glass standard vials with screw caps and Teflon-coated septum

7.0 Reagents and Standards

7.1 Document reagent/standards and reagent/standard preparation in TALS using the reagent module as described in SOP TA-QA-0619.

7.2 Methanol, Baker Purge and Trap Grade or equivalent.

7.3 Neat standards used for surrogate/standard solution preparation should be purchased from Aldrich or other certified supplier. Analyte list below:

7.3.1 1-Bromo-4-fluoro-benzene

7.3.2 α,α,α-Trifluorotoluene

7.4 Surrogate: A 500 mg/L standard is prepared by adding 46.7-uL 1-Bromo-4-fluorobenzene to 150.0-mL of the Internal Standard for Initial Calibration solution.

7.5 *Soil* TFT Stock Solution: A 10,000 mg/L solution is prepared by diluting 421.0-uL α,α,α-Trifluorotoluene to 50.00-mL in methanol.

7.6 *Soil* TFT Preservation/*Surrogate* Solution: A 4.00 mg/L TFT solution is prepared by diluting 1.00-mL TFT Stock Solution to 1.00 L in methanol.

7.7 Water TFT Surrogate Solution: A 400 mg/L TFT solution is prepared by diluting 33.65-uL α,α,α-Trifluorotoluene to 100-mL in methanol.

7.8 WA VPH Standard mix: 20,000 mg/L purchased as certified mixture from Ultra Scientific or other equivalent supplier.

7.9 Retention Time Standard: A 20.0 mg/L solution is prepared by diluting to 50.00-mL in methanol as follows:

RT Standard Component	Volume of Source (uL)	Final Volume (mL)	Final Concentration (mg/L)
WA VPH	50.0	50.00	20.0

2-Methylpentane	1.00	50.00	20.0
1,2,4-Trimethylbenzene	1.00	50.00	20.0

7.10 Unleaded Gasoline Composite Standard: 50,000-ug/mL purchased from Restek or other certified supplier.

7.11 GRO ICAL/CCAL Solution: A 1,000 mg/L solution is prepared by diluting 4-mL Unleaded Gasoline Composite Standard to 200-mL in methanol.

7.12 GRO Working Solution (ICV/LCS): A 50,000-ug/mL solution purchased from Accustandard or other certified supplier. This standard is purchased from a separate manufacturer than the ICAL/CCAL standard.

7.13 GRO Initial Calibration: An initial calibration is prepared using 9 calibration points as follows:

Level	Volume of GRO ICAL/CCAL Stock (uL)	Volume of Surr. Stock (uL)	Volume of Methanol (uL)	Final Volume (mL)	Final Concentration (ug/L)
1	5.0	5.0	2500	100.0	50.0
2	10.0	10.0	2500	100.0	100.0
3	25.0	15.0	2500	100.0	250.0
4	50.0	20.0	2450	100.0	500.0
5*	100.0	25.0	2400	100.0	1000.0
6	500.0	37.5	2000	100.0	5000.0
7	1000.0	50.0	1500	100.0	10,000
8	1500.0	--	1000	100.0	15,000
9	2500.0	--	--	100.0	25,000
* This level is used as the Continuing Calibration Verification (CCV) standard.					

7.14 A GRO initial calibration verification standard is prepared at the 1,000-ug/L concentration by diluting the GRO working solution in ASTM Type II water in a 100-mL volumetric flask as follows:

Level	Volume of GRO ICAL/CCAL Stock (uL)	Volume of Surr. Stock (uL)	Volume of Methanol (uL)	Final Volume (mL)	Final Concentration (ug/L)
ICV	50.0	20.0	2500	100.0	1100.0

7.15 Managers/supervisors or a designee are expected to check their areas on a monthly basis for expired standards/reagents and dispose of them according to SOP TA-EHS-0036.

8.0 Sample Collection, Preservation, Shipment and Storage

8.1 All samples are to be stored at 0-6℃ in 43-mL VOA vials or jars with Teflon-lined caps. Water samples should be preserved to a pH <2.0 with HCl. Unpreserved soil samples should be preserved in methanol within 48 hours of collection.

Document Uncontrolled When Printed

8.2 The holding time for preserved waters and soil (from date of collection to date of analysis is 14 days. Unpreserved waters should be analyzed within 7 days of collection. AK101 GRO 5035 field preserved samples have a 28-day holding time limit.

8.3 VOA vials are inverted to check for air bubbles. If at all possible, samples should not be opened prior to analysis.

8.4 Field preserved soil sampling procedure. Soil samples must be collected in appropriately sized containers and submerged in methanol or methanol containing surrogate at 1:1 ratio. The tare weight of the sample container, the weight of the methanol, and the weight of the sample must be known in order to accurately quantitate gasoline range organics. Soil samples must be stored below 25℃. Methanol preser ved soil samples must be analyzed within 28 days of collection.

9.0 Quality Control

9.1 The minimum quality controls (QC), acceptance criteria, and corrective actions are described in this section. When processing samples in the laboratory, use the LIMS QC program code and special instructions to determine specific QC requirements that apply.

9.1.1 The laboratory's standard QC requirements, the process of establishing control limits, and the use of control charts are described more completely in the TestAmerica Seattle QAM.

9.1.2 Project-specific requirements can override the requirements presented in this section when there is a written agreement between the laboratory and the client, and the source of those requirements should be described in the project documents. Project-specific requirements are communicated to the analyst via special instructions in the LIMS and may also come in the form of email or written notifications distributed at "project kick off" meetings.

9.1.3 Any QC result that fails to meet control criteria must be documented in a Nonconformance Memo (NCM). The NCM is approved by the supervisor and then automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group also receives NCMs by e-mail for tracking and trending purposes. The NCM process is described in more detail in SOP TA-QA-0610. This is in addition to the corrective actions described in the following sections.

9.2 Batch Definition

Batches are defined at the sample preparation stage. The batch is a set of up to 20 samples of the same matrix, plus required QC samples, processed using the same procedures and reagents within the same time period. Batches should be kept together through the whole analytical process as far as possible, but it is not mandatory to analyze prepared extracts on the same instrument or in the same sequence. The method blank must be run on each instrument and in each analytical batch.

9.3 Method Blanks

For each batch of samples, analyze a method blank. The method blank is analyzed after the calibration standards and before any samples. For aqueous samples, the method blank consists of reagent water. For solid samples, the method blank consists of 10 mL of Reagent ID: V-4TFT_EX_xxxxx and ten grams of muffled Ottawa sand (reagent ID VOA-Sand_xxxxx). Additional surrogates and internal standard are added automatically by the autosampler at the time of analysis. The method blank is carried through the entire analytical procedure.

Document Uncontrolled When Printed

Acceptance Criteria: The method blank must not contain any analyte of interest at or above one-half the reporting limit or above 10% of the measured concentration of that analyte in the associated samples, whichever is higher.

The method blank must have acceptable surrogate recoveries.

Corrective Actions: Reanalysis of samples associated with an unacceptable method blank is required when reportable concentrations are determined in the associated samples.

If there is no target analyte greater than the RL (less than one half the RL for LaMP and DoD clients) in the samples associated with an unacceptable method blank, the data may be reported with qualifiers *and an NCM*.

If surrogate recoveries in the blank are not acceptable, the data must be evaluated to determine if the method blank has served the purpose of demonstrating that the analysis is free of contamination. If surrogate recoveries are low and there are reportable analytes in the associated samples, re-extraction of the blank and affected samples will normally be required. Consultation with the client should take place.

If reanalysis of the batch is not possible due to limited sample volume or other constraints, the method blank is reported, all affected analytes in the associated samples are flagged with a "B", and appropriate comments may be made in a narrative to provide further documentation.

9.4 Laboratory Control Samples (LCS)

An LCS is analyzed for each batch. An LCS Duplicate (LCSD) is performed only when insufficient sample is available for the MS/MSD or when requested by the client/project/contract. The LCS is analyzed after the calibration standard, and normally before any samples. The LCS is prepared from a different source than are the calibration standards. The LCS contains all the required analytes of interest (See Table 5), and must contain the same analytes as the matrix spike.

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. Recovery limits are updated at a set frequency by QA and are stored in the LIMS

Corrective Actions: If any analyte or surrogate is outside established control limits as described above, the system is out of control and corrective action must occur. Corrective action will normally be re-preparation and reanalysis of the batch.

If the batch is not re-extracted and reanalyzed, the reasons for accepting the batch must be clearly presented in the project

Document Uncontrolled When Printed

records (via NCMs and the case narrative) and in the final report. Examples of acceptable reasons for not reanalyzing might be that the matrix spike and matrix spike duplicate are acceptable, and sample surrogate recoveries are good, demonstrating that the problem was confined to the LCS. This type of justification should be reviewed and documented with the client before reporting. (LaMP: Bias high recoveries– NCM and flag ND samples)

If re-extraction and reanalysis of the batch is not possible due to limited sample volume or other constraints, the LCS is reported, all associated samples are flagged, and appropriate comments are made in a narrative to provide further documentation.

9.5 Matrix Spike and Matrix Spike Duplicate (MS/MSD)

For each QC batch, analyze a matrix spike and matrix spike duplicate if sufficient sample volume is received. If an MS/MSD is not possible due to limited sample, then an LCS duplicate should be analyzed whenever requested by the client. The RPD between the LCS and LCSD is compared to the established acceptance limit. Spiking compounds and levels are given in Table 5. The matrix spike/duplicate must be analyzed at the same base dilution as the unspiked sample, even if the matrix spike compounds will be diluted out, dilutions (beyond the base dilution if necessary) of MS/MSD analyses are not required unless there are specific client instructions to do so. If necessary, this requirement will be passed to the laboratory through the PM by means of the mechanisms described in section 9.1.3 of this SOP.

LaMP: MS required if >10 samples. The client consultant is to identify the sample to be used.

Acceptance Criteria: The MS/MSD 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. The relative percent difference (RPD) between the MS and the MSD must be less than the established RPD limit, which is based on statistical analysis of historical data. MS/MSD recovery and RPD limits are updated at a regular frequency by QA and are stored in the LIMS.

Corrective Actions: If any individual recovery or RPD falls outside the acceptable range, corrective action must occur. The initial corrective action will be to check the recovery of that analyte in the LCS. Generally, if the recovery of the analyte in the LCS is within limits, then the laboratory operation is in control and analysis may proceed. The reasons for accepting the batch must be documented.

If the recovery for any component is outside QC limits for both the matrix spike/matrix spike duplicate and the LCS, the laboratory is out of control and corrective action must be taken. Corrective action will normally include reanalysis of the batch, except in cases where a high bias is indicated and no target is detected above the reporting limit in any associated sample.

If an MS/MSD is not possible due to limited sample, then an LCS duplicate should be analyzed whenever requested by the client.

The RPD between the LCS and LCSD is compared to the established acceptance limit.

9.6 Surrogates

Every sample, blank, and QC sample is spiked with surrogates. Surrogate recoveries in samples, blanks, and QC samples must be assessed to ensure that recoveries are within established limits. The compounds included in the surrogate spiking solutions are listed in Table 3.

Acceptance Criteria: Acceptance limits for surrogate recoveries are set at ± 3 standard deviations around the historical mean. Surrogate recovery limits are updated at a fixed frequency by QA and stored in the LIMS.

Corrective Actions: If any surrogates are outside limits, the following corrective actions must take place (except for dilutions):

- Check all calculations for error.
- Ensure that instrument performance is acceptable.
- Recalculate the data and/or reanalyze if either of the above checks reveal a problem.
- Re-prepare and reanalyze the sample or flag the data as "Estimated Concentration" if neither of the above resolves the problem.

The decision to reanalyze or flag the data should be made in consultation with the client. It is necessary to re-prepare/reanalyze a sample only once to demonstrate that poor surrogate recovery is due to matrix effect, unless the analyst believes that the repeated out of control results are not due to matrix effect.

If the surrogates are out of control for the sample, matrix spike, and matrix spike duplicate, then matrix effect has been demonstrated for that sample and re-preparation/reanalysis is not necessary. If the sample is out of control and the MS and/or MSD is in control, then reanalysis or flagging of the data is required.

NOTE: For LaMP samples, if the surrogate percent recovery fails, the recovery must be confirmed by re-extraction and reanalysis with the following exceptions:

- The lab has unequivocally demonstrated a sample matrix effect and informed the LaMP representative.
- The recovery exceeds control limits and all target analytes in the sample are non-detect.

9.7 If batch QC samples or trip blanks are re-analyzed to confirm a recovery or result, and an improvement in results would cause the re-analysis to be reported, then the associated client samples must also be re-analyzed. The only exception to this protocol would be if an obvious analytical problem occurred during the initial analysis (i.e. no internal standard added, bent autosampler needle, etc).

9.8 Any extra QC that is analyzed in a batch or sequence must be evaluated using the same criteria as the corresponding QC above.

Document Uncontrolled When Printed

10.0 Procedure

One-time procedural variations are allowed only if deemed necessary in the professional judgment of management to accommodate variation in sample matrix, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using an NCM. The NCM is approved by the supervisor and then automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA department also receives NCMs by e-mail for tracking and trending purposes. The NCM process is described in more detail in SOP # TA-QA-0610. The NCM shall be filed in the project file and addressed in the case narrative.

10.1 Sample Preparation

10.1.1 Check the Balance logbook to determine if the daily calibration check has been completed. If it has not, the analyst must perform this check according to SOP TA-QA-0014.

10.1.2 Soil Extraction

10.1.2.1 Method 5035 Laboratory preserved:

10.1.2.2 *Weigh 10 g of the sample into a tared 20 mL scintillation vial. Record scintillation vial lot number, the sample weight, the methanol lot number, the preparation date and the analyst who prepared it in the 5035 preparation batch in TALS. Sample weights are calculated in the laboratory by adding the tare weight of the scintillation vial to the "Tare Weight" column and then entering the weight of the scintillation vial plus sample into the "Vial & Sample" column of the preparation batch sheet for the corresponding sample container ID. This can be done by either a direct read from the balance in the volatiles prep area (preferred method), or by manually entering the weight. If the samples were preserved in MeOH at another TALS lab the calculated initial sample weight can be manually entered into the "Initial Amount" column of the preparation batch sheet for the corresponding sample container ID.*

10.1.2.2.1 To prepare the blank spike (LCS)/blank spike duplicate (LCSD), add 80-uL of GRO Working Solution and 10-mL of the TFT Preservation Solution to 10 g muffled Ottawa sand. (NOTE: The same metal spatulas to weigh soil samples must be used for measuring out the Ottawa sand).

10.1.2.2.2 To prepare the matrix spike (MS)/matrix spike duplicate (MSD), add 80-uL of GRO Working Solution and 10-mL of the TFT Preservation Solution to 10 g pre-weighed soil samples. To prepare the method blank (MB); add 10-mL of the TFT Preservation Solution to 10 g Ottawa sand.

10.1.2.2.3 *All spiking solutions must be added immediately to the sample after the addition of the TFT Preservation Solution.*

10.1.2.3 Vortex the samples for the extraction batch for approximately 10 to 30 seconds to break up any large clumps in the extraction vials. If after 30 seconds a pellet still remains, vortex for an additional 30 seconds. If pellet still remains, further vortex mixing is not recommended, proceed to next step. It should be noted that the MB and LCS must

Document Uncontrolled When Printed

be vortex mixed the same amount of time as the longest associated sample.

10.1.2.4 After all samples have been vortex mixed, place all samples for the extraction batch into shaker box and set timer for five minutes. It is recommended that the caps of all vials are checked and tightened before placing in shaker box to prevent leaking. If samples are present which still contain pelletized sample after vortex mixing in step 10.1.2.3, set the timer for 10 to 15 minutes. It is not recommended to shake samples for more than 15 minutes. If a sample still contains pelletized sample after shaking for 15 minutes, vortex the sample for an additional 15 seconds and shake entire batch for additional 5 minutes. Any pelletized sample remaining after second shaking is noted in batch record and an "other anomaly" NCM is generated to accompany the extraction batch.

10.1.2.5 After samples are shaken, place samples four at a time into centrifuge with inserts for scintillation vials. Spin samples for a sufficient time to create a transparent but not necessarily uncolored layer of methanol extract above the extracted material. The time will vary depending on the nature and particle size of the extracted material. Three to five minutes at 50% speed is usually sufficient. Again it should be noted that the MB and LCS must be centrifuged at the same rate and for the same time as the longest centrifuged associated sample.

10.1.2.6 Extracts are stored in the scintillation vial used for extraction and are stored at 0-6°C. The extracts are removed from cold storage and are allowed to return to ambient temperature prior to analysis.

10.1.2.7 Method 5035 Field preserved:

10.1.2.8 All soil samples for AK101 analysis must be field preserved and any analysis performed with less than 48 hours allowed for the sample to equilibrate in the methanol (from time of sampling to analysis) noted in an NCM in the analytical batch.

10.1.2.9 Each containers tare weight is recorded *in the TALS preparation batch. Most containers will contain a bar code with the tare weight information that can be scanned for automatic entry into the tare weight entry field in the preparation batch. Sample weights are calculated in the laboratory by adding the received weight of the sample jar to the "Vial & Sample" column of the preparation batch sheet for the corresponding sample container ID. This can be done by either a direct read from the balance in the volatiles prep area (preferred method), or by manually entering the weight. If the samples received are in 4 oz jars with 25 mls of methanol (AK samples) the calculated initial weight of the sample must be adjusted to correct for the weight of the methanol which is not included in the container tare weight. TALS will perform this calculation, however the analyst must enter a "1" into the "Tare Incl MeOH" column of the preparation batch sheet for the corresponding sample container ID.*

10.1.2.9.1 To prepare the blank spike (LCS)/blank spike duplicate (LCSD), add 80-uL of GRO Working Solution and 10-mL of the TFT

Document Uncontrolled When Printed

Preservation Solution to 10 g muffled Ottawa sand. *All spiking solutions must be added immediately to the sample after the addition of the TFT Preservation Solution.*

10.1.2.9.2 To prepare the **matrix spike** (MS)/**matrix spike duplicate** (MSD), add a 1075-uL aliquot of sample methanol extract and 25-uL of GRO Working Solution to 42.1-mL of ASTM Type II water contained in appropriately labeled 43 mL VOA vials.

10.1.2.9.3 To prepare the **method blank** (MB), add 1075-uL of the TFT Preservation Solution (section 7.8) to 42.1-mL of ASTM Type II water contained in an appropriately labeled 43 mL VOA vial.

10.1.2.10 *Vortex the samples for the extraction batch for approximately 10 to 30 seconds to break up any large clumps in the extraction vials. If after 30 seconds a pellet still remains, vortex for an additional 30 seconds. If pellet still remains, further vortex mixing is not recommended, proceed to next step. It should be noted that the MB and LCS must be vortex mixed the same amount of time as the longest associated sample.*

10.1.2.11 *After all samples have been vortex mixed, place all samples for the extraction batch into shaker box and set timer for five minutes. It is recommended that the caps of all vials are checked and tightened before placing in shaker box to prevent leaking. If samples are present which still contain pelletized sample after vortex mixing in step 10.1.2.3, set the timer for 10 to 15 minutes. It is not recommended to shake samples for more than 15 minutes. If a sample still contains pelletized sample after shaking for 15 minutes, vortex the sample for an additional 15 seconds and shake entire batch for additional 5 minutes. Any pelletized sample remaining after second shaking is noted in batch record and an "other anomaly" NCM is generated to accompany the extraction batch.*

10.1.2.12 *After samples are shaken, place samples four at a time into centrifuge with inserts for scintillation vials. Spin samples for a sufficient time to create a transparent but not necessarily uncolored layer of methanol extract above the extracted material. The time will vary depending on the nature and particle size of the extracted material. Three to five minutes at 50% speed is usually sufficient. Again it should be noted that the MB and LCS must be centrifuged at the same rate and for the same time as the longest centrifuged associated sample.*

10.1.2.13 *Extracts are stored in the scintillation vial used for extraction and are stored at 0-6°C. The extracts are removed from cold storage and are allowed to return to ambient temperature prior to analysis.*

10.1.2.14 *The MB, LCS and any received samples which appear to contain "clumps" of sample which could be broken up with agitation are vortex mixed for up to 1 minute. If no samples require agitation, only the MB and LCS are vortex mixed for approximately 15 seconds to ensure mixing of the sand and added solutions.*

10.1.3 Extract preparation for analysis

10.1.3.1 A 1075-uL aliquot of each methanol extract is added to 42.1-mL of ASTM Type II water contained in appropriately labeled 43 mL VOA vials.

10.1.3.2 If the water solution becomes milky or cloudy in appearance, this is an indication of potential high concentrations of target and/or non-target compounds and a higher dilution may be prepared at this time.

10.1.4 Dry Weight.

10.1.4.1 Percent solids (dry weight) is determined by weighing approximately 10 grams of sample, completely drying the sample and then re-weighing and recording the difference in weight. SOP TA-WC-0160 describes this procedure in further detail.

10.1.5 Water Prep

10.1.5.1 All aqueous samples will be spiked with 10.75-uL of Water Surrogate Solution (section 7.7) and analyzed as received.

10.1.5.2 Samples may be screened for gasoline by analyzing a spare VOA, if available.

10.1.5.3 To prepare the Laboratory Control Sample /Laboratory Control Sample Duplicate (LCS/LCSD), add 25-uL of Water Surrogate Solution and 23.25-uL of GRO Working Solution to a 100-mL volumetric flask partially filled with and brought to final volume with ASTM Type II water. The volumetric is then gently inverted several times to ensure homogenization and transferred to two appropriately labeled VOA vials.

10.1.5.4 To prepare the matrix spike/matrix spike duplicate (MS/MSD), add 10-uL of GRO Working Solution to each of two additional samples provided before adding surrogate. To prepare the method blank (MB), add 12.5-uL of the Water Surrogate Solution to a 50-mL volumetric flask partially filled with and then brought to volume with ASTM Type II water. The volumetric is then gently inverted several times to ensure homogenization and transferred to an appropriately labeled VOA vial.

10.1.5.5 All aqueous sample VOA vials should be inverted to check for headspace/air bubbles prior to analysis. If multiple VOA vials are submitted for analysis, the one with the least amount of headspace should be used first. Any headspace is measured and documented in an NCM in the analytical batch.

10.1.5.6 For AK101 analysis, amber glass vials should be used for aqueous samples. If clear glass vials are submitted for analysis, the samples should be protected from light.

10.2 **Calibration**

10.3 Recommended Instrument Conditions

10.3.1 Gas Chromatograph Suggested Temperature Program

The following temperature programs vary with the column type used.

In-use column dimensions and serial numbers are recorded in each instrument's maintenance logbook.

Document Uncontrolled When Printed

Sample Analysis

Injector Temperature:	230˚C
Detector Temperature:	250˚C
Initial Temperature:	45˚C
Initial Hold Time:	3.5 minutes
Temperature Program:	45℃ to 75 °C at 15°C/min hold for 0.50 minutes 75°C to 130°C at 20°C/min hold for 0.50 minutes 130°C to 230°C at 25°C/min hold for 1.75 minutes Total run time is 15.00 minutes.
Final Temperature:	184 ˚C
Final Temperature:	230 ˚C
Final Hold Time:	1.75 minutes

Note: These conditions can vary by instrument. This is only a guideline. Actual instrument conditions are posted in each maintenance logbook.

10.4 Initial Calibration

10.4.1 A series of five or more initial calibration standards is prepared and analyzed for the target compounds and each surrogate compound. Nominal calibration levels for GRO are listed in sections 7.13 and 7.14. Other calibration levels may be used depending on the capabilities of the specific instrument or program requirements. Calibration levels below the reporting limit may be removed provided that there is a minimum of five calibration points, and the lowest standard is at or below the TestAmerica Seattle reporting limit.

10.4.1.1 For GRO the following carbon ranges are determined:

AK101: The area including n-Hexane to the start of n-Decane

8015B: Toluene through n-Dodecane, inclusive

Hawaii: Hexane through n-Dodecane, inclusive

California 8015B: 2-methylpentane through 1,2,4-trimethylbenzene

NWTPH-Gx: at a minimum Toluene through 1-Methylnaphthalene inclusive is integrated and plotted against the known concentration of gasoline standard added.

10.4.2 The same purge volume must be used for calibration and sample analysis, and the low level standard must be at or below the reporting limit.

10.4.3 It may be necessary to analyze more than one set of calibration standards to encompass all of the analytes required for some tests.

10.4.4 Calibration is valid when the following conditions have been met: a correlation coefficient (r) value ≥0.995 for each target analyte, a higher order polynomial that is continuous and monotonic with a coefficient of determination (r^2) ≥ 0.990, or the mean RSD of each target analyte is less than 15 %. NOTE: When using a higher order polynomial, there must be an additional calibration standard for each degree beyond linearity (i.e. 5 for linear, 6 for quadratic, etc.). Recalibration must occur when either the continuing calibration standard value falls outside +/-20 % of the true value twice consecutively or, the linear coefficient (r) falls below 0.995, or other conditions such as a major instrument changes warrant recalibration.

Document Uncontrolled When Printed

10.4.5 Initial surrogate calibration is performed by average RF of all standards used unless matrix effects are observed. Calculation is performed using average of response factors when less than or equal to 15 %, linear regression with coefficient value (r) >0.995 for each target analyte, or higher order polynomial that is continuous and monotonic with a coefficient value (r) > 0.995. Recalibration occurs when surrogate recoveries consistently fail to meet established control limits.

10.4.6 See Corporate SOP CA-Q-S-005 for information on acceptable initial calibration models and associated algorithms.

10.4.7 Initial Calibration Verification (ICV)

Once the initial calibration has been evaluated and determined to be valid, the calibration must be verified with initial calibration verification (ICV) using a standard prepared from a source other than the calibration solutions. Multiple levels of ICV may be needed to validate all compounds in the initial calibration curve. Acceptance limits are as follows:

For 8015, the ICV must be <15% Drift.

For NWTPH-Gx, the ICV must be <20% Drift.

For AK101, the ICV must be <25% Drift.

If the %Drift falls outside acceptance criteria, assess the system for possible problems (eg. Standard degradation, etc.), re-prepare the ICV and re-analyze. If the second ICV also fails, corrective action is required (e.g., System maintenance, re-preparing intermediate standards, etc.) and the calibration must be re-prepared and re-analyzed. An acceptable ICV must be achieved before analysis.

10.4.8 A daily secondary source QC check standard (LCS) is performed using a Certified Gasoline with certified individual analytes standard from a source separate from that used for the calibration. Quantitation is performed identically as samples and standards and the result compared to the true value. Acceptance criteria for each parameter is determined by performance based empirical data.

10.4.8.1 For all DoD projects a second source calibration verification will be performed immediately following the initial calibration. The value for all analytes must be within +/- 20% of the expected value. This criterion must be met before any samples are analyzed.

10.4.9 A retention time standard (7.12) is analyzed each analytical day and every 12 hours for 8015B. The standard concentration is approximately 500 ug/L and contains at a minimum n-Hexane, Toluene, n-Decane, n-Dodecane, and 1-Methylnaphthalene. Adjustments in the retention time windows for the respective hydrocarbon groups are made, if necessary, prior to any standard or sample quantitation. See Attachment 2 for an example retention time standard chromatogram.

The default retention time ranges are established as follows:

Gasoline Range Organics NWTPH-Gx:	Toluene to 1-Methylnapthalene
Gasoline Range Organics AK101:	Hexane to the start of Decane
Gasoline Range Organics CA 8015:	2-methylpentane to 1,2,4-Trimethylbenzene
Gasoline Range Organics HI 8015:	Hexane to Dodecane

10.4.9.1 For all DoD projects the retention time window position will be established for each analyte and surrogate once per ICAL and at the beginning of the

analytical shift. The position must be set using the midpoint standard of the calibration curve or the value in the CCV run at the beginning of the analytical shift.

10.5 Continuing Calibration

10.5.1 The initial calibration must be verified every twelve hours.

10.5.2 A continuing calibration check standard is analyzed at the beginning of each analytical sequence and at the end of each analytical sequence or every 10 samples, whichever is more frequent. The percent recovery must be within +/-15 %, 20 % or 25 % of the true value for 8015 California-G/Hawaii-G, NWTPH-Gx, and AK101, respectively. If the CCV recoveries exceed the method specified upper %Recovery limits and there are no associated sample detections above the RL, the data may be qualified and reported as the system has shown a potentially high bias (this must be done in consultation with the client). If the CCV recoveries exceed method specified %D, the CCV may be re-prepared and re-analyzed. If two sequential CCVs fail the system must be re-calibrated and all samples analyzed since the last acceptable CCV must be re-analyzed.

10.5.2.1 For BP LaMP projects CCV is run at beginning and end of sequence and every 10 samples. Recoveries should be between 85% and 115%.

10.5.2.2 Instrument response should be monitored daily by recording the absolute response of an individual peak. The internal standard is the peak chosen for these instruments. The absolute response for this peak, in the CCVRT, needs to be documented in the maintenance log book on a daily basis.

10.5.3 Once the above criteria have been met, sample analysis may begin.

10.6 **Sample Analysis**

10.6.1 Analysis - All samples are injected into the GC using the auto sampler and the purge and trap system to increase reproducibility. Data is acquired from the GC detector on the Chrom data acquisition system. The resultant area amounts are compared to the calibration curve, and the concentration of each analyte is calculated.

10.6.2 The presence of Gasoline is indicated if compounds are detected in the appropriate hydrocarbon range.

10.6.3 Refer to section 11.15 of SOP MV-0312 for dilution procedures.

10.6.4 An example analysis sequence log is provided as Attachment 1.

10.6.5 **pH for aqueous samples**

Immediately after analysis or immediately after opening the sealed VOA vial and obtaining the necessary aliquots for dilutions, the analyst must check the pH of aqueous samples with narrow range pH paper (0-2.5) to ensure the pH is <2. Record the pH of aqueous samples on the analytical batch sheets. In those cases where the pH is >2, initiate a non-conformance report and qualify the data, noting if the sample(s) was analyzed outside of the shortened seven-day hold time.

10.7 **Data Reduction and Review**

10.7.1 Upon completion of the analytical sequence:

10.7.1.1 Review chromatograms online and determine whether manual data manipulations are necessary.

10.7.1.2 Manual Integrations

All manual integrations must be justified and documented. See Corporate SOP CA-Q-S-002 for requirements for manual integration.

10.7.1.3 Manual integrations are processed using Chrom, which stores the before and after chromatograms and the reason for the change, and attaches the analyst's electronic signature.

10.7.1.4 Confirm that run logs have printed on them the instrument ID, the analyst **and the method** used. If this is not printed on the run logs, this must be entered by hand prior to completing the package.

10.7.1.5 Update the sequence log.

10.7.2 Compile the raw data for all the samples and QC samples in a batch. The analytical batch is defined as containing no more than 20 field samples.

10.7.2.1 Perform a level 1 data review and document the review on the data review checklist (GC and GCMS Data Review Checklist).

10.7.2.2 Submit the data package and review checklist to the peer reviewer for the level 2 review. The data review process is explained in SOP TA-QA-0635.

10.8 Instrument Maintenance

10.8.1 All maintenance and repairs need to be documented in the instrument's maintenance logbook. The logbook must include the instrument name, serial number for each major component (e.g., GC, autosampler, column) and the date of start-up. When an instrument is not capable of analyzing samples, it needs to be tagged "Out of Service". Logbook entries must include a description of the problem and what actions were taken to address the problem. After an instrument has undergone maintenance or repairs, the system is evaluated using a tune, CCV or ICAL. If the evaluation is successful, the analyst documents in the logbook that the "System returned to control as indicated by a passing CCV" (or ICAL, MB, tune, etc as may be the case).

If columns were replaced during maintenance procedures the specific make, model and serial numbers of the columns installed need to be entered in the instruments maintenance logbook.

See section 11.15 of SOP MV-0312 for additional details about instrument maintenance.

10.9 Troubleshooting

10.9.1 *To ensure optimum performance of the FID, you must keep it clean and free of dust and deposits. Symptoms such as reduced sensitivity and increased noise indicate that detector needs cleaning.*

10.9.2 *Baseline noise:*

10.9.2.1 *Contaminated injector and or column. Clean the injector and solvent rinse the column.*

10.9.2.2 *The column is inserted into the flame of the FID. Reinstall the column.*

10.9.2.3 *Incorrect combustion gases or flow rates. Check and reset the gases at their proper values.*

Document Uncontrolled When Printed

10.9.2.4 *Physical defect in the detector. Clean or replace parts as necessary.*

10.9.2.5 *Defective detector board. Consult the instruction manual or contact the GC manufacturer.*

10.9.3 *Baseline spiking:*

10.9.3.1 *Particulate matter passing through the detector. Clean the detector per section 9.7.4.*

10.9.3.2 *Loose connections on cables or circuit boards (usually random spiking). Clean and repair the electrical connections as needed.*

10.9.4 *Baseline Drift (Upward):*

10.9.4.1 *GC or column contamination. Clean the injector. Solvent rinse the column.*

10.9.4.2 *Damaged stationary phase. Replace the column. Determine the cause of the damage to prevent future problems.*

10.9.5 *Baseline Drift (Downward):*

10.9.5.1 *Incomplete conditioning of the column. Condition the column until a stable baseline is obtained.*

10.9.5.2 *Unequilibrated detector. Allow the detector enough time to equilibrate.*

10.9.6 *Irregular Peak Shapes or Sizes*

10.9.6.1 *No peaks.*

10.9.6.1.1 *Plugged syringe. Clean or replace the syringe.*

10.9.6.1.2 *Broken column. Replace or reinstall the column.*

10.9.6.1.3 *Detector gases improperly set or not on. Check and reset the detector gases.*

10.9.6.1.4 *Very low or no carrier gas flow. Immediately lower the column temperature to 35-45C. Measure and verify the carrier gas flow rate. Check for leaks.*

10.9.6.2 *Tailing Peaks.*

10.9.6.2.1 *Active injector liner or column. Clean or replace liner. Replace the column if it is damaged.*

10.9.6.2.2 *Contaminated injector liner or column. Clean or replace the injector liner. Solvent rinse the column.*

10.9.6.2.3 *Poorly installed column, liner or union. Check and verify the installation of each fitting. Reinstall the column if needed.*

10.9.6.2.4 *Poorly cut column end. Re-cut and reinstall the column.*

10.9.6.3 *Rounded or Flat Topped Peaks.*

10.9.6.3.1 *Exceeding the range of the integrator. Dilute the sample.*

10.9.7 *Retention Time Shifts*

10.9.7.1 *Leak in the injector, especially the septum. Find and repair the leak. Change the septum.*

Document Uncontrolled When Printed

10.9.7.2 *Contaminated column. Solvent rinse the column.*

11.0 Calculations

11.1 Accuracy

ICV / CCV, LCS % Recovery = $\frac{\text{observed concentration}}{\text{known concentration}}$ x 100

MS % Recovery = $\frac{\text{(spiked sample) - (unspiked sample)}}{\text{spiked concentration}}$ x 100

11.2 Precision (RPD)

Matrix Duplicate (MD) = $\frac{|\text{orig. sample value - dup. sample value}|}{[(\text{orig. sample value + dup. sample value})/2]}$ x 100

11.3 Response Factor (RF)

$$RF = \frac{A_x C_{is}}{A_{is} C_x}$$

Where:

A_x = Area of the characteristic ion for the compound to be measured.
A_{is} = Area of the characteristic ion for the specific internal standard.
C_{is} = Concentration of the specific internal standard, ng.
C_x = Concentration of the compound being measured, ng.

11.4 Standard Deviation (SD)

$$SD = \sqrt{\frac{\sum_{i=1}^{n}\left(X_i - \overline{X}\right)^2}{n-1}}$$

Where:

X_i = Value of X at i through n.
n = Number of points.
$\overline{X}$ = Average value of Xi.

11.5 Percent Relative Standard Deviation (%RSD)

$$\%RSD = \frac{SD}{\overline{RF}} \times 100\%$$

Where $\overline{RF}$ is the mean of RF values for the calibration.

Document Uncontrolled When Printed

11.6 Percent Drift between the initial calibration and the continuing calibration:

$$\%Drift = \frac{C_{expected} - C_{found}}{C_{expected}} \times 100\%$$

Where:

$C_{expected}$ = Known concentration in standard.
C_{found} = Measured concentration using selected quantitation method.

11.7 Concentration = mg/kg or L = $\frac{C \times V \times D}{W}$

Where:
C = sample concentration in extract (ppm)
V = Volume of extract (mL)
D = Dilution Factor

W = Weight/Volume of sample aliquot extracted (grams or mLs)

NOTE: All dry weight corrections are made in LIMS at the time the final report is prepared.

11.8 Calculation of Results for Methanol Extracts

$$\text{Sample Conc (ug/Kg)} = [\text{On Column (ug/L)}] \times \frac{\text{Extraction Final Volume (mL)}}{\text{Amount of Soil Sample (g)}} \times \frac{\text{VOA Vial Volume (mL)}}{\text{Amt of MeOH Extract (mL)}} \times \frac{\text{1L (CF)}}{\text{1000mL}} \times \frac{\text{1000g (CF)}}{\text{1Kg}}$$

VOA vial volume is 43 ml and when the extract doesn't require a dilution, 1.075 mL of the methanol extract is used. So, the equation becomes:

$$\text{Sample Conc (ug/Kg)} = [\text{On Column (ug/L)}] \times \frac{\text{Extraction Final Volume (mL)}^{1}}{\text{Amount of Soil Sample (g)}^{2}} \times \frac{\text{43 (mL)}}{\text{1.075 (mL)}} \times \frac{\text{1L (CF)}}{\text{1000mL}} \times \frac{\text{1000g (CF)}}{\text{1Kg}}$$

[1]Extract Final Volume, miscible solvent corrected (mL) = ((g of samples * % moisture/100) + ml of MeOH) * 40 (used when % Moisture of the soil sample is greater than 10%).

[2]Amount of Soil, dry-weight corrected (g) = sample mass (g) * (100 - % moisture/100)

When the Extraction Final Volume is 10mL, the Soil Extract Volume expressed on Form 1 will be 400mL. [10 * (43/1.075)]
When the Extraction Final Volume is 25mL, the Soil Extract Volume expressed on Form 1 will be 1000mL. [25 * (43/1.075)]

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 (see SOP TA-QA-0602). 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.

Document Uncontrolled When Printed

12.2 Demonstration of Capabilities

Analyst initial and continuing Demonstrations of Capability (DOC) are performed before any client samples are analyzed and are updated annually. See SOP TA-QA-0617 for details.

12.3 Training Requirements

See SOP TA-QA-0608 for detailed training requirements.

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

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 SOP TA-EHS-0036.

14.1 Waste Streams Produced by the Method

14.1.1 Methanol with trace levels of volatile analytes described by this method is temporarily stored in appropriately constructed (i.e. plastic) satellite waste containers labeled "Hazardous Waste." When filled, the container is bulked into the mixed solvent waste drum in the waste disposal warehouse. This waste stream is sent out for fuel blending.

14.1.2 VOA vials are collected in large plastic satellite waste bins marked "Hazardous Waste." At or before the waste reaches 55 gallons, the contents are transferred to the sample disposal area where the vials are bulked into a 55 gallon waste barrel and sent out for incineration.

14.1.3 Expired Standards. Expired standards are collected in satellite containers marked "Hazardous Waste." At or before the containers reach 55 gallons the containers are taken to the waste warehouse where they are bulked into an expired standards lab pack and sent out for incineration.

15.0 References / Cross-References

15.1 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition, Methods 5035A, 5030B, and 8015B.

15.2 Method AK101 for the Determination of Gasoline range organics Version 4-8-02.

15.3 NWTPH-Gx Volatile Petroleum Products Method for Soil and Water. Washington DOE Publication No. ECY 97-602, June 1997.

15.4 Department of Defense Quality Systems Manual for Environmental Laboratories, prepared by DoD Environmental Quality Workgroup, Final Version 4.2, October 2010.

16.0 Method Modifications:

Item	Method	Modification
1	NWTPH-Gx	The method states one duplicate and one method blank per 10 samples. TestAmerica Seattle batches 2 duplicate sets (if sufficient sample exists), one LCS/LCSD set, and one method blank per 20 samples
2	NWTPH-Gx	The method states 5 g of soil is to be added to 10-mL Methanol followed by a 100-uL aliquot added to 5-mL water. TestAmerica Seattle extracts 10 g of soil with 10-mL Methanol followed by an 1075-uL aliquot added to 43-mL water
3	8015B	GRO range organics are quantitated via GC/MS TIC as opposed to FID, were appropriate

17.0 Attachments

Attachment 1: Example Instrument Sequence

Attachment 2: Example Chromatogram for RT Standard

Table 1: Reporting Limits

Table 2: Surrogate Standards

Table 3: LCS and Matrix Spike Compounds

18.0 Revision History

- Revision 12, dated 30 April 2014.
  - Removed all references to BTEX by GCMS.
  - More clearly defined GRO ranges for the different methods in section 3.1.
  - Added computer hardware and software to section 6.1.
  - Added clear instructions on the preparation procedures for MeOH 5035 Soil samples, section 10.1.2.
  - Added troubleshooting, section 10.9

- Revision 11, dated 13 March 2013.
  - Added section 10.1.2.3.1 to better describe the preparation procedures and documentation.
  - Added section 14.1 Waste Streams Produced by the Method.

- Revision 10, dated 12 October 2012.
  - Added an elaborated calculation for methanolic extracts 11.8

- Revision 9, dated 20 June 2011
  - Specified software in section 6.1
  - Updated standard information in section 7
  - Incorporated ROMDs 00019 and 00026 in sections 6.1 and 10.4
  - Added hold time information for aromatics in unpreserved water samples, section 8
  - Incorporated ROMD 00025 in Section 9.4 and 9.5
  - Incorporated ROMD 00020 in section 10.4
  - Incorporated ROMD 00022 in section 10.6.8
  - Updated surrogated Methanol reagent name section 9.3
  - Changed 5% to 10% per QSM 4.2 section 9.3

- Added spiking criteria per LaMP sections 10.1.2.1.4 and 10.1.2.2.3
- Adjusted reagent water volume from 43 to 42.1 mL in sections 10.1.2.1.6, 10.1.2.2.2 and 10.1.2.2.5 for final volume of 43 mL.
- Added section 10.1.2.2.6 specifying field preservation for AK101.
- Added sections 10.1.3.5 and 10.1.3.6 to address headspace and glass for Aq.
- Added reference to corporate tuning policy section 10.5
- Added two consecutive CCV failures section 10.6.5
- Added explicit ICV section 10.6.9
- Added more detail/condensed CCV analysis in sections 10.7.2 and 10.7.2.1
- Incorporated ROMD 00024 in section 10.7.2
- Incorporated ROMD 00033 in section 10.7.2.2
- Added dilution procedures (by reference) in section 10.8.4.
- Added pH section 10.8.6
- Added data reduction/data review (10.9) and maintenance (10.10) sections
- Corrected section/table references and grammar where necessary.

- Revision 8, dated 16 April 2010
  - Added documentation of standards/reagents and standard/reagent preparation Section 7.1
  - Added removal of expired standards Section 7.24.
  - Added LaMP matrix spike requirements Section 9.5
  - Added BP LaMP surrogate requirements, Section 9.6.
  - Added criteria for additional QC, Section 9.7.
  - Added daily balance check to Section 10.1.1.
  - Added BP LaMP CCV criteria Section 10.7.2.2
  - Updated DoD Table (Attachment)
  - Integration for TestAmerica Bothell and TestAmerica Tacoma operations.
- Revision 7, dated 17 December 2008
  - Integration for TestAmerica and STL operations.
  - This revision is a complete rewrite and an expansion of scope.

Document Uncontrolled When Printed

Attachment 1. Example Instrument Sequence

Page 1

Sequence Log

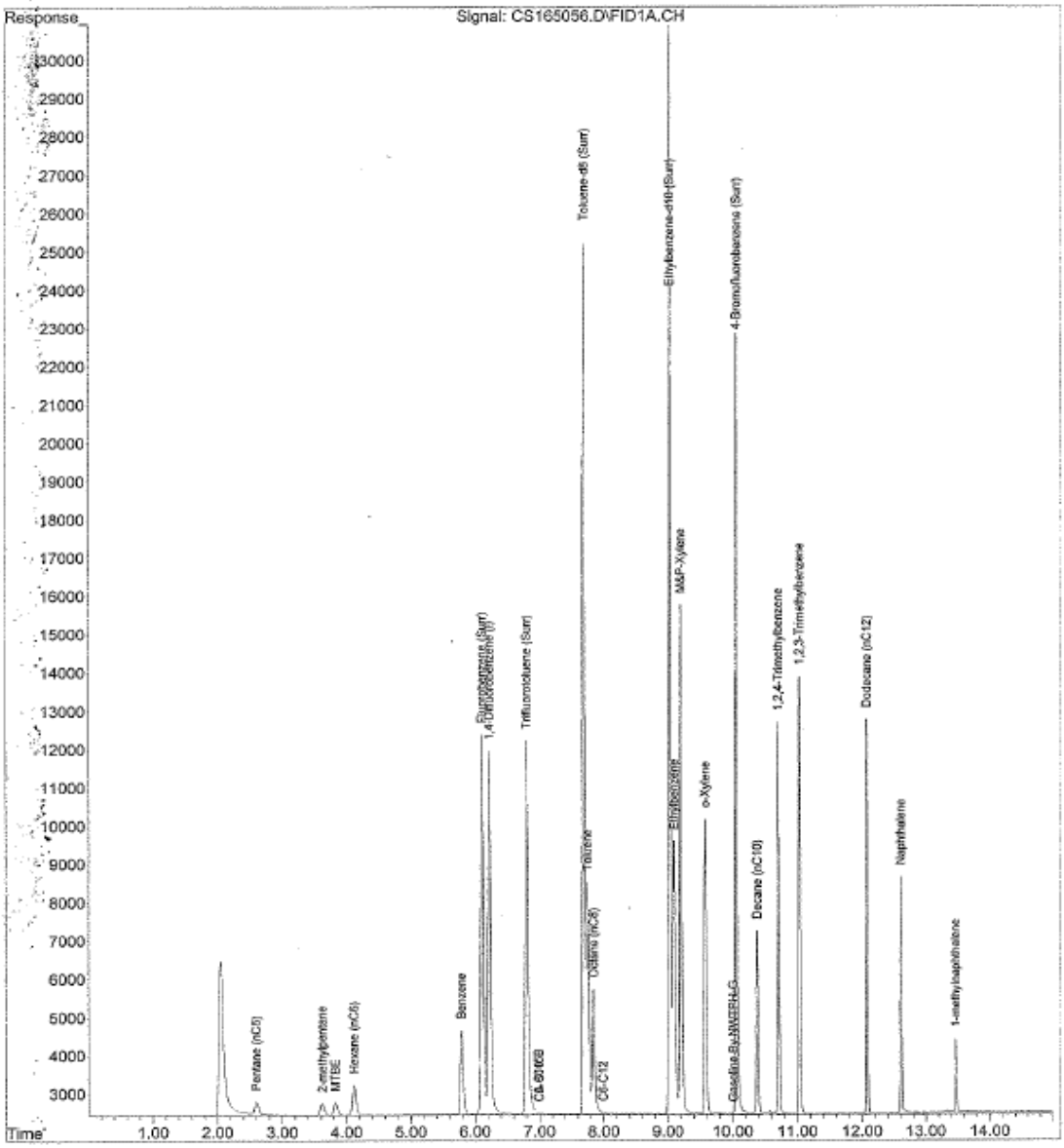
Directory : i:\1\DATA\01092009

#	Filename	Sample Name	Date/Time
1	ms176640.d	Fife Rinse	01/09/09 11:43
2	ms176641.d	Rinse/Tune	01/09/09 12:04
3	ms176642.d	RT standard	01/09/09 12:26
4	ms176643.d	1000 ug/L GRO ccal	01/09/09 12:47
5	ms176644.d	25 ug/L 8260 ccal	01/09/09 13:08
6	ms176645.d	MB 580-39682/1-A	01/09/09 13:30
7	ms176646.d	LCS 580-39682/2-A	01/09/09 13:51
8	ms176647.d	580-12421-E-1-A	01/09/09 14:12
9	ms176648.d	580-12421-E-3-A	01/09/09 14:34
10	ms176649.d	580-12421-E-3-B MS	01/09/09 14:55
11	ms176650.d	580-12421-E-3-C MSD	01/09/09 15:17
12	ms176651.d	580-12421-B-5-A	01/09/09 15:38
13	ms176652.d	Rinse/Tune	01/09/09 16:00
14	ms176653.d	1000 ug/L GRO ccal	01/09/09 16:21

Company Confidential & Proprietary

Attachment 2. Example Chromatogram for RT Standard

```
File        : I:\2\DATA\04282006\CS165056.D
Operator    : jc
Acquired    : 4-28-2006 09:33:19 AM using AcqMethod GBTEX.M
Instrument  :   Instrumen
Sample Name: rt std
Misc Info   : 1369-18-1
Vial Number: 3
```



Document Uncontrolled When Printed

Table 1. Reporting Limits

Compound	CAS Number	Reporting Limits[1]	
		Water (µg/L)	Soil (mg/kg)
Gasoline Range Organics, NWTPH-Gx	STL00228	100	4.0
Gasoline Range Organics, AK101	8006-61-9	50	4.0
Gasoline Range Organics, Hawaii-Gx	STL00061	50	2.0
Gasoline Range Organics, CA 8015B	STL00215	50	4.0

[1] Reporting limits listed for soil/sediment are based on wet weight. The reporting limits calculated by the laboratory for soil/sediment, calculated on dry weight basis, will be higher.

Table 2. Surrogate Standards

Surrogate Compounds	Standard Concentration (mg/L)
Trifluorotoluene	4000
4-Bromofluorobenzene	4000

NOTES:

1) Recovery and precision limits for the surrogates are generated from historical data and are maintained by the QA department.

Table 3. LCS and Matrix Spike Compounds

Compound	Standard Concentration (mg/L)
Gasoline Range Organics	5500

NOTES:

1) Recovery and precision limits for the LCS, MS, and MSD are generated from historical data and are maintained by the QA department.

Company Confidential & Proprietary

Quality Assurance Manual

TestAmerica Seattle
5755 8th Avenue East
Tacoma, WA 98424
Phone No. (253) 922-2310
Fax No. (253) 922-5047

www.testamericainc.com

Copyright Information:
This documentation has been prepared by TestAmerica Laboratories, Inc. and its affiliates ("TestAmerica"), solely for their own use and the use of their customers in evaluating their qualifications and capabilities in connection with a particular project. The user of this document agrees by its acceptance to return it to TestAmerica upon request and not to reproduce, copy, lend, or otherwise disclose its contents, directly or indirectly, and not to use it for any other purpose other than that for which it was specifically provided. The user also agrees that where consultants or other outside parties are involved in the evaluation process, access to these documents shall not be given to said parties unless those parties also specifically agree to these conditions.

THIS DOCUMENT CONTAINS VALUABLE CONFIDENTIAL AND PROPRIETARY INFORMATION. DISCLOSURE, USE OR REPRODUCTION OF THESE MATERIALS WITHOUT THE WRITTEN AUTHORIZATION OF TESTAMERICA IS STRICTLY PROHIBITED. THIS UNPUBLISHED WORK BY TESTAMERICA IS PROTECTED BY STATE AND FEDERAL LAW OF THE UNITED STATES. IF PUBLICATION OF THIS WORK SHOULD OCCUR THE FOLLOWING NOTICE SHALL APPLY:

©COPYRIGHT 2012 TESTAMERICA LABORATORIES, INC. ALL RIGHTS RESERVED.

Facility Distribution No. Uncontrolled **Distributed To:**_____

Title Page:

Quality Assurance Manual
Approval Signatures

Signatures on File

Laboratory Director – Heather Prater	Date
Quality Assurance Manager – Terri Torres	Date
Environmental Health & Safety – Sandra Langway	Date
Project Manager Supervisor – Kris Allen	Date
Volatile Organic Technical Manager – Bisrat Tadesse	Date
Inorganic Technical Manager – Stan Palmquist	Date
Semivolatile Technical Manager – Colin McKean	Date

SECTION 2. TABLE OF CONTENTS

Sec. No.	Title	2009 TNI Standard Reference	ISO/IEC 17025:2005 (E) Reference	Page No.
-	QUALITY ASSURANCE MANUAL	V1M2 Sec. 4.2.8.3		1
1.0	TITLE PAGE			2
2.0	TABLE OF CONTENTS	V1M2 Secs. 4.2.8.3-4.2.8.4		3
3.0	INTRODUCTION, SCOPE AND APPLICABILITY	V1M2 Sec. 4.2.8.4		13
3.1	Introduction And Compliance References	V1M2 Secs. 1.1; 1.2; 2.0; 3.2; 4.1.2; 4.2.4	4.1.2; 4.2.4	13
3.2	Terms And Definitions	V1M2 Secs. 3.0; 4.2.4	4.2.4	13
3.3	Scope / Fields Of Testing	V1M2 Secs. 1.2; 4.2.4	4.1.2; 4.2.4	14
3.4	Management Of The Manual	V1M2 Secs. 4.2.1; 4.2.7; 4.3.3.2; 4.3.3.3	4.2.1; 4.2.7; 4.3.3.2; 4.3.3.3	14
4.0	MANAGEMENT REQUIREMENTS	V1M2 Sec. 4		15
4.1	Overview	V1M2 Secs. 4.1.1, 4.1.3; 4.1.5	4.1.1; 4.1.3; 4.1.5; 4.2.6	15
4.2	Roles And Responsibilities	V1M2 Secs. 4.1.4; 4.1.5; 4.1.6; 4.2.1; 4.2.6; 5.2.4	4.1.3; 4.1.5; 4.1.6; 4.2.1; 4.2.6; 5.2.4	15
4.3	Deputies	V1M2 Secs. 4.1.5; 4.1.7.2; 4.2.7	4.1.5; 4.2.7	23
5.0	QUALITY SYSTEM			27
5.1	Quality Policy Statement	V1M2 Secs. 4.1.5; 4.2.2; 4.2.3; 4.2.8.3	4.1.5; 4.2.2; 4.2.3	27
5.2	Ethics And Data Integrity	V1M2 Secs. 4.1.5; 4.16; 4.2.2; 4.2.8.1; 5.2.7	4.1.5; 4.2.2	27
5.3	Quality System Documentation	V1M2 Secs. 4.1.5; 4.2.2; 4.2.5	4.2.2; 4.2.5	28
5.4	QA/QC Objectives For The Measurement Of Data	V1M2 Sec. 4.2.2	4.1.5; 4.2.2	29
5.5	Criteria For Quality Indicators			31
5.6	Statistical Quality Control			31
5.7	Quality System Metrics			31
6.0	DOCUMENT CONTROL	V1M2 Secs. 4.2.7; 4.3.1; 4.3.2.2 ; 4.3.3.3; 4.3.3.4	4.2.7; 4.3.1; 4.3.2.2; 4.3.3.3; 4.3.3.4	32
6.1	Overview			32
6.2	Document Approval And Issue	V1M2 Secs. 4.3.2; 4.3.2.1-4.3.2.3; 4.3.3.1	4.3.2.1; 4.3.2.2; 4.3.2.3; 4.3.3.1	32

Sec. No.	Title	2009 TNI Standard Reference	ISO/IEC 17025:2005 (E) Reference	Page No.
6.3	Procedures For Document Control Policy	V1M2 Secs. 4.3.2.1–4.3.2.2; 4.3.3.1	4.3.2.1; 4.3.2.2; 4.3.3.1	33
6.4	Obsolete Documents	V1M2 Secs. 4.3.2.1–4.3.2.2	4.3.2.1; 4.3.2.2	33
7.0	SERVICE TO THE CLIENT	V1M2 Secs. 4.4.1 - 4.4.4	4.4.1; 4.4.2; 4.4.3; 4.4.4	33
7.1	Overview	V1M2 Secs. 4.4.5; 4.5.5; 5.7.1	4.4.5; 5.7.1	33
7.2	Review Sequence And Key Personnel	V1M2 Sec. 4.4.5	4.4.5	34
7.3	Documentation	V1M2 Sec. 5.7.1	5.7.1	35
7.4	Special Services	V1M2 Secs. 4.7.1-4.7.2	4.7.1; 4.7.2	36
7.5	Client Communication	V1M2 Secs. 4.7.1-4.7.2	4.7.1; 4.7.2	37
7.6	Reporting	V1M2 Secs. 4.7.1-4.7.2	4.7.1; 4.7.2	37
7.7	Client Surveys	V1M2 Secs. 4.7.1-4.7.2	4.7.1; 4.7.2	37
8.0	SUBCONTRACTING OF TESTS	V1M2 Secs. 4.4.3; 4.5.4	4.4.3; 4.5.4	37
8.1	Overview	V1M2 Secs. 4.5.1 - 4.5.3; 4.5.5; 5.3.1	4.5.1; 4.5.2; 4.5.3; 5.3.1	37
8.2	Qualifying And Monitoring Subcontractors	V1M2 Secs. 4.5.1; 4.5.2; 4.5.3; 4.5.5	4.5.1; 4.5.2; 4.5.3	38
8.3	Oversight And Reporting	V1M2 Sec. 4.5.5		39
8.4	Contingency Planning			40
9.0	PURCHASING SERVICES AND SUPPLIES	V1M2 Sec. 4.6.1	4.6.1	42
9.1	Overview	V1M2 Secs. 4.6.2; 4.6.3; 4.6.4	4.6.2; 4.6.3; 4.6.4	42
9.2	Glassware	V1M2 Sec. 5.5.13.1		42
9.3	Reagents, Standards & Supplies	V1M2 Secs. 4.6.2; 4.6.3; 4.6.4	4.6.2; 4.6.3; 4.6.4	42
9.4	Purchase Of Equipment / Instruments / Software			44
9.5	Services			45
9.6	Suppliers			45
10.0	COMPLAINTS	V1M2 Sec. 4.8	4.8	46
10.1	Overview			46
10.2	External Complaints			46
10.3	Internal Complaints			47
10.4	Management Review			47
11.0	CONTROL OF NON-CONFORMING WORK	V1M2 Secs. 4.9.1; 5.10.5	4.9.1; 5.10.5	47
11.1	Overview	V1M2 Secs. 4.9.1; 4.11.3; 4.11.5	4.9.1; 4.11.3; 4.11.5	47

Sec. No.	Title	2009 TNI Standard Reference	ISO/IEC 17025:2005 (E) Reference	Page No.
11.2	Responsibilities And Authorities	V1M2 Secs. 4.9.1; 4.11.3; 4.11.5; 5.2.7	4.9.1; 4.11.3; 4.11.5	48
11.3	Evaluation Of Significance And Actions Taken	V1M2 Secs. 4.9.1; 4.11.3; 4.11.5	4.9.1; 4.11.3; 4.11.5	48
11.4	Prevention Of Nonconforming Work	V1M2 Secs. 4.9.4; 4.11.2	4.9.2; 4.11.2	49
11.5	Method Suspension / Restriction (Stop Work Procedures)	V1M2 Secs. 4.9.1; 4.9.2; 4.11.5	4.9.1; 4.9.2; 4.11.5	49
12.0	CORRECTIVE ACTION	V1M2 Sec. 4.11		50
12.1	Overview	V1M2 Secs. 4.9.2; 4.11.1; 4.11.2	4.9.2; 4.11.1; 4.11.2	50
12.2	General	V1M2 Sec. 4.11.2; 4.11.3	4.11.2; 4.11.3	50
12.3	Closed Loop Corrective Action Process	V1M2 Sec. 4.11.2; 4.11.3; 4.11.4; 4.11.6; 4.11.7; 4.12.2	4.11.2; 4.11.3; 4.11.4; 4.12.2	51
12.4	Technical Corrective Actions	V1M2 Sec. 4.11.6		53
12.5	Basic Corrections	V1M2 Secs. 4.11.1; 4.13.2.3	4.11.1; 4.13.2.3	53
13.0	PREVENTIVE ACTION / IMPROVEMENT	V1M2 Secs. 4.10; 4.12.1; 4.12.2	4.10; 4.12.1; 4.12.2	58
13.1	Overview	V1M2 Secs. 4.15.1; 4.15.2	4.15.1; 4.15.2	58
13.2	Management Of Change			59
14.0	CONTROL OF RECORDS	V1M2 Secs. 4.2.7; 4.13.1.1; 4.13.3	4.2.7; 4.13.1.1	59
14.1	Overview	V1M2 Secs. 4.13.1.1; 4.13.1.2; 4.13.1.3; 4.13.1.4; 4.13.2.1; 4.13.2.2; 4.13.2.3; 4.13.3	4.13.1.1; 4.13.1.2; 4.13.1.3; 4.13.1.4; 4.13.2.1; 4.13.2.2; 4.13.2.3	59
14.2	Technical And Analytical Records	V1M2 Sec. 4.13.2.2 - 4.13.2.3	4.13.2.2; 4.13.2.3	62
14.3	Laboratory Support Activities			63
14.4	Administrative Records			64
14.5	Records Management, Storage And Disposal	V1M2 Sec. 4.13.3		64
15.0	AUDITS			65
15.1	Internal Audits	V1M2 Sec. 4.2.8.1; 4.14; 4.14.1; 4.14.2 ; 4.14.3; 4.14.5; 5.9.1; 5.9.2	4.14.1; 4.14.2; 4.14.3; 5.9.1; 5.9.2	65
15.2	External Audits	V1M2 Secs.4.14.2; 4.14.3	4.14.2; 4.14.3; 4.14.4	67

Sec. No.	Title	2009 TNI Standard Reference	ISO/IEC 17025:2005 (E) Reference	Page No.
15.3	Audit Findings	V1M2 Secs. 4.14.2; 4.14.3; 4.14.5		68
16.0	MANAGEMENT REVIEWS	V1M2 Sec. 4.1.6; 4.15; 4.15.1; 4.15.2	4.1.6; 4.15.1; 4.15.2	68
16.1	Quality Assurance Report			68
16.2	Annual Management Review	V1M2 Sec. 4.2.2; 4.15.3	4.2.2	69
16.3	Potential Integrity Related Managerial Reviews			70
17.0	PERSONNEL	V1M2 Secs. 5.2; 5.2.1	5.2.1	70
17.1	Overview	V1M2 Secs. 5.2.2; 5.2.3; 5.2.5	5.2.2; 5.2.3; 5.2.5	70
17.2	Education And Experience Requirements For Technical Personnel	V1M2 Secs. 5.2.1; 5.2.3; 5.2.4	5.2.1; 5.2.3; 5.2.4	71
17.3	Training	V1M2 Sec. 5.2.5	5.2.5	72
17.4	Data Integrity And Ethics Training Program	V1M2 Sec. 4.2.8.1; 5.2.7		73
18.0	ACCOMMODATIONS AND ENVIRONMENTAL CONDITIONS	V1M2 Sec. 5.3		74
18.1	Overview	V1M2 Secs. 5.3.1; 5.3.3; 5.3.4; 5.3.5	5.3.1; 5.3.3; 5.3.4; 5.3.5	74
18.2	Environment	V1M2 Secs. 5.3.1; 5.3.2; 5.3.3; 5.3.4; 5.3.5	5.3.1; 5.3.2; 5.3.3; 5.3.4; 5.3.5	75
18.3	Work Areas	V1M2 Secs. 5.3.3; 5.3.4; 5.3.5	5.3.3; 5.3.4; 5.3.5	75
18.4	Floor Plan			76
18.5	Building Security	V1M2 Sec. 5.3.4	5.3.4	76
19.0	TEST METHODS AND METHOD VALIDATION	V1M2 Sec. 5.4.1	5.4.1	65
19.1	Overview	V1M2 Sec. 5.4.1	5.4.1; 5.4.5.1	76
19.2	Standard Operating Procedures (Sops)	V1M2 Secs. 4.2.8.5; 4.3.3.1; 5.4.2	4.3.3.1; 5.4.2	76
19.3	Laboratory Methods Manual	V1M2 Sec. 4.2.8.5		77

Sec. No.	Title	2009 TNI Standard Reference	ISO/IEC 17025:2005 (E) Reference	Page No.
19.4	Selection Of Methods	V1M2 Secs. 4.13.3; 5.4.1; 5.4.2; 5.4.3. V1M4 Secs. 1.4; 1.5.1; 1.6.1; 1.6.2; 1.6.2.1; 1.6.2.2	5.4.1; 5.4.2; 5.4.3; 5.4.4; 5.4.5.1; 5.4.5.2; 5.4.5.3	77
19.5	Laboratory Developed Methods And Non-Standard Methods	V1M2 Sec. 5.4.2. V1M4 Sec. 1.5.1	5.4.2; 5.4.4; 5.4.5.2; 5.4.5.3	81
19.6	Validation Of Methods	V1M2 Sec. 5.4.2. V1M4 Secs. 1.5.1; 1.5.2; 1.5.2.1; 1.5.2.2; 1.5.3	5.4.2; 5.4.4; 5.4.5.2; 5.4.5.3	81
19.7	Method Detection Limits (mdl) / Limits Of Detection (LOD)	V1M2 Sec. 5.9.3. V1M4 Secs. 1.5.2; 1.5.2.1; 1.5.2.2	5.4.5.3	82
19.8	Instrument Detection Limits (Idl)	V1M2 Sec. 5.9.3		83
19.9	Verification Of Detection And Reporting Limits	V1M2 Sec. 5.9.3. V1M4 Sec. 1.5.2.1		83
19.10	Retention Time Windows	V1M2 Sec. 5.9.3		84
19.11	Evaluation Of Selectivity	V1M2 Sec. 5.9.3. V1M4 Sec. 1.5.4; 1.7.3.6		84
19.12	Estimation Of Uncertainty Of Measurement	V1M2 Sec. 5.1.1; 5.1.2; 5.4.6	5.1.1; 5.1.2; 5.4.6.1; 5.4.6.2; 5.4.6.3	84
19.13	Sample Reanalysis Guidelines	V1M2 Sec 5.9.1	5.9.1	85
19.14	Control Of Data	V1M2 Secs. 5.4.7.1; 5.4.7.2; 5.9.1	5.4.7.1; 5.4.7.2; 5.9.1	85
20.0	EQUIPMENT and CALIBRATIONS	V1M2 Secs. 5.5.4; 5.5.5; 5.5.6	5.5.4; 5.5.5; 5.5.6; 5.6.1	93
20.1	Overview	V1M2 Secs. 5.5.1; 5.5.2; 5.5.3; 5.5.5; 5.5.10	5.5.1; 5.5.2; 5.5.3; 5.5.5; 5.5.10; 5.6.1	93
20.2	Preventive Maintenance	V1M2 Secs. 5.5.1; 5.5.3; 5.5.7; 5.5.9	5.5.1; 5.5.3; 5.5.7; 5.5.9; 5.6.1	93
20.3	Support Equipment	V1M2 Secs. 5.5.10; 5.5.11; 5.5.13.1	5.5.10; 5.5.11; 5.6.2.1.2; 5.6.2.2.1; 5.6.2.2.2	94
20.4	Instrument Calibrations	V1M2 Secs. 5.5.8; 5.5.10; 5.6.3.1. V1M4 Sec. 1.7.1.1; 1.7.2	5.5.8; 5.5.9; 5.5.10; 5.6.1; 5.6.2; 5.6.3.1	96
20.5	Tentatively Identified Compounds (TICS) – GC/MS Analysis			99

Sec. No.	Title	2009 TNI Standard Reference	ISO/IEC 17025:2005 (E) Reference	Page No.
20.6	Gc/Ms Tuning			99
21.0	MEASUREMENT TRACEABILITY			104
21.1	Overview	V1M2 Sec. 5.6.3.1	5.6.2.1.2; 5.6.2.2.2; 5.6.3.1	104
21.2	NIST-Traceable Weights And Thermometers	V1M2 Secs. 5.5.13.1; 5.6.3.1; 5.6.3.2	5.6.3.1; 5.6.3.2	104
21.3	Reference Standards / Materials	V1M2 Secs. 5.6.3.1; 5.6.3.2; 5.6.3.3; 5.6.3.4; 5.6.4.1; 5.6.4.2; 5.9.1; 5.9.3	5.6.3.1; 5.6.3.2; 5.6.3.3; 5.6.3.4; 5.9.1	104
21.4	Documentation And Labeling Of Standards, Reagents, And Reference Materials	V1M2 Secs. 5.6.4.2; 5.9.3		105
22.0	SAMPLING			107
22.1	Overview	V1M2 Secs. 5.7.1; 5.7.3	5.7.1; 5.7.3	107
22.2	Sampling Containers			107
22.3	Definition Of Holding Time			108
22.4	Sampling Containers, Preservation Requirements, Holding Times			108
22.5	Sample Aliquots / Subsampling	V1M2 Sec. 5.7.1	5.7.1	108
23.0	HANDLING OF SAMPLES	V1M2 Sec. 5.8.1	5.8.1	108
23.1	Chain Of Custody (COC)	V1M2 Secs. 5.7.2; 5.7.4; 5.8.4; 5.8.7.5; 5.8.8; 5.9.1	5.7.2; 5.8.4; 5.9.1	108
0	Sample **Receipt**	V1M2 Secs. 5.8.1; 5.8.2; 5.8.3; 5.8.5; 5.8.7.3; 5.8.7.4; 5.8.7.5	5.8.2; 5.8.3	110
23.3	Sample Acceptance Policy	V1M2 Secs. 5.8.6; 5.8.7.2		111
23.4	Sample Storage	V1M2 Secs. 5.7.4; 5.8.4	5.8.4	111
23.5	Hazardous Samples And Foreign Soils			112
23.6	Sample Shipping	V1M2 Sec. 5.8.2	5.8.2	112
23.7	Sample Disposal			113
24.0	ASSURING THE QUALITY OF TEST RESULTS			119
24.1	Overview	V1M2 Secs. 5.9.2; 5.9.3	5.9.2	119
24.2	Controls	V1M2 Secs. 5.9.2; 5.9.3	5.9.2	119

Sec. No.	Title	2009 TNI Standard Reference	ISO/IEC 17025:2005 (E) Reference	Page No.
24.3	Negative Controls	V1M2 Secs. 5.9.2; 5.9.3 V1M4 Secs. 1.7.3; 1.7.3.1; 1.7.4.1	5.9.2	119
24.4	Positive Controls	V1M2 Secs 5.9.2; 5.9.3. V1M4 Secs. 1.7.3; 1.7.3.2; 1.7.3.2.1; 1.7.3.2.2; 1.7.3.2.3	5.9.2	120
24.5	Sample Matrix Controls	V1M2 Secs. 5.9.2; 5.9.3. V1M4 Secs. 1.7.3 ; 1.7.3.3; 1.7.3.3.1; 1.7.3.3.2; 1.7.3.3.3	5.9.2	122
24.6	Acceptance Criteria (Control Limits)	V1M2 Sec. 5.9.3. V1M4 Secs. 1.7.4.2; 1.7.4.3		122
24.7	Additonal Procedures To Assure Quality Control	V1M2 Sec. 5.9.3. V1M4 Sec. 1.7.3.4		125
25.0	REPORTING RESULTS			125
25.1	Overview	-V1M2 Secs. 5.10.1; 5.10.2; 5.10.8	5.10.1; 5.10.2; 5.10.8	125
25.2	Test Reports	V1M2 Secs. 5.10.1; 5.10.2; 5.10.3.1; 5.10.3.2; 5.10.5; 5.10.6; 5.10.7; 5.10.8; 5.10.10; 5.10.11	5.10.1; 5.10.2; 5.10.3.1; 5.10.3.2; 5.10.5; 5.10.6; 5.10.7; 5.10.8	125
25.3	Reporting Level Or Report Type	V1M2 Secs. 5.10.1; 5.10.7; 5.10.8	5.10.1; 5.10.7; 5.10.8	128
25.4	Supplemental Information For Test	V1M2 Secs. 5.10.1; 5.10.3.1; 5.10.5	5.10.1; 5.10.3.1; 5.10.5	128
25.5	Environmental Testing Obtained From Subcontractors	V1M2 Secs. 4.5.5; 5.10.1; 5.10.6	5.10.1; 5.10.6	129
25.6	Client Confidentiality	V1M2 Secs. 4.1.5; 5.10.7	4.1.5; 5.10.7	129
25.7	Format Of Reports	V1M2 Sec. 5.10.8	5.10.8	130
25.8	Amendments To Test Reports	V1M2 Sec. 5.10.9	5.10.1; 5.10.9	130
25.9	Policies On Client Requests For Amendments	V1M2 Secs. 5.9.1; 5.10.9	5.9.1; 5.10.1; 5.10.5; 5.10.9	130

LIST OF TABLES

Table No.	Title	2009 TNI Standard Reference	ISO/IEC 17025:2005 (E) Reference	Page No.
12-1	Example – General Corrective Action Procedures	V1M2 Sec. 4.11.6. V1M4 Sec. 1.7.4.1	4.11.2	55
14-1	Record Index		4.13.1.1	59
14-2	Example: Special Record Retention Requirements			61
15-1	Types Of Internal Audits And Frequency		4.14.1	66
20-1	Example: Instrumentation List		5.5.4; 5.5.5	99
20-2	Example: Software List			100
20-3	Example: Schedule Of Routine Maintenance			101
24-1	Example – Negative Controls			119
24-3	Sample Matrix Control			122

LIST OF FIGURES

Figure No.	Title	2009 TNI Standard Reference	ISO/IEC 17025:2005(E) Reference	Page No.
4-1	Corporate And Laboratory Organization Chart	V1M2 Sec. 4.1.5	4.1.3; 4.1.5; 4.2.6	255
8-1	Example - Subcontracted Sample Form			41
12-1	Example - Non Conformance Memo			54
19-1	Example - Demonstration Of Capability Documentation			91
19-2	Example: Work Flow			92
23-1	Example: Chain Of Custody (COC)			114
23-2	Example: Sample Acceptance Policy	V1M2 Sec. 5.8.6; 5.8.7.1. V1M4 Sec. 1.7.5		115
23-3	Example: Cooler Receipt Form		5.8.3	118

LIST OF APPENDICES

Appendix No.	Title	Page No.
1	Laboratory Floor Plan	132
2	Glossary/Acronyms	133
3	Laboratory Certifications, Accreditations, Validations	141
4	Summary Of Calibration, QC And Corrective Action Procedures	142

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
CA-Q-S-009	Root Cause Analysis
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
TA-QA-0528	Document Control
TA-QA-0529	Complaint Resolution
TA-QA-0530	Management of Change
TA-QA-0506	Archiving Reports and Report File Maintenance
TA-QA-0608	Employee Training Procedures
TA-QA-0500	Standard Operating Procedures
TA-QA-0617	Analyst Demonstration of Capability
TA-QA-0602	Method Detection Limit Studies and Limits of Detection
TA-IP-0226	Multi Incremental Subsampling of Soils and Sediments
TA-QA-0028	Subsampling of Solid Samples
TA-QA-0001	Sample Receipt & Login
TA-QA-0610	Laboratory Corrective Action Procedures
TA-QA-0635	Data Review
TA-QA-0014	Selection and Use of Laboratory Balances
TA-QA-0024	Use, Calibration and Maintenance of Laboratory Thermometers
TA-QA-0016	Volumetric Verification
TA-QA-0610	Laboratory Corrective Action Procedures
TA-QA-0619	Receipt, Storage and Verification of Standards
TA-QA-0600	Quality Control Charting and Establishing

SECTION 3. INTRODUCTION, SCOPE AND APPLICABILITY

3.1 Introduction and Compliance References

TestAmerica Seattle'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.
- 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 drinking water, effluent water, groundwater, hazardous waste, sludge, sediments and soils. The Quality Assurance Program contains specific procedures and methods to test samples of differing matrices for chemical and physical 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 the Laboratory Information Management System (LIMS)**.** 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 **annually** by senior laboratory management to assure that it reflects current practices and meets the requirements of the laboratory's clients and regulators as well as the CQMP. Occasionally, the manual may need changes in order to meet new or changing regulations and operations. The QA Manager will review the changes in the normal course of business and incorporate changes into revised sections of the document. All updates will be reviewed by the senior laboratory management staff. The laboratory updates and approves such changes according to our Document Control procedures (refer to SOP No. TA-QA-0528).

SECTION 4. MANAGEMENT REQUIREMENTS

4.1 Overview

TestAmerica Seattle 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 Executive 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 Seattle 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 Seattle laboratory.

4.2.2 Laboratory Director

TestAmerica Seattle's Laboratory Director is responsible for the overall quality, safety, financial, technical, human resource and service performance of the whole laboratory and reports to their respective GM. The Laboratory Director provides the resources necessary to implement and maintain an effective and comprehensive Quality Assurance and Data Integrity Program.

Specific responsibilities include, but are not limited to:

- Provides one or more technical managers for the appropriate fields of testing. The name(s) of the Technical Director will be included in the national database. If the Technical Director is absent for a period of time exceeding 15 consecutive calendar days, the Laboratory Director must designate another full time staff member meeting the qualifications of the Technical Director to temporarily perform this function. If the absence exceeds 65 consecutive calendar days, the primary accrediting authority must be notified in writing.
- 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 that 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.
- Reviews and approves the laboratory specific QAM, policies, SOPs prior to their implementation and ensures all approved procedures are implemented and adhered to.
- Pursues and maintains appropriate laboratory certification and contract approvals. Supports ISO 17025 requirements. Organizes bid activities for prospective new projects and clients.
- Ensures client specific reporting and quality control requirements are met.
- Captains the management team, consisting of the QA Manager, the Technical Director(s), and the Operations Manager as direct reports.
- Annually assesses the effectiveness of the QMP and QAM within the laboratory.

4.2.3 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 the QA officers to accomplish specific responsibilities, which include, but are not limited to:

- Compliance with ISO 17025
- Compliance with DoD ELAP
- 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 monitors 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.
- Evaluation of the thoroughness and effectiveness of training.

4.2.4 Technical Managers or Designees

The Technical Managers report directly to the Laboratory Director. He/she is 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:

- Compliance with ISO 17025
- Compliance with DoD ELAP

- 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.
- 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.
- Coordinates audit responses with the QA Manager.

4.2.5 LIMS Administration/IT Support

LIMS Administration and IT support are managed at the corporate level. In general, the duties of the corporate LIMS administration team consist of:

- Establishes and maintains the laboratory information system (LIMS) for tracking all samples in the laboratory.
- Updates and enhances LIMS.

- Develops expertise in the requirements described in Good Automated Laboratory Practices (GALP) EPA 2185, 1995 Edition, in order to ensure compliance.
- Programs and tests software modifications/changes.
- Coordinates testing to ensure that all LIMS software accurately performs its intended functions. Testing is performed and documented after installation or when modifications/ changes are made.
- 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.
- Develops and verifies security practices to assure the integrity of LIMS data. Identifies threats, potential threats, and future threats.
- Maintains awareness of any environmental conditions of the facility housing the LIMS that may compromise LIMS raw data and informs management.
- LIMS database back-up once daily.

4.2.6 Hazardous Waste Coordinator

The Hazardous Waste Coordinator reports directly to the Laboratory Director. The duties consist of:

- Managing laboratory generated hazardous waste in accordance with appropriate regulations.
- 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

4.2.8 Department Managers

The Department Manager duties at TestAmerica Seattle are performed by the Technical Managers. The responsibilities of the department managers are listed below:

- Ensure that analysts in their department adhere to applicable SOPs and the QA Manual. They perform frequent SOP and QA Manual review to determine if analysts are in compliance and if new, modified, and optimized measures are feasible and should be added to these documents.
- With regard to analysts, participates in the selection, training (as documented in Section 8.1), development of performance objectives and standards of performance, appraisal (measurement of objectives), scheduling, counseling, discipline, and motivation of analysts and documents these activities in accordance with systems developed by the QA and Personnel Departments. They evaluate staffing sufficiency and overtime needs. Training consists of familiarization with SOP, QC, Safety, and computer systems.

- Encourage the development of analysts to become cross-trained in various methods and/or operate multiple instruments efficiently while performing maintenance and documentation, self-supervise, and function as a department team.
- Provide guidance to analysts in resolving problems encountered daily during sample prep/analysis in conjunction with the QA Manager. Each is responsible for 100% of the data review and documentation, non-conformance and CPAR issues, the timely and accurate completion of performance evaluation samples and MDLs, for his/her department.
- Works with the Project Managers and Analysts/Technicians to ensure that the requirements of project are met in a timely manner.
- Ensure all logbooks are maintained, current, and properly labeled or archived.
- Responsible for meeting quality requirements and reporting all non-conformance conditions to the QA Manager and/or Laboratory Director.
- Ensure that preventive maintenance is performed and documented 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.
- Conduct efficiency and cost control evaluations on an ongoing basis to determine optimization of labor, supplies, overtime, first-run yield, capacity (designed vs. demonstrated), second- and third-generation production techniques/instruments, and long-term needs for budgetary planning.
- Develop, implement, and enhance calibration programs.
- Provide written responses and facilitates implementing corrective actions for external and internal audit issues.

4.2.9 Laboratory Analysts

Laboratory analysts are responsible for conducting analysis and performing all tasks assigned to them by the department manager, group leader or supervisor. The responsibilities of the analysts are listed below:

- Perform analyses by adhering to analytical and quality control protocols prescribed by current SOPs, this QA Manual, and project-specific plans honestly, accurately, timely, safely, and in the most cost-effective manner.
- Document standard and sample preparation, instrument calibration and maintenance, data calculations, sample matrix effects, and any observed non-conformance on worklists, benchsheets, lab notebooks and/or the Non-Conformance Database.
- Report all non-conformance situations, instrument problems, matrix problems and QC failures, which might affect the reliability of the data, to their supervisor, the Technical Director, and/or the QA Manager or member of QA staff.
- Ensures sample and data integrity by adhering to internal chain-of-custody procedures.

- Perform 100% review of the data generated prior to entering and submitting for secondary level review.
- Suggest method improvements to their supervisor, the Technical Director, and the QA Manager. These improvements, if approved, will be incorporated. Ideas for the optimum performance of their assigned area, for example, through the proper cleaning and maintenance of the assigned instruments and equipment, are encouraged.
- 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.

4.2.10 Safety Officer

The Safety Officer reports to the Laboratory Director and ensures that systems are maintained for the safe operation of the laboratory. The Safety Officer is responsible to:

- 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.
- Manages facility maintenance.

4.2.11 Sample Receiving Staff

The Sample Receiving Technicians report to the Sample Receiving Supervisor; the Supervisor reports to the Operations Manager. The responsibilities are outlined below:

- Ensures implementation of proper sample receipt procedures, including maintenance of chain-of-custody.
- Reports nonconformances associated with condition-upon-receipt of samples.
- Logs incoming samples into the LIMS.
- Ensures that all samples are stored in the proper environment.
- Ensure the verification of data entry from login.
- Responsible for meeting quality requirements including documenting preservation.
- Responsible for ensuring the timely and correct shipment of sample containers, including proper preservatives and instructions, to clients
- Assists Environmental Health and Safety staff with sample disposal.

4.2.12 Project Manager Supervisor

The Project Manager Supervisor reports to the Client Services 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.
- Has signature authority for laboratory reports.
- Assesses and assures customer satisfaction.
- Provides feedback to management on changing customer needs.
- Works with the Department Managers and/or Analysts/Technicians to ensure the requirements of projects are met in a timely manner.
- Organizes bid activities for prospective new projects and clients.

4.2.13 Project Managers

The Project Managers report to the Customer Service Manager and serve as the interface between the laboratory and the clients. With the overall goal of total client satisfaction, the functions of this position are outlined below:

- Responsible to ensure that clients receive the proper sampling supplies.
- Accountable for response to client inquiries concerning sample status.
- Responsible for assistance to clients regarding the resolution of problems concerning COC.
- Ensuring that client specifications, when known, are met by communicating project and quality assurance requirements to the laboratory. Prepares a Quality Assurance Summary (QAS) or equivalent summary form as needed.
- 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.
- Approves customer requested variances to methods and to standard laboratory protocols.
- Responsible for discussing with client any project-related problems, resolving service issues, and coordinating technical details with the laboratory staff.
- Reports client inquiries involving data quality issues or data acceptability to the facility, CSM, Laboratory Director, QA Manager, and to the appropriate 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. Reviews project data packages for completeness and compliance to client needs and have signature authority for final reports.
- Inform clients of data package-related problems and resolve service issues.
- Coordinate requests for sample containers and other services (data packages).
- Prepares re-issue requests for project data.
- Organizes bid activities for prospective new projects and clients.
- Has signature authority for laboratory reports.

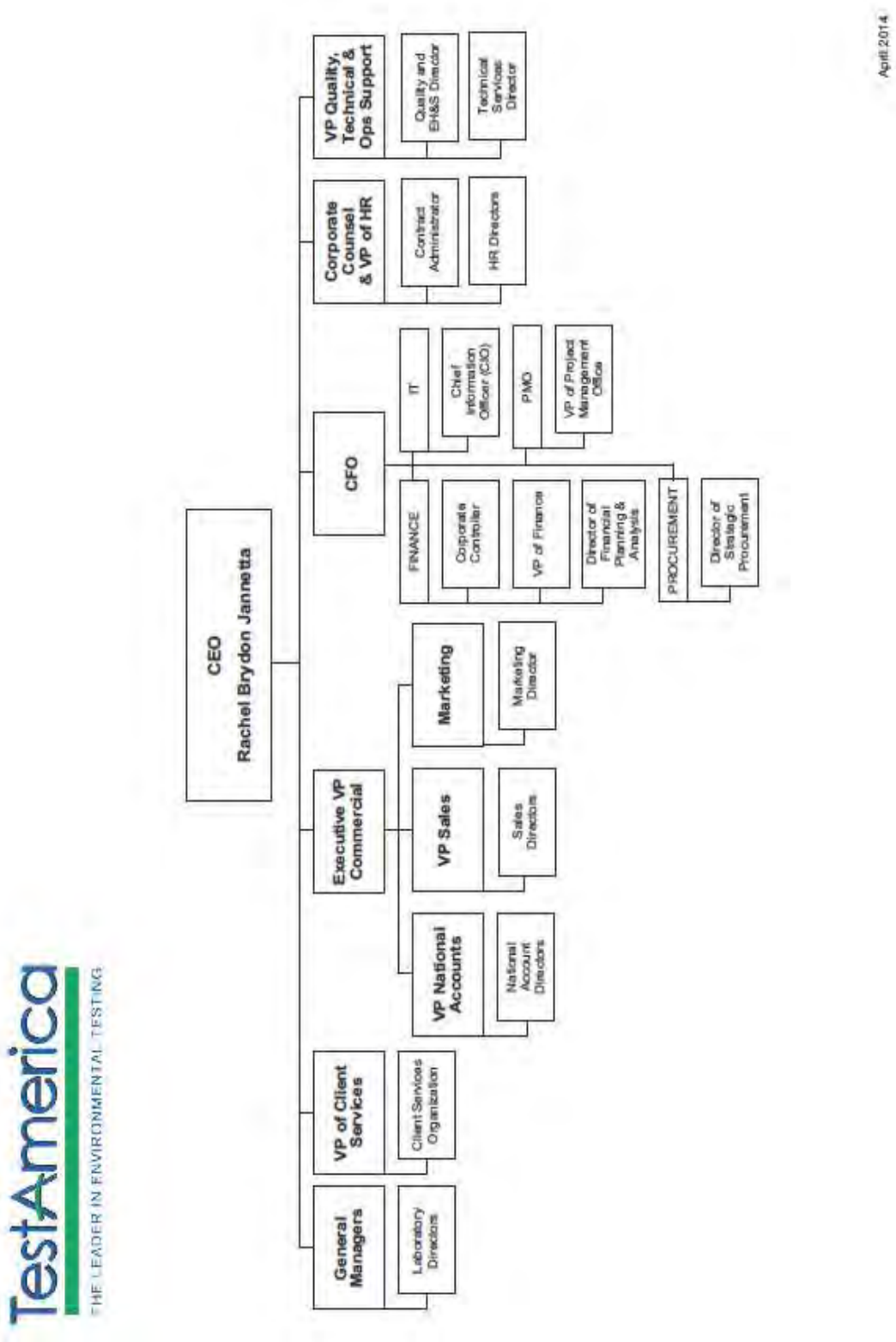
4.3 Deputies

The following table defines who assumes the responsibilities of key personnel in their absence:

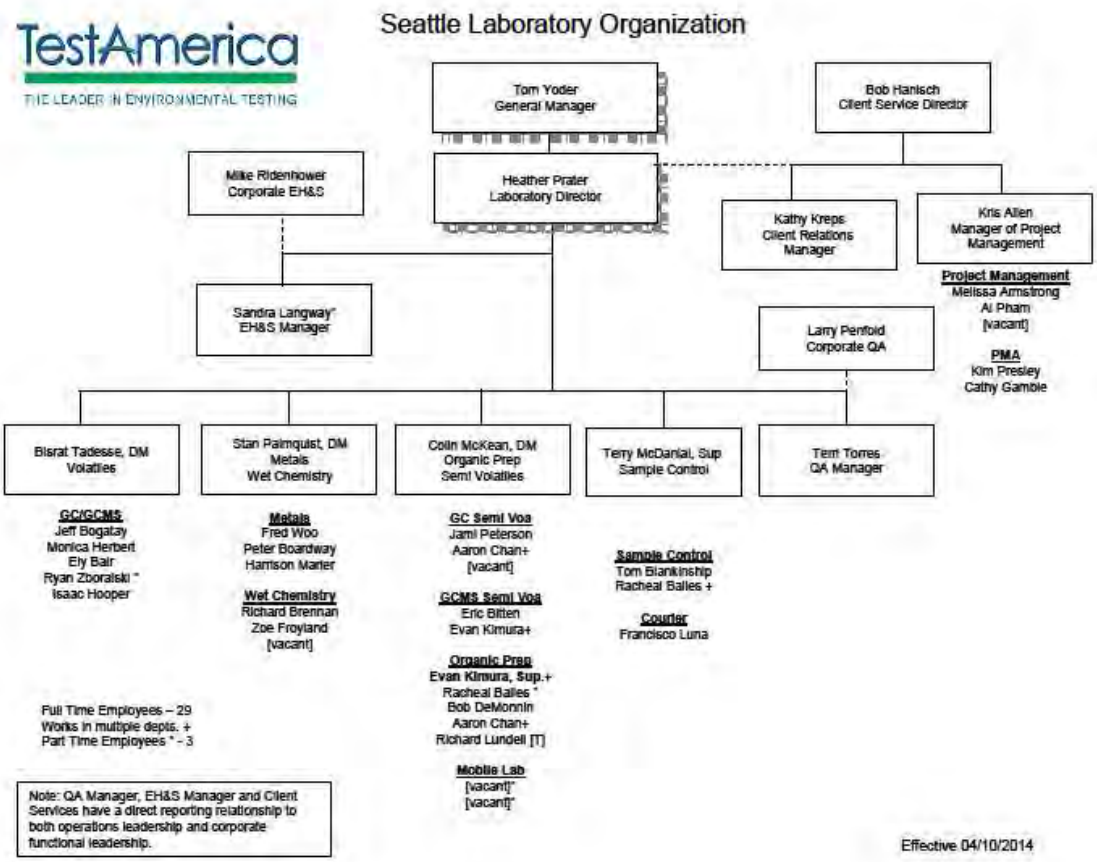
Key Personnel	Deputy
Laboratory Director	QA Manager
QA Manager	Laboratory Director
Inorganic Technical Manager	Laboratory Director

Key Personnel	Deputy
Semivolatile Technical Manager	Laboratory Director
Volatile Technical Manager	Laboratory Director
EHS Coordinator	Laboratory Director
Project Manager Supervisor	Laboratory Director

Figure 4-1. Corporate and Laboratory Organization Charts



TestAmerica Seattle
Organization Chart



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 in the Laboratory Information Management System 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 SOP TA-QA-0600 Control Charting and Establishing Method Warning and Action Limits.

5.6 Statistical Quality Control

Statistically-derived precision and accuracy limits are required by selected methods (such as SW-846) and programs [such as the Ohio Voluntary Action Plan (VAP)]. The laboratory routinely utilizes statistically-derived limits to evaluate method performance and determine when corrective action is appropriate. The analysts are instructed to use the current limits in the laboratory (dated and approved by the 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 laboratory via the 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

As the QC limits are calculated, QC charts are generated showing warning and control limits for the purpose of evaluating trends. The QA Manager evaluates these to determine if adjustments need to be made or for corrective actions to methods. All findings are documented and kept on file. (Refer to SOP TA-QA-0600 Control Charting and Establishing Method Warning and Action Limits.)

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 procedures are defined in SOPs TA-QA-0528, (Document Control) and TA-QA-0506 (Document Archiving).

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 nonconformance memos/corrective action reports. 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 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 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 No. TA-QA-0528 (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 QAM folder for the applicable revision.

For changes to SOPs, refer to SOP No. TA-QA-0500, Standard Operating Procedures. 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 TA-QA-0528 (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. TA-QA-0528 (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
- Customer Services Manager
- Laboratory Operations Manager
- Laboratory and/or Corporate Technical Managers / Directors
- Corporate Information Technology Managers
- Account Executives
- Laboratory and/or Corporate Quality
- Laboratory Environmental Health and Safety Managers
- The Laboratory Director reviews the formal laboratory quote and makes final acceptance for their facility.

The Sales Director, Legal Contracts Director or Account Executive then submits the final proposal to the client.

In the event that one of the above personnel is not available to review the contract, his or her back-up will fulfill the review requirements.

The Legal & Contracts Director maintains copies of all signed contracts**.** The Laboratory Director also maintains copies of all signed contracts on site.

7.3 Documentation

Appropriate records are maintained for every contract or work request. All stages of the contract review process are documented and include records of any significant changes. The reviewed contracts or QAPPs, along with records identifying issues or changes (at a minimum the Additional Project Notes from the quote) will be scanned and stored on the Public server in the QA folder.

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 and all actions agreed upon between the PM and the client are officially documented with a follow-up email.

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 communicated to the laboratory during production or status meetings. These changes are also updated in 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 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).

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.

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 develop 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-NELAC accredited work where required.

Project Managers (PMs), Project Manager Supervior, 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 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;
- 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, 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 or CSM responsible for the project must advise and obtain client consent to the subcontract as appropriate, and provide the scope of work to ensure that the proper requirements are made a part of the subcontract and are made known to the subcontractor.

Prior to sending samples to the subcontracted laboratory, the PM confirms their certification status to determine if it's current and scope-inclusive. The information is documented on a Subcontracted Sample Form (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-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**_____
- 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 completes the Material Request Sheet when requesting reagents, standards, or supplies that are not stocked in on-site consignment system that contains items approved for laboratory use.

The analyst must provide the master item number (from the master item list that has been approved by the Technical Manager), 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 Manager prior to placing the order. The authorized requisitioning agent places the order.

9.3.2 Receiving

It is the responsibility of the receiving clerk 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 QA office**.**

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. 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 megohm-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 Manager 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 stored as scanned images in the LIMS Reagent module. 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 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 Technical 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 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 the 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.

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 Manager 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 laboratory SOP TA-QA-0529 (Client Complaint Resolution).

10.2 External Complaints

An employee that receives a complaint initiates the complaint resolution process by first documenting the complaint according to SOP TA-QA-0529.

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 supervisor for resolution. The supervisor may elect to discuss it with the Technical 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 laboratory's 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 Technical Manager 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-NELAC 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, a 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, 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), Corporate Quality Director 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 and Operations Manager.

The Laboratory Director and/or Operations Manager 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, Operations Manager, Technical 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 Memoranda (NCM) and Corrective Action Reports (CAR) (refer to SOP No. TA-QA-601 and 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 Memorandum (NCM) - is used to document the following types of corrective actions:

- Deviations from an established procedure or SOP
- QC outside of limits (non-matrix related)
- 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 NCMs.
- Issues found while reviewing NCMs that warrant further investigation.
- Internal and external audit findings.
- Failed or unacceptable PT results.
- Corrective actions that cross multiple departments in the laboratory.
- Systematic reporting / calculation errors
- Client complaints
- Data recall investigations
- Identified poor process or method performance trends
- Excessive revised reports
- Health and Safety violations

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. (refer to SOP No. TA-QA-0610 for more detail)

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 Technical 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.

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 arrive at the root cause. Also, 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, thus curtailing issues that could plague the laboratory or operation. (refer to SOP No. CA-Q-S-009 for more detail)

12.3.4 Monitoring of the Corrective Actions

- The Technical 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 Operations Manager and Laboratory Director to ensure final acceptable resolution is achieved and documented appropriately.
- Each NCM and CAR is entered into a database for tracking purposes and a monthly summary of all corrective actions is printed out for review to aid in ensuring that the corrective actions have taken effect.
- The QA Manager reviews monthly NCMs and 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 NCM.

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 and 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 - Non Conformance Memo

01/09/2008 **NonConformance Memo Report** Page 1 of 1

NCM ID: 580-5190 **Date Opened:** 11/30/2007

NCM Type: CCV - %D, High, Sample ND **Date Closed:**

Lab Section: Pesticides and PCBs **Date Verified:**

Narrative

The closing continuing calibration verification (CCV) for Endosulfan II recovered above the upper control limit. The samples associated with this CCV were non-detects for the affected analytes; therefore, the data have been reported.

Affected Items

Description	Project Manager
580-8247-E-1-A	Curbow, Heather
LCS 580-26165/2-A	Curbow, Heather
580-8247-C-2-A	Curbow, Heather
580-8247-E-1-A	Curbow, Heather
LCS 580-26165/2-A	Curbow, Heather
MB 580-26165/1-A	Curbow, Heather
LCSD 580-26165/3-A	Curbow, Heather
580-8247-C-2-A	Curbow, Heather
MB 580-26165/1-A	Curbow, Heather
LCSD 580-26165/3-A	Curbow, Heather

Notifications

User Full Name	Date Received	Date	Notice Level	Verification Type
Mott, Christina M	11/30/2007		Level 1	Review

Job Case Narratives

Job: 580-J8247-1

Job Narrative
580-J8247-1

Comments
No additional comments.

Receipt
All samples were received in good condition within temperature requirements.

GC/MS VOA
No analytical or quality issues were noted.

GC/MS Semi VOA
No analytical or quality issues were noted.

GC Semi VOA
Method 8081: The RPD between the results of the two columns for heptachlor and DDT was >40%. This is due to matrix effect; the lower of the two values (presumably free from matrix interference) is reported as primary.

No other analytical or quality issues were noted.

Metals
No analytical or quality issues were noted.

General Chemistry
No analytical or quality issues were noted.

Organic Prep
No analytical or quality issues were noted.

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, Technical Manager(s))	- 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, Technical Manager(s))	- % 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 LIMS.	- 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 LIMS.	- 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, Technical 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.
Internal / External Audits (QA Manager, Technical 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.

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, Technical Managers, 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 Lab SOP TA-QA-0610 Laboratory Corrective Action Procedures.
Client Complaints (Project Managers, Lab Director, 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, Technical Manager(s))	- 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, Technical Manager(s))	- 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 or for DoD projects the method blank should be below ½ the RL. 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 QA 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 procedures, 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 changes covered under this system include: Facility Changes, Major Accreditation Changes, Addition or Deletion to Division's Capabilities or Instrumentation, Key Personnel Changes, Laboratory Information Management System (LIMS) changes. This process is discussed in further detail in SOP TA-QA-0530, Management of Change.

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, in this instance the electronic copy will be considered to be the vital record). Technical records are maintained by the Technical Managers.

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*

	Record Types [1]:	**Retention Time:**
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, Permits	**Indefinitely**
	Employee Handbook	Indefinitely
	Personnel files, Employee Signature & Initials, Administrative Training Records (e.g., Ethics)	Refer to HR Manual
	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 or the Iron Mountain data storage facility that provide 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 archived off-site are stored in a secure location where a record is maintained of any entry into the storage facility. Whether on-site or off-site storage is used, logs are maintained in each storage box to note removal and return of records. Retention of records are maintained on-site at the laboratory for at least 6 months after their generation and moved

offsite for the remainder of the required storage time. Records are maintained for a minimum of five years unless otherwise specified by a client or regulatory requirement.

For raw data and project records, record retention shall be calculated from the date the project report is issued. For other records, such as Controlled Documents, QA, or Administrative Records, the retention time is calculated from the date the record is formally retired. Records related to the programs listed in Table 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)
Alaska	10 years
Louisiana – All	10 years
Navy Facilities Engineering Service Center (NFESC)	10 years
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. Additional information can also be reference in SOP TA-QA-0506 Archiving Data and Reports.

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 COC is stored with the invoice and the work order

sheet generated by the LIMS in a job folder. The chain of custody would indicate the name of the sampler. If any sampling notes are provided with a work order, they are kept with this package.

- 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 as per SOP TA-QA-0506 Archiving Reports and Report File Maintenance). Instrument data is stored sequentially by instrument. A given day's analyses are maintained in the order of the analysis. Run logs are maintained for each instrument or method; a copy of each day's run log or instrument sequence is stored with the data to aid in re-constructing an analytical sequence. Where an analysis is performed without an instrument, bound logbooks or bench sheets are used to record and file data. Standard and reagent information is recorded in logbooks or entered into the LIMS for each method as required.

- Changes to hardcopy records shall follow the procedures outlined in Section 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. The procedure for this verification can be found in SOP TA-QA-0506 Archiving Reports and Report File Maintenance.

- 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 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 or in the LIMS.
- Instrumentation identification and instrument operating conditions/parameters. Operating conditions/parameters are typically recorded in instrument maintenance logs where available.
- analysis type**;**
- all manual calculations and manual integrations**;**
- analyst's or operator's initials/signature**;**
- sample preparation including cleanup, separation protocols, incubation periods or subculture, ID codes, volumes, weights, instrument printouts, meter readings, calculations, reagents**;**
- test results**;**
- standard and reagent origin, receipt, preparation, and use**;**
- calibration criteria, frequency and acceptance criteria**;**
- data and statistical calculations, review, confirmation, interpretation, assessment and reporting conventions**;**
- quality control protocols and assessment**;**
- electronic data security, software documentation and verification, software and hardware audits, backups, and records of any changes to automated data entries; and
- Method performance criteria including expected quality control requirements. These are indicated both in the LIMS and on specific analytical report formats**.**

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 maintained in the LIMS – no logbooks are used to record that data**.** 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: **100% 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 NELAC 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, NELAC quality systems, client and state requirements, and the effectiveness of the internal controls of the analytical process, including but not limited to data review, quality controls, preventive action and corrective action. The completeness of earlier corrective actions is assessed 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 Method Audits

Method 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. Method audits will include all methods annually.

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 Technical Manager or qualified designee annually. 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, and Soil.

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 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, Technical Managers, Operation Manager, their Quality Director as well as the General Manager. All aspects of the QA system are reviewed to evaluate the suitability of policies and procedures. During the course of the year, the Laboratory Director, General Manager or Corporate QA may request that additional information be added to the report.

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, Technical Managers, Operation Manager, 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 is 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 Support, 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 Managers 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
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

Specialty	Education	Experience
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 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 violations). 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 Employee Training SOP (TA-QA-0608).

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 *fixed* laboratory is a 20,000 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 mobile laboratory is a 23 ft van or client provided work space designed to accommodate an efficient workflow and to provide a safe and comfortable work environment for employees.

The *fixed and mobile* laboratories *are* 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 *at fixed laboratory*), 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. *The fixed* laboratory HVAC and deionized water systems are designed to minimize potential trace contaminants.

The *fixed* 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 *fixed* 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 temperature levels in the laboratory.

When any of the method or regulatory required environmental conditions change to a point where they may adversely affect test results, analytical testing will be discontinued until the environmental conditions are returned to the required levels.

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

The fixed laboratory floor plan can be found in Appendix 1.

18.5 Building Security

Building/Van 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 the laboratory's SOP TA-QA-0500 Standard Operating Procedures.
- 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
- 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
- Plumb, Jr., R.H. 1981, Procedures for Handling and Chemical Analysis of Sediment and Water Samples, Technical Report EPA/C E-81-1. US Army Engineering Waterways Experiment Station, Vicksburg, MS.
- Recommended Protocols for Measuring Conventional Sediment Variables in Puget Sound, April 2003.
- Recommended Guidelines for Measuring Organic Compounds in Puget Sound Waters, Sediment and Tissue Samples, April 1997.
- Recommended Guidelines for Measuring Metals in Puget Sound Waters, Sediment and Tissue Samples, April 1997.

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 (Analyst DOC, Lab SOP # TA-QA-0617) 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 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 or the laboratory's SOP No. TA-QA-0602 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 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 TA-QA-0602 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. 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 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 uncertainties at approximately the 99% confidence level with a coverage factor of k = 3. As an example, for a reported result of 1.0 mg/l with an LCS recovery range of 50 to 150%, the estimated uncertainty in the result would be 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 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 Area Technical Manager or 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 SOP TA-IT-0523 Data Integrity and Security. The laboratory is currently running the TALS system 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 Sequel 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 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 Technical Director and 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 outlined in several SOPs and SOP TA-QA-0635 Procedure for Peer Review of Analytical Data and Project Management Review of Reports to ensure that reported

data are free from calculation and transcription errors and that QC parameters have been reviewed and evaluated before data is reported. The laboratory also utilizes corporate SOP CA-Q-S-002 Acceptable Manual Integration Practices to ensure the authenticity of the data. 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 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.

19.14.4.7 A visual summary of the flow of samples and information through the laboratory, as well as data review and validation, is presented in Figure 19-2.

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,

Document Uncontrolled When Printed

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

TestAmerica Seattle
Analyst Demonstration of Capability-Certification Statement

Initial: _____
Continuing: _____

Date of Preparation: _____ Date of Analysis: _____

Analyst: _____ Analyst: _____

Matrix: Water Soil Sediment Other (Specify):_____

Reference method:_____

Reference SOP:_____

Parameters:_____

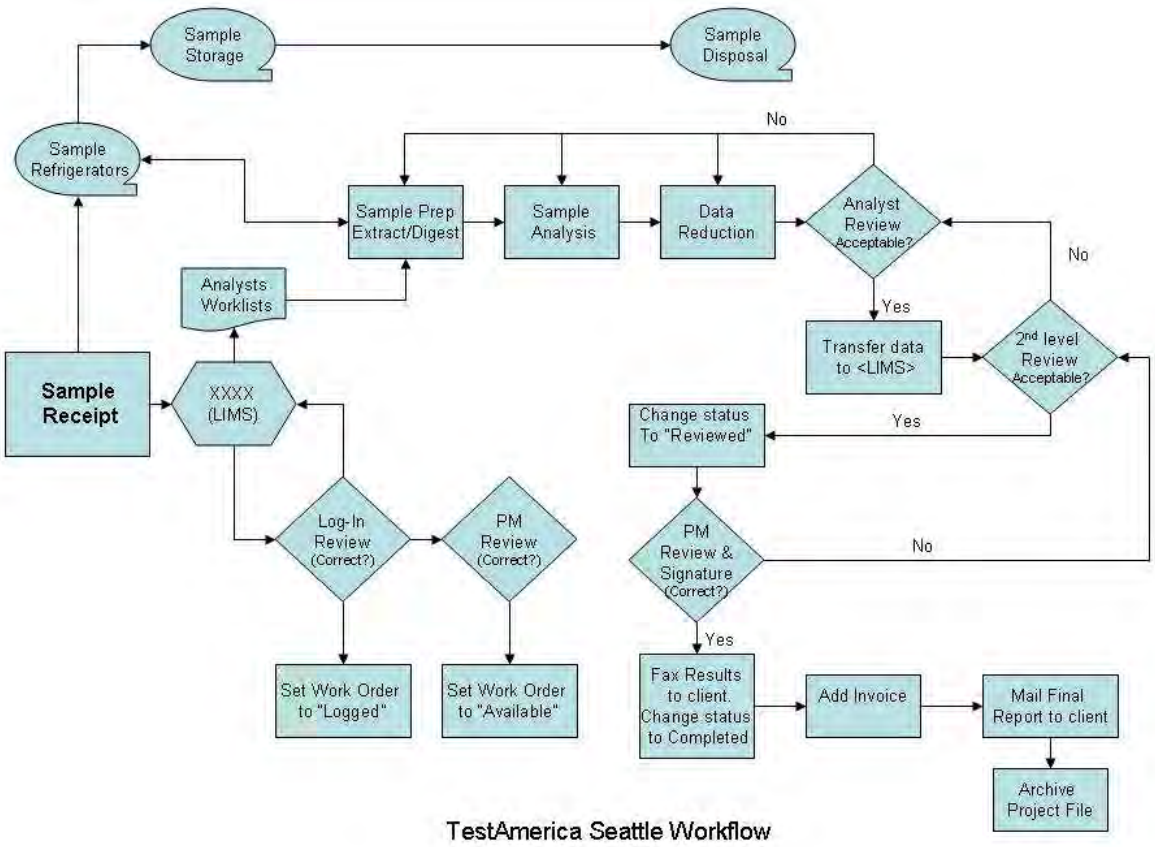
We, the undersigned, CERTIFY that:

- ❑ The analyst identified above, using the cited test method with the specification in the cited SOP, which is in use at this facility for the analyses of samples under the Nation Environmental Laboratory Accreditation Program, have met Demonstration of Capability.
- ❑ The test method was performed by the analyst identified on this certification following the TestAmerica Seattle SOP.
- ❑ A copy of the laboratory specific SOP is available for all personnel on-site.
- ❑ The data associated with the demonstration of capability are true, accurate, complete, and self-explanatory (*).
- ❑ All raw data including a copy of this certification form) necessary to reconstruct and validate these analyses have been retained at the facility, and that the associated information is well organized and available for review by authorized assessors.

Name	Analyst, Signature	Date
Name	Technical Director, Signature	Date
Name	QA Manager, Signature	Date

* True: consistent with supporting data
Accurate: Based on good laboratory practices with sound scientific principles/practices
Complete: includes the results of all supporting performance testing
Self-explanatory: Data properly labeled and stored so that the results are clean and require no additional explanation.

Figure 19-2. Example: Work Flow



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. A list of software is provided in Table 20-2.

Equipment is only operated by authorized and trained personnel. Manufacturer 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 20-3 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 are also outlined in analytical SOPs or instrument manuals. (Note: for some equipment, the log used to monitor performance is also the maintenance log. Multiple pieces of equipment may share the same log as long as it is clear as to which instrument is associated with an entry.)

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, 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 in the QA office. Refer to SOP TA-QA-0014 Selecting and Using Balances for details.

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 at temperatures bracketing the range of use. 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 SOP TA-QA-0024 Use, Calibration, and Maintenance of Laboratory Thermometers.

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. As a DOD-approved lab, sample storage is monitored 7 days a week.

Ovens, water baths and incubators are monitored on days of use.

All of this equipment has a unique identification number, and is assigned a unique thermometer for monitoring.

Sample storage refrigerator temperatures are kept between > 0ºC and ≤ 6 ºC.

Specific temperature settings/ranges for other refrigerators, ovens, water baths, 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 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 SOP TA-QA-0016 Volumetric Verification.

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.

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). 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.

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).

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 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 and reported 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. Alternatively, a reporting limit standard may be analyzed to demonstrate that the laboratory can still support non-detects at their reporting limit**.**

Samples reported by either of the conditions identified above will be appropriately flagged.

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

GC	GC/MS	Ion Trap GC/MS/MS	ICP	ICPMS	CVAA	AutoAnalyzer	IC	TOC
13	15	2	2	2	2	2	2	2

Table 20-2. Example: Software List

Lab Section	Software Title and Version
All (Data Processing and Reporting)	LIMS - TALS (TestAmerica) Ver 1.0.1.330 or higher
ECD, Fuels 8270 and VOA (Data Processing)	CHROM (TestAmerica) Ver 1.2 or higher
Data Acquistion	
ECD	Environmental Chemstation G1701DA Version D.00.01.27
	Envronmental Chemstation MSD Chemstation D.02.00.275
FUELS	Environmental Chemstation G1701DA Version D.00.01.27
	EnviroQuant ChemStation G1701BA Version B.01.00.
8270	Environmental ChemStation MSD ChemStation D.01.00 Build 75 26-Aug-2003.
	Environmental Chemstation G1701DA Version D.00.01.27 08-Nov-2002
	Environmental ChemStation MSD ChemStation E.01.00.237
	EnviroQuant ChemStation G1702BA Version B.01.00.
	Varian Saturn Software Data System
	Finnigan Magnum
	Finnigan GCQ
VOA	Environmental ChemStation MSD ChemStation D.01.00.275.
	EnviroQuant Chemstation G1701BA Version B.01.00.
	Environmental ChemStation MSD ChemStation E.02.00.493.
	Environmental ChemStation MSD ChemStation D.02.00.275.
	Environmental ChemStation MSD ChemStation D.03.00.552.
	Environmental ChemStation G1701DA Version D.00.01.27.
	EnviroQuant ChemStation G1701AA Version A.03.00.
	Environmental ChemStation G1701CA Version C.00.00 21-Dec-1999
Metals	ELAN Version 3.0 Hotfix 3 (Build 3, 0, 6, 48d)
	Agilent G1834B ICP-MS ChemStation B.03.03
	Perkin Elmer WinLab32 for ICP Version 4.0.0.0305
	Thermo iTeVA – Analyst Version 2.2.0.51
	Perkin Elmer WinLab32 for ICP Version 4.0.0.0305
	WinHg Runner 1.4 CT Rev 0.286
Wet Chem	
Lachat QuikChem	Omnion Program version 3.0.220.04
Astoria Pacific	EnviroQuant Chemstation G1701BA Version B.01.00
Dionex DX-500	Dionex Peaknet Run PeakNet 5.11
Dionex ICS 2000	Chromeleon Client Version 6.60 Build1428
Mitsubishi TOX-100	TOX-100 Versions 2.17 Mitsubishi Chemical Corp
OI Analytical	OI Analytical WinTOC for Solids 3.0
Teledyne Tekmar	TOCTalk Ver 4.2.105.
LECO C632	*LECO C632 Carbon Determination Ver 1.60*

Table 20-3. Example: Schedule of Routine Maintenance

Instrument	Procedure	Frequency
Leeman Mercury Analyzer	Check tubing for wear Fill rinse tank with 10% HCl Change drying tube Fill reductant bottle with 10% Stannous Chloride	Daily Daily As required 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 Weekly Weekly Daily Weekly Weekly Monthly Monthly Monthly Monthly
Ion chromatograph	Check seals for leakage Replace seals/valves/lamps Replace suppressor Replace column Clean source/analyzer	Daily As required As required As required As required
UV-Vis Spectrophotometer	Clean ambient flow cell Precision check/alignment of flow cell Wavelength verification check	As required As required Semi-annually
TOC Analyzer	Check gas flow Check fluid level (IC reservoirs) Replace “O” rings Check needle Replace scrubbers (halogen and CO_2) Replace catalyst	Daily Daily As required Daily Yearly As required
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

Instrument	Procedure	Frequency
Hewlett Packard 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
Gas Chromatograph	Compare standard response to previous day or since last initial calibration Check carrier gas flow rate in column Check temp. of detector, inlet, column oven Septum replacement Check system for gas leaks with SNOOP Check for loose/frayed wires and insulation ½"Bake injector/column Change/remove sections of guard column Replace connectors/liners Change/replace column(s)	Daily Daily via use of known compound retention Daily As required W/cylinder change as required Monthly As Required As Required As Required As Required
Electron Capture Detector (ECD)	Detector wipe test (Ni-63) Detector cleaning	Semi-annually As required
Flame Ionization Detector (FID)	Detector cleaning	As required
Balances	Class "S" traceable weight check Clean pan and check if level Field service	Daily, when used Daily At least Annually
Conductivity Meter	0.01M KCl calibration Conductivity cell cleaning	Daily As required
Turbidimeter	Check light bulb	Daily, when used
Deionized/Distilled Water	Conductivity Point Sources Daily conductivity check Check deionizer light Monitor for VOA's System cleaning Replace cartridge & large mixed bed resins	Water Quality SOP UQA-035 Daily Daily As required As required
Drying Ovens	Temperature monitoring Temperature adjustments	Daily As required
Refrigerators/ Freezers	Temperature monitoring Temperature adjustment Defrosting/cleaning	Daily As required As required
Vacuum Pumps/ Air Compressor	Drained Belts checked Lubricated	Weekly Monthly Semi-annually
pH/Specific Ion Meter	Calibration/check slope Clean electrode	Daily As required

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
Zero Headspace Extractors	Verify rotation speed Check for leakage Vendor repair	As required Annually As required
TCLP Extractors	Verify rotation speed	Quarterly

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, 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), or another accreditation organization that is a signatory to a MRA (Mutual Recognition Arrangement) of one or more of the following cooperations – ILAC (International Laboratory Accreditation Cooperation) or APLAC (Asia –Pacific Laboratory Accreditation Cooperation). A calibration 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 reference standards, to the extent available, are purchased from vendors that are accredited to ISO Guide 34 and ISO/IEC 17025. All reference standards from commercial vendors shall be accompanied with a certificate that includes at least the following information:*

- *Manufacturer*
- *Analytes or parameters calibrated*
- *Identification or lot number*
- *Calibration method*
- *Concentration with associated uncertainties*
- *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. 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 has 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 LIMS. 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. These records are maintained in the LIMS. For detailed information on documentation and labeling, please refer to SOP TA-QA-0619, Preparation, Storage, and Verification of Standards.

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 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 (from LIMS)
- 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 in the LIMS and MSD Sheets**.**

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 laboratory SOP TA-QA-0619, Preparation, Storage, and Verification of Standards.

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 SOP TA-QA-0028, Subsampling of Solid Samples.

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 the 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.

23.2 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 on an NCM or the login Checklist in LIMS 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):

Example: 580 - 9608 - A - 1

Location ID | Login ID | Container Occurrence | Sample Number

In the example above, the Location ID code identifies the lab (580 = TestAmerica Seattle), the Login ID or Job Number (unique to a particular client/job occurrence) is 9608 and 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: 580 - 9608 - A - 1 - **A** ← **Secondary Container Occurrence**

Example: 580-9608-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);
- 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 TA-QA-0001 (Sample Receiving and Login).

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. Aqueous samples for metals testing are typically stored at ambient temperature**.** 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. The refrigerator blanks are logged into the LIMS and treated as normal samples with the data stored and archived in the LIMS.

Analysts and technicians retrieve the sample container allocated to their analysis from the designated storage location and place them on carts, analyze the sample, and return the remaining sample or empty container to the storage location 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 designated storage locations for two to four weeks after analysis, which meets or exceeds most sample holding times. After two to four weeks the samples are moved to dry room temperature, sample archive area where they are stored for an additional four weeks before they are disposed of. This eight week holding period allows samples to be checked if a discrepancy or question arises. Special arrangements may be made to store samples for longer periods of time. This extended holding period allows additional metal analyses to be performed on the archived sample and assists clients in dealing with legal matters or regulatory issues**.**

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 isolated areas designated for hazardous waste or foreign soil samples only. 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. Foreign soil samples are sent out for incineration by a USDA-approved waste disposal facility.

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: TA-EHS-0036). All procedures in the laboratory Environmental, Health and Safety Manual are followed during disposal. Samples are normally maintained in the laboratory no longer than ninety days from receipt unless otherwise requested. Unused portions of samples found or suspected to be hazardous according to state or federal guidelines may be returned to the client upon completion of the analytical work.

If a sample is part of a known litigation, the affected legal authority, sample data user, and/or submitter of the sample must participate in the decision about the sample's disposal. All documentation and correspondence concerning the disposal decision process must be kept on file. Pertinent information includes the date of disposal, nature of disposal (such as sample depletion, hazardous waste facility disposal or return to client), names of individuals who conducted the arrangements and physically completed the task. The laboratory will remove or deface sample labels prior to disposal unless this is accomplished through the disposal method (e.g., samples are incinerated). A Waste Disposal Record should be completed.

Figure 23-1. Example: Chain of Custody (COC)

Seattle
5755 8th Street East
Tacoma, WA 98424
253-922-2310

Chain of Custody Record

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

TestAmerica Laboratories, Inc.

Client Contact:	Project Manager:	Site Contact:	Date:	COC No:
Your Company Name here	Tel/Fax:	Lab Contact:	Carrier:	_____ of _____ COCs
Address:	Analysis Turnaround Time			Job No:
City/State/Zip:	Calendar (C) or Work Days (W) _____			
(xxx) xxx-xxxx Phone:	TAT if different from Below _____			
(xxx) xxx-xxxx FAX:	☐ 2 weeks			SDG No:
Project Name:	☐ 1 week			
Site:	☐ 2 days			
P O #	☐ 1 day			

Sample Identification	Sample Date	Sample Time	Sample Type	Matrix	# of Cont.	Filtered Sample																Sample Specific Notes:

Preservation Used: 1= Ice, 2= HCl; 3= H2SO4; 4=HNO3; 5=NaOH; 6= Other _____

Possible Hazard Identification
☐ *Non-Hazard* ☐ *Flammable* ☐ *Skin Irritant* *Poison B* ☐ *Unknown* ☐

Sample Disposal (A fee may be assessed if samples are retained longer than 1 month)
☐ *Return To Client* ☐ *Disposal By Lab* ☐ *Archive For* _____ *Months*

Special Instructions/QC Requirements & Comments:

Relinquished by:	Company:	Date/Time:	Received by:	Company:	Date/Time:
Relinquished by:	Company:	Date/Time:	Received by:	Company:	Date/Time:
Relinquished by:	Company:	Date/Time:	Received by:	Company:	Date/Time:

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.
   - 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. See Lab Sampling Guide.

4) Samples must be preserved according to the requirements of the requested analytical method (See Sampling Guide**.**

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 $\leq$ 10 ^{o}C), the samples must arrive within $\pm$ 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 (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 (Monday unless a holiday). Samples will remain refrigerated and sealed until the time of analysis. The actual times of all "field" sample analyses are noted on the "Short Hold Time Detail Report" 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 and do not seal very well and are prone to intrusion from the water from melted ice.
- Water samples would be best if wrapped with bubble-wrap or paper (newspaper, or paper towels work) and then placed in plastic zip-lock bags.
- Fill extra cooler space with bubble wrap.

Figure 23-3. Example: Cooler Receipt Form

Login Sample Receipt Checklist

Client: Client X

Job Number: 580-43036-1

Login Number: 43036
List Number: 1
Creator: Blankinship, Tom X

List Source: TestAmerica Seattle

Question	Answer	Comment
Radioactivity wasn't checked or is </= background as measured by a survey meter.	True	
The cooler's custody seal, if present, is intact.	True	
Sample custody seals, if present, are intact.	True	
The cooler or samples do not appear to have been compromised or tampered with.	True	
Samples were received on ice.	True	
Cooler Temperature is acceptable.	True	
Cooler Temperature is recorded.	True	
COC is present.	True	
COC is filled out in ink and legible.	True	
COC is filled out with all pertinent information.	True	
Is the Field Sampler's name present on COC?	True	
There are no discrepancies between the containers received and the COC.	True	
Samples are received within Holding Time.	True	
Sample containers have legible labels.	True	
Containers are not broken or leaking.	True	
Sample collection date/times are provided.	True	
Appropriate sample containers are used.	True	
Sample bottles are completely filled.	True	
Sample Preservation Verified.	True	
There is sufficient vol. for all requested analyses, incl. any requested MS/MSDs	True	
Containers requiring zero headspace have no headspace or bubble is <6mm (1/4").	N/A	
Multiphasic samples are not present.	True	
Samples do not require splitting or compositing.	True	
Residual Chlorine Checked.	N/A	

TestAmerica Seattle

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. Quality control samples are to be treated in the exact same manner as the associated field samples being tested. 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;
	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 185%.

- 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. See SOP TA-QA-0600 Quality Control Charting and Establishing Warning and Action Limits.

One example: The QA department generates a Quality Control Limit Summary that contains tables that summarize the precision and accuracy acceptability limits for analyses performed at TestAmerica Seattle. This summary includes an effective date, is updated each time new limits are generated and is located in the LIMS**.** Unless otherwise noted, limits within these tables are laboratory generated. The analysts are instructed to use the current limits in the laboratory (dated and approved by the Technical Director and QA Manager) and entered into the Laboratory Information Management System (LIMS). The Quality Assurance department maintains an archive of all limits used within the laboratory.

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. If agredd to by the client, 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.

Or, for NELAC and Department Of Defense (DOD) work, there are an allowable number of Marginal Exceedances (ME):

<11 analytes	0 marginal exceedances are allowed.
11 – 30 Analytes	1 marginal exceedance is allowed
31-50 Analytes	2 marginal exceedances are allowed
51-70 Analytes	3 marginal exceedances are allowed
71-90 Analytes	4 marginal exceedances are allowed
> 90 Analytes	5 marginal exceedances are allowed

- Marginal exceedances are recovery exceedances between 3 SD and 4 SD from the mean recovery limit (TNI).

- Marginal exceedances must be random. If the same analyte exceeds the LCS control limit repeatedly, it is an indication of a systematic problem. The source of the error must be located and corrective action taken. The laboratory has a system to monitor marginal exceedances to ensure that they are random.

Though marginal exceedences may be allowed, the data must still be qualified to indicate it is outside of the normal limits.

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 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.

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. job 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).

- Any COCs involved with Subcontracting are included.
- In most cases, the applicable COC is paginated as an integral part of the report.
- Any additional addenda to the report are also paginated as an integral part of the report (eg. Sample Receiving Checklist).

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 (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 statement that the report shall not be reproduced except in full, without prior express written approval by the laboratory coordinator.

25.2.19 A signature and title of the person(s) accepting responsibility for the content of the report and date of issue. Signatories are appointed by the Lab Director.

25.2.20 When NELAC 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.

25.2.21 Where applicable, a narrative to the report that explains the issue(s) and corrective action(s) taken in the event that a specific accreditation or certification requirement was not met.

25.2.22 When soil samples are analyzed, a specific identification as to whether soils are reported on a “wet weight” or “dry weight” basis.

25.2.23 Appropriate laboratory certification number for the state of origin of the sample, if applicable.

25.2.24 If only part of the report is provided to the client (client requests some results before all of it is complete), it must be clearly indicated on the report (e.g., preliminary). A complete report must be sent once all of the work has been completed.

25.2.25 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.

25.2.26 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 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 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.
- Level III contains all the information supplied in Level II, but presented on the CLP-like summary forms, and relevant calibration information. A Level II report is not included, unless specifically requested. No raw data is provided.
- Level IV is the same as Level III with the addition of all raw supporting data.

In addition to the various levels of QC packaging, the laboratory also provides reports in 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 Seattle offers a variety of EDD formats including Environmental Restoration Information Management System (ERPIMS), Staged Electronic Data Deliverable (SEDD) Environmental Quality Information System (EQuIS), Electronic Deliverable Format (EDF), Excel and custom 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 report is issued solely for the use of the person or company to whom it is addressed. Any use, copying or disclosure other than by the intended recipient is unauthorized. If you have received this report in error, please notify the sender immediately at 253-922-2310 and destroy this report immediately.

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 job number followed by the appropriate revision number. 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 "Revision" with the appropriate number 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

Key Areas *

1. Admin/HR
2. QA Offices
3. Project Management
4. Sample Control
5. Metals & Mercury Lab
6. Wet / General Chem
7. Metals Prep
8. Organics Prep
9. GC-MS SVOA
10. GC SVOA
11. GC & GC-MS VOA
12. Sample Disposal
13. GeoTech, Storage, Warehouse
14. Conference Room

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 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 ~~is~~ 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
LOQV - Limit of Quantitation Check Standard
MDL – Method Detection Limit
MDLV – MDL Verification Check Standard
MRL – Method Reporting Limit
MS – Matrix Spike
MSD – Matrix Spike Duplicate
MSDS - Material Safety Data Sheet
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 Seattle 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:

Organization	Lab ID Number
DoD ELAP	L2236
ISO 17025	L2236
Alaska	UST-022
California (NELAP)	01115CA
Montana	(UST – no number)
Oregon (NELAP)	WA100007
Washington	C553
USDA Soil Permit	P330-11-00222
USFWS Tissue Import Permit	LE192332-0

The certificates and parameter lists (which may differ) for each organization are available, upon request, from a laboratory representative or may be found on the corporate web site, the laboratory's public server, the final report review table, and in the following offices: QA, marketing, and project management.

Appendix 4: Summary of Calibration, QC and Corrective Action Procedures for GC Organics

Method	QC Check	Frequency	Acceptance Criteria[3]	Corrective Action[4]
SW8081A SW8082	Minimum five-point initial calibration for all target analytes[2]	Initial calibration prior to sample analysis. Perform instrument re-calibration once per year minimum.	Linear regression correlation coefficient $r \geq 0.990$, $r^2 \geq 0.990$. RSD of CF ≤ 20% **For DoD:** Linear regression correlation coefficient $r \geq 0.995$, $r^2 \geq 0.990$. RSD of CF ≤ 20%	Correct problem then repeat initial calibration
	Initial calibration verification (ICV) must be from a 2nd source	Once immediately following initial calibration	All target analytes within 15% of expected value. **For DoD:** All target analytes within 20% of expected value.	Correct problem then repeat initial calibration
	Continuing calibration verification (CCV)	Before sample analysis, after every 10 samples, and at the end of the analysis sequence	All analytes within 15% of expected value and within the RT Window. **For DoD:** All target analytes within 20% of expected value.	Correct problem then repeat initial CCV (re-calibrate if necessary) and re-analyze all samples since last successful CCV.
	Breakdown check (Endrin and DDT)[1]	Before sample analysis	Degradation ≤15% for either Endrin or DDT.	Inlet/column maintenance; repeat breakdown check and re-analyze all samples since last successful breakdown check.
	Method blank	One per analytical prep batch, not to exceed 20 samples in a batch.	No analytes detected ≥ ½ RL or MDL, whichever is greater[5]	Correct problem then re-prep[6] and analyze method blank and all samples processed with the contaminated blank
	LCS for all analytes, must be from a 2nd source.	One per prep batch, not to exceed 20 samples in a batch.	See Control Limits	Re-prep[6] and analyze the LCS and all samples in the affected analytical batch
	Surrogate(s)	Every sample, spike, standard, and method blank	See Control Limits	Check system, re-inject, re-extract[6]
	MS/MSD, must be from a 2nd source. Rotate Aroclors each quarter.	One per batch per matrix, if insufficient sample for MS/MSD, then a LCS/LCSD will be analyzed.	See Control Limits	None (LCS is used to determine if data is acceptable).
	Second-column confirmation	100% for all positive results	Same as for initial or primary column analysis	Same as for initial or primary column analysis. If the relative % difference of results between the 2 columns is greater than 40%, a comment should be placed in LIMS.
	Retention time window calculated for each analyte (see section 9 for how to calculate RTWs).	System set-up, with each new column or major instrument maintenance. Update the mid-RTW at the start of the run or daily.	Each analyte of the LCS, MS/MSD and CCV must be within the calculated RTW.	Correct the problem and re-process or re-analyze samples. If questions, see the supervisor or Technical Manager.
	MDL verification	Minimum yearly **For DOD:** Minimum quarterly	Detectable	Re-evaluate MDL standard used and MDL; see Technical Manager.
	LOQ verification	**For DOD:** Minimum quarterly	Detectable	Re-evaluate LOQ standard used and LOQ; see Technical Manager.

1 --8081A only

Document Uncontrolled When Printed

2 - Method 8082, a five-point calibration is only analyzed for Aroclors 1016 and 1260.
3 - This is a summary of the acceptance criteria, refer to the method SOP for specific or more information.
4 - All abnormalities must be noted in an NCM.
5 - Report all target compounds identified in the method blank above the MDL.
6 - If unable to re-extract the samples because of insufficient sample volume or holding time has expired, then place a comment on the benchsheet and in LIMS.

Company Confidential & Proprietary

Appendix 4: Summary of Calibration, QC and Corrective Action Procedures for GC Organics

Method	QC Check	Frequency	Acceptance Criteria[3]	Corrective Action[4]
EPA608	Minimum three-point (preferably five) initial calibration for all target analytes	Initial calibration prior to sample analysis. Perform instrument re-calibration once per year minimum.	**RSD of CF ≤ 10%** Linear regression - correlation coefficient r ≥ 0.995, r^2 ≥ 0.990.	Correct problem then repeat initial calibration
	Initial calibration verification (ICV) must be from a 2nd source.	Immediately following initial calibration	All analytes within 15% of expected value	Correct problem then repeat initial calibration
	Continuing calibration verification (CCV)	Before sample analysis, and at the end of the analysis sequence	All analytes within **15% of expected value** and within the RTW. 608 (not 608.2[5]) must be within 15% of the true value.	Correct problem then repeat initial CCV (re-calibrate if necessary) and re-analyze all samples since last successful CCV.
	Breakdown check (Endrin and DDT)[1]	Before sample analysis	Degradation ≤15% for either Endrin or DDT.	Inlet/column maintenance; repeat breakdown check and re-analyze all samples since last successful breakdown check.
	Method blank	One per analytical prep batch, **not to exceed 10 samples in a batch**.	No analytes detected ≥ ½ RL or MDL, whichever is greater[6]	Correct problem then re-prep[7] and analyze method blank and all samples processed with the contaminated blank
	LCS (QC check standard) must be from a 2nd source.	One per prep batch, **not to exceed 10 samples in a batch.**	See Control Limits	Re-prep[7] and analyze the LCS and all samples in the affected analytical batch
	Surrogate(s)	Every sample, spiked sample, standard, and method blank	See Control Limits	Check system, re-inject, re-extract[7]
	MS must be from a 2nd source.	One per batch per matrix, 10%, if insufficient sample for MS, then an additional LCS will be analyzed.	See Control Limits	All target compounds should be reported, and any compounds that are outside criteria must be within criteria in the LCS.
	Second-column confirmation	100% for all positive results	Same as for initial or primary column analysis	Same as for initial or primary column analysis. If the relative % difference of results between the 2 columns is greater than 40%, a comment should be placed in LIMS.
	Retention time window calculated for each analyte (see section 9 for how to calculate RTWs).	System set-up, with each new column or major instrument maintenance. Update the mid-RTW at the start of the run or as needed.	Each analyte of the LCS, MS/MSD and CCV must be within the calculated RTW.	Correct the problem and re-process or re-analyze samples. If questions, see the supervisor or Technical Manager.
	MDL verification	Minimum yearly	Detectable	Re-evaluate MDL standard used and MDL; see Technical Manager.

3 - This is a summary of the acceptance criteria, refer to the method SOP for specific or more information.
4 - All abnormalities must be noted in an NCM.
5 – 608.2 compounds are Methoxychlor and Permethrin.
6 - Report all target compounds identified in the method blank above the MDL.
7 - If unable to re-extract the samples because of insufficient sample volume or holding time has expired, then place a comment on the benchsheet and in LIMS.

Appendix 4: Summary of Calibration, QC and Corrective Action Procedures for GC/MS Organics

Method	QC Check	Frequency	Acceptance Criteria[2]	Corrective Action[3]
SW8260B SW8270C SW8151mod	Check of mass spectral ion intensities[1], i.e., Tune	Prior to initial calibration or Continuing calibration verification, every 12 hours	Refer to criteria listed in the method SOP for Tune criteria, including DDT, Benzidine and Pentachlorophenol requirements for 8270.	Retune the instrument and verify (instrument maintenance may be needed).
SW8260B SW8270C	Minimum five-point initial calibration for all target analytes	Initial calibration prior to sample analysis. Perform instrument re-calibration once per year minimum.	VOCs: SPCCs average RF ≥ 0.30 or 0.1 depending on the compound and %RSD for RFs for CCCs ≤ 30% and all other target analytes %RSD for RF ≤ 15%.	Correct problem then repeat initial calibration
			SVOCs: SPCCs average RF ≥ 0.050 and %RSD for RFs for CCCs ≤ 30% and all other target analytes %RSD for RF ≤ 15%.	Correct problem then repeat initial calibration
			option (if %RSD is > 15%)–linear regression $r^2 \geq 0.99$, r ≥ 0.995.	If the calibration is not considered linear by either %RSD or linear regression, then correct the problem and re-calibrate.
	Initial calibration verification (ICV) must be from a 2nd source.	Immediately following five-point initial calibration	All CCCs within 20% of expected value. **For DoD:** All analytes within 20% of expected value. See limits in SOP for poor-performing and other non-standard HSL compounds.	Correct problem then repeat initial calibration
	Relative Retention time window	Each sample	Relative retention time (RRT) of the analyte within 0.06 RRT units of the RRT of the internal standard	Correct problem then reprocess or re-analyze all samples analyzed since the last retention time check
	Continuing calibration verification (CCV)	Daily, before sample analysis and every 12 hours of analysis time	VOCs: SPCCs average RF ≥ 0.30 or 0.1 depending on the compound; **For DoD:** SPCCs average RF ≥ 0.050; CCCs: ≤20% difference (when using RFs) or drift (when using least squares regression). See limits in SOP for poor-performing and other non-standard HSL compounds.	Correct problem then repeat initial calibration and re-analyze all samples since last successful CCV.
			SVOCs: SPCCs average RF ≥ 0.050; and	

Method	QC Check	Frequency	Acceptance Criteria[2]	Corrective Action[3]
	Continuing calibration check		CCCs: ≤20% difference (when using RFs) or drift (when using least squares regression) for standard list analytes, ≤35% for non-standard analytes, and ≤55% for poor performing analytes. **For DoD:** SPCCs average RF ≥ 0.050; CCCs: ≤20% difference (when using RFs) or drift (when using least squares regression). See limits in SOP for poor-performing and other non-standard HSL compounds.	
	Method blank	One per analytical prep batch	No analytes detected ≥ ½ RL or MDL, whichever is greater[4]	Correct problem then re-prep[5] and analyze method blank and all samples processed with the contaminated blank
	Internal Standards	Every sample/standard and blank	Retention time ±30 seconds from retention time of the mid-point std. in the CCV/ICAL (sample/standard). EICP area within -50% to +100% of ICAL mid-point std for the CCV and –50% to +100% of the prior CCV for the samples. **For DoD:** EICP area within -50% to +100% of ICAL mid-point std for the CCV and samples.	Inspect mass spectrometer and GC for malfunctions; mandatory re-analysis of samples analyzed while system was malfunctioning (dilution of the sample may be required, see the supervisor or the Technical Manager for advice).
	LCS for all analytes must be from a 2nd source.	One per prep batch, not to exceed the 20 samples in a batch.	See Control Limits Manual	Correct problem then re-prep[5] and analyze the LCS and all samples in the affected analytical batch
	MS/MSD must be from a 2nd source.	One per batch per matrix, if insufficient sample for MS/MSD, then a LCS/LCSD will be analyzed.	See Control Limits Manual	None (the LCS is used to evaluate to determine if the batch is acceptable).
	Surrogate(s)	Every sample, spike, standard, and blank	See Control Limits Manual	Check system, re-analyze, re-prep[5]
	MDL verification	Minimum yearly **For DOD:** Minimum quarterly	Detectable	Re-evaluate MDL standard used and MDL; see Technical Manager.
	LOQ verification	**For DOD:** Minimum quarterly	Detectable	Re-evaluate LOQ standard used and LOQ; see Technical Manager.
SW8260B	pH check	All 8260 water samples.	pH ≤2.	If the pH is > 2, then comment the data, in the PIPE database, and LIMS.
	Residual chlorine check (North Carolina samples only)	Each sample.	Residual chlorine should be negative.	If the residual chlorine is positive, then comment in the data and LIMS.

Document Uncontrolled When Printed

1 - SW8260B requires BFB; SW8270C requires DFTPP
2 - This is a summary of the acceptance criteria, refer to the method SOP for specific or more information.
3 - All abnormalities must be noted in an NCM.
4 - Report all target compounds identified in the method blank above the MDL.
5 - If unable to re-prep samples because of insufficient sample volume or the holding time has expired, then note in an NCM.

Company Confidential & Proprietary

Appendix 4: Summary of Calibration, QC and Corrective Action Procedures for GC/MS Organics

Method	QC Check	Frequency	Acceptance Criteria[2]	Corrective Action[3]
EPA624 EPA625	Check of mass spectral ion intensities[1] (i.e. Tune)	Prior to initial calibration or Continuing calibration verification every 12 hours.	Refer to criteria listed in the method SOP for Tune requirements including DDT, Benzidine and Pentachlorophenol criteria for 625.	Retune instrument and verify instrument maintenance may be needed.
	Minimum three-point initial calibration for all target analytes.	Initial calibration prior to sample analysis. Perform instrument re-calibration once per year minimum.	%RSD < 35%, if %RSD is > 35% then linear regression is used (for linear regression $r^2 \geq 0.99$), $r \geq 0.995$.	If the calibration is not considered linear by either %RSD or linear regression, then correct problem then repeat initial calibration
	Initial calibration verification (ICV), 20 ug/L, must be from a 2nd source. May be the same as the LCS.	Immediately following initial calibration	See Control Limits Manual.	Correct problem then repeat initial calibration
	Relative Retention time window	Each sample	Retention time (RT) of the analyte within 30 seconds of the RT (± 0.25 min. RTW is used) of the target.	Correct problem then reprocess or re-analyze all samples analyzed since the last retention time check
EPA625	Continuing calibration verification (CCV)	Daily, before sample analysis and every 12 hours of analysis time.	All calibration analytes within 20% of expected value	Correct problem then repeat initial calibration and re-analyze all samples since last successful CCV.
EPA624			See limits in SOP	
EPA624 EPA625	Method blank	One per prep batch (not to exceed 20 samples per batch).	No analytes detected ≥ ½ RL or MDL, whichever is greater[4]	Correct problem then re-prep[5] and analyze method blank and all samples processed with the contaminated blank
	LCS for all analytes, 20 ug/L, must be from a 2nd source. May be the same as the ICV.	One per prep batch (not to exceed 20 samples per batch) or daily.	See Control Limits Manual	Correct problem then re-prep[5] and analyze the LCS and all samples in the affected analytical batch
	MS must be from a 2nd source.	One per batch of 20 per matrix, if insufficient sample for MS, then a-duplicate LCS will be analyzed.	See Control Limits Manual	All target compounds should be reported, and any compound that is outside criteria must be within criteria in the LCS.
	Surrogate(s)	Every sample, spiked sample, standard, and method blank	See Control Limits Manual	Correct problem then re-prep[5] and analyze sample
EPA624 EPA625	Internal Standards	Every sample/standard	Retention time ±30 seconds from retention time of the mid-point std. in the CCV/ICAL (sample/standard). EICP area within -50% to +100% of ICAL mid-point std for the CCV and –50% to +100% of the prior CCV for the samples.	Inspect mass spectrometer and GC for malfunctions; mandatory re-analysis of samples analyzed while system was malfunctioning (dilution of the sample may be required, see the supervisor or the Technical Manager for advice).
EPA624	pH check	All 624 samples after analysis	pH should be ≤ 2.	If the pH is > 2, then comment the data, in the PIPE database, and LIMS.

Method	QC Check	Frequency	Acceptance Criteria[2]	Corrective Action[3]
EPA624	Residual chlorine check (North Carolina samples only)	All samples after analysis	Residual chlorine should be negative.	If the residual chlorine is positive, then comment the data and LIMS.
	MDL verification	Minimum yearly	Detectable	Re-evaluate MDL standard used and MDL; see Technical Manager.

1 - 624 requires BFB; 625 requires DFTPP
2 - This is summary of the acceptance criteria, refer to the method SOP for specific or more information.
3 - All abnormalities must be noted in an NCM.
4 - Report all target compounds identified in the method blank above the MDL.
5 - If unable to re-prep samples because of insufficient sample volume or holding time has expired, then place a comment on the benchsheet and in LIMS.

Appendix 4: Summary of Calibration, QC and Corrective Action Procedures for GC Organics

Method	QC Check	Frequency	Acceptance Criteria[1]	Corrective Action[2]
SW8015[5] SW8021	Five-point initial calibration for all target analytes	Initial calibration prior to sample analysis. Perform instrument re-calibration once per year minimum.	RSD of CF ≤ 20% Linear – least squares regression r^2 ≥ 0.99, r ≥ 0.995	Correct problem then repeat initial calibration
	Initial calibration verification (ICV), must be from a 2nd source.	Immediately following five-point initial calibration	All analytes within 15% of expected value	Correct problem then repeat initial calibration
	LCS for all analytes must be from a 2nd source.	One per prep batch, not to exceed 20 samples in a batch.	See Control Limits	Re-prep[4] and analyze the LCS and all samples in the affected analytical batch
	Continuing calibration verification (CCV)	Before sample analysis, after every 10 samples, and at the end of the analysis sequence	All analytes within 15% of expected value and within the RTW. **For DoD:** All analytes within 20% of expected value and within the RTW.	Correct problem then repeat initial CCV (re-calibrate if necessary) and re-analyze all samples since last successful CCV.
	Method blank	One per analytical prep batch, not to exceed 20 samples in a batch.	No analytes detected ≥ ½ RL or MDL, whichever is greater[3]	Correct problem then re-prep[4] and analyze method blank and all samples processed with the contaminated blank
	Surrogate	Every sample, spiked sample, standard, and method blank	See Control Limits	Check system, re-analyze, re-prep[4]
	MS/MSD must be from a 2nd source.	One per batch per matrix, if insufficient sample for MS/MSD, then a LCS/LCSD will be analyzed.	See Control Limits	None (LCS is used to determine if data is acceptable).
	GC/MS confirmation.	At the clients request or analyst judgment.		
	Retention time window calculated for each analyte (see section 9 for how to calculate RTWs).	System set-up, with each new column or major instrument maintenance. Update the mid-RTW as the start of the run or daily.	Each analyte of the LCS, MS/MSD and CCV must be within the calculated RTW.	Correct the problem and re-process or re-analyze samples. For questions, see the supervisor or Technical Manager.
	MDL verification	Minimum yearly **For DoD:** Minimum quarterly	Detectable	Re-evaluate MDL standard used and MDL; see Technical Manager.
	LOQ verification	**For DoD:** Minimum quarterly	Detectable	Re-evaluate LOQ standard used and LOQ; see Technical Manager.
SW8021	pH Check	All water samples after analysis.	pH should be less than 2.	If pH is > 2, then place a comment on the benchsheet and in LIMS.
MT-VPH MT-EPH WA-VPH WA-EPH NWTPH-EPH NWTPH-VPH	Five-point initial calibration for all target analytes	Initial calibration prior to sample analysis. Perform instrument re-calibration once per year minimum.	RSD of CF ≤ 20% Linear – least squares regression r^2 ≥ 0.99, r ≥ 0.995	Correct problem then repeat initial calibration
	Initial calibration verification (ICV) or Petroleum Performance Check Standard	Immediately following five-point initial calibration	Must be within 70-130% recovery.	Correct problem then repeat initial calibration
	Retention time window calculated for each analyte (see section 9 for how to calculate RTWs).	System set-up, with each new column or major instrument maintenance. Update the mid-RTW as the start of the run or daily.	Each analyte of the LCS, MS/MSD and CCV must be within the calculated RTW.	Correct the problem and re-process or re-analyze samples. For questions, see the supervisor or Technical Manager.

Method	QC Check	Frequency	Acceptance Criteria[1]	Corrective Action[2]
	Continuing calibration verification (CCV)	Before sample analysis, after every 10 samples, and at the end of the analysis sequence	All analytes within 20% of expected value and within the RTW.	Correct problem then repeat initial CCV (re-calibrate if necessary) and re-analyze all samples since last successful CCV.
	Method blank	One per analytical prep batch, not to exceed 20 samples in a batch.	No analytes detected ≥ RL	Correct problem then re-prep[4] and analyze method blank and all samples processed with the contaminated blank
	LCS for all analytes must be from a 2nd source.	One per prep batch, not to exceed 20 samples in a batch.	Must be within 70-130% recovery.	Re-prep[4] and analyze the LCS and all samples in the affected analytical batch
	MS/MSD must be from a 2nd source.	One per batch per matrix, if insufficient sample for MS/MSD, then a LCS/LCSD will be analyzed.	Must be within 50-150% recovery. **For WA-VPH and WA-EPH:** Must be within 70-130% recovery.	None (LCS is used to determine if data is acceptable).
	Duplicate	Each batch, less than 20	±25%	None
	Surrogate	Every sample, spiked sample, standard, and method blank	Low end aliphatic compounds must be within 30-150% recovery and high end aliphatic compounds must be within 50-150% recovery. **For WA-VPH and WA-EPH:** Must be within 60-140% recovery	Check system, re-analyze, re-prep[4]
	MDL and MDL verification	Minimum yearly	Detectable	Re-evaluate MDL standard used and MDL; see Technical Manager.
SW8011 EPA504.1	Five-point initial calibration for all target analytes (calibration standards should be prepped as the samples). Three point minimum for 504.1.	Initial calibration prior to sample analysis. Perform instrument re-calibration once per year minimum.	RSD of CF of each analyte ≤ 20% **RSD of CF < 10% for Method 8011** Linear – $r^2 \geq 0.990$, $r \geq 0.995$	Correct problem then repeat initial calibration
	Initial calibration verification (ICV) must be from a 2nd source.	Immediately following initial calibration	All analytes within 20% of expected value	Correct problem then repeat initial calibration
	Continuing calibration verification (CCV)	Before sample analysis, after every 10 samples, and at the end of the analysis sequence	All analytes within 10% of expected value and within the RTW.	Repeat once. If subsequent CCV fails, correct problem, re-calibrate and re-analyze all samples since last successful CCV.
	Continuing calibration blank (CCB)	After each CCV	No analytes ≥ ½ RL	Correct problem and re-analyze all samples since last successful CCB, unless contamination is <10X the conc present in the sample
	Method blank	One per analytical prep batch, not to exceed 10 samples in a batch.	No analytes ≥ ½ RL	Correct problem then re-prep[3] and analyze method blank and all samples processed with the contaminated blank
	LCS for all analytes must be from a 2nd source.	One per prep batch, not to exceed 10 samples in a batch.	All analytes within 30% of expected value.	Re-prep[3] and analyze the LCS and all samples in the affected analytical batch

Method	QC Check	Frequency	Acceptance Criteria[1]	Corrective Action[2]
	Surrogate	Every sample, spike, standard, and method blank	See Control Limits	Check system, re-inject, re-extract[3]
	MS/MSD must be from a 2nd source.	One per batch per matrix, if insufficient sample for MS/MSD, then a LCS/LCSD will be analyzed.	See Control Limits	(LCS is used to determine if data is acceptable). Add comments in LIMS.
	Second-column confirmation	100% for all positive results	Same as for initial or primary column analysis	Same as for initial or primary column analysis. If the relative % difference of results between the 2 columns is greater than 40%, a comment should be placed in LIMS.
	Retention time window calculated for each analyte (see section 20.1 for how to calculate RTW's).	System set-up, with each new column or major instrument maintenance. Update the mid-RTW as the start of the run or daily.	Each analyte of the LCS, MS/MSD and CCV must be within the calculated RTW.	Correct the problem and re-process or re-analyze samples. For questions, see the supervisor or Technical Manager.
	MDL / MDL verification	Minimum yearly **For DOD:** Minimum quarterly	MDL<Spike<10X MDL Detected	Re-evaluate MDL standard used and MDL; see Technical Manager.
	LOQ verification	**For DOD:** Minimum quarterly	Detectable	Re-evaluate LOQ standard used and LOQ; see Technical Manager.

1 - This is a summary of the acceptance criteria, refer to the method SOP for specific or more information.
2 - All abnormalities must be noted on the data and in an NCM.
3 - Report all target compounds identified in the method blank above the MDL.
4 - If unable to re-prep the samples because of insufficient sample volume or holding time has expired, then note in an NCM.
5 - For GRO and DRO, see state specific SOP/Method for acceptance criteria. If there is not a specific method for that state, then follow the acceptance criteria in this table.

Appendix 4: Summary of Calibration, QC and Corrective Action Procedures for Method SW6010

Method	QC Check	Frequency	Acceptance Criteria	Corrective Action
SW6010B	Initial calibration (minimum 1 standard and a blank)	Daily initial calibration prior to sample analysis.	N/A	N/A
	Second-source calibration verification (ICV)	Daily after initial calibration	All analytes within 10% of expected value	Correct problem then repeat initial calibration
	RL Standard (CRI)	Daily after initial calibration	All others: within 50% of expected value. **For DoD:** within 20% of expected value.	Re-analyzed RL Standard, correct problem no samples may be analyzed without a valid RL Standard.
	Calibration blank (CB)	After every continuing calibration verification	Must be <RL. **For DoD:** Must be < LOD	Correct problem then analyze calibration blank and previous 10 samples
	Continuing calibration verification (CCV)	Before sample analysis, after every 10 samples, and at the end of the analysis sequence	All analytes within 10% of expected value and RSD of replicate integrations <5%	Repeat calibration and re-analyze all samples since last successful calibration
	Method blank	One per prep batch	No analytes detected ≥ ½ RL or MDL, whichever is greater[1] **For DoD:** Must be < ½ RL	Correct problem then re-prep and analyze method blank and all samples processed with the contaminated blank
	Interference check solution (ICS)	At the beginning of an analytical run	ICS-A: Absolute value of concentration for all non-spiked analytes < LOD (unless they are a verified trace impurity form one of the spike analytes) ICS-AB: Within 20% of expected value	Terminate analysis; correct problem; re-analyze ICS; re-analyze all affected samples
	LCS	One per prep batch	See Control Limits	Correct problem then re-prep and analyze the LCS and all samples in the affected analytical batch
	MS/MSD	One per batch per matrix	See Control Limits	None
	Dilution test	Each new sample matrix	1:5 dilution must agree within 10% of the original determination	Perform post digestion spike addition
	Post digestion spike addition	When dilution test fails	Recovery within 25% of expected results	None
	MDL verification	Minimum yearly **For DOD:** Minimum quarterly	Detectable	Re-evaluate MDL standard used and MDL; see Technical Manager.
	LOQ verification	**For DOD:** Minimum quarterly	Detectable	Re-evaluate LOQ standard used and LOQ; see Technical Manager.

1 – Report all targets identified in the method blank above the MDL.

Appendix 4: Summary of Calibration, QC and Corrective Action Procedures for Method SW6020

Method	QC Check	Frequency	Acceptance Criteria	Corrective Action
SW6020	Initial calibration (minimum 1 standard and a blank)	Daily initial calibration prior to sample analysis.	If more than one calibration standard used, r ≥ 0.995	Correct problem then repeat initial calibration
	Second-source calibration verification (ICV)	Daily after initial calibration	All analytes within 10% of expected value	Correct problem then repeat initial calibration
	RL Standard (CRI)	Daily after initial calibration	All others: within 50% of expected value. **For DoD:** within 20% of expected value.	Re-analyzed RL Standard, correct problem no samples may be analyzed without a valid RL Standard.
	Calibration blank (CB)	After every continuing calibration verification	Must be <RL. **For DoD:** Must be < LOD	Correct problem then analyze calibration blank and previous 10 samples
	Continuing calibration verification (CCV)	Before sample analysis, after every 10 samples, and at the end of the analysis sequence	All analytes within 10% of expected value and RSD of replicate integrations <5%	Repeat calibration and re-analyze all samples since last successful calibration
	Linear Dynamic Range	Every 6 months	Within 10% of expected value	N/A
	Method blank	One per prep batch	No analytes detected ≥ RL or MDL, whichever is greater[1] **For DoD:** Must be < ½ RL	Correct problem then re-prep and analyze method blank and all samples processed with the contaminated blank
	Interference check solution (ICS)	At the beginning of an analytical run	ICS-A: Absolute value of concentration for all non-spiked analytes < LOD (unless they are a verified trace impurity form one of the spike analytes) ICS-AB: Within 20% of expected value	Terminate analysis; correct problem; re-analyze ICS; re-analyze all affected samples
	Internal standards (IS)	Every sample	IS intensity within 30-120% of intensity of the IS in the initial calibration	Perform corrective action as described in 6020.
	RL standard	Daily, after one point initial calibration	Within 20% of expected value	Correct problem then re-analyze samples.
	LCS	One per prep batch	See Control Limits	Correct problem then re-prep and analyze the LCS and all samples in the affected analytical batch
	MS/MSD	One per batch per matrix	See Control Limits	None
	Dilution test	Each new sample matrix	1:5 dilution must agree within 10% of the original determination	Perform post digestion spike addition
	Post digestion spike addition	When dilution test fails	Recovery within 25% of expected results	None.
	MDL verification	Minimum yearly **For DOD:** Minimum quarterly	Detectable	Re-evaluate MDL standard used and MDL; see Technical Manager.
	LOQ verification	**For DOD:** Minimum quarterly	Detectable	Re-evaluate LOQ standard used and LOQ; see Technical Manager.

1 – Report all targets identified in the method blank above the MDL.

Appendix 4: Summary of Calibration, QC and Corrective Action Procedures for Method SW7196

Method	QC Check	Frequency	Acceptance Criteria	Corrective Action
SW7196A	Initial calibration (minimum three standards and a blank)	Initial calibration prior to sample analysis.	$r^2 \geq 0.99$, $r \geq 0.995$ for linear regression	Correct problem then repeat initial calibration
	Second-source calibration verification (ICV)	Immediately following initial calibration	All analytes within 10% of expected value	Correct problem then repeat initial calibration
	Continuing calibration verification (CCV)	Beginning and after every 10 samples and at the end of the analysis sequence	All analytes within 20% of expected value	Correct problem then repeat initial calibration and re-analyze all samples since last successful calibration
	Verification check to ensure lack of reducing condition and/or interference	Once for every sample matrix analyzed	Spike recovery between 85-115%	If check indicates interference, dilute and re-analyze sample persistent interference indicates the need to use and alternate method
	Method blank	One per prep batch	No analytes detected ≥ ½ RL or MDL, whichever is greater[1]	Correct problem then re-prep and analyze method blank and all samples processed with the contaminated blank
	MS/MSD	One per 20 samples per matrix	See Control Limits Manual	none
	LCS	One per batch	See Control Limits Manual	Re-prep, re-analyze all affected samples.
	MDL verification	Minimum yearly	Detectable	Re-evaluate MDL standard used and MDL; see Technical Manager.

1 - Report all targets identified in the method blank above the MDL.

Appendix 4: Summary of Calibration, QC and Corrective Action Procedures for Method SW7470/SW7471

Method	QC Check	Frequency	Acceptance Criteria	Corrective Action
SW7470A SW7471A	Initial calibration (minimum 5 standards and a blank)	Daily initial calibration prior to sample analysis. Perform instrument re-calibration once per year minimum.	$r^2 \geq 0.99$, $r \geq 0.995$ for linear regression	Correct problem then repeat initial calibration. If calibration fails again, re-digest the entire digestion batch.
	Second-source calibration verification (ICV)	Immediately following initial daily calibration	Analytes within 10% of expected value	Correct problem then repeat initial calibration. If calibration fails again, re-digest the entire digestion batch.
	Calibration blank	Once per initial daily calibration	No analytes detected ≥ MDL	Correct problem then re-digest and re-analyze calibration and entire digestion batch
	Continuing calibration verification (CCV)	Before sample analysis, after every 10 samples, and at the end of the analysis sequence	Analytes within 20% of expected value	Correct problem then repeat all QC and samples since last successful calibration. If the CCV fails again upon reanalysis, reprep the entire digestion batch.
	Method blank	One per prep batch	No analytes detected ≥ RL **For DoD:** Must be < ½ RL	Correct problem then re-prep and analyze method blank, all samples, and QC processed with the contaminated blank
	LCS	One per prep batch	See Control Limits Manual	Correct problem then re-prep and analyze the LCS, all samples, and QC in the affected analytical batch
	MS/MSD	One per batch per matrix	See Control Limits Manual	None
	MDL verification	Minimum yearly **For DOD:** Minimum quarterly	Detectable	Re-evaluate MDL standard used and MDL; see Technical Manager.
	LOQ verification	**For DOD:** Minimum quarterly	Detectable	Re-evaluate LOQ standard used and LOQ; see Technical Manager.

1 - Report all targets identified in the method blank above the MDL.

Appendix 4: Summary of Calibration, QC and Corrective Action Procedures for Method SW9010/SW9012/SW9014

Method	QC Check	Frequency	Acceptance Criteria	Corrective Action
SW9010 SW9012A SW9014	Initial calibration (six standards and a calibration blank)	Initial daily calibration prior to sample analysis. Perform instrument re-calibration once per year minimum.	$r^2 \geq 0.99$, $r \geq 0.995$ for linear regression	Correct problem then repeat initial calibration
	Distilled standards (one high and one low)	Once per calibration	Analyte within 10% of true value. **For DoD:** Analyte within 15% of true value.	Correct problem then repeat distilled standards
	Second-source calibration verification (ICV)	Immediately following initial daily calibration	Analyte within 15% of expected value.	Correct problem then repeat initial calibration
	Continuing calibration verification (CCV)	Beginning and after every 10 samples and at the end of the analysis sequence	Analyte within 15% of expected value	Correct problem then repeat initial Continuing calibration verification and re-analyze all samples since last successful Continuing calibration verification
	Calibration blank	Once per initial daily calibration	No analytes detected ≥ RL **For DoD:** Must be < ½ RL	Correct problem then re-analyze calibration blank and all samples associated with blank
	Method blank	One per prep batch	No analyte detected ≥ RL or MDL, whichever is greater[1] **For DoD:** Must be < ½ RL	Correct problem then re-prep and analyze method blank and all samples processed with the contaminated blank
	LCS	One per batch per matrix	See Control Limits in LIMS	Re-prep, re-run affected samples
	MS/MSD	One per batch per matrix	See Control Limits in LIMS	None
	MDL verification	Minimum quarterly **For DOD:** Minimum quarterly	Detectable	Re-evaluate MDL standard used and MDL; see Technical Manager.
	LOQ verification	**For DOD:** Minimum quarterly	Detectable	Re-evaluate LOQ standard used and LOQ; see Technical Manager.

Appendix 4: Summary of Calibration, QC and Corrective Action Procedures for Mercury

Method	QC Check	Frequency	Acceptance Criteria	Corrective Action
EPA245.1	Initial calibration (minimum 5 standards and a blank)	Daily initial calibration prior to sample analysis. Perform instrument re-calibration once per year minimum.	$r^2 \geq 0.99$, $r \geq 0.995$ for linear regression	Correct problem then repeat initial calibration
	Second-source calibration verification (ICV)	Immediately following five-point initial calibration	Analyte within 5% of expected value	Correct problem then repeat initial calibration
	Calibration blank	Once per initial daily calibration	No analytes detected ≥ MDL	Correct problem then re-analyze calibration blank and all samples associated with blank
	Continuing calibration verification (CCV)	Before sample analysis, after every 10 samples, and at the end of the analysis sequence	Analyte within 10% of true value	Correct problem then repeat calibration and re-analyze all samples and QC since last successful calibration
	LCS	One per prep batch	All analytes within 15% of expected value	Correct problem then re-prep and analyze the LCS, all samples, and QC in the affected analytical batch
	Matrix Spike/Matrix Spike Duplicate	One per batch or 10 samples	All analytes within 25% of expected value	None
	Method Blank	One per batch	No analytes > ½ RL	Reprep
	MDL verification	Minimum yearly	Detectable	Re-evaluate MDL standard used and MDL; see Technical Manager.

1 - Report all targets identified in the method blank above the MDL.

Appendix 4: Summary of Calibration, QC and Corrective Action Procedures for ICP Metals

Method	QC Check	Frequency	Acceptance Criteria	Corrective Action
EPA200.7	Initial calibration (minimum 1 standard and a blank)	Daily initial calibration prior to sample analysis.	N/A	N/A
	Second-source calibration verification (ICV/QCS)	Each calibration	Value of all analytes within 5% of expected value	Correct problem then repeat initial calibration
	Linear Dynamic Range	Once annually	All analytes within 10% of expected value	Calibration range lowered to meet LDR results
	Calibration blank	After every Continuing calibration verification	No analytes detected (< IDL or MDL, whichever is greater)	Correct problem then analyze calibration blank and previous 10 samples
	Continuing calibration verification (CCV/IPC)	Before sample analysis, after every 10 samples, and at the end of the analysis sequence	All analytes within 10%	Repeat calibration and re-analyze all samples since last successful calibration
	Method blank	One per prep batch	No analytes detected, < 10% of sample conc OR < 2.2 x MDL[1]	Correct problem then re-prep and analyze method blank and all samples processed with the contaminated blank
	Interference check solution (ICSA/ACSAB)	At the beginning of an analytical run, daily	Monitor for interferences.	Terminate analysis; correct problem; re-analyze ICS; re-analyze all affected samples
	LCS/LFB	One per prep batch	All analytes within 15% of expected value	Correct problem then re-prep and analyze the LCS and all samples in the affected analytical batch
	Dilution test	Each new sample matrix	1:5 dilution must agree within 10% of the original determination for concentrations > 50 X IDL	Perform post digestion spike addition
	Post digestion spike addition	When dilution test fails	Recovery within 15% of expected results	Correct problem then re-analyze post digestion spike addition
	Matrix Spike/Matrix Spike Duplicate	One per batch of 10 samples	All analytes within 30% of expected value	None
	MDL verification	Minimum yearly	Detectable	Re-evaluate MDL standard used and MDL; see Technical Manager.

1 - Report all targets identified in the method blank above the MDL.

Appendix 4: Summary of Calibration, QC and Corrective Action Procedures for ICP-MS Metals

Method	QC Check	Frequency	Acceptance Criteria	Corrective Action
EPA200.8	Initial calibration (minimum 1 standard and a blank)	Daily initial calibration prior to sample analysis.	N/A	N/A
	Second-source calibration verification (ICV/QCS)	Each calibration	Value of all analytes within 10% of expected value	Correct problem then repeat initial calibration
	Calibration blank (ICB)	After every Continuing calibration verification	No analytes detected (< RL)	Correct problem then analyze calibration blank and previous 10 samples
	Continuing calibration verification (CCV)	Before sample analysis, after every 10 samples, and at the end of the analysis sequence	All analytes within 10%	Repeat calibration and re-analyze all samples since last successful calibration
	Method Blank/Laboratory Reagent Blank	One per prep batch	No analytes detected, < 10% of sample conc. OR < 2.2xMDL[1]	Correct problem then re-prep and analyze method blank and all samples processed with the contaminated blank
	Interference check solution (ICSA/ICSAB)	At the beginning of an analytical run, daily	Monitor for possible interferences.	Terminate analysis; correct problem; re-analyze ICS; re-analyze all affected samples
	LCS/LFB	One per prep batch	All analytes within 15% of expected value	Correct problem then re-prep and analyze the LCS and all samples in the affected analytical batch
	Internal Standard	Every sample	IS intensity within 60-125% of intensity of the IS in the initial calibration	Perform corrective action as described in 200.8.
	Matrix Spike/Matrix Spike Duplicate (MS/MSD)	One per batch of 10 samples	All analytes within 30% of expected value	None
	MDL verification	Minimum yearly	Detectable	Re-evaluate MDL standard used and MDL; see Technical Manager.

1 - Report all targets identified in the method blank above the MDL.

Document Uncontrolled When Printed

Appendix 4: Summary of Calibration, QC and Corrective Action Procedures for Gravimetric Analyses

Method	QC Check	Frequency	Acceptance Criteria	Corrective Action
EPA160.1 (TDS) SM2540 C (TDS) EPA160.2 (TSS) SM2540D (TSS) EPA160.3 (TS) SM2540B (TS) EPA160.4 (TVS)	Verification standard– single standard (if available)	Each batch	±10%	Repeat
	Method blank	Each batch	No analytes detected ≥ RL	Repeat, rerun
	Duplicate	Each batch, less than 20	±20%	None

Appendix 4: Summary of Calibration, QC and Corrective Action Procedures for Titrimetric Analyses

Method	QC Check	Frequency	Acceptance Criteria	Corrective Action
EPA310.1: Alkalinity. SM2320: HCO_3^-, CO_3^{-2}.	Verification standard– single standard (if available)	Each batch	±10%	Repeat, check
	Method blank	Each batch	No analyte detected ≥ RL.	Repeat batch
	Duplicate	Each batch	±20%	None

Appendix 4: Summary of Calibration, QC and Corrective Action Procedures for Electrometric Analyses

Method	QC Check	Frequency	Acceptance Criteria	Corrective Action
EPA405.1: BOD[1], CBOD[1]. EPA120.1: Cond[1]. SM2510B: Cond[1]. EPA150.1: pH. SW9040B, 9045C:pH. EPA180.1: Turbidity.	Calibration Curve – minimum of 5 standards	Initial Calibration. Perform re-calibration once per year minimum	±10%, $r^2 \geq 0.99$, $r \geq 0.995$.	Recalibrate
	Independent calibration verification (second source) (ICV)	Immediately after initial calibration	±10%	Recalibrate
	Continuing calibration verification (CCV)	Beginning, every 10 samples, and end of batch	±10%	Rerun
	Method blank	Each batch	No analyte detected ≥ ½ report limit or MDL, whichever is greater[2]	Reprep
	LCS	Each batch	±10%	Rerun batch
	MS/MSD	Each batch	± 20%	None
	Duplicate	When spike not available	±20%	None
	MDL verification	Minimum yearly	Detectable	Re-evaluate MDL standard used and MDL; see Technical Manager.

1 - Calibration curve does not apply.

2 - Report all targets identified in the method blank above the MDL.

Appendix 4: Summary of Calibration, QC and Corrective Action Procedures for Spectrophotometric Analyses

Method	QC Check	Frequency	Acceptance Criteria	Corrective Action
EPA350.1: NH_3. EPA410.4: COD. EPA335.4: CN. EPA353.2 NO_2/NO_3 EPA365.1 SM4500-P E T. Phos. EPA415.1: TOC. SM4500-CN^-I: WAD CN EPA9060: TOC. SM4500-Cr D Hexchrome SM4500-N_{org} C	Calibration curve – minimum 5 point	Initial. Perform re-calibration once per year minimum.	RSD <10%, $r^2 \geq 0.99$, $r \geq 0.995$	Recalibrate
	Independent calibration verification – mid-level, second-source required (ICV)	Immediately following initial calibration.	See Control Limits in LIMS	Recalibrate
	Continuing calibration verification (CCV)	Beginning, every 10 samples, and at end of sequence	See Control Limits in LIMS	Correct, recalibrate
	Method blank	Each use	No analyte detected ≥ ½ report limit or MDL, whichever is greater[1]	Reprep, rerun
	MS/MSD	Each batch, less than 20	See Control Limits in LIMS	None
	LCS	Each batch	See Control Limits in LIMS	Rerun
	MDL verification	Minimum yearly	Detectable	Re-evaluate MDL standard used and MDL; see Technical Manager.

Appendix 4: Summary of Calibration, QC and Corrective Action Procedures for Ion Chromatographic Analyses

Method	QC Check	Frequency	Acceptance Criteria	Corrective Action
EPA300: SW9056A: Bromide Chloride Fluoride Nitrate Nitrite Sulfate.	Calibration Curve – Minimum 5-point calibration	Initial calibration. Perform instrument re-calibration once per year minimum.	RSD ± 10%, $r^2 \geq 0.99$, $r \geq 0.995$.	Recalibrate
	Calibration verification (ICV), second source	Immediately following initial calibration	±10%	Recalibrate
	Continuing calibration verification (CCV)	Each use, beginning, every 10 samples, end of batch	± 10%	Rerun affected samples
	Method blank	Each batch	No analyte detected ≥ ½ report limit or MDL, whichever is greater[3]	Rerun batch
	LCS	Each batch	±10%	Rerun batch
	MS/MSD[1]	Each batch	±20%	None, use LCS
	Duplicate[2]	Each batch	±30%	None
	MDL verification	Minimum yearly **For DOD:** Minimum quarterly	Detectable	Re-evaluate MDL standard used and MDL; see Technical Manager.
	LOQ verification	**For DOD:** Minimum quarterly	Detectable	Re-evaluate LOQ standard used and LOQ; see Technical Manager.

1 - Only applies to EPA300, SW9056.
2 - Oil only.
3 - Report all targets identified in the method blank above the MDL.

Appendix 4: Summary of Calibration, QC and Corrective Action Procedures for Physical Analyses

Method	QC Check	Frequency	Acceptance Criteria	Corrective Action
SW1020A: Flash Point. EPA160.5: Settleable Solids.	Method blank	Each batch (if applicable to the method)	No analyte detected ≥ ½ report limit or MDL, whichever is greater[1]	Repeat, rerun
	Two standards for Flash Point One standard for BTU 1 Known for Settleable Solids	Each batch	See Control Limits in LIMS	Rerun batch
	Duplicate	Each batch	See Control Limits in LIMS	None

1 - Report all targets identified in the method blank above the MDL

Document Uncontrolled When Printed

Appendix 4: Summary of Calibration, QC and Corrective Action Procedures for Oil & Grease Analyses

Method	QC Check	Frequency	Acceptance Criteria	Corrective Action
EPA1664A	Verification standard	Single standard	±10% PAR standard	Rerun
	Method blank	Each batch	No analyte detected ≥ report limit	Repeat batch
	LCS	Each batch	See Control Limits	Repeat batch
	Duplicate	Each batch	See Control Limits	
	MS/MSD	Each batch	See Control Limits	None, use LCS
	LOQ verification	**For DOD:** Minimum quarterly	Detectable	Re-evaluate LOQ standard used and LOQ; see Technical Manager.

Analysis Group Description	Method Description	Method Code
SOIL	Northwest - Semi-Volatile Petroleum Products (GC)	NWTPH_Dx

Analyte Description	CAS Number	RL - Limit	RL - Units	MDL - Limit	MDL - Units	LCSREC - Recovery Low	LCSREC - Recovery High	LCSREC - Units	LCSRPD - Precision	LCSRPD - Units
o-Terphenyl	84-15-1	0.8	mg/Kg	0.09	mg/Kg			%		%
#2 Diesel (C10-C24)	STL00163	25	mg/Kg	5.7	mg/Kg	64	127	%	16	%
Motor Oil (>C24-C36)	STL00299	50	mg/Kg	9.1	mg/Kg	70	125	%	17	%

SOIL	Microwave Extraction	3546
SOIL	Semivolatile Organic Compounds (GC/MS)	8270D

Analyte Description	CAS Number	RL - Limit	RL - Units	MDL - Limit	MDL - Units	LCSREC - Recovery Low	LCSREC - Recovery High	LCSREC - Units	LCSRPD - Precision	LCSRPD - Units
2-Fluorophenol (Surr)	367-12-4	2000	ug/Kg	18.7	ug/Kg			%		%
Phenol-d5 (Surr)	4165-62-2	2000	ug/Kg	29.8	ug/Kg			%		%
Nitrobenzene-d5 (Surr)	4165-60-0	1000	ug/Kg	9.2	ug/Kg			%		%
2-Fluorobiphenyl	321-60-8	1000	ug/Kg	12.6	ug/Kg			%		%
2,4,6-Tribromophenol (Surr)	118-79-6	2000	ug/Kg	53	ug/Kg			%		%
Terphenyl-d14 (Surr)	1718-51-0	1000	ug/Kg	31	ug/Kg			%		%
Phenol	108-95-2	100	ug/Kg	15	ug/Kg	66	126	%	26	%
Bis(2-chloroethyl)ether	111-44-4	100	ug/Kg	15	ug/Kg	57	122	%	60	%
2-Chlorophenol	95-57-8	100	ug/Kg	15	ug/Kg	65	125	%	27	%
1,3-Dichlorobenzene	541-73-1	50	ug/Kg	15	ug/Kg	64	124	%	60	%
1,4-Dichlorobenzene	106-46-7	50	ug/Kg	15	ug/Kg	62	132	%	32	%
Benzyl alcohol	100-51-6	100	ug/Kg	15	ug/Kg	42	147	%	60	%
1,2-Dichlorobenzene	95-50-1	55	ug/Kg	15	ug/Kg	68	118	%	60	%
2-Methylphenol	95-48-7	100	ug/Kg	15	ug/Kg	56	121	%	25	%
3 & 4 Methylphenol	15831-10-4	200	ug/Kg	15	ug/Kg	61	126	%	27	%
N-Nitrosodi-n-propylamine	621-64-7	100	ug/Kg	15	ug/Kg	52	127	%	28	%
Hexachloroethane	67-72-1	100	ug/Kg	15	ug/Kg	56	131	%	60	%
Nitrobenzene	98-95-3	100	ug/Kg	34	ug/Kg	59	134	%	60	%
Isophorone	78-59-1	100	ug/Kg	5	ug/Kg	53	118	%	60	%
2-Nitrophenol	88-75-5	100	ug/Kg	15	ug/Kg	58	128	%	60	%
2,4-Dimethylphenol	105-67-9	100	ug/Kg	15	ug/Kg	58	133	%	60	%
Benzoic acid	65-85-0	2500	ug/Kg	750	ug/Kg	10	130	%	60	%
Bis(2-chloroethoxy)methane	111-91-1	100	ug/Kg	5	ug/Kg	63	128	%	60	%
2,4-Dichlorophenol	120-83-2	100	ug/Kg	15	ug/Kg	59	124	%	60	%
1,2,4-Trichlorobenzene	120-82-1	50	ug/Kg	15	ug/Kg	63	128	%	28	%
Naphthalene	91-20-3	20	ug/Kg	5	ug/Kg	64	129	%	26	%
4-Chloroaniline	106-47-8	100	ug/Kg	15	ug/Kg	20	181	%	60	%
Hexachlorobutadiene	87-68-3	50	ug/Kg	15	ug/Kg	59	134	%	60	%
4-Chloro-3-methylphenol	59-50-7	100	ug/Kg	15	ug/Kg	58	128	%	27	%
2-Methylnaphthalene	91-57-6	20	ug/Kg	5	ug/Kg	65	125	%	27	%
Hexachlorocyclopentadiene	77-47-4	100	ug/Kg	10	ug/Kg	30	132	%	60	%

2,4,6-Trichlorophenol	88-06-2	150	ug/Kg	15	ug/Kg	66	131	%	60	%
2,4,5-Trichlorophenol	95-95-4	100	ug/Kg	15	ug/Kg	64	124	%	60	%
2-Chloronaphthalene	91-58-7	20	ug/Kg	5	ug/Kg	69	129	%	25	%
2-Nitroaniline	88-74-4	100	ug/Kg	15	ug/Kg	58	133	%	60	%
Dimethyl phthalate	131-11-3	100	ug/Kg	5	ug/Kg	65	125	%	60	%
Acenaphthylene	208-96-8	20	ug/Kg	5	ug/Kg	69	129	%	28	%
2,6-Dinitrotoluene	606-20-2	100	ug/Kg	15	ug/Kg	65	125	%	60	%
3-Nitroaniline	99-09-2	100	ug/Kg	15	ug/Kg	80	165	%	60	%
Acenaphthene	83-32-9	20	ug/Kg	5	ug/Kg	65	130	%	27	%
2,4-Dinitrophenol	51-28-5	1000	ug/Kg	200	ug/Kg	53	168	%	60	%
4-Nitrophenol	100-02-7	1000	ug/Kg	250	ug/Kg	47	172	%	33	%
Dibenzofuran	132-64-9	100	ug/Kg	5	ug/Kg	70	125	%	60	%
2,4-Dinitrotoluene	121-14-2	100	ug/Kg	15	ug/Kg	57	122	%	31	%
Diethyl phthalate	84-66-2	200	ug/Kg	15	ug/Kg	64	129	%	26	%
4-Chlorophenyl phenyl ether	7005-72-3	100	ug/Kg	15	ug/Kg	65	130	%	60	%
Fluorene	86-73-7	20	ug/Kg	5	ug/Kg	68	128	%	31	%
4-Nitroaniline	100-01-6	100	ug/Kg	20	ug/Kg	70	150	%	60	%
4,6-Dinitro-2-methylphenol	534-52-1	1000	ug/Kg	100	ug/Kg	38	143	%	60	%
N-Nitrosodiphenylamine	86-30-6	50	ug/Kg	5	ug/Kg	88	153	%	60	%
4-Bromophenyl phenyl ether	101-55-3	100	ug/Kg	15	ug/Kg	64	134	%	60	%
Hexachlorobenzene	118-74-1	50	ug/Kg	5	ug/Kg	61	136	%	60	%
Pentachlorophenol	87-86-5	200	ug/Kg	20	ug/Kg	29	124	%	68	%
Phenanthrene	85-01-8	20	ug/Kg	5	ug/Kg	65	125	%	28	%
Anthracene	120-12-7	20	ug/Kg	5	ug/Kg	73	123	%	27	%
Di-n-butyl phthalate	84-74-2	500	ug/Kg	50	ug/Kg	69	124	%	60	%
Fluoranthene	206-44-0	20	ug/Kg	5	ug/Kg	61	121	%	36	%
Pyrene	129-00-0	20	ug/Kg	5	ug/Kg	54	134	%	31	%
Butyl benzyl phthalate	85-68-7	200	ug/Kg	50	ug/Kg	65	140	%	60	%
3,3'-Dichlorobenzidine	91-94-1	200	ug/Kg	30	ug/Kg	73	163	%	60	%
Benzo[a]anthracene	56-55-3	20	ug/Kg	5	ug/Kg	64	124	%	27	%
Chrysene	218-01-9	25	ug/Kg	5	ug/Kg	71	126	%	26	%
Bis(2-ethylhexyl) phthalate	117-81-7	600	ug/Kg	50	ug/Kg	64	144	%	60	%
Di-n-octyl phthalate	117-84-0	500	ug/Kg	5	ug/Kg	58	148	%	31	%
Benzo[a]pyrene	50-32-8	30	ug/Kg	5	ug/Kg	68	128	%	30	%
Indeno[1,2,3-cd]pyrene	193-39-5	40	ug/Kg	5	ug/Kg	59	139	%	29	%
Dibenz(a,h)anthracene	53-70-3	40	ug/Kg	5	ug/Kg	57	142	%	30	%
Benzo[g,h,i]perylene	191-24-2	25	ug/Kg	5	ug/Kg	57	142	%	28	%
Carbazole	86-74-8	100	ug/Kg	5	ug/Kg	88	158	%	60	%
1-Methylnaphthalene	90-12-0	30	ug/Kg	5	ug/Kg	48	148	%	30	%
Benzo[b]fluoranthene	205-99-2	20	ug/Kg	5	ug/Kg	66	136	%	31	%
Benzo[k]fluoranthene	207-08-9	25	ug/Kg	5	ug/Kg	63	143	%	31	%
bis(chloroisopropyl) ether	108-60-1	250	ug/Kg	15	ug/Kg	44	140	%	60	%

SOIL	Ultrasonic Extraction	3550B
SOIL	Volatile Organic Compounds by GC/MS (MeOH Preserved)	8260C

Analyte Description	CAS Number	RL - Limit	RL - Units	MDL - Limit	MDL - Units	LCSREC - Recovery Low	LCSREC - Recovery High	LCSREC - Units	LCSRPD - Precision	LCSRPD - Units
Toluene-d8 (Surr)	2037-26-5	100	ug/Kg	3.2	ug/Kg			%		%
Dichlorodifluoromethane	75-71-8	40	ug/Kg	6.5	ug/Kg	38	150	%	26	%
Chloromethane	74-87-3	100	ug/Kg	10.1	ug/Kg	55	136	%	26	%
Vinyl chloride	75-01-4	16	ug/Kg	7.1	ug/Kg	67	131	%	22	%
Bromomethane	74-83-9	140	ug/Kg	13.4	ug/Kg	57	148	%	29	%
Chloroethane	75-00-3	400	ug/Kg	15.9	ug/Kg	48	167	%	53	%
Trichlorofluoromethane	75-69-4	40	ug/Kg	5.9	ug/Kg	47	165	%	54	%
1,1-Dichloroethene	75-35-4	20	ug/Kg	8.8	ug/Kg	70	133	%	23	%
Methylene Chloride	75-09-2	25	ug/Kg	11.5	ug/Kg	57	146	%	21	%
trans-1,2-Dichloroethene	156-60-5	40	ug/Kg	6.1	ug/Kg	76	131	%	18	%
1,1-Dichloroethane	75-34-3	40	ug/Kg	4.2	ug/Kg	70	128	%	21	%
2,2-Dichloropropane	594-20-7	40	ug/Kg	4.8	ug/Kg	56	144	%	21	%
cis-1,2-Dichloroethene	156-59-2	40	ug/Kg	4.9	ug/Kg	70	130	%	19	%
Bromochloromethane	74-97-5	40	ug/Kg	4.6	ug/Kg	78	123	%	19	%
Chloroform	67-66-3	40	ug/Kg	4.2	ug/Kg	78	125	%	17	%
1,1,1-Trichloroethane	71-55-6	40	ug/Kg	5.6	ug/Kg	63	135	%	20	%
Carbon tetrachloride	56-23-5	20	ug/Kg	3.8	ug/Kg	59	145	%	19	%
1,1-Dichloropropene	563-58-6	40	ug/Kg	5.3	ug/Kg	77	125	%	16	%
Benzene	71-43-2	16	ug/Kg	3.5	ug/Kg	70	128	%	19	%
1,2-Dichloroethane	107-06-2	16	ug/Kg	3.3	ug/Kg	71	128	%	18	%
Trichloroethene	79-01-6	16	ug/Kg	3.1	ug/Kg	83	124	%	17	%
1,2-Dichloropropane	78-87-5	12	ug/Kg	2.4	ug/Kg	76	161	%	15	%
Dibromomethane	74-95-3	40	ug/Kg	4.1	ug/Kg	78	126	%	18	%
Bromodichloromethane	75-27-4	40	ug/Kg	1.4	ug/Kg	58	133	%	19	%
cis-1,3-Dichloropropene	10061-01-5	16	ug/Kg	1.8	ug/Kg	69	129	%	19	%
Toluene	108-88-3	40	ug/Kg	2.6	ug/Kg	75	126	%	19	%
trans-1,3-Dichloropropene	10061-02-6	16	ug/Kg	2.4	ug/Kg	72	129	%	20	%
1,1,2-Trichloroethane	79-00-5	12	ug/Kg	2.8	ug/Kg	77	124	%	18	%
Tetrachloroethene	127-18-4	20	ug/Kg	1.3	ug/Kg	56	155	%	27	%
1,3-Dichloropropane	142-28-9	40	ug/Kg	2.3	ug/Kg	77	123	%	19	%
Dibromochloromethane	124-48-1	20	ug/Kg	1	ug/Kg	42	129	%	23	%
1,2-Dibromoethane	106-93-4	16	ug/Kg	3.4	ug/Kg	69	126	%	21	%
Chlorobenzene	108-90-7	40	ug/Kg	2.1	ug/Kg	75	120	%	21	%
Ethylbenzene	100-41-4	40	ug/Kg	2	ug/Kg	78	126	%	23	%
1,1,1,2-Tetrachloroethane	630-20-6	40	ug/Kg	1.1	ug/Kg	72	123	%	20	%
1,1,2,2-Tetrachloroethane	79-34-5	10	ug/Kg	2.3	ug/Kg	73	125	%	22	%
m-Xylene & p-Xylene	179601-23-1	40	ug/Kg	3	ug/Kg	78	126	%	23	%

o-Xylene	95-47-6	40	ug/Kg	3	ug/Kg	77	127	%	22	%
Styrene	100-42-5	40	ug/Kg	2.4	ug/Kg	79	127	%	21	%
Bromoform	75-25-2	40	ug/Kg	2.2	ug/Kg	50	124	%	25	%
Isopropylbenzene	98-82-8	40	ug/Kg	2.6	ug/Kg	79	127	%	20	%
Bromobenzene	108-86-1	40	ug/Kg	2.4	ug/Kg	80	120	%	19	%
N-Propylbenzene	103-65-1	40	ug/Kg	2.6	ug/Kg	81	127	%	20	%
1,2,3-Trichloropropane	96-18-4	40	ug/Kg	3.8	ug/Kg	77	123	%	23	%
2-Chlorotoluene	95-49-8	40	ug/Kg	3.4	ug/Kg	79	122	%	18	%
1,3,5-Trimethylbenzene	108-67-8	40	ug/Kg	2.9	ug/Kg	80	125	%	18	%
4-Chlorotoluene	106-43-4	40	ug/Kg	3	ug/Kg	80	122	%	18	%
t-Butylbenzene	98-06-6	40	ug/Kg	3.1	ug/Kg	71	136	%	27	%
1,2,4-Trimethylbenzene	95-63-6	40	ug/Kg	3.3	ug/Kg	79	124	%	18	%
sec-Butylbenzene	135-98-8	40	ug/Kg	2.8	ug/Kg	78	128	%	17	%
1,3-Dichlorobenzene	541-73-1	40	ug/Kg	3.1	ug/Kg	79	119	%	17	%
4-Isopropyltoluene	99-87-6	40	ug/Kg	2.8	ug/Kg	78	126	%	18	%
1,4-Dichlorobenzene	106-46-7	40	ug/Kg	2	ug/Kg	79	117	%	18	%
n-Butylbenzene	104-51-8	40	ug/Kg	3.5	ug/Kg	78	128	%	17	%
1,2-Dichlorobenzene	95-50-1	40	ug/Kg	3.2	ug/Kg	79	117	%	17	%
1,2-Dibromo-3-Chloropropane	96-12-8	200	ug/Kg	2.6	ug/Kg	53	132	%	27	%
1,2,4-Trichlorobenzene	120-82-1	40	ug/Kg	3.9	ug/Kg	61	130	%	22	%
1,2,3-Trichlorobenzene	87-61-6	40	ug/Kg	7.8	ug/Kg	61	130	%	23	%
Hexachlorobutadiene	87-68-3	40	ug/Kg	3.3	ug/Kg	68	134	%	21	%
Naphthalene	91-20-3	40	ug/Kg	6	ug/Kg	14	170	%	50	%
4-Bromofluorobenzene (Surr)	460-00-4	100	ug/Kg	2.4	ug/Kg			%		%
Dibromofluoromethane (Surr)	1868-53-7	100	ug/Kg	3.3	ug/Kg			%		%
Trifluorotoluene (Surr)	98-08-8	100	ug/Kg	9.8	ug/Kg			%		%
1,2-Dichloroethane-d4 (Surr)	17060-07-0	100	ug/Kg	3.3	ug/Kg			%		%
Methyl tert-butyl ether	1634-04-4	40	ug/Kg	6	ug/Kg	65	125	%	30	%

SOIL	Closed System Purge and Trap	5035A_M
SOIL	Northwest - Volatile Petroleum Products (GC)	NWTPH_Gx

Analyte Description	CAS Number	RL - Limit	RL - Units	MDL - Limit	MDL - Units	LCSREC - Recovery Low	LCSREC - Recovery High	LCSREC - Units	LCSRPD - Precision	LCSRPD - Units
4-Bromofluorobenzene (Surr)	460-00-4	0.5	mg/Kg	0.095	mg/Kg			%		%
Gasoline	STL00228	4	mg/Kg	0.5	mg/Kg	68	120	%	25	%
Trifluorotoluene (Surr)	98-08-8	0.5	mg/Kg	0.095	mg/Kg			%		%

SOIL	Closed System Purge and Trap	5035A_FM
SOIL	Volatile Organic Compounds by GC/MS (Frozen)	8260C

Analyte Description	CAS Number	RL - Limit	RL - Units	MDL - Limit	MDL - Units	LCSREC - Recovery Low	LCSREC - Recovery High	LCSREC - Units	LCSRPD - Precision	LCSRPD - Units
Toluene-d8 (Surr)	2037-26-5	5	ug/Kg	0.16	ug/Kg			%		%
Dichlorodifluoromethane	75-71-8	1	ug/Kg	0.3	ug/Kg	38	150	%	26	%

Chloromethane	74-87-3	1	ug/Kg	0.3	ug/Kg	55	136	%	26	%
Vinyl chloride	75-01-4	1	ug/Kg	0.3	ug/Kg	67	131	%	22	%
Bromomethane	74-83-9	1	ug/Kg	0.4	ug/Kg	57	148	%	29	%
Chloroethane	75-00-3	1	ug/Kg	0.2	ug/Kg	48	167	%	53	%
Trichlorofluoromethane	75-69-4	1	ug/Kg	0.3	ug/Kg	47	165	%	54	%
1,1-Dichloroethene	75-35-4	5	ug/Kg	0.2	ug/Kg	70	133	%	23	%
Methylene Chloride	75-09-2	15	ug/Kg	3	ug/Kg	57	146	%	21	%
trans-1,2-Dichloroethene	156-60-5	1	ug/Kg	0.4	ug/Kg	76	131	%	18	%
1,1-Dichloroethane	75-34-3	1	ug/Kg	0.4	ug/Kg	70	128	%	21	%
2,2-Dichloropropane	594-20-7	5	ug/Kg	0.3	ug/Kg	56	144	%	21	%
cis-1,2-Dichloroethene	156-59-2	1	ug/Kg	0.3	ug/Kg	70	130	%	19	%
Bromochloromethane	74-97-5	2	ug/Kg	0.5	ug/Kg	78	123	%	19	%
Chloroform	67-66-3	1	ug/Kg	0.3	ug/Kg	78	125	%	17	%
1,1,1-Trichloroethane	71-55-6	1	ug/Kg	0.3	ug/Kg	63	135	%	20	%
Carbon tetrachloride	56-23-5	1	ug/Kg	0.3	ug/Kg	59	145	%	19	%
1,1-Dichloropropene	563-58-6	1	ug/Kg	0.3	ug/Kg	77	125	%	16	%
Benzene	71-43-2	1	ug/Kg	0.3	ug/Kg	70	128	%	19	%
1,2-Dichloroethane	107-06-2	1	ug/Kg	0.4	ug/Kg	71	128	%	18	%
Trichloroethene	79-01-6	1	ug/Kg	0.3	ug/Kg	83	124	%	17	%
1,2-Dichloropropane	78-87-5	1	ug/Kg	0.4	ug/Kg	76	161	%	15	%
Dibromomethane	74-95-3	1	ug/Kg	0.3	ug/Kg	78	126	%	18	%
Bromodichloromethane	75-27-4	1	ug/Kg	0.4	ug/Kg	58	133	%	19	%
cis-1,3-Dichloropropene	10061-01-5	1	ug/Kg	0.2	ug/Kg	69	129	%	19	%
Toluene	108-88-3	2	ug/Kg	0.3	ug/Kg	75	126	%	19	%
trans-1,3-Dichloropropene	10061-02-6	1	ug/Kg	0.2	ug/Kg	72	129	%	20	%
1,1,2-Trichloroethane	79-00-5	2	ug/Kg	0.5	ug/Kg	77	124	%	18	%
Tetrachloroethene	127-18-4	1	ug/Kg	0.4	ug/Kg	56	155	%	27	%
1,3-Dichloropropane	142-28-9	2	ug/Kg	0.5	ug/Kg	77	123	%	19	%
Dibromochloromethane	124-48-1	2	ug/Kg	0.5	ug/Kg	42	129	%	23	%
1,2-Dibromoethane	106-93-4	1	ug/Kg	0.2	ug/Kg	69	126	%	21	%
Chlorobenzene	108-90-7	1	ug/Kg	0.4	ug/Kg	75	120	%	21	%
Ethylbenzene	100-41-4	1	ug/Kg	0.4	ug/Kg	78	126	%	23	%
1,1,1,2-Tetrachloroethane	630-20-6	1	ug/Kg	0.4	ug/Kg	72	123	%	20	%
1,1,2,2-Tetrachloroethane	79-34-5	2	ug/Kg	0.9	ug/Kg	73	125	%	22	%
m-Xylene & p-Xylene	179601-23-1	2	ug/Kg	0.2	ug/Kg	78	126	%	23	%
o-Xylene	95-47-6	2	ug/Kg	0.5	ug/Kg	77	127	%	22	%
Styrene	100-42-5	2	ug/Kg	0.2	ug/Kg	79	127	%	21	%
Bromoform	75-25-2	1	ug/Kg	0.3	ug/Kg	50	124	%	25	%
Isopropylbenzene	98-82-8	2	ug/Kg	0.2	ug/Kg	79	127	%	20	%
Bromobenzene	108-86-1	2	ug/Kg	0.5	ug/Kg	80	120	%	19	%
N-Propylbenzene	103-65-1	2	ug/Kg	0.5	ug/Kg	81	127	%	20	%
1,2,3-Trichloropropane	96-18-4	1	ug/Kg	0.3	ug/Kg	77	123	%	23	%

2-Chlorotoluene	95-49-8	2	ug/Kg	0.5	ug/Kg	79	122	%	18	%
1,3,5-Trimethylbenzene	108-67-8	5	ug/Kg	0.5	ug/Kg	80	125	%	18	%
4-Chlorotoluene	106-43-4	2	ug/Kg	0.5	ug/Kg	80	122	%	18	%
t-Butylbenzene	98-06-6	2	ug/Kg	0.2	ug/Kg	71	136	%	27	%
1,2,4-Trimethylbenzene	95-63-6	2	ug/Kg	0.4	ug/Kg	79	124	%	18	%
sec-Butylbenzene	135-98-8	2	ug/Kg	0.5	ug/Kg	78	128	%	17	%
1,3-Dichlorobenzene	541-73-1	2	ug/Kg	0.5	ug/Kg	79	119	%	17	%
4-Isopropyltoluene	99-87-6	2	ug/Kg	0.4	ug/Kg	78	126	%	18	%
1,4-Dichlorobenzene	106-46-7	1	ug/Kg	0.2	ug/Kg	79	117	%	18	%
n-Butylbenzene	104-51-8	2	ug/Kg	0.2	ug/Kg	78	128	%	17	%
1,2-Dichlorobenzene	95-50-1	2	ug/Kg	0.6	ug/Kg	79	117	%	17	%
1,2-Dibromo-3-Chloropropane	96-12-8	2	ug/Kg	0.3	ug/Kg	53	132	%	27	%
1,2,4-Trichlorobenzene	120-82-1	2	ug/Kg	0.4	ug/Kg	61	130	%	22	%
1,2,3-Trichlorobenzene	87-61-6	2	ug/Kg	0.6	ug/Kg	61	130	%	23	%
Hexachlorobutadiene	87-68-3	2	ug/Kg	0.6	ug/Kg	68	134	%	21	%
Naphthalene	91-20-3	5	ug/Kg	0.5	ug/Kg	14	170	%	50	%
4-Bromofluorobenzene (Surr)	460-00-4	5	ug/Kg	0.19	ug/Kg			%		%
Dibromofluoromethane (Surr)	1868-53-7	5	ug/Kg	0.38	ug/Kg			%		%
Trifluorotoluene (Surr)	98-08-8	5	ug/Kg	0.21	ug/Kg			%		%
1,2-Dichloroethane-d4 (Surr)	17060-07-0	5	ug/Kg	0.32	ug/Kg			%		%
Methyl tert-butyl ether	1634-04-4	1	ug/Kg	0.3	ug/Kg	65	125	%	30	%

SOIL	Closed System Purge and Trap	5035A_FW
SOIL	Metals (ICP/MS)	6020A

Analyte Description	CAS Number	RL - Limit	RL - Units	MDL - Limit	MDL - Units	LCSREC - Recovery Low	LCSREC - Recovery High	LCSREC - Units	LCSRPD - Precision	LCSRPD - Units
Arsenic	7440-38-2	0.5	mg/Kg	0.18	mg/Kg	80	120	%	20	%
Barium	7440-39-3	0.2	mg/Kg	0.03	mg/Kg	80	120	%	20	%
Cadmium	7440-43-9	0.2	mg/Kg	0.008	mg/Kg	80	120	%	20	%
Chromium	7440-47-3	0.2	mg/Kg	0.113	mg/Kg	80	120	%	20	%
Lead	7439-92-1	0.2	mg/Kg	0.013	mg/Kg	80	120	%	20	%
Selenium	7782-49-2	0.7	mg/Kg	0.202	mg/Kg	80	120	%	20	%
Silver	7440-22-4	0.2	mg/Kg	0.012	mg/Kg	80	120	%	20	%

SOIL	Preparation, Metals	3050B
SOIL	Mercury (CVAA)	7471A

Analyte Description	CAS Number	RL - Limit	RL - Units	MDL - Limit	MDL - Units	LCSREC - Recovery Low	LCSREC - Recovery High	LCSREC - Units	LCSRPD - Precision	LCSRPD - Units
Mercury	7439-97-6	0.02	mg/Kg	0.0063	mg/Kg	80	120	%	20	%

SOIL	Preparation, Mercury	7471A_Prep
WATER	Volatile Organic Compounds by GC/MS	8260C

Analyte Description	CAS Number	RL - Limit	RL - Units	MDL - Limit	MDL - Units	LCSREC - Recovery Low	LCSREC - Recovery High	LCSREC - Units	LCSRPD - Precision	LCSRPD - Units
Toluene-d8 (Surr)	2037-26-5	25	ug/L	0.13	ug/L			%		%
Dichlorodifluoromethane	75-71-8	1	ug/L	0.31	ug/L	30	155	%	30	%
Chloromethane	74-87-3	5	ug/L	0.19	ug/L	40	125	%	30	%
Vinyl chloride	75-01-4	1	ug/L	0.22	ug/L	50	145	%	30	%
Bromomethane	74-83-9	5	ug/L	0.27	ug/L	30	145	%	30	%
Chloroethane	75-00-3	5	ug/L	0.4	ug/L	60	135	%	30	%
Trichlorofluoromethane	75-69-4	1	ug/L	0.26	ug/L	60	145	%	30	%
1,1-Dichloroethene	75-35-4	1	ug/L	0.33	ug/L	70	130	%	30	%
Methylene Chloride	75-09-2	3	ug/L	1.34	ug/L	55	140	%	30	%
trans-1,2-Dichloroethene	156-60-5	1	ug/L	0.24	ug/L	60	140	%	30	%
1,1-Dichloroethane	75-34-3	1	ug/L	0.17	ug/L	70	135	%	30	%
2,2-Dichloropropane	594-20-7	1	ug/L	0.2	ug/L	70	135	%	30	%
cis-1,2-Dichloroethene	156-59-2	1	ug/L	0.21	ug/L	70	125	%	30	%
Bromochloromethane	74-97-5	1	ug/L	0.29	ug/L	65	130	%	30	%
Chloroform	67-66-3	1	ug/L	0.17	ug/L	65	135	%	30	%
1,1,1-Trichloroethane	71-55-6	1	ug/L	0.26	ug/L	65	130	%	30	%
Carbon tetrachloride	56-23-5	1	ug/L	0.24	ug/L	65	140	%	30	%
1,1-Dichloropropene	563-58-6	1	ug/L	0.12	ug/L	75	130	%	30	%
Benzene	71-43-2	1	ug/L	0.14	ug/L	80	120	%	30	%
1,2-Dichloroethane	107-06-2	1	ug/L	0.16	ug/L	70	130	%	30	%
Trichloroethene	79-01-6	1	ug/L	0.12	ug/L	70	125	%	30	%
1,2-Dichloropropane	78-87-5	1	ug/L	0.18	ug/L	75	125	%	30	%
Dibromomethane	74-95-3	1	ug/L	0.14	ug/L	75	125	%	30	%
Bromodichloromethane	75-27-4	1	ug/L	0.13	ug/L	75	120	%	30	%
cis-1,3-Dichloropropene	10061-01-5	1	ug/L	0.2	ug/L	70	130	%	30	%
Toluene	108-88-3	1	ug/L	0.16	ug/L	75	120	%	30	%
trans-1,3-Dichloropropene	10061-02-6	1	ug/L	0.16	ug/L	55	140	%	30	%
1,1,2-Trichloroethane	79-00-5	1	ug/L	0.24	ug/L	75	125	%	30	%
Tetrachloroethene	127-18-4	1	ug/L	0.21	ug/L	45	150	%	30	%
1,3-Dichloropropane	142-28-9	1	ug/L	0.15	ug/L	75	125	%	30	%
Dibromochloromethane	124-48-1	1	ug/L	0.2	ug/L	60	135	%	30	%
1,2-Dibromoethane	106-93-4	1	ug/L	0.15	ug/L	80	120	%	30	%
Chlorobenzene	108-90-7	1	ug/L	0.11	ug/L	80	120	%	30	%
Ethylbenzene	100-41-4	1	ug/L	0.13	ug/L	75	125	%	30	%
1,1,1,2-Tetrachloroethane	630-20-6	1	ug/L	0.18	ug/L	80	130	%	30	%
1,1,2,2-Tetrachloroethane	79-34-5	1	ug/L	0.24	ug/L	65	130	%	30	%
m-Xylene & p-Xylene	179601-23-1	2	ug/L	0.13	ug/L	75	130	%	30	%
o-Xylene	95-47-6	1	ug/L	0.12	ug/L	80	120	%	30	%
Styrene	100-42-5	5	ug/L	0.62	ug/L	65	135	%	30	%
Bromoform	75-25-2	1	ug/L	0.21	ug/L	70	130	%	30	%
Isopropylbenzene	98-82-8	1	ug/L	0.3	ug/L	75	125	%	30	%

Bromobenzene	108-86-1	1	ug/L	0.11	ug/L	75	125	%	30	%
N-Propylbenzene	103-65-1	1	ug/L	0.13	ug/L	70	130	%	30	%
1,2,3-Trichloropropane	96-18-4	2	ug/L	0.41	ug/L	75	125	%	30	%
2-Chlorotoluene	95-49-8	1	ug/L	0.14	ug/L	75	125	%	30	%
1,3,5-Trimethylbenzene	108-67-8	1	ug/L	0.13	ug/L	75	130	%	30	%
4-Chlorotoluene	106-43-4	1	ug/L	0.16	ug/L	75	130	%	30	%
t-Butylbenzene	98-06-6	1	ug/L	0.18	ug/L	70	130	%	30	%
1,2,4-Trimethylbenzene	95-63-6	1	ug/L	0.11	ug/L	75	130	%	30	%
sec-Butylbenzene	135-98-8	1	ug/L	0.15	ug/L	70	125	%	30	%
1,3-Dichlorobenzene	541-73-1	1	ug/L	0.15	ug/L	75	125	%	30	%
4-Isopropyltoluene	99-87-6	1	ug/L	0.13	ug/L	75	130	%	30	%
1,4-Dichlorobenzene	106-46-7	1	ug/L	0.13	ug/L	75	125	%	30	%
n-Butylbenzene	104-51-8	2	ug/L	0.63	ug/L	70	135	%	30	%
1,2-Dichlorobenzene	95-50-1	1	ug/L	0.14	ug/L	70	120	%	30	%
1,2-Dibromo-3-Chloropropane	96-12-8	2	ug/L	0.4	ug/L	50	130	%	30	%
1,2,4-Trichlorobenzene	120-82-1	1	ug/L	0.23	ug/L	65	135	%	30	%
1,2,3-Trichlorobenzene	87-61-6	1	ug/L	0.14	ug/L	55	140	%	30	%
Hexachlorobutadiene	87-68-3	1	ug/L	0.13	ug/L	50	140	%	30	%
Naphthalene	91-20-3	3	ug/L	0.26	ug/L	55	140	%	30	%
4-Bromofluorobenzene (Surr)	460-00-4	5	ug/L	0.17	ug/L			%		%
Dibromofluoromethane (Surr)	1868-53-7	5	ug/L	0.19	ug/L					
Trifluorotoluene (Surr)	98-08-8	5	ug/L	1.23	ug/L			%		%
1,2-Dichloroethane-d4 (Surr)	17060-07-0	5	ug/L	0.2	ug/L			%		
Methyl tert-butyl ether	1634-04-4	1	ug/L	0.17	ug/L	65	125	%	30	%

WATER	Purge and Trap	5030B

WATER	Northwest - Semi-Volatile Petroleum Products (GC)	NWTPH_Dx

Analyte Description	CAS Number	RL - Limit	RL - Units	MDL - Limit	MDL - Units	LCSREC - Recovery Low	LCSREC - Recovery High	LCSREC - Units	LCSRPD - Precision	LCSRPD - Units
o-Terphenyl	84-15-1	0.008	mg/L	0.00018	mg/L			%		%
#2 Diesel (C10-C24)	STL00163	0.125	mg/L	0.019	mg/L	59	120	%	27	%
Motor Oil (>C24-C36)	STL00299	0.25	mg/L	0.029	mg/L	71	140	%	27	%

WATER	Liquid-Liquid Extraction (Separatory Funnel)	3510C_LVI_14d
WATER	Northwest - Volatile Petroleum Products (GC)	NWTPH_Gx

Analyte Description	CAS Number	RL - Limit	RL - Units	MDL - Limit	MDL - Units	LCSREC - Recovery Low	LCSREC - Recovery High	LCSREC - Units	LCSRPD - Precision	LCSRPD - Units
4-Bromofluorobenzene (Surr)	460-00-4	0.001	mg/L	0.00032	mg/L			%		%
Gasoline	STL00228	0.05	mg/L	0.01	mg/L	79	110	%	20	%
Trifluorotoluene (Surr)	98-08-8	0.001	mg/L	0.0005	mg/L			%		%

WATER	Purge and Trap	5030B

WATER	Semivolatile Organic Compounds (GC/MS)	8270D

Analyte Description	CAS Number	RL - Limit	RL - Units	MDL - Limit	MDL - Units	LCSREC - Recovery Low	LCSREC - Recovery High	LCSREC - Units	LCSRPD - Precision	LCSRPD - Units
2-Fluorophenol (Surr)	367-12-4	0.4	ug/L	0.0968	ug/L			%		%
Phenol-d5 (Surr)	4165-62-2	0.4	ug/L	0.0176	ug/L			%		%
Nitrobenzene-d5 (Surr)	4165-60-0	0.2	ug/L	0.0172	ug/L			%		%
2-Fluorobiphenyl	321-60-8	0.2	ug/L	0.0092	ug/L			%		%
2,4,6-Tribromophenol (Surr)	118-79-6	0.4	ug/L	0.0746	ug/L	60	140	%	20	%
Terphenyl-d14 (Surr)	1718-51-0	0.2	ug/L	0.009	ug/L			%		%
Phenol	108-95-2	0.6	ug/L	0.1	ug/L	53	130	%	20	%
Bis(2-chloroethyl)ether	111-44-4	0.4	ug/L	0.1	ug/L	55	125	%	20	%
2-Chlorophenol	95-57-8	0.4	ug/L	0.1	ug/L	57	125	%	20	%
1,3-Dichlorobenzene	541-73-1	0.4	ug/L	0.1	ug/L	40	125	%	20	%
1,4-Dichlorobenzene	106-46-7	0.4	ug/L	0.1	ug/L	40	125	%	20	%
Benzyl alcohol	100-51-6	0.4	ug/L	0.1	ug/L	41	144	%	20	%
1,2-Dichlorobenzene	95-50-1	0.4	ug/L	0.1	ug/L	44	125	%	20	%
2-Methylphenol	95-48-7	0.4	ug/L	0.1	ug/L	60	130	%	20	%
3 & 4 Methylphenol	15831-10-4	0.8	ug/L	0.1	ug/L	60	130	%	20	%
N-Nitrosodi-n-propylamine	621-64-7	0.4	ug/L	0.1	ug/L	60	120	%	20	%
Hexachloroethane	67-72-1	0.6	ug/L	0.1	ug/L	30	125	%	20	%
Nitrobenzene	98-95-3	0.4	ug/L	0.1	ug/L	62	125	%	20	%
Isophorone	78-59-1	0.4	ug/L	0.1	ug/L	64	125	%	20	%
2-Nitrophenol	88-75-5	0.4	ug/L	0.1	ug/L	55	140	%	20	%
2,4-Dimethylphenol	105-67-9	2	ug/L	0.3	ug/L	30	135	%	20	%
Benzoic acid	65-85-0	3	ug/L	0.6	ug/L	20	144	%	20	%
Bis(2-chloroethoxy)methane	111-91-1	0.4	ug/L	0.1	ug/L	59	125	%	20	%
2,4-Dichlorophenol	120-83-2	0.4	ug/L	0.1	ug/L	50	140	%	20	%
1,2,4-Trichlorobenzene	120-82-1	0.4	ug/L	0.1	ug/L	40	125	%	20	%
Naphthalene	91-20-3	0.4	ug/L	0.1	ug/L	56	125	%	20	%
4-Chloroaniline	106-47-8	0.4	ug/L	0.1	ug/L	20	150	%	20	%
Hexachlorobutadiene	87-68-3	0.6	ug/L	0.1	ug/L	25	125	%	20	%
4-Chloro-3-methylphenol	59-50-7	0.4	ug/L	0.1	ug/L	65	145	%	20	%
2-Methylnaphthalene	91-57-6	0.2	ug/L	0.02	ug/L	56	125	%	20	%
Hexachlorocyclopentadiene	77-47-4	2	ug/L	0.1	ug/L	20	125	%	20	%
2,4,6-Trichlorophenol	88-06-2	0.6	ug/L	0.1	ug/L	55	140	%	20	%
2,4,5-Trichlorophenol	95-95-4	0.4	ug/L	0.1	ug/L	66	130	%	20	%
2-Chloronaphthalene	91-58-7	0.06	ug/L	0.02	ug/L	55	125	%	20	%
2-Nitroaniline	88-74-4	0.4	ug/L	0.1	ug/L	52	140	%	20	%
Dimethyl phthalate	131-11-3	0.4	ug/L	0.1	ug/L	65	155	%	20	%
Acenaphthylene	208-96-8	0.08	ug/L	0.02	ug/L	62	125	%	20	%
2,6-Dinitrotoluene	606-20-2	0.4	ug/L	0.1	ug/L	67	134	%	20	%
3-Nitroaniline	99-09-2	0.4	ug/L	0.12	ug/L	22	124	%	20	%

Acenaphthene	83-32-9	0.1	ug/L	0.02	ug/L	63	125	%	20	%
2,4-Dinitrophenol	51-28-5	5	ug/L	1	ug/L	24	146	%	20	%
4-Nitrophenol	100-02-7	3	ug/L	1	ug/L	35	153	%	20	%
Dibenzofuran	132-64-9	0.4	ug/L	0.1	ug/L	60	125	%	20	%
2,4-Dinitrotoluene	121-14-2	0.4	ug/L	0.1	ug/L	73	126	%	20	%
Diethyl phthalate	84-66-2	0.4	ug/L	0.1	ug/L	60	150	%	20	%
4-Chlorophenyl phenyl ether	7005-72-3	0.4	ug/L	0.1	ug/L	59	125	%	20	%
Fluorene	86-73-7	0.06	ug/L	0.02	ug/L	69	125	%	20	%
4-Nitroaniline	100-01-6	0.6	ug/L	0.1	ug/L	49	125	%	20	%
4,6-Dinitro-2-methylphenol	534-52-1	4	ug/L	1	ug/L	50	136	%	20	%
N-Nitrosodiphenylamine	86-30-6	0.4	ug/L	0.1	ug/L	40	135	%	20	%
4-Bromophenyl phenyl ether	101-55-3	0.4	ug/L	0.1	ug/L	62	132	%	20	%
Hexachlorobenzene	118-74-1	0.4	ug/L	0.1	ug/L	61	125	%	20	%
Pentachlorophenol	87-86-5	0.7	ug/L	0.1	ug/L	20	145	%	20	%
Phenanthrene	85-01-8	0.08	ug/L	0.02	ug/L	70	125	%	20	%
Anthracene	120-12-7	0.04	ug/L	0.01	ug/L	50	125	%	20	%
Di-n-butyl phthalate	84-74-2	0.4	ug/L	0.13	ug/L	55	167	%	20	%
Fluoranthene	206-44-0	0.05	ug/L	0.013	ug/L	70	145	%	20	%
Pyrene	129-00-0	0.06	ug/L	0.013	ug/L	70	133	%	20	%
Butyl benzyl phthalate	85-68-7	0.6	ug/L	0.2	ug/L	60	167	%	20	%
3,3'-Dichlorobenzidine	91-94-1	2	ug/L	0.1	ug/L	20	175	%	20	%
Benzo[a]anthracene	56-55-3	0.06	ug/L	0.02	ug/L	65	125	%	20	%
Chrysene	218-01-9	0.04	ug/L	0.013	ug/L	70	125	%	20	%
Bis(2-ethylhexyl) phthalate	117-81-7	3	ug/L	1.18	ug/L	70	185	%	20	%
Di-n-octyl phthalate	117-84-0	0.4	ug/L	0.18	ug/L	55	150	%	20	%
Benzo[a]pyrene	50-32-8	0.04	ug/L	0.02	ug/L	45	125	%	20	%
Indeno[1,2,3-cd]pyrene	193-39-5	0.06	ug/L	0.02	ug/L	70	136	%	20	%
Dibenz(a,h)anthracene	53-70-3	0.06	ug/L	0.02	ug/L	69	154	%	20	%
Benzo[g,h,i]perylene	191-24-2	0.06	ug/L	0.02	ug/L	65	153	%	20	%
Carbazole	86-74-8	0.4	ug/L	0.1	ug/L	75	142	%	20	%
1-Methylnaphthalene	90-12-0	0.06	ug/L	0.03	ug/L	54	125	%	20	%
Benzo[b]fluoranthene	205-99-2	0.08	ug/L	0.02	ug/L	70	129	%	20	%
Benzo[k]fluoranthene	207-08-9	0.06	ug/L	0.02	ug/L	70	123	%	20	%
bis(chloroisopropyl) ether	108-60-1	0.4	ug/L	0.1	ug/L	44	130	%	20	%

WATER	Liquid-Liquid Extraction (Continuous)	3520C
WATER	EDB, DBCP, and 1,2,3-TCP (GC)	8011

Analyte Description	CAS Number	RL - Limit	RL - Units	MDL - Limit	MDL - Units	LCSREC - Recovery Low	LCSREC - Recovery High	LCSREC - Units	LCSRPD - Precision	LCSRPD - Units
Ethylene Dibromide	106-93-4	0.01	ug/L	0.002	ug/L	70	130	%	20	%
1,2-Dibromopropane	78-75-1	0.01	ug/L	0.003	ug/L			%		%

WATER	Microextraction	8011_Prep
WATER	Metals (ICP/MS)	6020A

	Analyte Description	CAS Number	RL - Limit	RL - Units	MDL - Limit	MDL - Units	LCSREC - Recovery Low	LCSREC - Recovery High	LCSREC - Units	LCSRPD - Precision	LCSRPD - Units
0.01875	Arsenic	7440-38-2	0.005	mg/L	0.00375	mg/L	80	120	%	20	%
0.00135	Barium	7440-39-3	0.006	mg/L	0.00027	mg/L	80	120	%	20	%
0.0007	Cadmium	7440-43-9	0.002	mg/L	0.00014	mg/L	80	120	%	20	%
0.00675	Chromium	7440-47-3	0.002	mg/L	0.00135	mg/L	80	120	%	20	%
0.00085	Lead	7439-92-1	0.002	mg/L	0.00017	mg/L	80	120	%	20	%
0.01775	Selenium	7782-49-2	0.005	mg/L	0.00355	mg/L	80	120	%	20	%
0.00075	Silver	7440-22-4	0.002	mg/L	0.00015	mg/L	80	120	%	20	%

WATER	Preparation, Total Recoverable or Dissolved Metals	3005A
WATER	Mercury (CVAA)	7470A

Analyte Description	CAS Number	RL - Limit	RL - Units	MDL - Limit	MDL - Units	LCSREC - Recovery Low	LCSREC - Recovery High	LCSREC - Units	LCSRPD - Precision	LCSRPD - Units
Mercury	7439-97-6	0.0002	mg/L	0.000041	mg/L	80	120	%	20	%

WATER	Preparation, Mercury	7470A_Prep

MSREC - Recovery Low	MSREC - Recovery High	MSREC - Units	MSRPD - Precision	MSRPD - Units	SUREC - Recovery Low	SUREC - Recovery High	SUREC - Units
		%		%	50	150	%
70	125	%	16	%			%
64	127	%	17	%			%

MSREC - Recovery Low	MSREC - Recovery High	MSREC - Units	MSRPD - Precision	MSRPD - Units	SUREC - Recovery Low	SUREC - Recovery High	SUREC - Units
		%		%	36	145	%
		%		%	38	149	%
		%		%	38	141	%
		%		%	42	140	%
		%		%	28	143	%
		%		%	42	151	%
70	140	%	26	%			%
70	125	%	60	%			%
65	135	%	27	%			%
75	125	%	60	%			%
75	125	%	32	%			%
55	125	%	60	%			%
75	125	%	60	%			%
75	130	%	25	%			%
75	130	%	27	%			%
75	140	%	28	%			%
75	125	%	60	%			%
75	125	%	60	%			%
75	125	%	60	%			%
65	140	%	60	%			%
65	140	%	60	%			%
20	175	%	60	%			%
75	125	%	60	%			%
65	130	%	60	%			%
70	125	%	28	%			%
75	125	%	26	%			%
20	160	%	60	%			%
75	125	%	60	%			%
75	135	%	27	%			%
75	125	%	27	%			%
30	125	%	60	%			%

65	140	%	60	%			%
75	125	%	60	%			%
75	125	%	25	%			%
75	135	%	60	%			%
60	160	%	60	%			%
75	125	%	28	%			%
75	125	%	60	%			%
60	140	%	60	%			%
75	125	%	27	%			%
60	135	%	60	%			%
65	125	%	33	%			%
75	125	%	60	%			%
75	125	%	31	%			%
60	155	%	26	%			%
75	125	%	60	%			%
75	125	%	31	%			%
65	125	%	60	%			%
50	135	%	60	%			%
75	125	%	60	%			%
75	125	%	60	%			%
75	125	%	60	%			%
55	125	%	68	%			%
75	125	%	28	%			%
75	125	%	27	%			%
55	145	%	60	%			%
70	125	%	36	%			%
75	125	%	31	%			%
55	145	%	60	%			%
20	160	%	60	%			%
75	125	%	27	%			%
75	125	%	26	%			%
55	145	%	60	%			%
55	145	%	31	%			%
75	125	%	30	%			%
75	125	%	29	%			%
75	125	%	30	%			%
75	125	%	28	%			%
75	125	%	60	%			%
75	125	%	30	%			%
75	125	%	31	%			%
75	125	%	31	%			%
75	125	%	60	%			

MSREC - Recovery Low	MSREC - Recovery High	MSREC - Units	MSRPD - Precision	MSRPD - Units	SUREC - Recovery Low	SUREC - Recovery High	SUREC - Units
		%		%	80	120	%
35	135	%	30	%			%
50	130	%	30	%			%
60	125	%	30	%			%
30	160	%	30	%			%
40	155	%	30	%			%
25	185	%	30	%			%
65	135	%	30	%			%
55	140	%	30	%			%
65	135	%	30	%			%
75	125	%	30	%			%
65	135	%	30	%			%
65	125	%	30	%			%
70	125	%	30	%			%
70	125	%	30	%			%
70	135	%	30	%			%
65	135	%	30	%			%
70	135	%	30	%			%
75	125	%	30	%			%
70	135	%	30	%			%
75	125	%	30	%			%
70	120	%	30	%			%
75	130	%	30	%			%
70	130	%	30	%			%
70	125	%	30	%			%
70	125	%	30	%			%
65	125	%	30	%			%
60	125	%	30	%			%
65	140	%	30	%			%
75	125	%	30	%			%
65	130	%	30	%			%
70	125	%	30	%			%
75	125	%	30	%			%
75	125	%	30	%			%
75	125	%	30	%			%
55	130	%	30	%			%
80	125	%	30	%			%

75	125	%	30	%			%
75	125	%	30	%			%
55	135	%	30	%			%
75	130	%	30	%			%
65	120	%	30	%			%
65	135	%	30	%			%
65	130	%	30	%			%
70	130	%	30	%			%
65	135	%	30	%			%
75	125	%	30	%			%
65	130	%	30	%			%
65	135	%	30	%			%
65	130	%	30	%			%
70	125	%	30	%			%
75	135	%	30	%			%
70	125	%	30	%			%
65	140	%	30	%			%
75	120	%	30	%			%
40	135	%	30	%			%
65	130	%	30	%			%
60	135	%	30	%			%
55	140	%	30	%			%
40	125	%	30	%			%
		%		%	70	120	%
				%	75	132	%
		%		%	65	140	%
		%		%	71	136	%
59	137	%	30	%			%

MSREC - Recovery Low	MSREC - Recovery High	MSREC - Units	MSRPD - Precision	MSRPD - Units	SUREC - Recovery Low	SUREC - Recovery High	SUREC - Units
		%		%	50	150	%
50	150	%	35	%			%
		%		%	50	150	%

MSREC - Recovery Low	MSREC - Recovery High	MSREC - Units	MSRPD - Precision	MSRPD - Units	SUREC - Recovery Low	SUREC - Recovery High	SUREC - Units
		%		%	80	120	%
35	135	%	30	%			%

50	130	%	30	%			%
60	125	%	30	%			%
30	160	%	30	%			%
40	155	%	30	%			%
25	185	%	30	%			%
65	135	%	30	%			%
55	140	%	30	%			%
65	135	%	30	%			%
75	125	%	30	%			%
65	135	%	30	%			%
65	125	%	30	%			%
70	125	%	30	%			%
70	125	%	30	%			%
70	135	%	30	%			%
65	135	%	30	%			%
70	135	%	30	%			%
75	125	%	30	%			%
70	135	%	30	%			%
75	125	%	30	%			%
70	120	%	30	%			%
75	130	%	30	%			%
70	130	%	30	%			%
70	125	%	30	%			%
70	125	%	30	%			%
65	125	%	30	%			%
60	125	%	30	%			%
65	140	%	30	%			%
75	125	%	30	%			%
65	130	%	30	%			%
70	125	%	30	%			%
75	125	%	30	%			%
75	125	%	30	%			%
75	125	%	30	%			%
55	130	%	30	%			%
80	125	%	30	%			%
75	125	%	30	%			%
75	125	%	30	%			%
55	135	%	30	%			%
75	130	%	30	%			%
65	120	%	30	%			%
65	135	%	30	%			%
65	130	%	30	%			%

70	130	%	30	%			%
65	135	%	30	%			%
75	125	%	30	%			%
65	130	%	30	%			%
65	135	%	30	%			%
65	130	%	30	%			%
70	125	%	30	%			%
75	135	%	30	%			%
70	125	%	30	%			%
65	140	%	30	%			%
75	120	%	30	%			%
40	135	%	30	%			%
65	130	%	30	%			%
60	135	%	30	%			%
55	140	%	30	%			%
40	125	%	30	%			%
		%		%	70	120	%
				%	75	132	%
		%		%	65	140	%
		%		%	71	136	%
59	137	%	30	%			%

MSREC - Recovery Low	MSREC - Recovery High	MSREC - Units	MSRPD - Precision	MSRPD - Units	SUREC - Recovery Low	SUREC - Recovery High	SUREC - Units
80	120	%	20	%			
80	120	%	20	%			
80	120	%	20	%			
80	120	%	20	%			
80	120	%	20	%			
80	120	%	20	%			
80	120	%	20	%			

MSREC - Recovery Low	MSREC - Recovery High	MSREC - Units	MSRPD - Precision	MSRPD - Units	SUREC - Recovery Low	SUREC - Recovery High	SUREC - Units
80	120	%	20	%			

MSREC - Recovery Low	MSREC - Recovery High	MSREC - Units	MSRPD - Precision	MSRPD - Units	SUREC - Recovery Low	SUREC - Recovery High	SUREC - Units
		%	30	%	85	120	%
30	155	%	30	%			%
40	125	%	30	%			%
50	145	%	30	%			%
30	145	%	30	%			%
60	135	%	30	%			%
60	145	%	30	%			%
70	130	%	30	%			%
55	140	%	30	%			%
60	140	%	30	%			%
70	135	%	30	%			%
70	135	%	30	%			%
70	125	%	30	%			%
65	130	%	30	%			%
65	135	%	30	%			%
65	130	%	30	%			%
65	140	%	30	%			%
75	130	%	30	%			%
80	120	%	30	%			%
70	130	%	30	%			%
70	125	%	30	%			%
75	125	%	30	%			%
75	125	%	30	%			%
75	120	%	30	%			%
70	130	%	30	%			%
75	120	%	30	%			%
55	140	%	30	%			%
75	125	%	30	%			%
45	150	%	30	%			%
75	125	%	30	%			%
60	135	%	30	%			%
80	120	%	30	%			%
80	120	%	30	%			%
75	125	%	30	%			%
80	130	%	30	%			%
65	130	%	30	%			%
75	130	%	30	%			%
80	120	%	30	%			%
65	135	%	30	%			%
70	130	%	30	%			%
75	125	%	30	%			%

75	125	%	30	%			%
70	130	%	30	%			%
75	125	%	30	%			%
75	125	%	30	%			%
75	130	%	30	%			%
75	130	%	30	%			%
70	130	%	30	%			%
75	130	%	30	%			%
70	125	%	30	%			%
75	125	%	30	%			%
75	130	%	30	%			%
75	125	%	30	%			%
70	135	%	30	%			%
70	120	%	30	%			%
50	130	%	30	%			%
65	135	%	30	%			%
55	140	%	30	%			%
50	140	%	30	%			%
55	140	%	30	%			%
		%	30	%	75	120	%
			30	%	85	115	%
		%	30	%	70	136	%
		%			70	120	%
65	125	%	30	%			%

MSREC - Recovery Low	MSREC - Recovery High	MSREC - Units	MSRPD - Precision	MSRPD - Units	SUREC - Recovery Low	SUREC - Recovery High	SUREC - Units
		%		%	50	150	%
59	120	%	27	%			%
71	140	%	27	%			%

MSREC - Recovery Low	MSREC - Recovery High	MSREC - Units	MSRPD - Precision	MSRPD - Units	SUREC - Recovery Low	SUREC - Recovery High	SUREC - Units
		%		%	50	150	%
50	150	%	35	%			%
		%		%	50	150	%

MSREC - Recovery Low	MSREC - Recovery High	MSREC - Units	MSRPD - Precision	MSRPD - Units	SUREC - Recovery Low	SUREC - Recovery High	SUREC - Units
		%		%	30	134	%
		%		%	52	120	%
		%		%	59	120	%
		%		%	50	120	%
60	140	%	20	%	44	125	%
		%		%	64	150	%
65	130	%	20	%			%
65	125	%	20	%			%
60	130	%	20	%			%
40	125	%	20	%			%
40	125	%	20	%			%
65	125	%	20	%			%
45	125	%	20	%			%
70	130	%	20	%			%
65	130	%	20	%			%
70	130	%	20	%			%
30	125	%	20	%			%
70	125	%	20	%			%
75	125	%	20	%			%
55	140	%	20	%			%
30	135	%	20	%			%
20	140	%	20	%			%
75	125	%	20	%			%
50	140	%	20	%			%
40	125	%	20	%			%
60	125	%	20	%			%
35	175	%	20	%			%
25	125	%	20	%			%
65	145	%	20	%			%
60	125	%	20	%			%
20	125	%	20	%			%
55	140	%	20	%			%
75	125	%	20	%			%
60	125	%	20	%			%
75	140	%	20	%			%
65	155	%	20	%			%
65	125	%	20	%			%
75	125	%	20	%			%
75	140	%	20	%			%

65	125	%	20	%			%
50	130	%	20	%			%
35	145	%	20	%			%
60	125	%	20	%			%
75	125	%	20	%			%
60	150	%	20	%			%
70	125	%	20	%			%
70	125	%	20	%			%
70	125	%	20	%			%
50	125	%	20	%			%
40	135	%	20	%			%
75	125	%	20	%			%
70	125	%	20	%			%
20	145	%	20	%			%
75	125	%	20	%			%
50	125	%	20	%			%
55	155	%	20	%			%
70	125	%	20	%			%
70	125	%	20	%			%
60	150	%	20	%			%
20	175	%	20	%			%
65	125	%	20	%			%
70	125	%	20	%			%
20	175	%	20	%			%
55	150	%	20	%			%
45	125	%	20	%			%
75	125	%	20	%			%
75	130	%	20	%			%
75	125	%	20	%			%
75	125	%	20	%			%
60	125	%	20	%			%
70	125	%	20	%			%
70	125	%	20	%			%
65	125	%	20	%			%

MSREC - Recovery Low	MSREC - Recovery High	MSREC - Units	MSRPD - Precision	MSRPD - Units	SUREC - Recovery Low	SUREC - Recovery High	SUREC - Units
70	130	%	20	%			%
		%		%	70	130	%

MSREC - Recovery Low	MSREC - Recovery High	MSREC - Units	MSRPD - Precision	MSRPD - Units	SUREC - Recovery Low	SUREC - Recovery High	SUREC - Units
80	120	%	20	%			
80	120	%	20	%			
80	120	%	20	%			
80	120	%	20	%			
80	120	%	20	%			
80	120	%	20	%			
80	120	%	20	%			

MSREC - Recovery Low	MSREC - Recovery High	MSREC - Units	MSRPD - Precision	MSRPD - Units	SUREC - Recovery Low	SUREC - Recovery High	SUREC - Units
80	120	%	20	%			

Seattle/Tacoma
5755 8th Street East

Tacoma, WA 98424
phone 253.922.2310 fax 253.922.5047

Chain of Custody Record

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

TestAmerica Laboratories, Inc.

Client Contact	**Project Manager: Julie Sharp-Dahl**	**Site Contact: Patrick Braley**	**Date:**	COC No:
Bristol Environmental Remediation Services, LLC	**Tel/Fax: (907) 743-9394**	**Lab Contact: Melissa Armstrong**	**Carrier:**	_____ of _____ COCs
111 W 16th Ave., Third Floor	**Analysis Turnaround Time**			Job No.
Anchorage, Alaska 99501	Calendar (C) or Work Days (W) _____			
(907) 563-0013 Phone	TAT if different from Below _____			
(907) 563-6713 Fax	☐ 2 weeks			SDG No.
Project Name:	☐ 1 week			
Site:	☐ 2 days			
P O #	☐ 1 day			

Sample Identification	**Sample Date**	**Sample Time**	**Sample Type**	**Matrix**	**# of Cont.**	**Filtered Sample**																		Sample Specific Notes:

Preservation Used: 1= Ice, 2= HCl; 3= H2SO4; 4=HNO3; 5=NaOH; 6= Other _____

Possible Hazard Identification
☐ *Non-Hazard* ☐ *Flammable* ☐ *Skin Irritant* *Poison B* ☐ *Unknown* ☐

Sample Disposal (A fee may be assessed if samples are retained longer than 1 month)
☐ *Return To Client* ☐ *Disposal By Lab* ☐ *Archive For ____1_____ Months*

Special Instructions/QC Requirements & Comments:

Relinquished by:	Company:	Date/Time:	Received by:	Company:	Date/Time:
Relinquished by:	Company:	Date/Time:	Received by:	Company:	Date/Time:
Relinquished by:	Company:	Date/Time:	Received by:	Company:	Date/Time:

APPENDIX B

QAPP Amendment Form

QAPP AMENDMENT FORM

Sample Alteration Form

Project Name and Number:_____
Material to be Sampled:_____

Measurement Parameter:_____

Standard Procedure for Field Collection & Laboratory Analysis (cite reference):

_____
_____
_____
_____

Reason for Change in Field Procedure or Analysis Variation:

_____
_____
_____
_____
_____

Variation from Field or Analytical Procedure:

_____
_____
_____
_____
_____

Special Equipment, Materials or Personnel Required:

_____
_____
_____
_____
_____

Initiators Name:_____ Date:_____

Project Manager: _____ Date:_____

QA Officer:_____Date:_____

APPENDIX C

Field Forms and Equipment Manuals

Bristol

ENVIRONMENTAL REMEDIATION SERVICES, LLC

BOREHOLE ID:

Site Name:

Site Location:

Date Started: Completed:

Drill Rig: Drill Company:

Driller: Borehole Dia. (ft):

Logged By:

Company:

Sample Method: Total Depth:

Client:

Job No:

Sketch Well Location:

Depth in Feet	Sample Interval	Blow Count	% Recovery	Sample ID	DESCRIPTION	PID (ppm)	WATER LEVEL	USCS	Estimated % Fines	Estimated % Sand	Estimated % Grave	REMARKS

Bristol

ENVIRONMENTAL
REMEDIATION SERVICES, LLC

Equipment Operator's Checklist

Company: _____ **Operator:** _____ **Date:** _____

Equipment Type: _____ **Model:** _____

Equipment Items	Good Condition	Needs Attention	Notes:
Steering Brakes			
Wheels, Tires, Tracks			
Horn, Back-up Alarm			
Seatbelt, Safety device			
Roll over Protection			
Fire Extinguisher			
Equip. Maintenance			
Any Other Deficiency			

Site Work Inspection

Site Work Inspection	None	Yes	Notes:
Material Obstructions			
Slip, Trip, & Fall Def.			
Ruts, Holes, Hazards			
Barricades / Perimeter			
Trenches/Excavations			
Overhead Power lines			
Traffic Exposures			
Any Other Deficiency			

Safety Comments:

_____

_____

_____

_____

_____

Signature: _____ **Date:** _____

Bristol
ENVIRONMENTAL
REMEDIATION SERVICES, LLC

GROUNDWATER LOW-FLOW PURGING FORM

Job Name _____ Well No.: _____

Job Number _____ Well Type: ☐ Monitor ☐ Extraction ☐ Other _____

Company _____ Well Material ☐ PVC ☐ St. Steel ☐ Other _____

_____ Date _____ Time: _____

Purged by _____ _____

(Signature)

WELL PURGING

PURGE VOLUME

Casing Diameter (D in inches):

☐ 2-inch ☐ 4-inch ☐ 6-inch ☐ Other _____

Total Depth of Casing (TD in feet BTOC): _____

Water Level Depth (WL in feet BTOC): _____

_____

PURGE METHOD

☐ Pump – Type: _____

☐ Submersible ☐ Centrifugal ☐ Bladder ☐ Peristaltic.

☐ Other – Type: _____

PUMP INTAKE SETTING

☐ Near Bottom ☐ Near Top ☐ Other

Depth in feet (BTOC): _____ Screen Interval in Feet (BTOC)

PURGE TIME

_____ Start _____ Stop _____ Elapsed

PURGE RATE

Initial _____ gpm Final _____ gpm

ACTUAL PURGE VOLUME

_____ gallons

FIELD PARAMETER MEASUREMENT

Minutes Since Pumping Began	Water Depth below MP	Pump Dial	Purge Rate (ml/min)	T ☐ °C ☐ °F	Specific Cond. (µS/cm)	pH	ORP (mV)	DO (mg/L)	Turbidity (NTU)	Cumulative Volume Purged

GROUNDWATER LOW-FLOW PURGING FORM (continued)

FIELD PARAMETER MEASUREMENT (Continued)

Minutes Since Pumping Began	Water Depth below MP	Pump Dial	Purge Rate (ml/min)	T ☐ °C ☐ °F	Specific Cond. (µS/cm)	pH	ORP (mV)	DO (mg/L)	Turbidity (NTU)	Cumulative Volume Purged

Bristol
ENVIRONMENTAL
REMEDIATION SERVICES, LLC

GROUNDWATER SAMPLING FORM

Well No.: _____

Well Type: ☐ Monitor ☐ Extraction ☐ Other _____

Job Name _____ Well Material ☐ PVC ☐ St. Steel ☐ Other _____

Job Number _____ Date _____ Time: _____

Recorded by _____ Sampled by _____
(Signature)

WELL PURGING

PURGE VOLUME

Casing Diameter (D in inches):

☐ 2-inch ☐ 4-inch ☐ 6-inch ☐ Other _____

Total Depth of Casing (TD in feet BTOC): _____

Water Level Depth (WL in feet BTOC): _____

Number of Well Volumes to be purged (# Vols)

☐ 3 ☐ 4 ☐ 5 ☐ 10 Other _____

PURGE METHOD

☐ Bailer – Type: _____

☐ Submersible ☐ Centrifugal ☐ Bladder Pump No. _____

☐ Other – Type: _____

PUMP INTAKE SETTING

☐ Near Bottom ☐ Near Top ☐ Other

Depth in feet (BTOC): _____ Screen Interval in Feet (BTOC)

from _____ to _____

PURGE VOLUME CALCULATION:

$$\underset{\text{TD (feet)}}{\_\_\_\_} - \underset{\text{WL (feet)}}{\_\_\_\_} \times \underset{\text{D (inches)}}{\_\_\_\_^2} \times \underset{\text{\# Vols}}{\_\_\_\_} \times 0.0408 = \underset{\text{Calculated Purge Volume}}{\_\_\_\_} \text{ gallons}$$

PURGE TIME

_____ Start _____ Stop _____ Elapsed

PURGE RATE

Initial _____ gpm Final _____ gpm

ACTUAL PURGE VOLUME

_____ gallons

FIELD PARAMETER MEASUREMENT

Minutes Since Pumping Began	pH	Cond. (µmhos/cm)	T ☐ °C ☐ °F	Other ____

Minutes Since Pumping Began	pH	Cond. (µmhos/cm)	T ☐ °C ☐ °F	Other ____
Meter Nos.				

Observations During Purging (Well Condition, Turbidity, Color, Odor): _____

Discharge Water Disposal: ☐ Sanitary Sewer ☐ Storm Sewer ☐ Other _____

GROUNDWATER SAMPLING FORM (continued)

WELL SAMPLING

SAMPLING METHOD

Casing Diameter (D in inches): ☐ Same As Above

☐ Bailer – Type: _____ ☐ Grab – Type _____

☐ Submersible ☐ Centrifugal ☐ Bladder Pump No. _____ ☐ Other – Type: _____

SAMPLING DISTRIBUTION

Sample Series: _____

Sample No.	Volume/Cont.	Analysis Requested	Preservatives	Lab	Comments

QUALITY CONTROL SAMPLES

Duplicate Samples

Original Sample No.	Duplicate Sample No.

Blank Samples

Type	Sample No.

Other Samples

Type	Sample No.

GROUNDWATER SAMPLING FORM
(To Accompany Low-Flow Purging Form)

Bristol
ENVIRONMENTAL
REMEDIATION SERVICES, LLC

Job Name _____

Job Number _____ Date _____ Time: _____

Recorded by _____ Sampled by _____
(Signature)

WELL INFORMATION

Well Number _____ **Well Location** _____

Casing Diameter (D in inches):

☐ 2-inch ☐ 4-inch ☐ 6-inch ☐ Other _____

Total Depth of Casing (TD in feet BTOC):

Water Level Depth (WL in feet BTOC):

WELL SAMPLING

SAMPLING METHOD

☐ Bailer – Type: _____ ☐ Grab – Type _____

☐ Submersible ☐ Centrifugal ☐ Bladder _____ ☐ Other – Type: _____

SAMPLING DISTRIBUTION

Sample No.	Volume	Analysis Requested	Preservatives	Lab	Comments

QUALITY CONTROL SAMPLES

Duplicate Samples

Original Sample No.	Duplicate Sample No.

Blank Samples

Type	Sample No.

Other Samples

Type	Sample No.

PHOTOGRAH LOG

Job#: _____ Project Manager: _____

Project Name: _____ Submitted by: _____

Photo Number	Date Photographed	Well ID	Direction of Photo	Photographer	Camera ID

Bristol
ENVIRONMENTAL
REMEDIATION SERVICES, LLC

111 W. 16th Avenue, Third Floor
Anchorage, Alaska 99501
907-563-0013 Phone
907-563-6713 Fax

SURFACE WATER/SEDIMENT FIELD FORM

Station No.: ______________
GPS coordinates ______________
____

Date: ______________
Start Time: ______________
End Time: ______________

Team:
Team Leader: ______________
Team Members: ______________
____
____

Sedt. Description: ______________

Flow:
Type of Flow Meter: ______________
Total Flow = ______________
Measurement Rating: Excellent, Good, Fair, Poor
Additional Information: ______________
Stream Width: ______________
Stream Depth: ______________

In-situ Field Parameters:
Meter Make and Model: ______________
Meter Number: ______________

Calibration Check:
Calibration Solution: ______________
Expiration Date: ______________
pH = ______________
Cond = ______________
Turb = ______________
Recalibrate: Y/N ______________

Stream Meter Readings:
pH = ______________
Cond = ______________
Turb = ______________
DO = ______________
Temp = ______________
Sal = ______________

Water Sample No.: ______________
Sedt. Sample No.: ______________
QC Sample: ______________

Number of Bottles ______________
Filtration: Y/N ______________
Analyses Requested: ______________

Additional Comments:

Weather:
Air Temperature ______________
Precipitation ______________
Wind ______________

A subsidiary of Bristol Bay Native Corporation

Bristol
ENVIRONMENTAL REMEDIATION SERVICES, LLC

111 W. 16th Avenue, Third Floor
Anchorage, AK 99501
phone (907) 563-0013
fax (907) 563-6713
www.bristol-companies.com

TOOLBOX SAFETY MEETING RECORD

DATE:_____

SUBJECTS:

1. _____
2. _____
3. _____
4. _____
5. _____
6. _____
7. _____
8. _____

	PRINTED NAME	SIGNATURE	COMPANY
1.			
2.			
3.			
4.			
5.			
6.			
7.			
8.			
9.			
10.			
11.			
12.			
13.			
14.			
15.			
16.			
17.			
18.			
19.			
20.			
21.			
22.			
23.			
24.			
25.			
26.			
27.			
28.			
29.			
30.			

Bristol

ENVIRONMENTAL
REMEDIATION SERVICES, LLC

WATER LEVEL FORM

page ___ of ____

Project: _____ Date: _____

Personnel: _____ Water Level Instrument: _____

Well Name	Measurement Time	Measuring Point	Depth to Water	Elevation of Measuring Point	Water Level Elevation	Comments

Bristol

ENVIRONMENTAL
REMEDIATION SERVICES, LLC

WATER LEVEL FORM

page ___ of ____

Well Name	Measurement Time	Measuring Point	Depth to Water	Elevation of Measuring Point	Water Level Elevation	Comments

Bristol

ENVIRONMENTAL
REMEDIATION SERVICES, LLC

WATER LEVEL FORM

page ___ of ____

Well Name	Measurement Time	Measuring Point	Depth to Water	Elevation of Measuring Point	Water Level Elevation	Comments

WELL CONSTRUCTION DETAILS

Bristol
ENVIRONMENTAL
REMEDIATION SERVICES, LLC

Site Name: _____ **Driller:** _____
Project Number: _____ **Geologist:** _____
Location: _____ **Client:** _____

Well ID: _____
Borehole ID: _____
Date Started: **Date Finished:**

STATIC WATER LEVEL (after completion)
_____ FT (TOC) Date/Time _____
_____ FT (TOC) Date/Time _____

WELL DIAGRAM - NOT TO SCALE

DEPTH
(FT. BGS)

ANNULAR SPACE DETAILS

Type of Annular Sealant (Grout): _____
Installation Method: _____
Type of Bentonite Seal: Granular / Pellet / Slurry (Choose One)
Number of Bags Used: _____
Installation Method: _____
Type of Sand Pack: _____
Installation Method: _____
Number of bags Used: _____

CONSTRUCTION DEPTHS (BGS)

Top of Casing: _____
Ground Surface: 0 feet
Top of Annular Sealant (Grout): _____
Top of Bentonite: _____
Top of Sand Pack: _____
Top of Screen: _____
Bottom of Well: _____
Bottom of Borehole: _____

CASING MEASUREMENTS

Diameter of Borehole (inches): _____
Diameter of Casing (inches): _____
Length of End Cap (inches): _____
Screen Length: _____
Total Length of Casing (feet): _____

WELL CONSTRUCTION MATERIALS

Monument Box: _____
Screen Slot Size: _____
Casing Type: _____

NOTES (added water, etc.)

Bristol
ENVIRONMENTAL
REMEDIATION SERVICES, LLC

WELL DEVELOPMENT FORM

Project: _____ Well No.: _____

Personnel: _____ Development Method _____ Date: _____

Time	Depth to Water (ft.)	Gallons Removed	Turbidity (Ntu)	pH	Temp ºC	E.C.	Recovery Rate Inches/min	Recovery Rate gpm	Observations

Total Gallons Removed _____

US ENVIRONMENTAL PROTECTION AGENCY REGION 8 - Chain Of Custody

Region 8 Laboratory
16194 W. 45th Drive
Golden, CO 80403
Fax: 303-312-7600

PROJECT NAME:

LSR #:

SAMPLER(S):
Name: Signature: Date:
Name: Signature: Date:

STATION ID.	STATION DESCRIPTION + NOTES	DATE	TIME	MATRIX	PRESERVATIVE	ANALYSIS							NUMBER OF CONTAINERS	Comments: BOTTLE ID.	LAB ID.

1-Relinquished By: Date/Time
2-Relinquished By: Date/Time
3-Relinquished By: Date/Time

1-Received By: Date/Time
2-Received By: Date/Time
3-Received By: Date/Time

PRESERVATIVES 1- HNO_3 2- H_3PO_4 3- H_2SO_4 4- HCl
5- $Na_2S_2O_3$ 6- Ice 7- Dry Ice 8- NaOH 9- Other_____

MATRIX
W- Water S- Soil Sed- Sediment Bio- Bio Tissue O- Other_____

Shaded Areas = Lab use only
Cooler Temperature =

Page ____ of ____

YSI **Environmental**

YSI 556 MPS

Multi Probe System

Operations Manual

Contents

1. Safety 1

1.1 General Information 1

2. General Information 6

2.1 Description 6
2.2 Unpacking the Instrument 7
2.3 Features of the YSI 556 Multi-Probe System 8
2.4 Batteries 9
2.5 Power On 14
2.6 Setting Display Contrast 14
2.7 Backlight 15
2.8 General Screen Features 15
2.9 Keypad Use 16
2.10 Instrument Reset 17
2.11 Menu Flowchart 18

3. Probe Module 18

3.1 Introduction 19
3.2 Unpacking the Probe Module 19
3.3 Features of the YSI 5563 Probe Module 20
3.4 Preparing the Probe Module 20
3.5 Transport/Calibration Cup 24
3.6 Instrument/Cable Connection 25

4. Sensors 26

5. Report 29

6. Calibrate 32

6.1 Getting Ready to Calibrate 32
6.2 Calibration Procedures 35
6.3 Return to Factory Settings. 52

7. Run 54

7.1 Real-Time Data 54

8. File 55

8.1 Accessing the File Screen 55
8.2 Directory 56
8.3 View File 58
8.4 Upload to PC 59
8.5 File Memory 63
8.6 Delete All Files 64

9. Logging 66
9.1 Accessing the Logging Setup Screen 66
9.2 Setting Logging Interval 67
9.3 Storing Barometer Readings 67
9.4 Creating a Site List 68
9.5 Editing a Site List 72
9.6 Logging Data Without a Site List 72
9.7 Logging Data with a Site List 75
9.8 Adding Data to Existing Files 77
10. System Setup 79
10.1 Accessing the System Setup Screen 79
10.2 Language Setting 80
10.3 Date and Time Setup 80
10.4 Data Filter 82
10.5 Shutoff Time 84
10.6 Comma Radix 85
10.7 ID 85
10.8 GLP Filename 85
10.9 TDS Constant 86
10.10 Barometer Units 87
10.11 Calibrate Barometer 88
11. Maintenance 90
11.1 Sensor Care and Maintenance 90
11.2 Upgrading YSI 556 MPS Software 96
12. Storage 99
12.1 General Recommendations for Short Term Storage 99
12.2 General Recommendations for Long Term Storage 99
13. Troubleshooting 102
14. Appendix A YSI 556 MPS Specifications 105
15. Appendix B Instrument Accessories 106
16. Appendix C Required Federal Communications Notice 107
17. Appendix D Health Safety 108
18. Appendix E Customer Service 111
18.1 Ordering and Technical Support 111
18.2 YSI Authorized Service Centers 111
18.3 Cleaning Instructions 111

18.4 Packing Procedure 112
18.5 Warranty 113

19. Appendix F Ferrite Bead Installation.....114

20. Appendix G EcoWatch.....116

20.1 Installing EcoWatch for Windows.....116
20.2 EcoWatch Tutorial.....116

21. Appendix H Calibration Record Information 127

21.1 Viewing the Calibration Record (.glp) File 127
21.2 Uploading the Calibration Record (.glp) File 127
21.3 Understanding the Calibration Record (.glp) File.....127

1. Safety

1.1 General Information

Read all safety information in this manual carefully before using the YSI 556 Multi-Probe System (MPS). Reagents that are used to calibrate and check this instrument may be hazardous to your health. Take a moment to review Appendix D Health and Safety.

⚠ **WARNING**

Warnings are used in this manual when misuse of the instrument could result in death or serious injury to a person.

⚠ **CAUTION**

Cautions are used in this manual when misuse of the instrument could result in mild or serious injury to a person and/or damage to equipment.

⚠ **IMPORTANT SAFETY INSTRUCTIONS!**

⚠ **SAVE THESE INSTRUCTIONS!**

⚠ In essence, the most important safety rule for use of the YSI 556 MPS is to utilize the instrument ONLY for purposes documented in this manual. This is particularly true of the YSI 6117 rechargeable battery pack that contains nickel metal hydride (NiMH) batteries. The user should be certain to read all of the safety precautions outlined below before using the instrument.

⚠ Batteries

⚠ This instrument is powered by alkaline or optional nickel-metal hydride batteries, which the user must remove and dispose of when the batteries no longer power the instrument. Disposal requirements vary by country and region, and users are expected to understand and follow the battery disposal requirements for their specific locale.

The circuit board in this instrument contains a manganese dioxide lithium "coin cell" battery that must be in place for continuity of power to memory devices on the board. This battery is not user serviceable or replaceable.

When appropriate, an authorized YSI service center will remove this battery and properly dispose of it, per service and repair policies.

⚠YSI Rechargeable Battery Pack Safety Information

⚠Restrictions on Usage

1. Never dispose of the battery pack in a fire.
2. Do not attempt to disassemble the YSI 6117 battery pack
3. Do not tamper with any of the electronic components or the batteries within the battery pack. Tampering with either the electronic circuitry or the batteries will result in the voiding of the warranty and the compromising of the system performance, but, more importantly, can cause safety hazards which result from overcharging such as overheating, venting of gas, and loss of corrosive electrolyte.
4. Do not charge the battery pack outside the 0–40°C temperature range.
5. Do not use or store the battery at high temperature, such as in strong direct sunlight, in cars during hot weather, or directly in front of heaters.
6. Do not expose the battery pack to water or allow the terminals to become damp.
7. Avoid striking or dropping the battery pack. If the pack appears to have sustained damage from these actions or malfunctions after an impact or drop, the user should not attempt to repair the unit. Instead, contact YSI Customer Service. Refer to *Appendix E Customer Service.*
8. If the battery pack is removed from the YSI 556 MPS, do not store it in pockets or packaging where metallic objects such as keys can short between the positive and negative terminals.

⚠Precautions for Users with Small Children.

Keep the battery pack out of reach of babies and small children.

⚠Danger Notifications – Misuse creates a STRONG possibility of death or serious injury.

FAILURE TO CAREFULLY OBSERVE THE FOLLOWING PROCEDURES AND PRECAUTIONS CAN RESULT IN LEAKAGE OF BATTERY FLUID, HEAT GENERATION, BURSTING, AND SERIOUS PERSONAL INJURY.

1. Never dispose of the battery pack in a fire or in heat.
2. Never allow the positive and negative terminals of the battery pack to become shorted or connected with electrically conductive materials. When the battery pack has been removed from the YSI 556 MPS, store it in a heavy plastic bag to prevent accidental shorting of the terminals.
3. Never disassemble the battery pack and do not tamper with any of the electronic components or the batteries within the battery pack. The battery pack is equipped with a variety of safety features. Accidental deactivation of any of these safety features can cause a serious hazard to the user.
4. The NiMH batteries in the battery pack contain a strong alkaline solution (electrolyte). The alkaline solution is extremely corrosive and will cause damage to skin or other tissues. If any fluid from the battery pack comes in contact with a user's eyes, immediately flush with clean water and consult a physician immediately. The alkaline solution can damage eyes and lead to permanent loss of eyesight.

⚠ Warning Notifications – Misuse creates a possibility of death or serious injury

1. Do not allow the battery pack to contact freshwater, seawater, or other oxidizing reagents that might cause rust and result in heat generation. If a battery becomes rusted, the gas release vent may no longer operate and this failure can result in bursting.
2. If electrolyte from the battery pack contacts the skin or clothing, thoroughly wash the area immediately with clean water. The battery fluid can irritate the skin.

⚠ Caution Notifications – Misuse creates a possibility of mild or serious injury or damage to the equipment.

1. Do not strike or drop the battery pack. If any impact damage to the battery pack is suspected, contact YSI Customer Service. Refer to *Appendix E Customer Service*.

2. Store the battery pack out of reach of babies and small children.
3. Store the battery pack between the temperatures of -20 and 30°C.
4. Before using the battery pack, be sure to read the operation manual and all precautions carefully. Then store this information carefully to use as a reference when the need arises.

⚠YSI 616 Cigarette Lighter Charger Safety Information

1. This section contains important safety and operating instructions for the YSI 556 MPS cigarette lighter battery charger (YSI 616; RadioShack Number 270-1533E). BE SURE TO SAVE THESE INSTRUCTIONS.
2. Before using the YSI 616 cigarette lighter charger, read all instructions and cautionary markings on battery charger, battery pack, and YSI 556 MPS.
3. Charge the YSI 6117 battery pack with the YSI 616 cigarette lighter charger ONLY when the YSI 6117 is installed in the YSI 556 MPS.
4. Do not expose charger to rain, moisture, or snow.
5. Use of an attachment not recommended or sold by the battery charger manufacturer may result in a risk of fire, electric shock, or injury to persons.
6. To reduce risk of damage to cigarette lighter and cord, pull by cigarette lighter rather than cord when disconnecting charger.
7. Make sure that the cord is located so that it will not be stepped on, tripped over, or otherwise subjected to damage or stress.
8. Do not operate charger with damaged cord or cigarette lighter connector – replace it immediately.
9. Do not operate charger if it has received a sharp blow, been dropped, or otherwise damaged in any way; contact YSI Customer Service. Refer to *Appendix E Customer Service*.
10. Do not disassemble charger other than to change the fuse as instructed. Replace the part or send it to YSI Product Service if repair is required (refer to *Appendix E Customer Service*). Incorrect reassembly may result in a risk of electric shock or fire.

11. To reduce risk of electric shock, unplug charger before attempting any maintenance or cleaning. Turning off controls will not reduce this risk.

⚠ YSI 556 MPS Water Leakage Safety Information

The YSI 556 MPS has been tested and shown to comply with IP67 criterion, i.e. submersion in 1 meter of water for 30 minutes with no leakage into either the battery compartment or the main case. However, if the instrument is submersed for periods of time in excess of 30 minutes, leakage may occur with subsequent damage to the batteries, the rechargeable battery pack circuitry, and/or the electronics in the main case.

If leakage into the battery compartment is observed when using alkaline C cells, remove batteries, dispose of batteries properly, and dry the battery compartment completely, ideally using compressed air. If corrosion is present on the battery terminals, contact YSI Customer Service for instructions. Refer to *Appendix E Customer Service*.

If leakage into the battery compartment is observed when using the YSI rechargeable battery pack, remove the battery assembly and set aside to dry. Return the battery pack to YSI Product Service for evaluation of possible damage. Finally dry the battery compartment completely, ideally using compressed air. If corrosion is present on the battery terminals, contact YSI Customer Service for instructions. Refer to *Appendix E Customer Service*.

⚠ **CAUTION:** If water has contacted the rechargeable battery pack, do not attempt to reuse it until it has been evaluated by YSI Product Service (refer to Appendix E Customer Service). Failure to follow this precaution can result in serious injury to the user.

If it is suspected that leakage into the main cavity of the case has occurred, remove the batteries immediately and return the instrument to YSI Product Service for damage assessment. Refer to *Appendix E Customer Service*.

⚠ **CAUTION:** Under no circumstances should the user attempt to open the main case.

2. General Information

2.1 Description

The rugged and reliable YSI 556 MPS (Multi-Probe System) combines the versatility of an easy-to-use, easy-to-read handheld unit with all the functionality of a multi-parameter system. Featuring a waterproof, impact-resistant case, the YSI 556 MPS simultaneously measures dissolved oxygen, conductivity, temperature, and optional pH and ORP. A simple cellular phone style keypad and large display make the instrument easy to use. The YSI 556 MPS is compatible with YSI EcoWatch™ for Windows™ software.

The YSI 556 MPS assists the user in conforming to Good Laboratory Practice (GLP) standards which help ensure that quality control/quality assurance methods are followed. Battery life is displayed with a fuel gauge, and the user can choose standard alkaline batteries or an optional rechargeable battery pack.

The 1.5 MB memory can store more than 49,000 data sets. Other options include a flow cell and barometer. The internal barometer can be user-calibrated and displayed along with other data, used in dissolved oxygen calibrations, and logged to memory for tracking changes in barometric pressure.

Features

- Waterproof -meets IP67 specifications
- Field-replaceable DO electrode module; pH and pH/ORP sensors
- Compatible with EcoWatch™ for Windows™ data analysis software
- Assists with Good Laboratory Practice Standards (GLP)
- Choice of DO membrane material for different applications
- Easy-to-use, screw-on cap DO membranes
- User-upgradeable software from YSI website
- Three-year warranty on the instrument; one-year on the probe modules
- Available with 4,10, and 20 m cable lengths
- Stores over 49,000 data sets, time and date stamped

- Auto temperature compensating display contrast
- Optional barometer
- Optional rechargeable battery pack or standard alkaline batteries

2.2 Unpacking the Instrument

1. Remove the instrument from the shipping box. Note that the probe module and sensors are shipped in a separate box and will be unpacked later in Section *3.2 Unpacking the Probe Module*

   NOTE: Do not discard any parts of supplies.

2. Use the packing list to ensure all items are present.

3. Visually inspect all components for damage.

   NOTE: If any parts are missing or damaged, contact your YSI Service Center immediately. Refer to Appendix E Customer Service or www.ysi.com.

2.3 Features of the YSI 556 Multi-Probe System

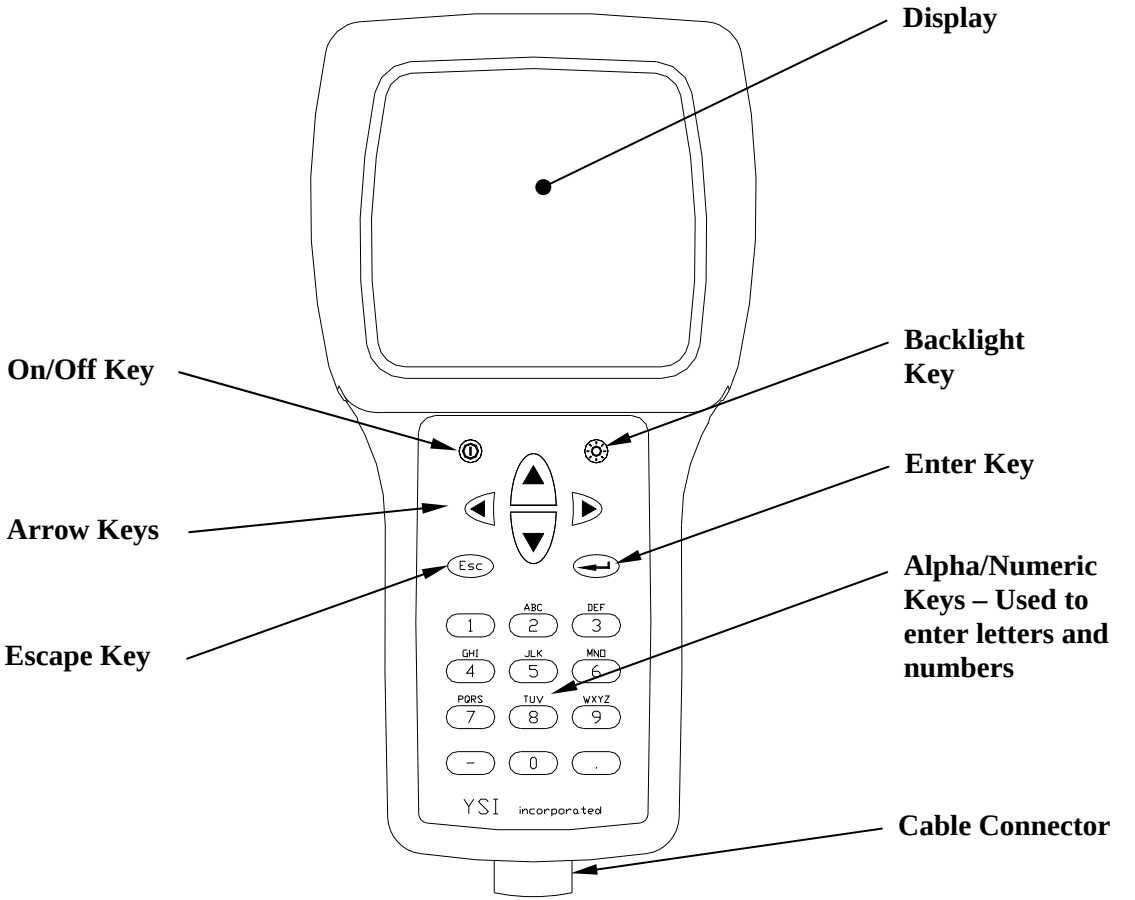


Figure 2.1 Front View of YSI 556 MPS

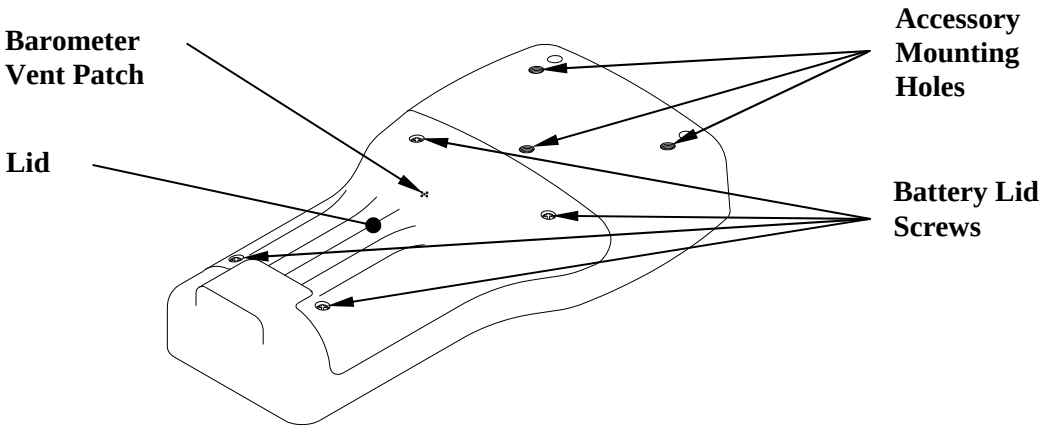


Figure 2.2 Back View of YSI 556 MPS

2.4 Batteries

2.4.1 Battery Life

Standard Alkaline Batteries

With the standard battery configuration of 4 alkaline C cells, the YSI 556 MPS will operate continuously for approximately 180 hours. Assuming a standard usage pattern when sampling of 3 hours of “on time” in a typical day, the alkaline cells will last approximately 60 days.

Optional Rechargeable Battery Pack

When fully charged, the optional rechargeable battery pack will provide approximately 50 hours of battery life.

2.4.2 Inserting 4 C Batteries

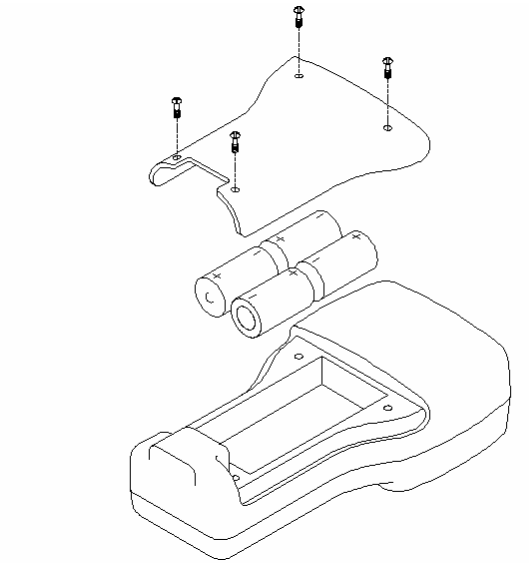


Figure 2.1 Inserting C Cells

⚠ **CAUTION: Install batteries properly to avoid damage to the instrument**.

1. Loosen the four screws in the battery lid on the back of the instrument using any screwdriver.
2. Remove the battery lid.
3. Insert four C batteries between the clips following the polarity (+ and -) labels on the bottom of the battery compartment.
4. Check gasket for proper placement on the battery lid.
5. Replace the battery lid and tighten the 4 screws securely and evenly.

NOTE: Do not over-tighten the screws.

2.4.3 Inserting Optional Rechargeable Battery Pack

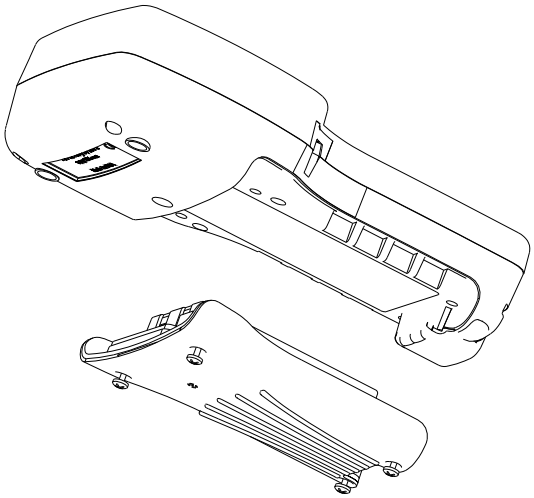


Figure 2.2 Inserting Battery Pack

⚠ **CAUTION: Read all cautions and warning that come with the battery pack before using the battery pack.**

1. Loosen the four screws in the battery lid on the back of the instrument using any screwdriver.

2. Remove the C battery lid and store for future use. Remove C batteries, if installed.

3. Install the rechargeable battery pack and lid and tighten the 4 screws securely and evenly.

NOTE: Do not over tighten the screws.

2.4.4 Charging the Optional Rechargeable Battery Pack

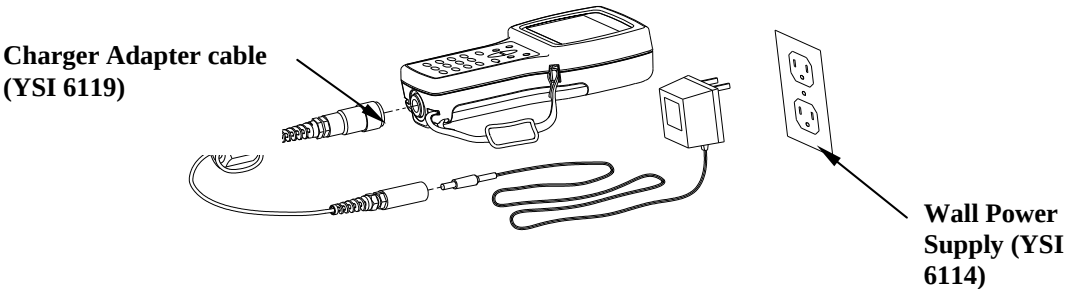


Figure 2.3 Charging the Battery Pack

⚠ **CAUTION: Do not use or store the battery pack at extreme temperatures such as in strong direct sunlight, in cars during hot weather or close to heaters.**

1. Install the rechargeable battery pack into the instrument as described in Section *2.4.3 Inserting Optional Rechargeable Battery Pack*.

2. Attach the charger adapter cable (YSI 6119) to the instrument.

NOTE: Wall power supplies for use in countries outside the US and Canada can be found in *Appendix B Instrument Accessories.*

3. Insert the barrel connector of the wall power supply into the barrel of the adapter cable.

⚠ **CAUTION: Do not charge the battery pack continuously for more than 48 hours.**

⚠ **CAUTION: Do not drop or expose to water.**

⚠ **CAUTION: Do no charge the battery pack at temperatures below 0°C or above 40°C.**

4. Plug the wall power supply into an AC power outlet for approximately 2 hours to obtain an 80% to 90% charge for 6 hours to get a full charge.

NOTE: The battery pack can be recharged whether the instrument is on or off.

2.4.5 Storing the Battery Pack

Remove the battery pack from the instrument when the instrument will not be used for extended periods of time to prevent over discharge of the battery pack.

Store the battery pack in a heavy plastic bag to prevent accidental shorting of the terminals. Store between –20 and 30°C.

2.4.6 Optional Cigarette Lighter Charger

⚠ **CAUTION: Read all warnings and cautions that come with the charger before using the charger.**

⚠ **CAUTION: Only use cigarette lighter charger when rechargeable battery pack is inserted into instrument.**

⚠ **CAUTION: Do not mishandle cigarette lighter charger. Do not expose to moisture.**

1. Plug the barrel connector of the cigarette lighter charger into the mating end of the YSI 6119 Charger Adapter Cable.

2. Attach the MS-19 end of the YSI 6119 Charger Adapter Cable to the instrument.

3. Make one of the following modifications to the other end of the charger:

Slide the adapter ring off the plug to use the device with an American or Japanese vehicle.

.

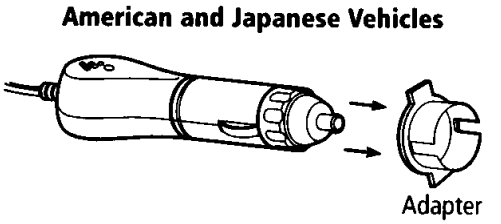


Figure 2.1 Charger Plug Adapter Use

Leave the adapter ring on the plug and position it so that the slots on the adapter ring line up with the plug's spring clips to use the device on a European vehicle.

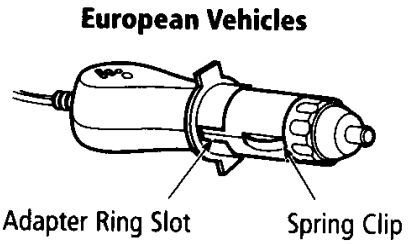


Figure 2.2 European Charger Plug Adapter Use

NOTE: If the charger stops working properly, refer to Section 13 *Troubleshooting.*

2.5 Power On

Press and release the on/off button in the upper left corner of the instrument keypad to turn the instrument on or off. See Figure2.1 Front View of YSI 556 MPS.

2.6 Setting Display Contrast

The display contrast automatically compensates for temperature changes. However, under extreme temperature conditions you may wish to optimize the display by manual adjustment as follows:

1. Press and *hold down* the backlight key in the upper right corner of the keypad and press the “up” arrow to increase (darken) the contrast.

2. Press and *hold down* the backlight key in the upper right corner of the keypad and press the “down” arrow to decrease (lighten) the contrast.

2.7 Backlight

Press and *release* the backlight key in the upper right corner of the keypad to turn the backlight on or off. See Figure 2.1 Front View of YSI 556 MPS.

NOTE: The backlight turns off automatically after two minutes of non-use.

2.8 General Screen Features

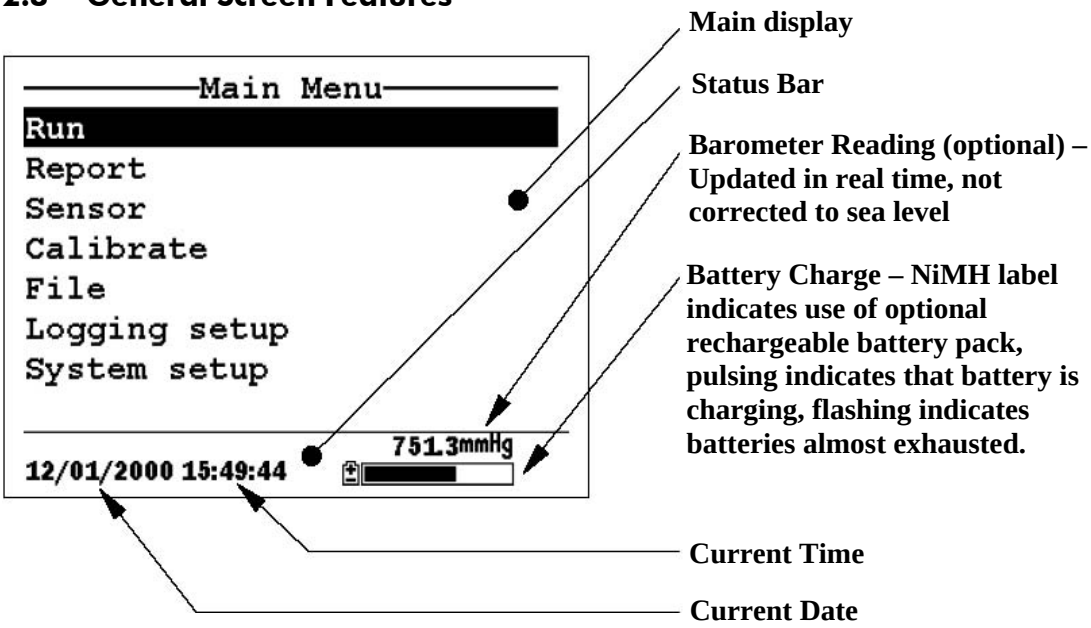


Figure 2.4 Main Screen Menu

2.9 Keypad Use

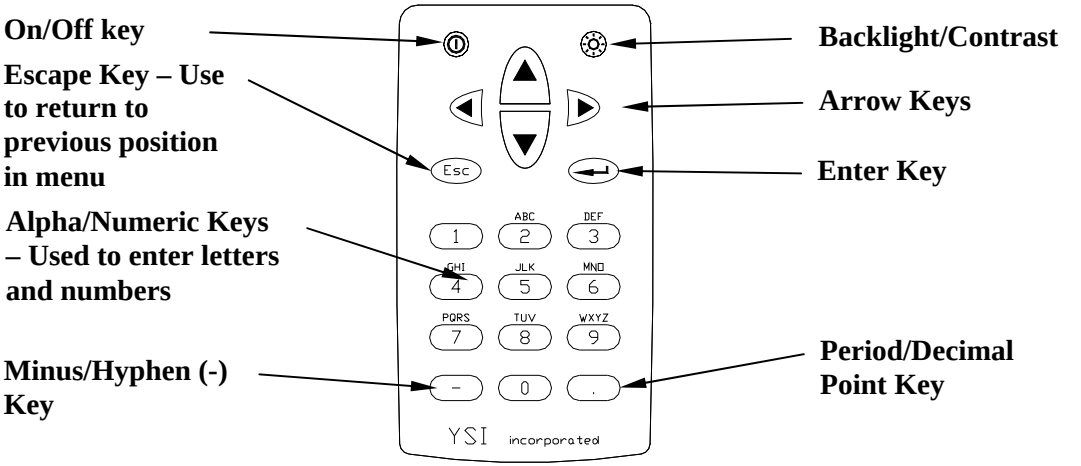


Figure 2.5 Keypad Features

KEY	LETTER/NUMBER
1	1
2	ABC2abc3
3	DEF3def3
4	GHI4ghi4
5	JKL5jkl5
6	MNO6mno6
7	PQRS7pqrs7
8	TUV8tuv8
9	WXYZ9wxyz9
0	0

Figure 2.6 Keypad Features

1. See Figure 2.10 Keypad Letters & Numbers and press the appropriate key repeatedly until letter or number desired appears in display.

NOTE: Press the key repeatedly in rapid succession to get to the desired letter or number. If you pause for more than a second, the cursor automatically scrolls to the right to prepare for the next input.

EXAMPLE 1: Press the **6** key *once* and *release* to display an uppercase “M”.

EXAMPLE 2: Press the **6** key *four times* and *release* to display the number “6”.

EXAMPLE 3: Press the **6** key *five times* and *stop* to display a lowercase “m”.

2. Press the left arrow key to go back and reenter a number or setter that needs to be changed.

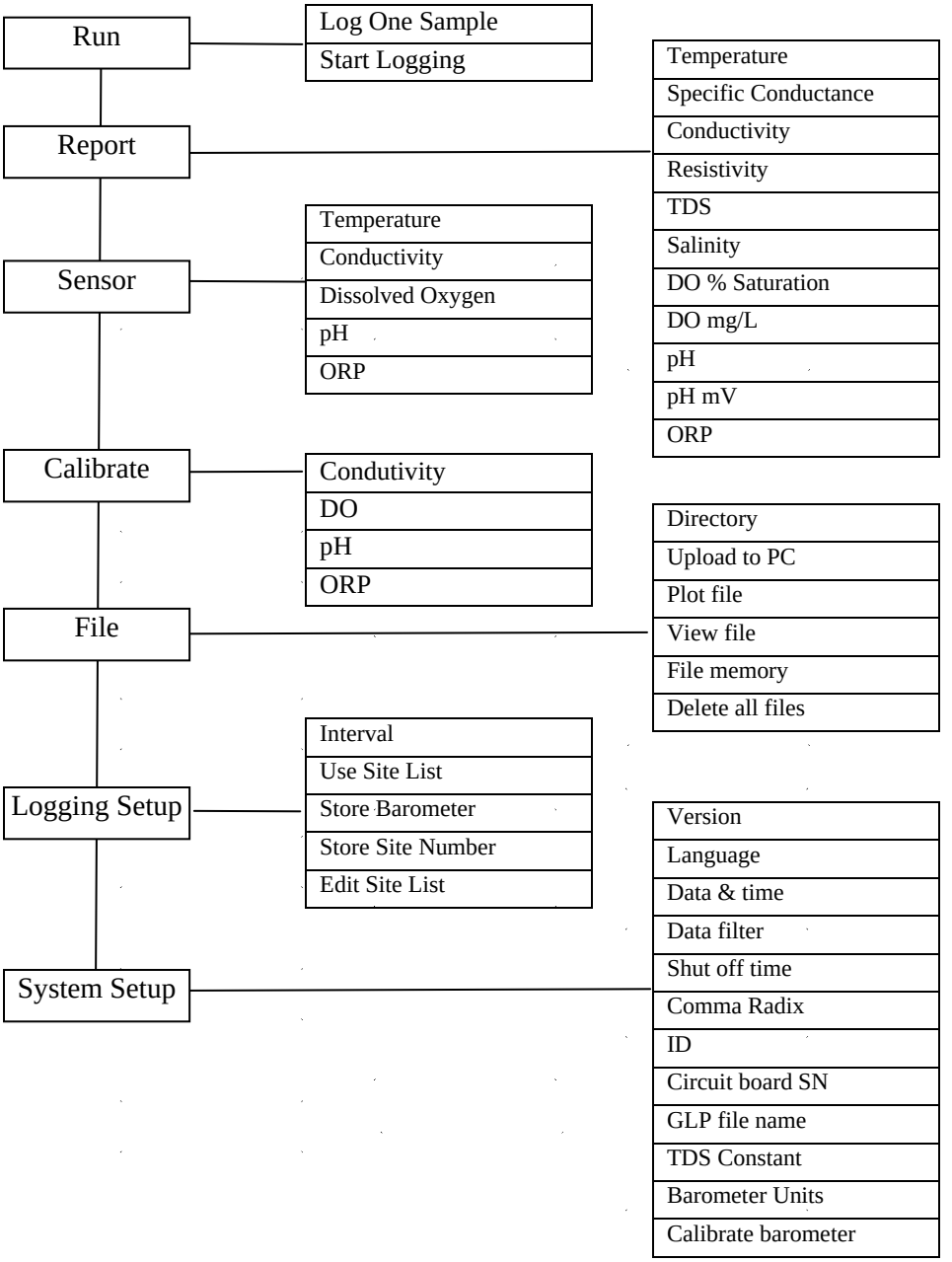
   Press the **Enter** key when your entry is complete.

   NOTE: The instrument software permits only numeric entries in many instances, such as when setting the clock or entering calibration parameters.

2.10 Instrument Reset

The YSI 556 MPS is characterized by sophisticated software that should provide trouble-free operation. However, as with all high-capability software packages, it is always possible that the user will encounter circumstances in which the instrument does not respond to keypad entry. If this occurs, the instrument function can easily be restored by removing and then reapplying battery power. Simply remove either your C-cells or rechargeable battery pack from the battery compartment, wait 30 seconds and then replace the batteries. See Section *2.4 Batteries* for battery removal/reinstallation instructions.

2.11 Menu Flowchart



3. Probe Module

3.1 Introduction

The YSI 5563 Probe module is used for measuring dissolved oxygen, temperature, conductivity, and optional pH and ORP. The probe module is rugged, with the sensors enclosed in a heavy duty probe sensor guard with attached sinking weight. A 4, 10 or 20 meter cable is directly connected to the probe module body making it waterproof. An MS-19 connector at the end of the cable makes the YSI 5563 fully compatible with the YSI 556 Multi-Probe System.

3.2 Unpacking the Probe Module

1. Remove the YSI 5563 Probe Module from the shipping boxes.

   NOTE: Do not discard any parts or supplies.

2. Use the packing list to ensure all items are present.

3. Visually inspect all components for damage.

NOTE: If any parts are missing or damaged, contact a YSI representative immediately. Refer to: *Appendix E Customer Service* o visit www.ysi.com.

3.3 Features of the YSI 5563 Probe Module

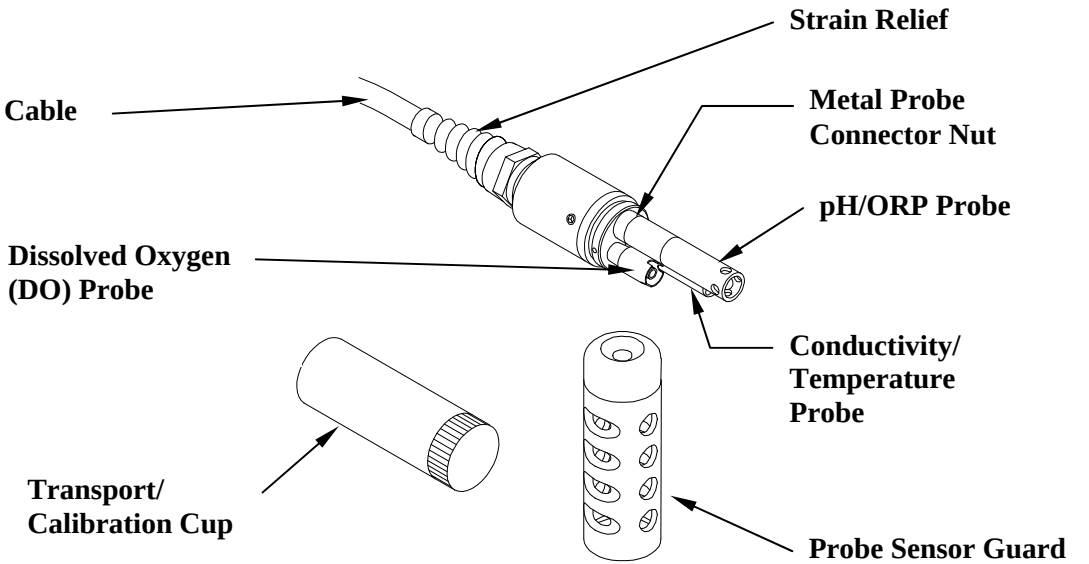


Figure 3.1 Probe Module

3.4 Preparing the Probe Module

To prepare the probe module for calibration and operation, you need to install the sensors into the connectors on the probe module bulkhead. In addition to sensor installation, you need to install a new DO membrane cap.

3.4.1 Sensor Installation

Whenever you install, remove or replace a sensor, it is extremely important that the entire probe module and all sensors be thoroughly dried prior to the removal of a sensor or a sensor port plug. This will prevent water from entering the port. Once you remove a sensor or plug, examine the connector inside the probe module sensor port. If any moisture is present, use compressed air to completely dry the connector. If the connector is corroded, return the probe module to your YSI Distributor or directly to YSI Customer Service. Refer to *Appendix E Customer Service*.

Conductivity/Temperature and pH, pH/ORP Sensor Installation

1. Unscrew and remove the probe sensor guard.

2. Using the sensor installation tool supplied in the YSI 5511 maintenance kit, unscrew and remove the sensor port plugs.

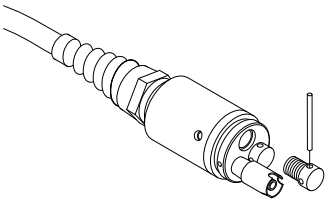


Figure 3.2 Port Plug Removal

3. Locate the port with the connector that corresponds to the sensor that is to be installed.

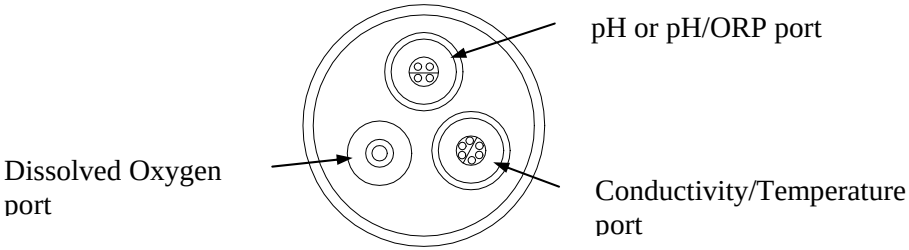


Figure 3.3 Sensor Port Identification

4. Apply a thin coat of o-ring lubricant (supplied in the YSI 5511 maintenance kit) to the o-rings on the connector side of the sensor (see Figure 3.4 O-ring Lubrication).

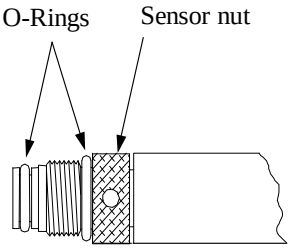


Figure 3.4 O-ring Lubrication

⚠ **CAUTION:** Make sure that there are NO contaminants between the o-ring and the sensor. Contaminants that are present under the o-ring may cause the o-ring to leak.

5. Be sure the probe module sensor port is free of moisture and then insert the sensor into the correct port. Gently rotate the sensor until the two connectors align.

6. With the connectors aligned, screw down the sensor nut using the sensor installation tool.

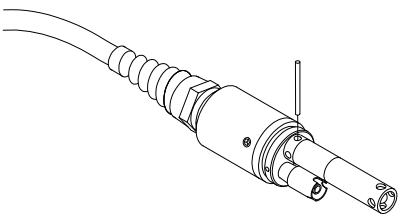


Figure 3.5 Sensor Installation

⚠ **CAUTION:** Do not cross thread the sensor nut. Tighten the nut until it is flush with the face of the probe module bulkhead. Do not over tighten.

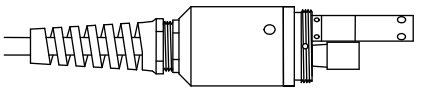


Figure 3.6 Bulkhead Seating

7. Repeat steps 3-6 for any other sensors.

8. Replace the probe sensor guard.

Dissolved Oxygen Sensor Installation

The YSI 5563 comes with the DO sensor already installed. Refer to Section *11.1.2 DO Sensor Replacement* for instructions on installing the YSI 559 Replaceable DO Module Kit.

3.4.2 Membrane Cap Selection

The YSI 5563 is shipped with a YSI 5909 kit that contains membrane caps made with 2 mil polyethylene (PE), a material which should be ideal for most field applications of the 556. However, YSI also offers membrane caps made with two other materials (1 mil polyethylene and 1 mil Teflon) which some users may also prefer. All membranes available for the 556/5563 system provide comparable accuracy if used properly. The difference between the two thicknesses of PE is found in the trade-off of flow dependence and response time as described below. Teflon is offered because some users may prefer to continue using the traditional membrane material used by YSI. To avoid confusion, the membrane caps are color coded as described below and can be ordered in kits as noted:

1 mil Teflon – Black Caps (Kit = YSI 5906)
1 mil Polyethylene (PE) – Yellow Caps (Kit = YSI 5908)
2 mil Polyethylene (PE) – Blue Caps (Kit = YSI 5909)

The 1 mil Teflon caps will offer traditional, reliable performance for most dissolved oxygen applications. The 1 mil PE caps will provide a significantly faster dissolved oxygen response (as long as your 556 Data Filter is set correctly as described below in Sections 10.2 and 10.3.1) while also giving readings which are significantly less flow dependent than the 1 mil Teflon caps. Finally, 2 mil PE caps will show a large reduction in flow dependence over 1 mil Teflon while not significantly increasing the response time. Generally, one of the PE caps is likely to provide better performance for your application.

IMPORTANT: No matter which type of membrane cap you select, you will have to confirm your selection in the 556 software from the Sensor menu as described in Section *4 Sensors*.

3.4.3 Membrane Cap Installation

NOTE: The YSI 5563 DO sensor (already installed in the probe module) was shipped dry. A shipping membrane was installed to protect the electrode. **A new membrane cap must be installed before the first use.**

1. Unscrew and remove the probe sensor guard.
2. Unscrew, remove, and discard the old membrane cap.
3. Thoroughly rinse the sensor tip with distilled water.
4. Prepare the electrolyte according to the directions on the electrolyte solution bottle.
5. Hold the new membrane cap and fill it at least ½ full with the electrolyte solution.
6. Screw the membrane cap onto the sensor moderately tight. A small amount of electrolyte should overflow.

⚠ **Caution:** Do not touch the membrane surface.

7. Screw the probe sensor guard on moderately tight.

3.5 Transport/Calibration Cup

The YSI 5563 Probe module has been supplied with a convenient transport/calibration cup. This cup is an ideal container for calibration of the different sensors, minimizing the amount of solution needed. Refer to Section *6 Calibrate*.

3.5.1 Transport/Calibration Cup Installation

1. Remove probe sensor guard, if already installed.

2. Ensure that an o-ring is installed in the o-ring groove on the threaded end of the probe module body.

3. Screw the transport/calibration cup on the threaded end of the probe module and securely tighten.

   NOTE: Do not over tighten as this could cause damage to the threaded portions.

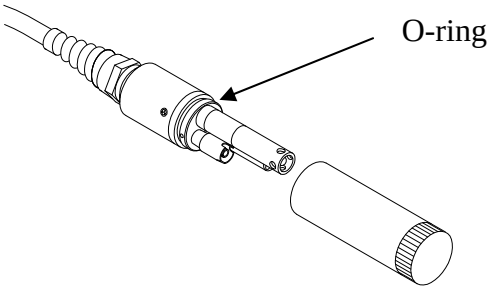


Figure 3.7 Transport/Calibration Cup Installation

3.6 Instrument/Cable Connection

Attach the cable to the instrument as follows:

1. Line up the pins and guides on the cable with the holes and indentations on the cable connector at the bottom of the YSI 556 instrument. See Figure 2.1 Front View of YSI 556 MPS.

2. Holding the cable firmly against the cable connector, turn the locking mechanism clockwise until it snaps into place.

Remove the cable from the instrument by turning the cable connector counterclockwise until the cable disengages from the instrument.

4. Sensors

The Sensors screen allows the user to enable or disable each of the sensors and select which membrane material will be used for the dissolved oxygen sensor. Disabled sensors will not be displayed on the screen in real time or logged to files.

1. Press the **On/off** key to display the run screen.

2. Press the **Escape** key to display the main menu screen.

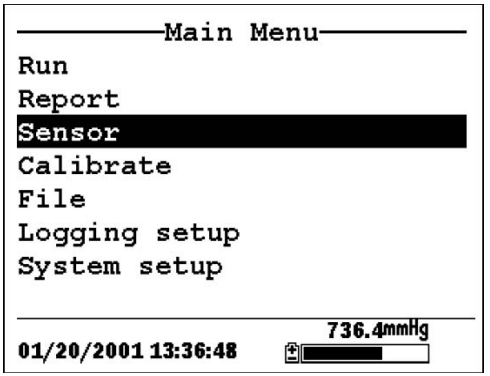


Figure 4.1 Main Menu Screen

3. Use the arrow keys to highlight the **Sensor** selection.

4. Press the **Enter** key to display the sensors enabled screen.

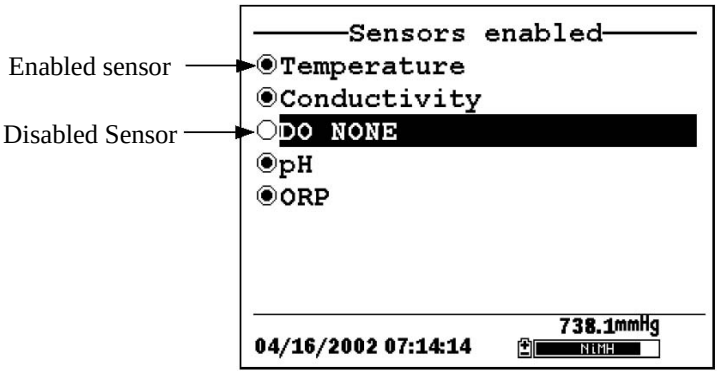


Figure 4.2 Sensors Enabled Screen Before DO Membrane Selection

A black dot to the left of a sensor indicates that sensor is enabled. Sensors with an empty circle are disabled.

Highlight the “DO None” entry as shown above and press **Enter** to display the membrane choice screen. Consult Section *3.4.2 Membrane Cap Selection* for information on the advantages of each type of membrane material. Blue membrane caps using 2 mil polyethylene (PE) were shipped with your YSI 5563 and are likely to be the best choice for most 556 field applications.

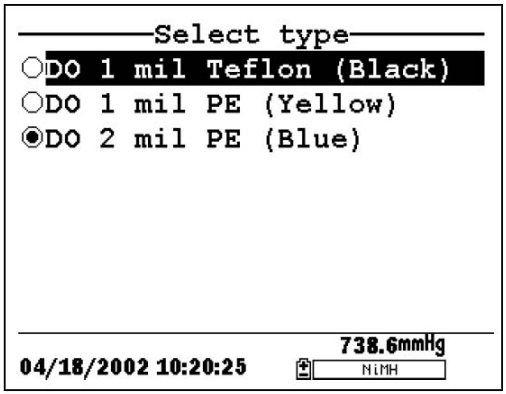


Figure 4.3 Membrane Selection Screen

Highlight the desired membrane choice – in this case, 2 mil PE - and press Enter to activate your selection with a dot to the left of the screen. Then press **Escape** to return to the Sensor menu that now shows your DO membrane selection.

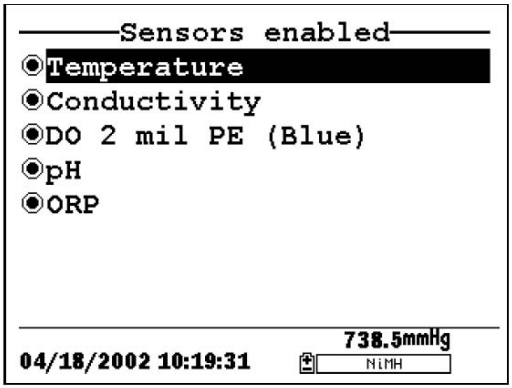


Figure 4.4 Sensors Enabled Screen After DO Membrane Selection

NOTE: The Temperature sensor cannot be disabled. Most other sensors require temperature compensation for accurate readings. In addition, the conductivity sensor must be activated in order to obtain accurate dissolved oxygen mg/L readings.

5. Use the arrow keys to highlight the sensor you want to change, then press the Enter key to enable or disable it.

6. Repeat step 5 for each sensor you want to change.

7. Press the Escape key to return to the main menu screen.

5. Report

The Report Setup screen allows the user to select which sample parameters and units the YSI 556 MPS will display on the screen. It does NOT determine which parameters are logged to memory. Refer to Section *4 Sensors.*

1. Press the **On/off** key to display the run screen.

2. Press the **Escape** key to display the main menu screen.

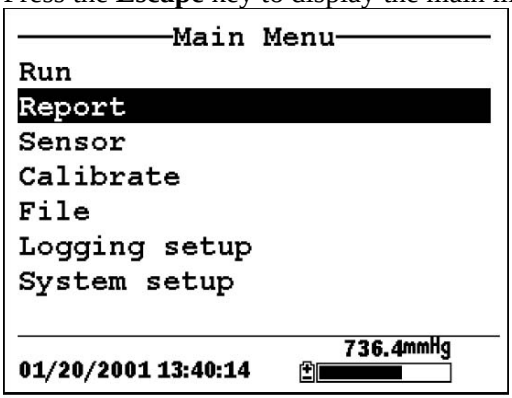


Figure 5.1 Main Menu

3. Use the arrow keys to highlight the **Report** selection.

4. Press the **Enter** key to display the report setup screen.

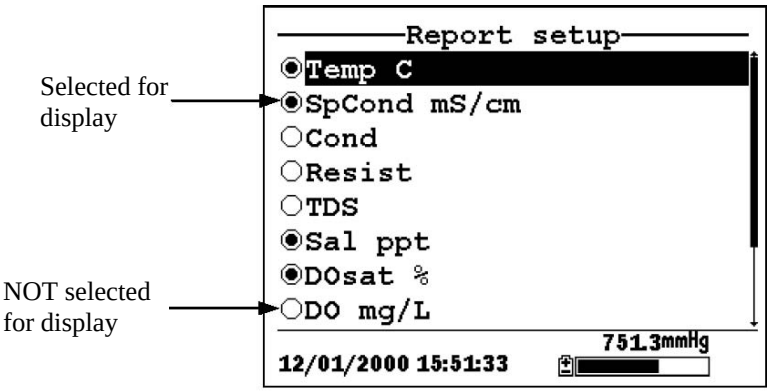


Figure 5.2 Report Setup Screen

NOTE: A black dot to the left of a parameter indicates that parameter is selected for display. Parameters with an empty circle will not be displayed.

NOTE: You may have to scroll down past the bottom of the screen to see all the parameters.

5. Use the arrow keys to highlight the parameter you want to change, then press the **Enter** key. If you can't find the parameter you want, even after scrolling down past the bottom of the screen, the sensor used for that parameter is disabled. Refer to Section *4 Sensors*.

6. If you selected Temperature, Specific Conductivity, Conductivity, Resistance or Total Dissolved Solids, the Units screen will appear.

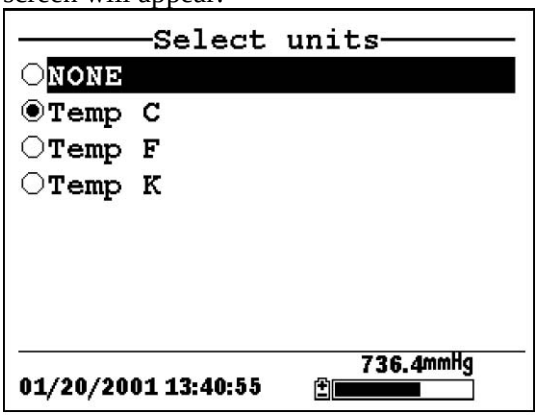


Figure 5.3 Units Screen

7. Use the arrow keys to select the units desired, then press the **Enter** key to return to the report setup screen.

8. Repeat steps 5 and 6 for each parameter you want to change.

NOTE: Specific Conductance (temperature compensated conductivity) is notated on the Run screen with a small ‘c’ after the units of measure.

All parameters may be enabled at the same time.

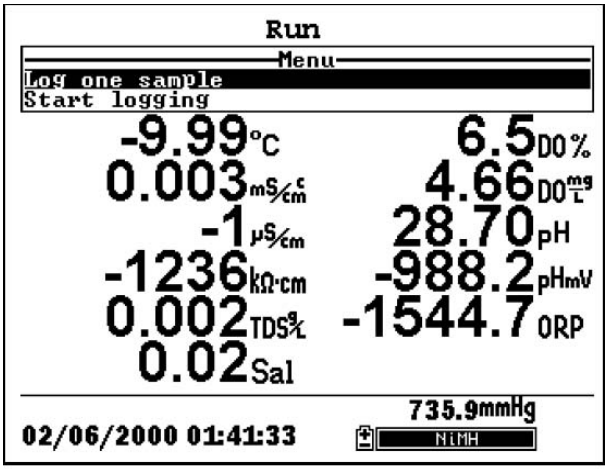


Figure 5.4 All Parameters Displayed

9. Press the **Escape** key to return to the Main menu screen.

6. Calibrate

All of the sensors, except temperature, require periodic calibration to assure high performance. You will find specific calibration procedures for all sensors that require calibration in the following sections. If a sensor listed is not installed in your probe module, skip that section and proceed to the next sensor until the calibration is complete.

⚠ **CAUTION:** Reagents that are used to calibrate and check this instrument may be hazardous to your health. Take a moment to review *Appendix D Health and Safety*. Some calibration standard solutions may require special handling.

6.1 Getting Ready to Calibrate

6.1.1 Containers Needed to Calibrate the Probe Module

The transport/calibration cup that comes with your probe module serves as a calibration chamber for all calibrations and minimizes the volume of calibration reagents required.

Instead of the transport/calibration cup, you may use laboratory glassware to perform calibrations. If you do not use the transport/calibration cup that is designed for the probe module, you are cautioned to do the following:

- ✓ Perform all calibrations with the Probe Sensor Guard installed. This protects the sensors from possible physical damage.
- ✓ Use a ring stand and clamp to secure the probe module body to prevent the module from falling over. Most laboratory glassware has convex bottoms.
- ✓ Ensure that all sensors are immersed in calibration solutions. Many of the calibrations factor in readings from other sensors (e.g., temperature sensor). The top vent hole of the conductivity sensor must also be immersed during some calibrations.

6.1.2 Calibration Tips

1. If you use the Transport/Calibration Cup for dissolved oxygen (DO) calibration, make certain to loosen the seal to allow pressure equilibration before calibration. The DO calibration is a water-saturated air calibration.

2. When calibrating pH, always calibrate with buffer 7 first, regardless if performing a 1, 2, or 3 point calibration

3. The key to successful calibration is to ensure that the sensors are completely submersed when calibration values are entered. Use recommended volumes when performing calibrations.

4. For maximum accuracy, use a small amount of previously used calibration solution to pre-rinse the probe module. You may wish to save old calibration standards for this purpose.

5. Fill a bucket with ambient temperature water to rinse the probe module between calibration solutions.

6. Have several clean, absorbent paper towels or cotton cloths available to dry the probe module between rinses and calibration solutions. Shake the excess rinse water off of the probe module, especially when the probe sensor guard is installed. Dry off the outside of the probe module and probe sensor guard. Making sure that the probe module is dry reduces carry-over contamination of calibrator solutions and increases the accuracy of the calibration.

7. If you are using laboratory glassware for calibration, you do not need to remove the probe sensor guard to rinse and dry the sensors between calibration solutions. The inaccuracy resulting from simply rinsing the sensor compartment and drying the outside of the guard is minimal.

8. If you are using laboratory glassware, remove the stainless steel weight from the bottom of the probe sensor guard by turning the weight counterclockwise. When the weight is removed, the calibration solutions have access to the sensors

without displacing a lot of fluid. This also reduces the amount of liquid that is carried between calibrations.

9. Make certain that port plugs are installed in all ports where sensors are not installed. It is extremely important to keep these electrical connectors dry.

6.1.3 Recommended Volumes

Follow these instructions to use the transport/calibration cup for calibration procedures.

- ✓ Ensure that an o-ring is installed in the o-ring groove of the transport/calibration cup bottom cap, and that the bottom cap is securely tightened.

  NOTE: Do not over-tighten as this could cause damage to the threaded portions.

- ✓ Remove the probe sensor guard, if it is installed.
- ✓ Remove the o-ring, if installed, from the probe module and inspect the installed o-ring on the probe module for obvious defects and, if necessary, replace it with the extra o-ring supplied.
- ✓ Some calibrations can be accomplished with the probe module upright or upside down. A separate clamp and stand, such as a ring stand, is required to support the probe module in the upside down position.
- ✓ To calibrate, follow the procedures in the next section, Calibration Procedures. The approximate volumes of the reagents are specified below for both the upright and upside down orientations.
- ✓ When using the Transport/Calibration Cup for dissolved oxygen % saturation calibration, make certain that the vessel is vented to the atmosphere by loosening the bottom cap or cup assembly and that approximately 1/8 inch (3 cm) of water is present in the cup.

Sensor to Calibrate	Upright	Upside Down
Conductivity	55ml	55ml
pH/ORP	30ml	60ml

Table 6.1 Calibration Volumes

6.2 Calibration Procedures

6.2.1 Accessing the Calibrate Screen

1. Press the **On/off** key to display the run screen.
2. Press the **Escape** key to display the main menu screen.
3. Use the arrow keys to highlight the **Calibrate** selection.

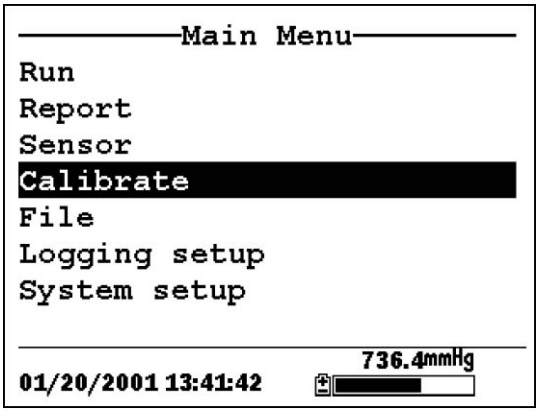


Figure 6.1 Main Menu

4. Press the **Enter** key. The Calibrate screen will be displayed.

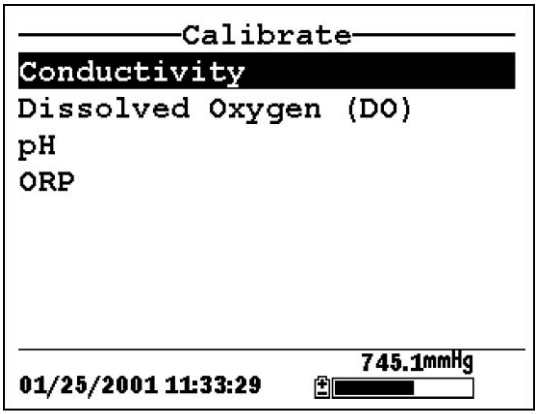


Figure 6.2 Calibrate Screen

6.2.2 Conductivity Calibration

This procedure calibrates specific conductance (recommended), conductivity and salinity. Calibrating any one option automatically calibrates the other two.

1. Go to the calibrate screen as described in Section *6.2.1Accessing the Calibrate Screen.*.

2. Use the arrow keys to highlight the **Conductivity** selection. See Figure 6.2 Calibrate Screen.

3. Press **Enter**. The Conductivity Calibration Screen is displayed.

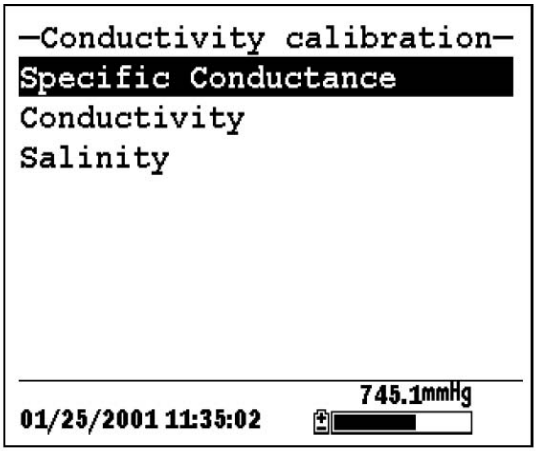


Figure 6.3 Conductivity Calibration Selection Screen

4. Use the arrow keys to highlight the Specific Conductance selection.

5. Press **Enter**. The Conductivity Calibration Entry Screen is displayed.

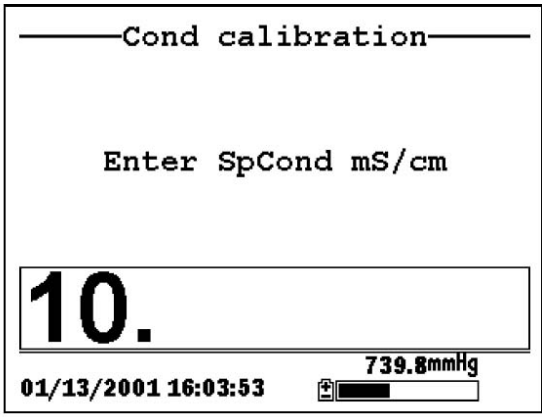


Figure 6.4 Conductivity Calibration Selection Screen

6. Place the correct amount of conductivity standard (see Table 6.1 Calibration Volumes) into a clean, dry or pre-rinsed transport/calibration cup.

⚠ **WARNING**: Calibration reagents may be hazardous to your health. See *Appendix D Health and Safety* for more information.

NOTE: For maximum accuracy, the conductivity standard you choose should be within the same conductivity range as the samples you are preparing to measure. However, we do not recommend using standards less than 1 mS/cm. For example:

- ✓ For fresh water use a 1 mS/cm conductivity standard.
- ✓ For brackish water use a 10 mS/cm conductivity standard.
- ✓ For seawater use a 50 mS/cm conductivity standard.

NOTE: Before proceeding, ensure that the sensor is as dry as possible. Ideally, rinse the conductivity sensor with a small amount of standard that can be discarded. Be certain that you avoid cross-contamination of solutions. Make certain that there are no salt deposits around the oxygen and pH/ORP sensors, particularly if you are employing standards of low conductivity.

7. Carefully immerse the sensor end of the probe module into the solution.

8. Gently rotate and/or move the probe module up and down to remove any bubbles from the conductivity cell.

NOTE: The sensor must be completely immersed past its vent hole. Using the recommended volumes from Table 6.1 Calibration Volumes, should ensure that the vent hole is covered.

9. Screw the transport/calibration cup on the threaded end of the probe module and securely tighten.

NOTE: Do not over tighten as this could cause damage to the threaded portions.

10. Use the keypad to enter the calibration value of the standard you are using.

NOTE: Be sure to enter the value in **mS/cm at 25°C**.

11. Press **Enter**. The Conductivity Calibration Screen is displayed.

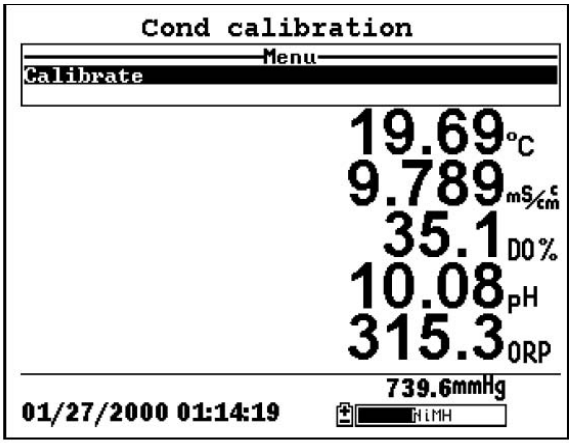


Figure 6.5 Conductivity Calibration Screen

12. Allow at least one minute for temperature equilibration before proceeding. The current values of all enabled sensors will appear on the screen and will change with time as they stabilize.

13. Observe the reading under Specific Conductance. When the reading shows no significant change for approximately 30 seconds, press **Enter**. The screen will indicate that the calibration has been accepted and prompt you to press **Enter** again to Continue.

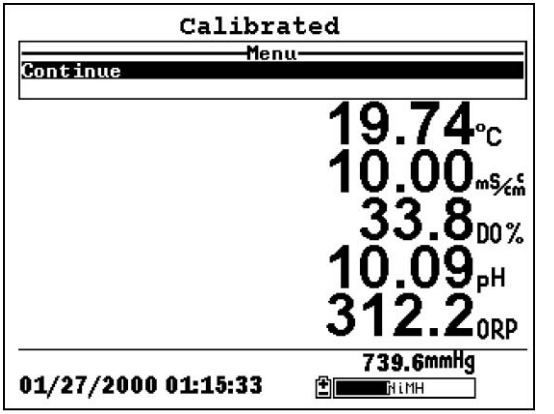


Figure 6.6 Calibrated

14. Press **Enter**. This returns you to the Conductivity Calibrate Selection Screen, See Figure 6.3 Conductivity Calibration Selection Screen..

15. Press **Escape** to return to the calibrate menu. See Figure 6.2 Calibrate Screen .

16. Rinse the probe module and sensors in tap or purified water and dry.

6.2.3 Dissolved Oxygen Calibration

This procedure calibrates dissolved oxygen. Calibrating any one option (% or mg/L) automatically calibrates the other.

1. Go to the calibrate screen as described in Section *6.2.1 Accessing the Calibrate Screen*.

NOTE: The instrument must be on for at least 10 - 15 minutes to polarize the DO sensor before calibrating.

2. Use the arrow keys to highlight the **Dissolved Oxygen** selection. See Figure 6.2 Calibrate Screen.

3. Press **Enter**. The dissolved oxygen calibration screen is displayed.

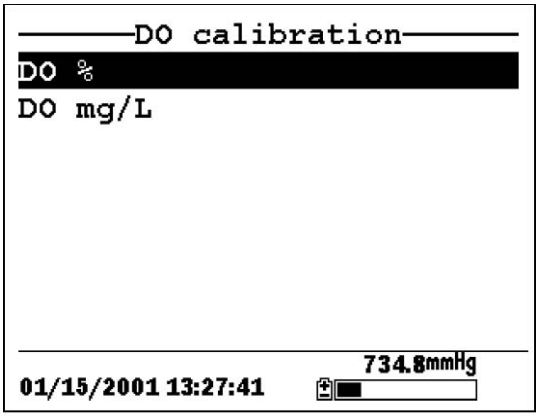


Figure 6.7 DO Calibration Screen

DO Calibration in % Saturation

1. Use the arrow keys to highlight the DO% selection.

2. Press **Enter**. The DO Barometric Pressure Entry Screen is displayed.

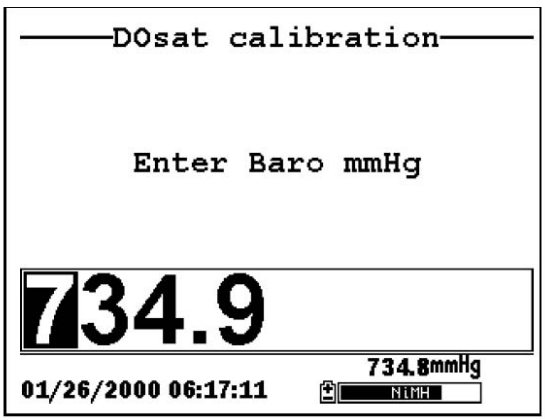


Figure 6.8 DO Barometric Pressure Entry Screen

3. Place approximately 3 mm (1/8 inch) of water in the bottom of the transport/calibration cup.

4. Place the probe module into the transport/calibration cup.

NOTE: Make sure that the DO and temperature sensors are **not** immersed in the water.

5. Engage only 1 or 2 threads of the transport/calibration cup to ensure the DO sensor is vented to the atmosphere.

6. Use the keypad to enter the current local barometric pressure.

NOTE: If the unit has the optional barometer, no entry is required.

NOTE: Barometer readings that appear in meteorological reports are generally corrected to sea level and must be uncorrected before use (refer to Section *10.10 Calibrate Barometer, Step 2*).

7. Press **Enter**. The DO% saturation calibration screen is displayed.

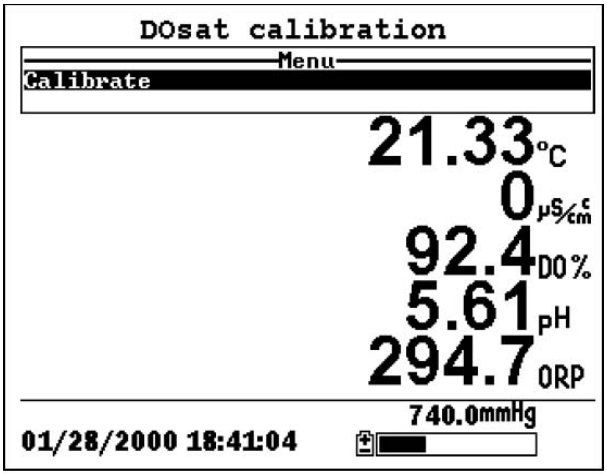


Figure 6.9 DO Sat Calibration Screen

8. Allow approximately ten minutes for the air in the transport/calibration cup to become water saturated and for the temperature to equilibrate before proceeding.

9. Observe the reading under DO %. When the reading shows no significant change for approximately 30 seconds, press **Enter**. The screen will indicate that the calibration has been accepted and prompt you to press **Enter** again to Continue. See Figure 6.6 Calibrated.

10. Press **Enter**. This returns you to the DO calibration screen, See Figure 6.7 DO Calibration Screen.

11. Press **Escape** to return to the calibrate menu. See Figure 6.2 Calibrate Screen.

12. Rinse the probe module and sensors in tap or purified water and dry.

DO Calibration in mg/L

DO calibration in mg/L is carried out in a water sample which has a known concentration of dissolved oxygen (usually determined by a Winkler titration).

1. Go to the DO calibrate screen as described in Section *6.2.3 Dissolved Oxygen Calibration*, steps 1 through 3.

2. Use the arrow keys to highlight the **DO mg/L** selection.

3. Press **Enter**. The DO mg/L Entry Screen is displayed.

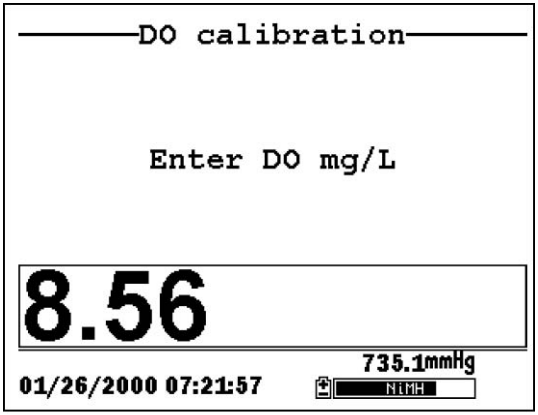


Figure 6.10 DO mg/L Entry Screen

4. Place the probe module in water with a known DO concentration.

NOTE: Be sure to completely immerse all the sensors.

5. Use the keypad to enter the known DO concentration of the water.

6. Press **Enter**. The Dissolved Oxygen mg/L Calibration Screen is displayed.

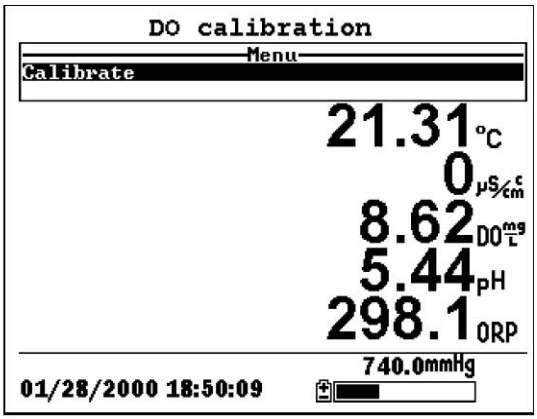


Figure 6.11 DO mg/L Calibration Screen

7. Stir the water with a stir bar, or by rapidly moving the probe module, to provide fresh sample to the DO sensor.

8. Allow at least one minute for temperature equilibration before proceeding. The current values of all enabled sensors will appear on the screen and will change with time as they stabilize.

9. Observe the DO mg/L reading, when the reading is stable (shows no significant change for approximately 30 seconds), press **Enter**. The screen will indicate that the calibration has been accepted and prompt you to press **Enter** again to Continue.

10. Press **Enter**. This returns you to the DO calibration screen. See Figure 6.7 DO Calibration Screen.

11. Press **Escape** to return to the calibrate menu. See Figure 6.2 Calibrate Screen.

12. Rinse the probe module and sensors in tap or purified water and dry.

6.2.4 pH Calibration

1. Go to the calibrate screen as described in *Section 6.2.1 Accessing the Calibrate Screen*.

2. Use the arrow keys to highlight the **pH** selection. See Figure 6.2 Calibrate Screen.

3. Press **Enter**. The pH calibration screen is displayed.

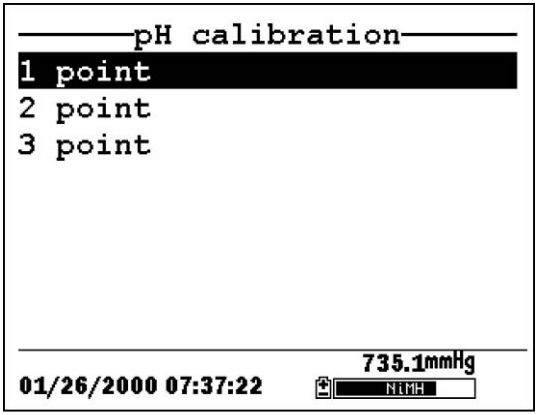


Figure 6.12 pH Calibration Screen

- Select the **1-point** option only if you are adjusting a previous calibration. If a 2-point or 3-point calibration has been performed previously, you can adjust the calibration by carrying out a one point calibration. The procedure for this calibration is the same as for a 2-point calibration, but the software will prompt you to select only one pH buffer.
- Select the **2-point** option to calibrate the pH sensor using only two calibration standards. Use this option if the media being monitored is known to be either basic or acidic. For example, if the pH of a pond is known to vary between 5.5 and 7, a two-point calibration with pH 7 and pH 4 buffers is sufficient. A three point calibration with an additional pH 10 buffer will not increase the accuracy of this measurement since the pH is not within this higher range.
- Select the **3-point** option to calibrate the pH sensor using three calibration solutions. In this procedure, the pH sensor is calibrated with a pH 7 buffer and two additional buffers. The 3-point calibration method assures maximum accuracy when the pH of the media to be monitored cannot be anticipated. The procedure for this calibration is the same as for a 2-point calibration, but the software will prompt you to select a third pH buffer.

4. Use the arrow keys to highlight the **2-point** selection.

5. Press **Enter**. The pH Entry Screen is displayed.

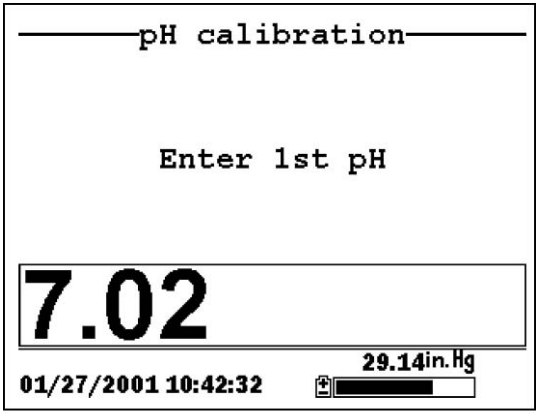


Figure 6.13 pH Entry Screen

6. Place the correct amount (see Table 6.1 Calibration Volumes) of pH buffer into a clean, dry or pre-rinsed transport/calibration cup.

NOTE: Always calibrate with buffer 7 first, regardless if performing a 1, 2, or 3 point calibration.

⚠ **WARNING**: Calibration reagents may be hazardous to your health. See *Appendix D Health and Safety* for more information.

NOTE: For maximum accuracy, the pH buffers you choose should be within the same pH range as the water you are preparing to sample.

NOTE: Before proceeding, ensure that the sensor is as dry as possible. Ideally, rinse the pH sensor with a small amount of buffer that can be discarded. Be certain that you avoid cross-contamination of buffers with other solutions.

7. Carefully immerse the sensor end of the probe module into the solution.

8. Gently rotate and/or move the probe module up and down to remove any bubbles from the pH sensor.

NOTE: The sensor must be completely immersed. Using the recommended volumes from Table 6.1 Calibration Volumes, should ensure that the sensor is covered.

9. Screw the transport/calibration cup on the threaded end of the probe module and securely tighten

NOTE: Do not over tighten as this could cause damage to the threaded portions.

10. Use the keypad to enter the calibration value of the buffer you are using **at the current temperature**.

NOTE: pH vs. temperature values are printed on the labels of all YSI pH buffers.

11. Press **Enter**. The pH calibration screen is displayed.

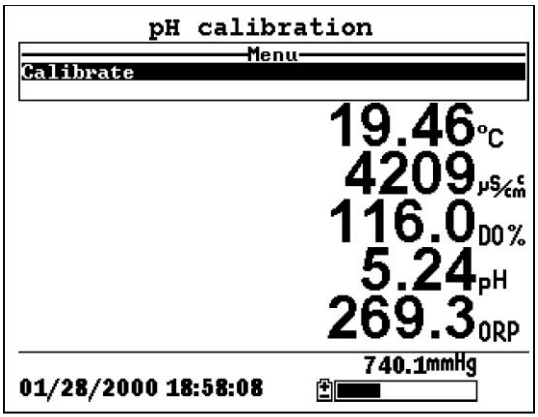


Figure 6.14 pH Calibration Screen

12. Allow at least one minute for temperature equilibration before proceeding. The current values of all enabled sensors will appear on the screen and will change with time as they stabilize.

13. Observe the reading under pH, when the reading shows no significant change for approximately 30 seconds, press **Enter**.

The screen will indicate that the calibration has been accepted and prompt you to press **Enter** again to Continue.

14. Press **Enter**. This returns you to the specified pH Calibration Screen, See Figure 6.13 pH Entry Screen.

15. Rinse the probe module, transport/calibration cup and sensors in tap or purified water and dry.

16. Repeat steps 6 through 13 above using a second pH buffer.

17. Press **Enter**. This returns you to the pH Calibration Screen, See Figure 6.12 pH Calibration Screen.

18. Press **Escape** to return to the calibrate menu. See Figure 6.2 Calibrate Screen.

19. Rinse the probe module and sensors in tap or purified water and dry.

6.2.5 ORP Calibration

1. Go to the calibrate screen as described in Section *6.2.1 Accessing the Calibrate Screen*.

2. Use the arrow keys to highlight the **ORP** selection. See Figure 6.2 Calibrate Screen..

3. Press **Enter**. The ORP calibration screen is displayed.

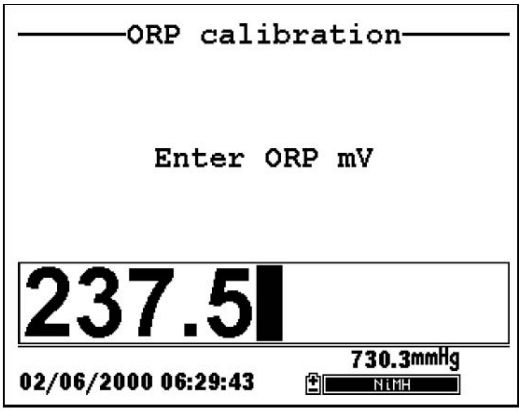


Figure 6.15 Specified ORP Calibration Screen

4. Place the correct amount (see Table 6.1 Calibration Volumes) of a known ORP solution (we recommend Zobell solution) into a clean, dry or pre-rinsed transport/calibration cup.

⚠ **WARNING**: Calibration reagents may be hazardous to your health. See *Appendix D Health and Safety* for more information.

NOTE: Before proceeding, ensure that the sensor is as dry as possible. Ideally, rinse the ORP sensor with a small amount of solution that can be discarded. Be certain that you avoid cross-contamination with other solutions.

5. Carefully immerse the sensor end of the probe module into the solution.

6. Gently rotate and/or move the probe module up and down to remove any bubbles from the ORP sensor.

NOTE: The sensor must be completely immersed. Using the recommended volumes from Table 6.1 Calibration Volumes should ensure that the sensor is covered.

7. Screw the transport/calibration cup on the threaded end of the probe module and securely tighten.

NOTE: Do not over tighten as this could cause damage to the threaded portions.

8. Use the keypad to enter the correct value of the calibration solution you are using at the current temperature. Refer to Table 6.2 Zobell Solution Values.

Temperature °C	Zobell Solution Value, mV
-5	270.0
0	263.5
5	257.0
10	250.5
15	244.0
20	237.5
25	231.0
30	224.5
35	218.0
40	211.5
45	205.0
50	198.5

Table 6.2 Zobell Solution Values

9. Press **Enter**. The ORP calibration screen is displayed.

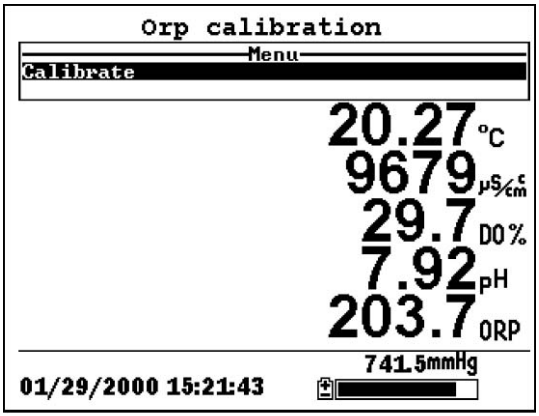


Figure 6.16 DO Cal Screen

10. Allow at least one minute for temperature equilibration before proceeding. The current values of all enabled sensors will appear on the screen and will change with time as they stabilize.

NOTE: Verify that the temperature reading matches the value you used in Table 6.2 Zobell Solution Values.

11. Observe the reading under ORP, when the reading shows no significant change for approximately 30 seconds, press **Enter**. The screen will indicate that the calibration has been accepted and prompt you to press **Enter** again to Continue.

12. Press **Enter**. This returns you to the Calibrate Screen. See Figure 6.2 Calibrate Screen.

13. Rinse the probe module and sensors in tap or purified water and dry.

6.3 Return to Factory Settings.

1. Go to the calibrate screen as described in Section *6.2.1 Accessing the Calibrate Screen*.

2. Use the arrow keys to highlight the **Conductivity** selection. See Figure 6.2 Calibrate Screen.

   NOTE: We will use the Conductivity sensor as an example; however, this process will work for any sensor.

3. Press **Enter.** The Conductivity Calibration Selection Screen is displayed. See Figure 6.3 Conductivity Calibration Selection Screen.

4. Use the arrow keys to highlight the **Specific Conductance** selection.

5. Press **Enter.** The Conductivity Calibration Entry Screen is displayed. See Figure 6.4 Conductivity Calibration Entry Screen.

6. Press and hold the **Enter** key down and press the **Escape** key.

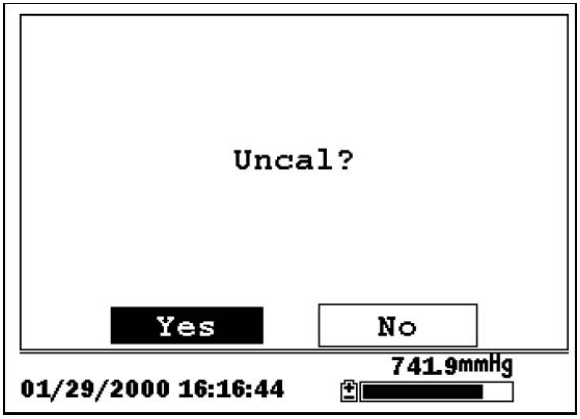


Figure 6.17 ORP Calibration Screen

7. Use the arrow keys to highlight the **YES** selection.

CAUTION: This returns a sensor to the factory settings. For example, in selecting to return specific conductance to the factory setting, salinity and conductivity will automatically return to their factory settings.

8. Press **Enter**. This returns you to the Conductivity Calibrate Selection Screen, See Figure 6.3 Conductivity Calibration Selection Screen. .

9. Press **Escape** to return to the calibrate menu. See Figure 6.2 Calibrate Screen.

7. Run

The Run screen displays data from the sensors in real-time and allows the user to log sample data to memory for later analysis. Refer to Section *9 Logging* for details on logging sample data.

7.1 Real-Time Data

NOTE: Before measuring samples you must prepare the probe module (refer to Section *3.4 Preparing the Probe Module*), attach the probe module to the instrument (refer to Section *3.6 Instrument/Cable Connection*) and calibrate the sensors (refer to Section *6 Calibrate*).

1. Press the On/off key.

OR select Run from the main menu to display the run screen.

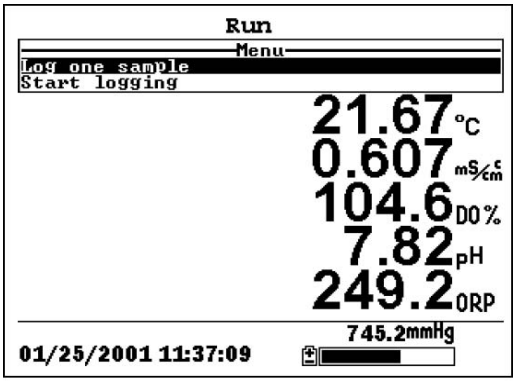


Figure 7.1 Run Screen

2. Make sure the probe sensor guard is installed.

3. Place the probe module in the sample. Be sure to completely immerse all the sensors.

4. Rapidly move the probe module through the sample to provide fresh sample to the DO sensor.

5. Watch the readings on the display until they are stable.

6. Refer to Section *9 Logging* for instructions on logging sample data.

8. File

The File menu allows the user to view, upload or delete sample data and calibration record files stored in the YSI 556 MPS.

8.1 Accessing the File Screen

1. Press the **On/off** key to display the run screen.

2. Press the **Escape** key to display the main menu screen.

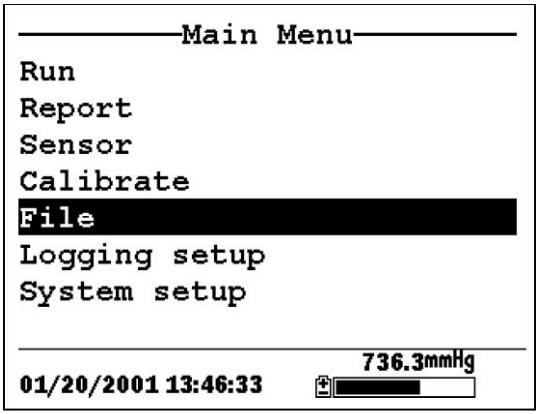


Figure 8.1 Main Menu Screen

3. Use the arrow keys to highlight the **File** selection.

4. Press the **Enter** key. The file screen is displayed.

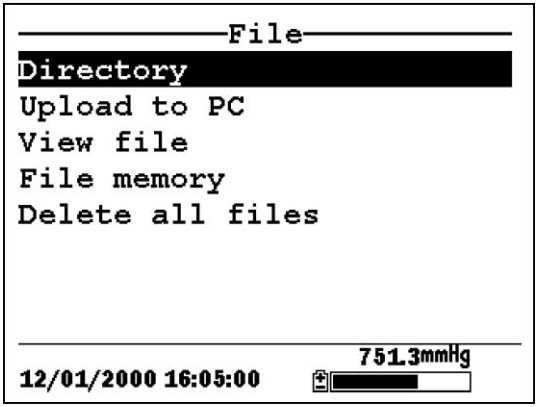


Figure 8.2 File Screen

8.2 Directory

1. Go to the file screen as described in Section *8.1 Accessing the File Screen*.

2. Use the arrow keys to highlight the **Directory** selection. See Figure 8.2 File Screen.

3. Press the **Enter** key. The file list screen is displayed.

NOTE: Files are listed in the order in which they are logged to memory. Sample Data files have the file extension **.dat**, while Calibration Record files have the file extension **.glp**.

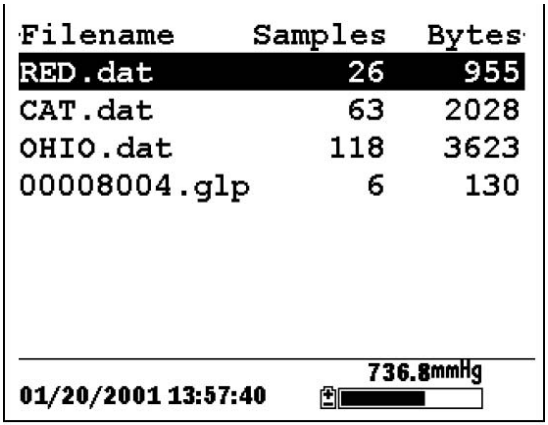


Figure 8.3 File List Screen

4. Use the arrow keys to highlight a file.

5. Press the **Enter** key. The file details screen is displayed.

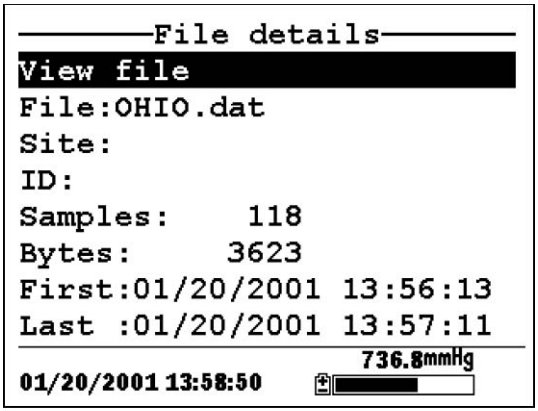


Figure 8.4 File Details Screen

6. Press the **Enter** key to view the file data. Refer to Section *8.3 View File* for details.

7. Press the **Escape** key repeatedly to return to the main menu screen.

8.3 View File

1. Go to the file screen as described in Section *8.1 Accessing the File Screen*. See Figure 8.2 File Screen.

2. Use the arrow keys to highlight the **View file** selection.

3. Press the **Enter** key. A list of files is displayed. See Figure 8.3 File List Screen.

4. Use the arrow keys to highlight an individual file.

   NOTE: You may have to scroll down to see all the files.

5. Press the **Enter** key. The file data is displayed with the file name at t8e top of the display.

   NOTE: If no file name was specified, the data is stored under the default name NONAME1.dat.

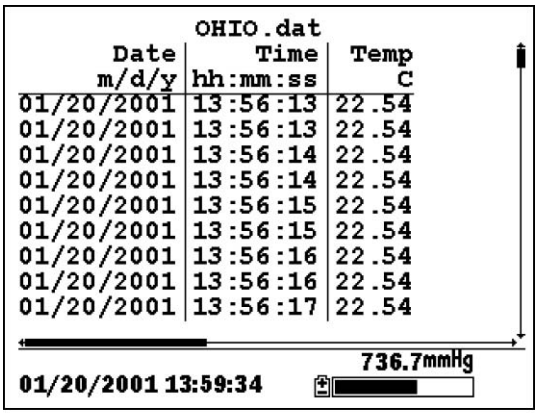


Figure 8.5 File Data Screen

6. Use the arrow keys to scroll horizontally and/or vertically to view all the data.

7. Press the **Escape** key repeatedly to return to the main menu screen.

8.4 Upload to PC

EcoWatch™ for Windows™ must be used as the PC software interface to the YSI 556 MPS. Refer to *Appendix G EcoWatch* for more information. EcoWatch for Windows® is available at no cost via a download from the YSI Web Site (www.ysi.com) or by contacting YSI Customer Support. Refer to *Appendix E Customer Service.*

8.4.1 Upload Setup

1. Disconnect the YSI 5563 Probe Module from the YSI 556 MPS instrument.

2. Connect the YSI 556 MPS to a serial (Comm) port of your computer via the 655173 PC Interface cable as shown in the following diagram:

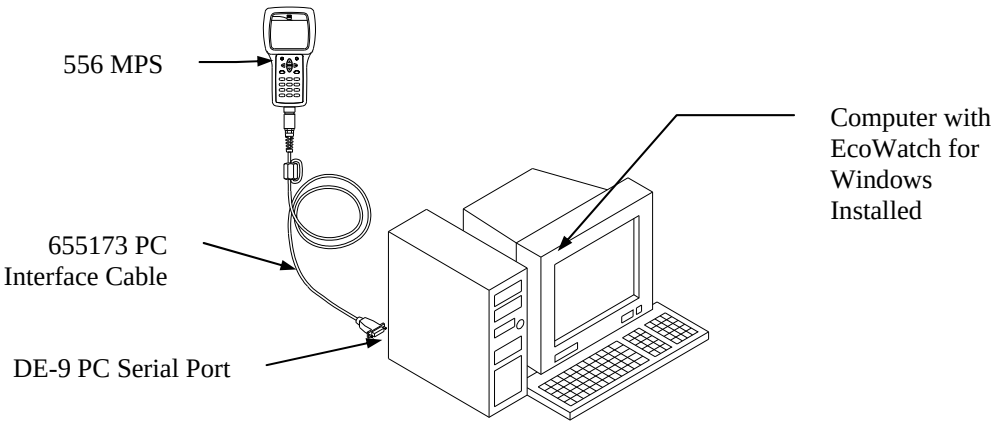


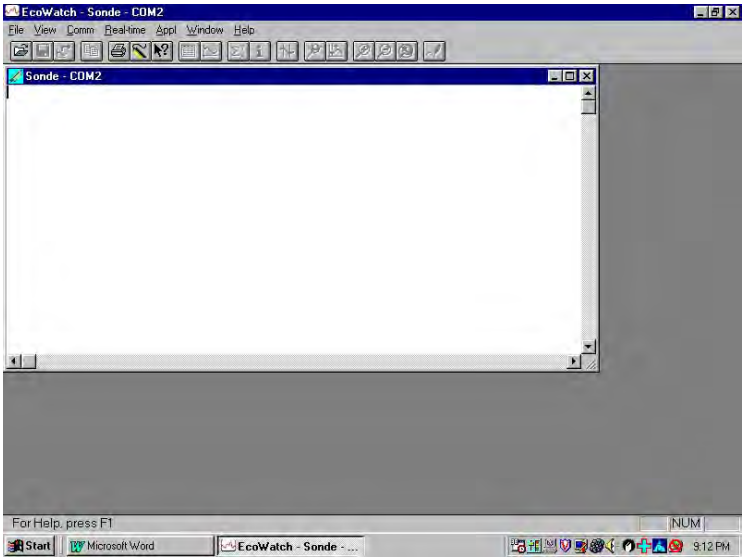
Figure 8.2 Computer/Instrument Interface

3. Open EcoWatch for Windows on your computer.

   NOTE: See *Appendix G EcoWatch* for installation instructions.

4. Click on the sonde/probe icon in the upper toolbar.

5. Set the Comm port number to match the port the YSI 556 MPS is connected to. After this setup procedure, the following screen will be present on your PC monitor:



8.4.2 Uploading a .DAT File

1. Setup the instrument as described in Section *8.4.1 Upload Setup*.

2. Go to the YSI 556 MPS file screen as described in Section *8.1 Accessing the File Screen.*

3. Use the arrow keys to highlight the **Upload to PC** selection. See Figure 8.2 File Screen.

4. Press the **Enter** key. The file list screen is displayed. See Figure 8.3 File List Screen.

5. Use the arrow keys to highlight the DAT file that you wish to transfer and press **Enter**, both the YSI 556 MPS and PC displays show the progress of the file transfer.

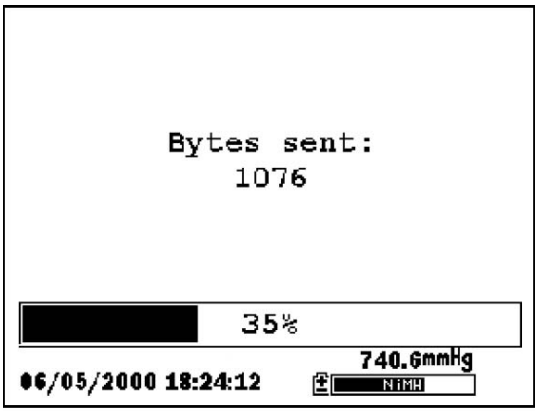
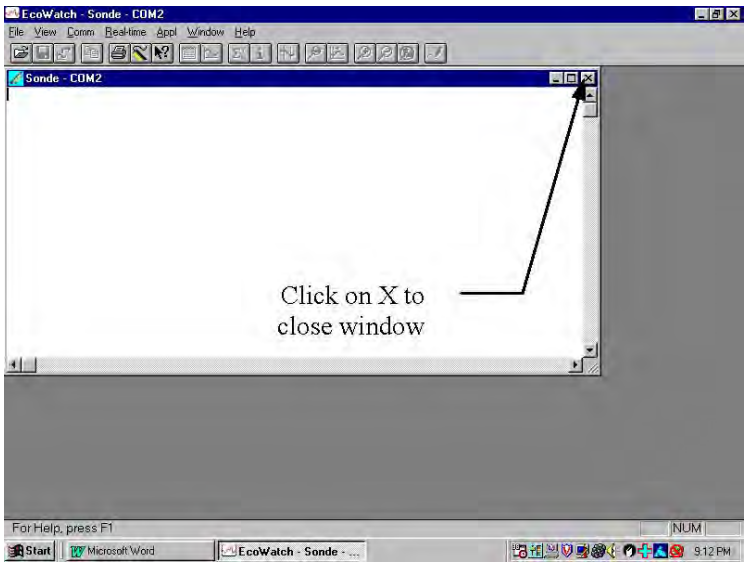


Figure 8.3 File Transfer Progress Screen

NOTE: After transfer, the file will be located in the C:\ECOWWIN\DATA folder of your PC, designated with a .DAT extension.

6. After the file transfer is complete, close the terminal window (small window on the PC) by clicking on the "X" at its upper right corner.



7. Press the **Escape** key on the YSI 556 MPS repeatedly to return

to the main menu screen.

8.4.3 Uploading a Calibration Record (.glp) File

For more information on the calibration record, refer to *Appendix H Calibration Record Information*.

1. Setup up the instrument as described in Section *8.4.1 Upload Setup*.

2. Go to the YSI 556 MPS file screen as described in Section

3. Use the arrow keys to highlight the Upload to PC selection. See Figure 8.2 File Screen.

4. Press the **Enter** key. The file list screen is displayed. See Figure 8.3 File List Screen.

5. Use the arrow keys to highlight the calibration record file that you wish to transfer and press **Enter.**

6. You will then be given a choice of uploading the file in three formats; **Binary, Comma & “” Delimited, and ASCII Text**.

   NOTE: The binary format is reserved for future YSI software packages.

7. Choose an option and press Enter, both the YSI 556 and PC displays show the progress of the file transfer.

   NOTE: After transfer, the file will be located in the C:\ECOWWIN\DATA folder of your PC, designated with the appropriate file extension.

   NOTE: To view the Calibration Record data after upload, simply open the .txt file in a general text editor such as Wordpad or Notepad.

8. After the file transfer is complete, close the terminal window (small window on the PC) by clicking on the “X” at its upper right corner.

9. Press the **Escape** key repeatedly to return to the main menu screen.

8.5 File Memory

1. Go to the file screen as described in Section *8.1 Accessing the File Screen*.

2. Use the arrow keys to highlight the **File memory** selection. See Figure 8.2 File Screen.

3. Press the **Enter** key. The file bytes used screen is displayed.

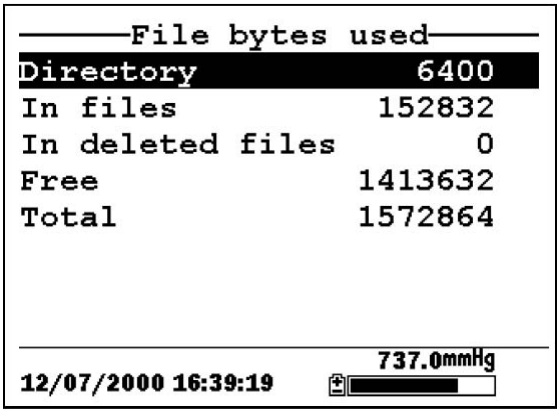


Figure 8.4 File Bytes Used Screen

4. The amount of free memory is listed in line 4 of the file bytes used screen.

   NOTE: If the amount of free memory is low, it may be time to delete all files (after first uploading all data to a PC). Refer to Section *8.6 Delete All Files*.

5. Press the **Escape** key repeatedly to return to the main menu screen.

8.6 Delete All Files

NOTE: It is not possible to delete individual files in order to free up memory. The only way to free up memory is to delete ALL files present. Take care to transfer all files to your computer (refer to Section 8.4 Upload to PC) before deleting them.

1. Go to the file screen as described in Section *8.1 Accessing the File Screen*.

2. Use the arrow keys to highlight the **Delete all files** selection. See Figure 8.2 File Screen.

3. Press the **Enter** key. The Delete all Files screen is displayed.

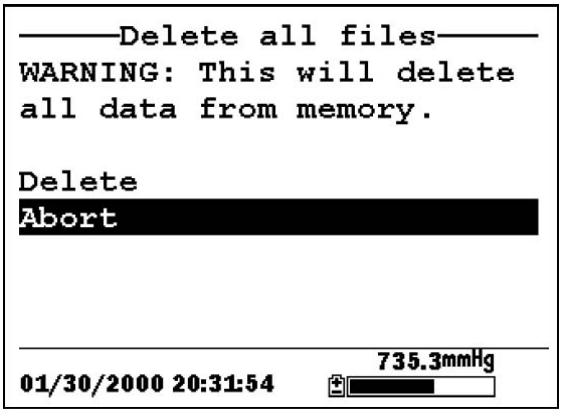


Figure 8.5 Delete All Files Screen

4. Use the arrow keys to highlight the **Delete** selection.

5. Press the **Enter** key.

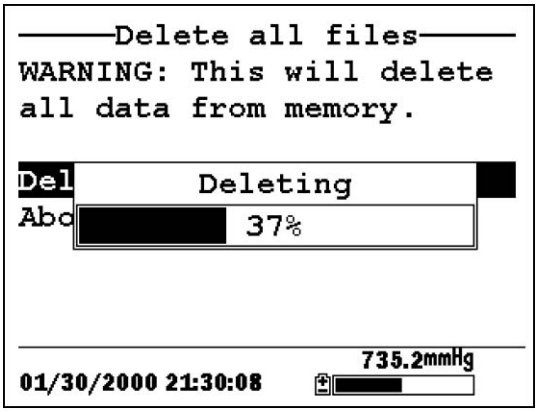


Figure 8.10 Deleting

The progress of file deletion is displayed in bar graph format.

NOTE: Deleting all files in the directory will not change any information in the site list.

6. Press the Escape key repeatedly to return to the main menu screen.

9. Logging

9.1 Accessing the Logging Setup Screen

1. Press the **On/off** key to display the run screen.

2. Press the **Escape** key to display the main menu screen.

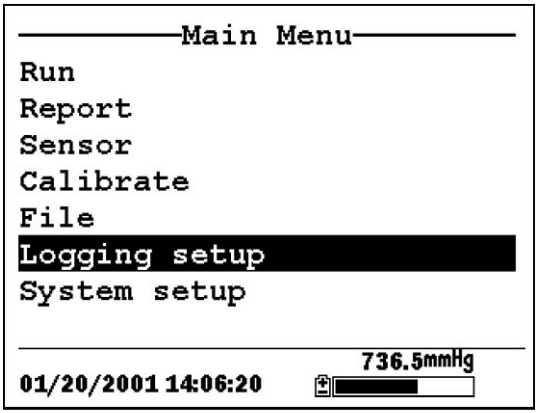


Figure 9.1 Main Menu

3. Use the arrow keys to highlight the **Logging setup** selection.

4. Press the **Enter** key. The logging setup screen is displayed.

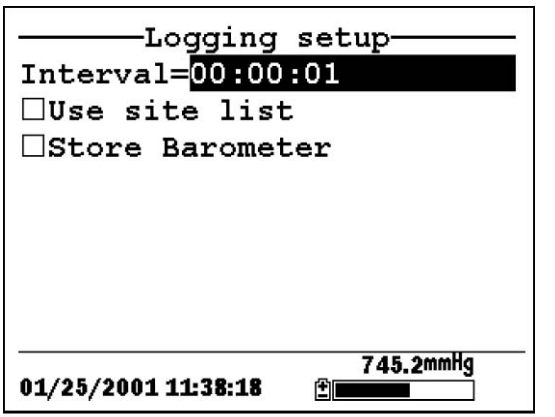


Figure 9.2 Setup Screen

9.2 Setting Logging Interval

Follow steps below to set the interval for logging a data stream.

NOTE: If you do not specify an interval, the instrument will use a default interval setting of 1 second.

NOTE: It is not necessary to set a logging interval when logging a single sample.

1. Go to the logging setup screen as described in Section *9.1 Accessing the Logging Setup Screen*.

2. Use the keypad to enter an interval between 1 second and 15 minutes. Refer to Section *2.9 Keypad Use*.

   NOTE: The interval field has hour, minute and second entry fields. Any entry over 1 hour will change automatically to a 15-minute setting.

3. Press the **Enter** key. The data stream interval is set.

4. Press the **Escape** key repeatedly to return to the main menu screen.

9.3 Storing Barometer Readings

NOTE: The **Store barometer** option is only available on instruments that are equipped with the optional barometer.

1. Go to the logging setup screen as described in Section *9.1 Accessing the Logging Setup Screen*.

2. Use the arrow keys to highlight the **Store barometer** selection. See Figure 9.2 Logging Setup Screen.

3. Press the **Enter** key until a check mark is entered in the box next to the store barometer selection if you want to log barometric readings.

OR press the **Enter** key until the box next to the barometer selection is empty if you do not want to log barometric readings.

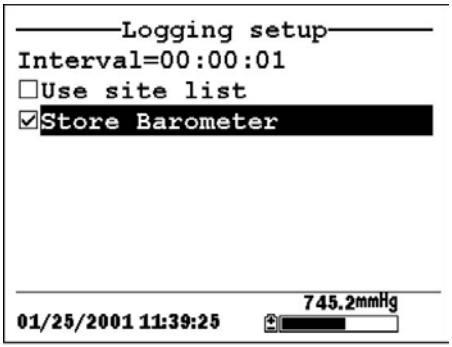


Figure 9.3 Store Barometer

4. Press the **Escape** key repeatedly to return to the main menu screen.

9.4 Creating a Site List

The site list option allows you to define file and site descriptions in the office or laboratory before moving to field logging studies. This is usually more convenient than entering the information at the site and is particularly valuable if you are visiting certain sites on a regular basis. The following section describes how to set up site lists which contain entries designated "Site Descriptions" that will be instantly available to the user in the field to facilitate the logging of data with pre-established naming of files and sites. There are two kinds of **Site Descriptions** available for use in Site lists:

• **Site Descriptions** associated with applications where data from a single site is always logged to a single file. This type is referred to as a "Single-Site Description" and is characterized by two parameters – a file name and a site name. Files logged to YSI 556 MPS memory under a **Single-Site Description** will be characterized primarily by the file name, but will also have the Site name attached, so that it is viewable in either the YSI 556 MPS **File directory** or in EcoWatch for Windows after upload to a PC

• **Site Descriptions** associated with applications where data from multiple sites are logged to a single file. This type is referred to as a "Multi-site Description" and is characterized by three parameters – a file name, a site name, and a site number. Files logged to YSI 556 MPS memory under a

Multi-site Description are characterized by a file name, but not a site name, since multiple sites are involved. However, each data point has a Site Number attached to it so that the user can easily determine the sampling site when viewing the data from the YSI 556 MPS **File** menu or processing the data in EcoWatch for Windows after upload to a PC.

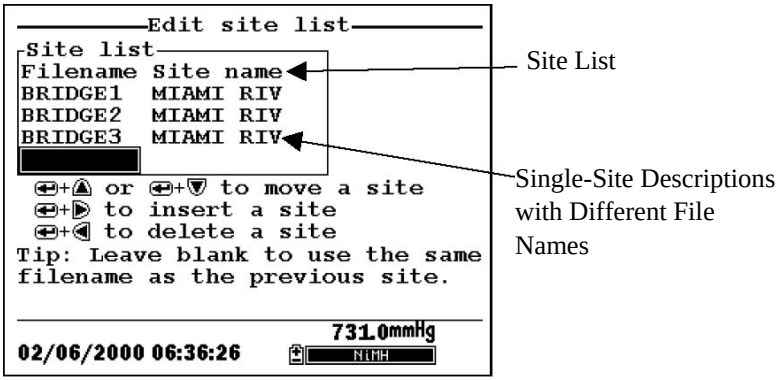


Figure 9.4 Single-Site Descriptions

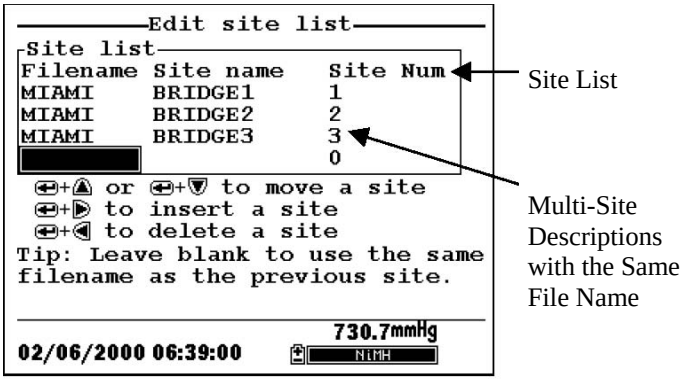


Figure 9.5 Multiple-Site Descriptions

NOTE: Site lists containing Single Site Descriptions are usually input with the designation **Store Site Number** INACTIVE in the YSI 556 MPS **Logging setup** menu. Thus, no site numbers appear in the first **Site list** example. Conversely, **Site lists** containing **Multi-Site Descriptions** MUST be input with the **Store Site Number** selection ACTIVE as shown in the second example.

To create a site list:

1. Go to the logging setup screen as described in Section *9.1 Accessing the Logging Setup Screen*.

2. Use the arrow keys to highlight the **Use site list** selection.

3. Press the **Enter** key. A check mark is entered in the box next to the use site list selection *and* two new entries appear on the logging setup screen. See Figure 9.6 Logging Setup Screen.

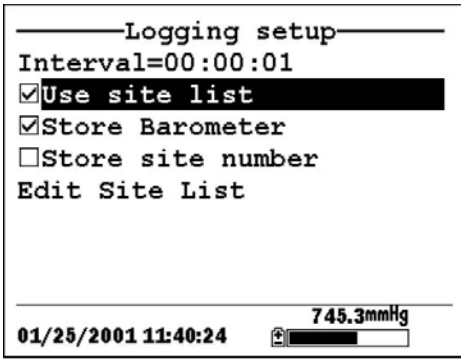


Figure 9.6 Logging Setup Screen

4. Use the arrow keys to highlight the **Store site number** selection.

5. If you are creating Multi-Site Descriptions (which require that the site **number** be stored in your data files), press the **Enter** key until a check mark appears in the box next to the store site number selection.

OR Press the **Enter** key until the box next to the store site number selection is empty, to create Single-Site Descriptions. The site **name** will be stored in the header of your data files.

6. Use the arrow keys to highlight the **Edit site list** selection.

7. Press the **Enter** key. The edit site list screen is displayed. See Figure 9.7 Edit Site List Screen. The **Filename** field is ready for input.

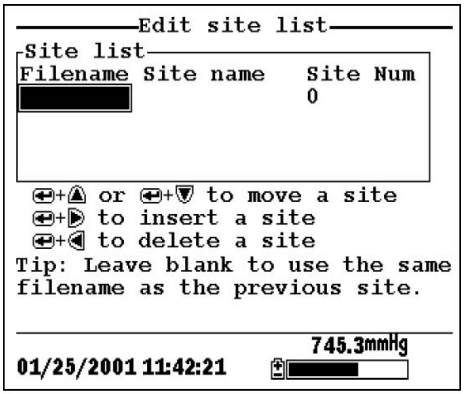


Figure 9.7 Edit Site List Screen

8. Use the keypad to enter a filename up to 8 characters in length. Refer to Section *2.9 Keypad Use*.

9. Press the **Enter** key. The cursor moves to the right for the entry of a **Site name**.

10. Use the keypad to enter a site name up to 11 characters in length. Refer to Section *2.9 Keypad Use*.

NOTE: If the store site number selection is *not* checked, skip to Step 13.

11. Press the **Enter** key. The cursor moves to the site number entry position.

12. Use the keypad to enter a site number up to 7 characters in length. Refer to Section *2.9 Keypad Use*.

13. Press **Enter**. The cursor moves to the next filename entry position.

14. Repeat Steps 8 to 13 until all filenames and sites have been entered.

15. Press **Escape** repeatedly to return to the main menu screen.

9.5 Editing a Site List

1. Go to the logging setup screen as described in Section *9.1 Accessing the Logging Setup Screen*.

2. Use the arrow keys to highlight the **Edit Site List** selection. See Figure 9.6 Logging Setup Screen.

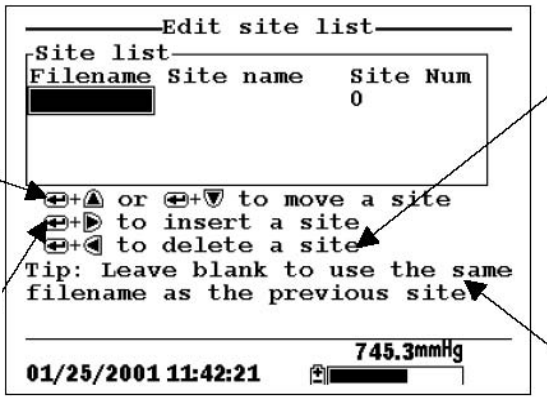
3. Press the **Enter** key. The edit site list screen is displayed.

4. Edit the site list using the keystrokes described below.

NOTE: Editing the site list will not have any effect on files stored in the instrument memory.

To MOVE a site:
Use the arrow keys to highlight a site. Press the Up or Down arrow key while holding down the Enter key.

To INSERT a site above another site:
Use the arrow keys to highlight the site. Press the Right arrow key while holding down the Enter key. Use keypad to input letters. Refer to Section 2.9 *Keypad Use.*



To DELETE a site:
Use the arrow keys to highlight a site. Press the Left arrow key while holding down the Enter key.

To use the same file name as the previous site: **Leave the filename blank**

Figure 9.1 Keystrokes for Editing Site List

9.6 Logging Data Without a Site List

1. Follow Steps 1 through 5 in Section 7.1 Real-Time Data.

2. Use the arrow keys to highlight the **Log one sample** selection on the run screen if only a single sample is being logged.

OR Use the arrow keys to highlight the **Start logging** selection on the run screen if a data stream is being logged.

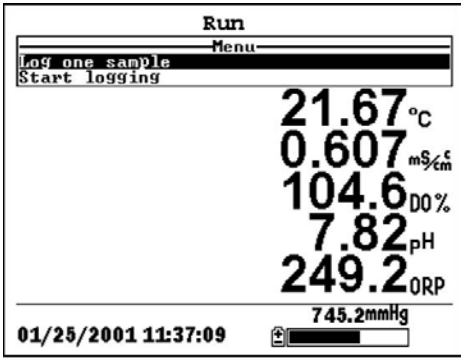


Figure 9.9 Run Screen

3. Press the **Enter** key. The Enter information screen is displayed.

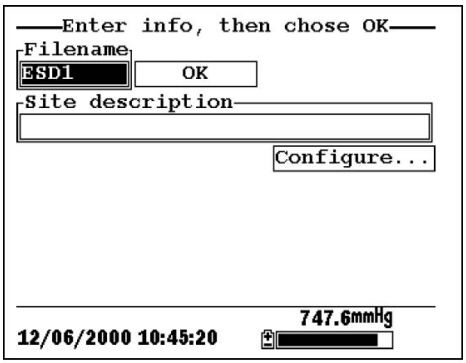


Figure 9.10 Enter Information Screen

NOTE: The last filename used will be displayed.

4. Use the keypad to enter a file name. Refer to Section *2.9 Keypad Use*.

NOTE: The instrument will assign a default file name of NONAME if no file name is specified.

5. Press the **Enter** key to input the file name.

6. Use the arrow keys to highlight the **Site description** field in the enter information screen.

   NOTE: Entering a Site Description is optional. You may leave the Site Description blank and skip to Step 9.

7. Use the keypad to enter a site description name. Refer to Section *2.9 Keypad Use*.

8. Press the **Enter** key to input the site description.

   NOTE: If you want to change the logging setup, such as sampling interval or storing the barometer reading, use the arrow keys to highlight the **Configure** field, press the **Enter** key, then refer to Section *9.2 Setting Logging Interval* or *9.3 Storing Barometer Readings* for details.

9. Use the arrow keys to highlight the **OK** field in the center of the information screen.

10. Press the **Enter** key to start logging.

    NOTE: If the parameter mismatch screen is displayed, refer to Section *9.8 Adding Data to Existing Files*.

11. If a single point is being logged, the header on the run screen changes momentarily from **Menu** to **Sample logged** to confirm that the point was successfully logged. Skip to Step

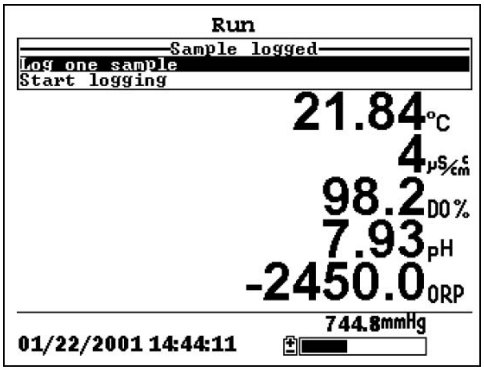


Figure 9.11 Sample Logged Screen

If a continuous stream of points is being logged, the start logging entry in the run screen changes from **Start logging** to **Stop logging**.

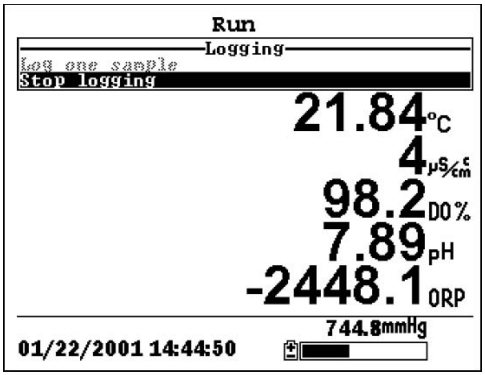


Figure 9.12 Logging Screen

12. At the end of the logging interval, press **Enter** to stop logging.

13. Refer to Section *8.3 View File* to view the data on the instrument display.

9.7 Logging Data with a Site List

1. If you have not already created a site list, refer to Section *9.4 Creating a Site List*.

2. Follow Steps 1 through 5 in Section 7.1 Real-Time Data.

3. Use the arrow keys to highlight the **Log one sample** selection on the run screen if only a single sample is being logged.

OR Use the arrow keys to highlight the **Start logging** selection on the run screen if a data stream is being logged. See Figure 9.9 Run Screen.

4. Press the **Enter** key. The Pick a site screen is displayed.

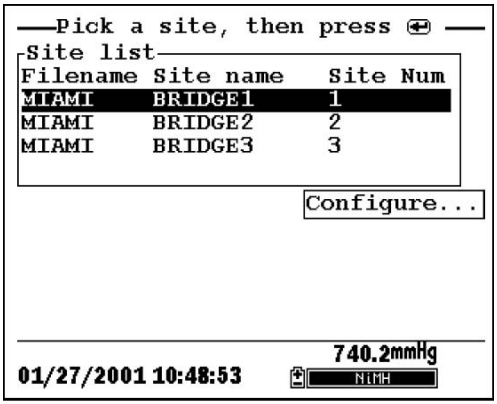


Figure 9.13 Pick a Site Screen

5. Use the arrow keys to highlight the **site** of your choice.

   NOTE: If the site of your choice is grayed out in the site list, refer to Section *9.8 Adding Data to Existing Files*.

   NOTE: Refer to Section *9.5 Editing a Site List* if you want to edit the site list.

6. Press the **Enter** key to start logging.

   NOTE: If the parameter mismatch screen is displayed, refer to Section 9.8 Adding Data to Existing Files.

7. If a single point is being logged, the header on the run screen changes momentarily from **Menu** to **Sample logged** to confirm that the point was successfully logged. See Figure 9.11 Sample Logged Screen. Skip to Step 9.

   If a continuous stream of points is being logged, the start logging entry in the run screen changes from **Start logging** to **Stop logging**. See Figure 9.12 Logging Screen.

8. At the end of the logging interval, press **Enter** to stop logging.

9. Refer to Section *8.3 View File* to view the data on the instrument display.

9.8 Adding Data to Existing Files

In order to add new data to an existing file, the current logging and sensor setup must be *exactly* the same as when the file was created. The following settings must be the same:

- **Sensors enabled** (refer to Section *4 Sensors*)
- **Store Barometer** (refer to Section *9.3 Storing Barometer Readings*)
- **Store Site Number** (refer to Section *9.4 Creating a Site List*)

If the current logging setup is not exactly the same as when the file was created, a parameter mismatch screen is displayed.

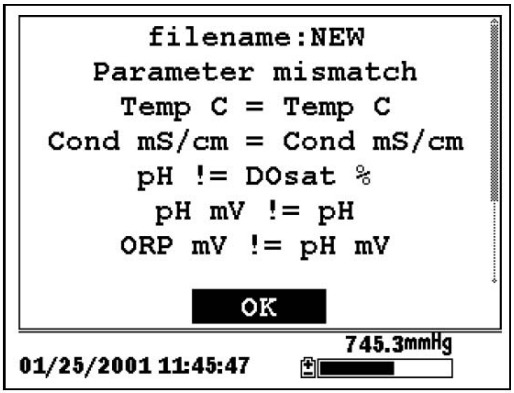


Figure 9.14 Parameter Mismatch Screen

NOTE: The right column shows parameters used when the file was created. The left column shows current parameters.

1. Press the **Down Arrow** key to scroll down and find the mismatch(es).

2. Use the following chart to resolve the mismatch(es).

Mismatch	**Action**	**Reference**
Sensor(s) missing from left column	Enable the missing sensor(s)	Section *4 Sensors*
Extra sensor(s) listed in left column	Disable the extra sensor(s)	Section *4 Sensors*
Barometer missing from left column, but present in right column	Enable the Store Barometer setting	Section *9.3 Storing Barometer Readings*
Barometer present in left column, but missing from right column	Disable the Store Barometer setting	Section *9.3 Storing Barometer Readings*
Store Site Number missing from left column, but present in right column	Enable the Store Site Number setting	Section *9.4 Creating a Site List*
Store Site Number present in left column, but missing from right column	Disable the Store Site Number setting	Section *9.4 Creating a Site List*

3. Return to Section *9.6 Logging Data without a Site List* or *9.7 Logging Data with a Site List*.

10. System Setup

The YSI 556 MPS has a number of features that are user-selectable or can be configured to meet the user's preferences. Most of these choices are found in the **System setup** menu.

10.1 Accessing the System Setup Screen

1. Press the **On/off** key to display the run screen. See Figure Front View of YSI 556 MPS.

2. Press the **Escape** key to display the main menu screen.

3. Use the arrow keys to highlight the **System setup** selection.

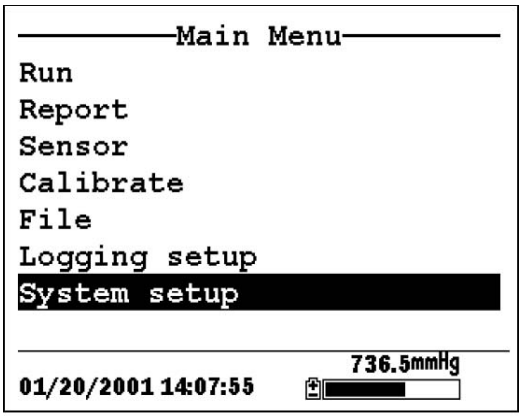


Figure 10.1 Main Menu

4. Press the **Enter** key. The system setup screen is displayed.

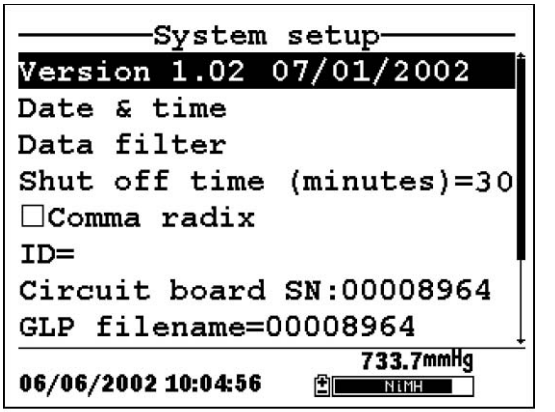


Figure 10.2 System Setup Screen

NOTE: The first line of the **System setup** menu shows the current software version of your YSI 556 MPS. As software enhancements are introduced, you will be able to upgrade your YSI 556 MPS from the YSI Web site. Refer to Section *11.2 Upgrading YSI 556 MPS* Software for details.

10.2 Language Setting

1. Go to the System Setup screen as described in Section *10.1 Accessing the System Setup Screen*.

2. Use the arrow keys to highlight **Language** on the System Setup screen. Press **Enter** to open the Language screen. .

3. Use the arrow keys to highlight your desired **Language**. Press **Enter**.

4. Press the Escape key repeatedly to return to the Main men

10.3 Date and Time Setup

1. Go to the system setup screen as described in Section *10.1 Accessing the System Setup Screen*.

2. Use the arrow keys to highlight the **Date & time** selection on the system setup screen. See Figure 10.2 System Setup Screen.

3. Press **Enter**. The date and time setup screen is displayed.

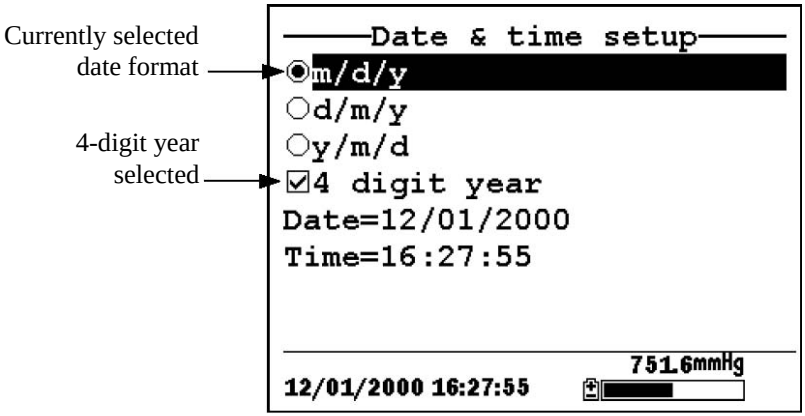


Figure 10.3 Date Setup Screen

NOTE: A black dot to the left of a date format indicates that format is selected.

4. Use the arrow keys to highlight your desired date format.

5. Press **Enter**.

6. Use the arrow keys to highlight the 4-digit year selection.

7. Press **Enter**. A check mark appears in the check box next to the 4-digit year selection.

   NOTE: If unchecked, a 2-digit year is used.

8. Use the arrow keys to highlight the **Date** selection.

9. Press **Enter**. A cursor appears over the first number in the date.

10. Enter the proper number from the keypad for the highlighted date digit. The cursor moves automatically to the next date digit. Refer to Section *2.9 Keypad Use* for more keypad information.

11. Repeat Step 10 until all date digits are correct.

12. Press **Enter** to input the specified date.

13. Use the arrow keys to highlight the **Time** selection.

14. Press **Enter**. A cursor appears over the first number in the time selection.

15. Enter the proper number from the keypad for the highlighted time digit. The cursor moves automatically to the next time digit.

    NOTE: Use military format when entering time. For example, 2:00 PM is entered as 14:00.

16. Repeat Step 15 until all time digits are correct.

17. Press **Enter** to input the correct time.

18. Press the **Escape** key repeatedly to return to the Main menu screen.

10.4 Data Filter

The Data Filter is a software filter that eliminates sensor noise and provides more stable readings.

NOTE: YSI recommends using the default values for the data filter for most field applications.

However, users who are primarily interested in a fast response from their dissolved oxygen sensor should consider a change of the default time constant setting of 8 seconds to one of 2 seconds. This change can be made according to the instructions in Section *10.3.1 Changing the Data Filter Settings* below. The disadvantage of lowering the time constant is that field pH readings may appear somewhat noisy if the cable is in motion.

10.4.1 Changing the Data Filter Settings

1. Go to the system setup screen as described in Section *10.1 Accessing the System Setup Screen*.

2. Use the arrow keys to highlight the **Data filter** selection. See Figure 10.1 Main Menu.

3. Press the **Enter** key. The Data filter setup screen is displayed.

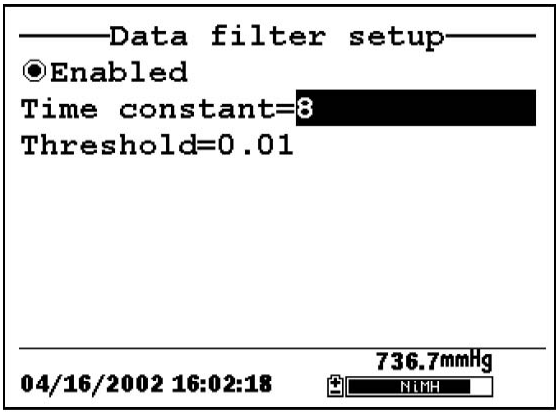


Figure 10.4 Data Filter Screen

4. With Enabled highlighted, press the **Enter** key to Enable or Disable the data filter. A black dot to the left of the selection indicates the data filter is enabled.

5. Use the arrow keys to highlight the **Time constant** field.

   NOTE: This value is the time constant in seconds for the software data filter. Increasing the time constant will result in greater filtering of the data, but will also slow down the apparent response of the sensors.

6. Use the keypad to enter a value. The default value is 8 and this value is ideal for most 556 field applications. As described in Section *10.3 Data Filter* above, users who wish to decrease the response time of the DO readings at the expense of some noise for the pH readings determined

concurrently, should change the Time Constant to a value of 2.

7. Press the **Enter** key to enter the time constant.

8. Use the arrow keys to highlight the **Threshold** field.

   NOTE: This value determines when the software data filter will engage/disengage, speeding the response to large changes in a reading. When the difference between two consecutive readings is larger than the threshold, then the reading is displayed unfiltered. When the difference between two consecutive readings drops below the threshold, readings will be filtered again.

9. Use the keypad to enter a value. The default value is 0.01.

10. Press the **Enter** key to enter the threshold.

11. Press the **Escape** key repeatedly to return to the Main menu screen.

10.5 Shutoff Time

The YSI 556 MPS shuts off automatically after 30 minutes of inactivity. The shut off time may be changed as described below.

1. Go to the system setup screen as described in Section *10.1 Accessing the System Setup Screen*.

2. Use the arrow keys to highlight the **Shutoff time** selection on the system setup screen. See Figure 10.2 System Setup Screen.

3. Use the keypad to enter a value from 0 to 60 minutes. The default value is 30.

   NOTE: To disable the automatic shutoff feature, enter a zero (0).

4. Press the **Enter** key to enter the correct shutoff time.

5. Press the **Escape** key repeatedly to return to the main menu screen.

10.6 Comma Radix

The user can toggle between a period (default) and comma for the radix mark by selecting this item and pressing the **Enter** key as follows:

1. Go to the system setup screen as described in Section *10.1 Accessing the System Setup Screen*.

2. Use the arrow keys to highlight the **Comma radix** selection on the system setup screen. See Figure 10.2 System Setup Screen.

3. Press the **Enter** key. A check mark appears in the check box next to the comma radix selection indicating that the radix mark is a comma.

10.7 ID

This selection allows you to enter an identification name/number for your YSI 556 MPS. This ID name/number is logged in the header of each file.

1. Go to the system setup screen as described in Section *10.1 Accessing the System Setup Screen*.

2. Use the arrow keys to highlight the **ID** selection. See Figure 10.1 Main Menu.

3. Use the keypad to enter an alphanumeric ID up to 15 characters in length. Refer to Section *2.9 Keypad Use*.

4. Press the **Enter** key to enter the ID.

5. Press the **Escape** key repeatedly to return to the main menu screen.

10.8 GLP Filename

This selection allows you to enter a different filename for the YSI 556 MPS Calibration Record file.

NOTE: The default filename is the “556 PC board Serial Number.glp.”

1. Go to the system setup screen as described in Section *10.1 Accessing the System Setup Screen*.
2. Use the arrow keys to highlight the **GLP Filename** selection. See Figure 10.1 Main Menu.
3. Use the keypad to enter a filename up to 8 characters in length. Refer to Section *2.9 Keypad Use*.
4. Press the **Enter** key to enter the new filename.
5. Press the **Escape** key repeatedly to return to the main menu screen.

10.9 TDS Constant

This selection allows you to set the constant used to calculate Total Dissolved Solids (TDS). TDS in g/L is calculated by multiplying this constant times the specific conductance in mS/cm.

10.9.1 Changing the TDS Constant

1. Go to the system setup screen as described in Section *10.1 Accessing the System Setup Screen*.
2. Use the arrow keys to highlight the **TDS Constant** selection. See Figure 10.1 Main Menu.
3. Use the keypad to enter a value. Refer to Section *2.9 Keypad Use*. The default value is 0.65.
4. Press the **Enter** key to enter the correct TDS constant.
5. Press the **Escape** key repeatedly to return to the main menu screen.

10.10 Barometer Units

The following information is only for instruments with the barometer option.

1. Go to the system setup screen as described in Section *10.1 Accessing the System Setup Screen*.

2. Use the arrow keys to highlight the **Barometer units** selection on the system setup screen. See Figure 10.2 System Setup Screen.

3. Press the **Enter** key. The Barometer units screen will appear.

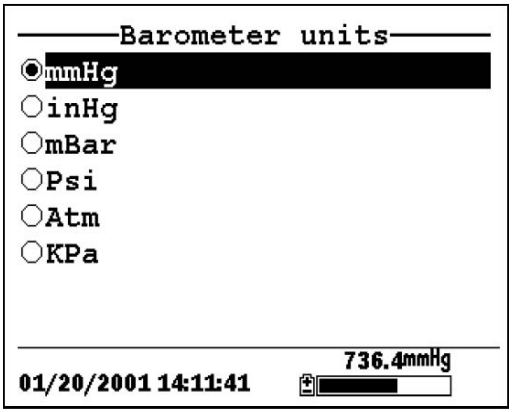


Figure 10.5 Data Filter Screen

A black dot indicates the currently selected units.

4. Use the arrow keys to highlight your desired barometric unit.

5. Press the **Enter** key to select your choice. A black dot will appear in the circle next to your selected units.

6. Press the **Escape** key repeatedly to return to the main menu screen.

10.11 Calibrate Barometer

The optional barometer has been factory calibrated to provide accurate readings. However, some sensor drift may occur over time, requiring occasional calibration by the user, as follows:

1. Determine your local barometric pressure from an independent laboratory barometer or from your local weather service.

2. If the barometric pressure (BP) reading is from your local weather station, reverse the equation that corrects it to sea level.

   NOTE: For this equation to be accurate, the barometric pressure units must be in mmHg.

   True BP = (Corrected BP) – [2.5 * (Local Altitude/100)]

3. Go to the system setup screen as described in Section *10.1 Accessing the System Setup Screen*.

4. Use the arrow keys to highlight the **Calibrate barometer** selection on the system setup screen. See Figure 10.2 System Setup Screen.

5. Press the **Enter** key. The Calibrate Barometer screen is displayed.

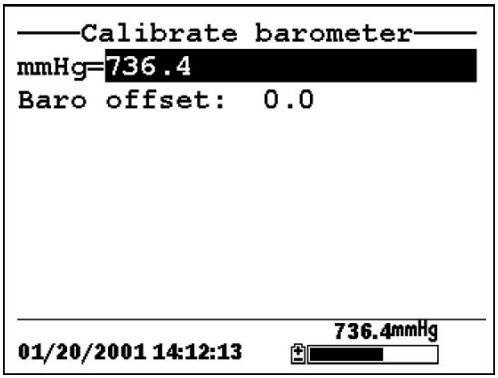


Figure 10.6 Barometer Calibration Screen

6. Use the keypad to input the known barometric pressure value as determined in Step 2.

7. Press the **Enter** key. The new barometer reading is displayed as well as the approximate offset from the factory reading.

   NOTE: To return the sensor to the factory setting, subtract the offset amount from the current setting and repeat Steps 5 to 7.

8. Press the **Escape** key repeatedly to return to the main menu screen.

11. Maintenance

11.1 Sensor Care and Maintenance

Once the sensors have been properly installed, remember that periodic cleaning and DO membrane changes are required.

11.1.1 DO Sensor

For best results, we recommend that the KCl solution and the membrane cap be changed at least once every 30 days.

1. It is important to recognize that oxygen dissolved in the sample is consumed during sensor operation. It is therefore essential that the sample be continuously stirred at the sensor tip. If stagnation occurs, your readings will be artificially low. Stirring may be accomplished by mechanically moving the sample around the sensor tip, or by rapidly moving the sensor through the sample. The rate of stirring should be at least 1 foot per second.

2. Membrane life depends on usage. Membranes will last a long time if installed properly and treated with care. Erratic readings are a result of loose, wrinkled, damaged, or fouled membranes, or from large (more than 1/8" diameter) bubbles in the electrolyte reservoir. If erratic readings or evidence of membrane damage occurs, you should replace the membrane and the electrolyte solution. The average replacement interval is two to four weeks.

3. If the membrane is coated with oxygen consuming (e.g. bacteria) or oxygen producing organisms (e.g. algae), erroneous readings may occur.

4. Chlorine, sulfur dioxide, nitric oxide, and nitrous oxide can affect readings by behaving like oxygen at the sensor. If you suspect erroneous readings, it may be necessary to determine if these gases are the cause.

5. Avoid any environment that contains substances that may attack the probe module and sensor materials. Some of these substances are concentrated acids, caustics, and strong solvents. The sensor materials that come in contact

with the sample include FEP Teflon, acrylic plastic, EPR rubber, stainless steel, epoxy, polyetherimide and the PVC cable covering.

6. It is possible for the silver anode, which is the entire silver body of the sensor, to become contaminated. This will prevent successful calibration. To restore the anode, refer to Section *11.1.1 DO Sensor, Silver Anode Cleaning.*

7. For correct sensor operation, the gold cathode must always be bright. If it is tarnished (which can result from contact with certain gases), or plated with silver (which can result from extended use with a loose or wrinkled membrane), the gold surface must be restored. To restore the cathode, refer to Section *11.1.1 DO Sensor, Gold Cathode Cleaning.*

8. To keep the electrolyte from drying out, store the sensor in the transport/calibration cup with at least 1/8″ of water.

Silver Anode Cleaning

After extended use, a thick layer of AgCl builds up on the silver anode reducing the sensitivity of the sensor. The anode must be cleaned to remove this layer and restore proper performance. The cleaning can be chemical or mechanical:

Chemical Cleaning: Remove the membrane cap and soak the entire anode section in a 14% ammonium hydroxide solution for 2 to 3 minutes, followed by a thorough rinsing with distilled or deionized water. The anode should then be thoroughly wiped with a wet paper towel to remove the residual layer from the anode.

Mechanical Cleaning: Sand off the dark layer from the silver anode with 400 grit wet/dry sandpaper. Wrap the sandpaper around the anode and twist the sensor. Rinse the anode with clean water after sanding, followed by wiping thoroughly with a wet paper towel.

NOTE: After cleaning, a new membrane cap must be installed. Refer to Section *3.4.3 Membrane Cap Installation***.**

Turn the instrument on and allow the system to stabilize for at least 30 minutes. If, after several hours, you are still unable to calibrate, contact your dealer or YSI Customer Service. Refer to *Appendix E Customer Service*.

Gold Cathode Cleaning

For correct sensor operation, the gold cathode must be textured properly. It can become tarnished or plated with silver after extended use. The gold cathode can be cleaned by using the adhesive backed sanding disc and tool provided in the YSI 5238 Probe Reconditioning Kit.

Using the sanding paper provided in the YSI 5238 Probe Reconditioning Kit, wet sand the gold with a twisting motion about 3 times or until all silver deposits are removed and the gold appears to have a matte finish. Rinse the cathode with clean water after sanding, followed by wiping thoroughly with a wet paper towel. If the cathode remains tarnished, contact your dealer or YSI Customer Service. Refer to *Appendix E Customer Service*.

NOTE: After cleaning, a new membrane cap must be installed. Refer to Section *3.4.3 Membrane Cap Installation***.**

11.1.2 DO Sensor Replacement

1. Remove the probe sensor guard.

⚠ **CAUTION:** Thoroughly dry the sensor so that no water enters the probe module sensor port when the sensor is removed.

2. Insert the long end of the hex key wrench into the small hole in the side of the probe module bulkhead. Turn the wrench counterclockwise and remove the screw. (You do not have to remove the screw all the way to release the sensor.)

3. Pull the old DO sensor module straight out of the probe module body.

   NOTE: The DO sensor is not threaded, it is keyed, so it cannot be removed by twisting.

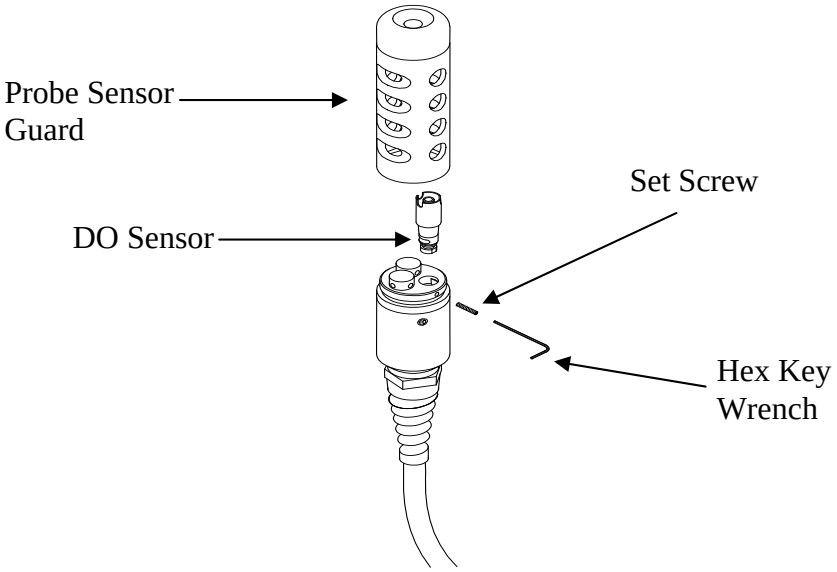


Figure 11.1 DO Sensor Replacement

4. Insert the new DO sensor module. Make sure that the inside of the probe module sensor port and the o-ring on the sensor are clean, with no contaminants, such as grease, dirt, or hair. The DO sensor is keyed, or has a flat side, so that it cannot be aligned improperly.

NOTE: Make sure the DO sensor bottoms out before the set screw is inserted.

5. Insert the set screw into the small hole in the side of the probe module bulkhead, and turn clockwise to rethread.

⚠ **CAUTION**: Make sure that you do not cross-thread the set screw. Use the hex key wrench to tighten the screw in properly, making sure that the screw does not stick out of the side of the probe module bulkhead. The probe sensor guard will not thread on properly and damage may result if the screw is allowed to stick out.

NOTE: The YSI 5563 DO sensor is shipped dry. A shipping membrane was installed to protect the electrode. A new membrane cap must be installed before the first use. Refer to Section 3.4.1 Sensor Installation.

11.1.3 YSI 5564 pH and 5565 Combination pH/ORP Sensor Cleaning

Cleaning is required whenever deposits or contaminants appear on the glass and/or platinum surfaces of these sensors or when the response of the sensor becomes slow.

1. Remove the sensor from the probe module.

2. Initially, simply use clean water and a soft clean cloth, lens cleaning tissue, or cotton swab to remove all foreign material from the glass bulb (YSI 5564 and YSI 5565) and platinum button (YSI 5565). Then use a moistened cotton swab to carefully remove any material that may be blocking the reference electrode junction of the sensor.

⚠ **CAUTION:** When using a cotton swab with the YSI 5564 or YSI 5565, be careful NOT to wedge the swab tip between the guard and the glass sensor. If necessary, remove cotton from the swab tip, so that the cotton can reach all parts of the sensor tip without stress.

NOTE: If good pH and/or ORP response is not restored by the above procedure, perform the following additional procedure:

1. Soak the sensor for 10-15 minutes in clean water containing a few drops of commercial dishwashing liquid.

2. GENTLY clean the glass bulb and platinum button by rubbing with a cotton swab soaked in the cleaning solution.

3. Rinse the sensor in clean water, wipe with a cotton swab saturated with clean water, and then re-rinse with clean water.

   NOTE: If good pH and/or ORP response is still not restored by the above procedure, perform the following additional procedure:

1. Soak the sensor for 30-60 minutes in one molar (1 M) hydrochloric acid (HCl). This reagent can be purchased from most distributors. Be sure to follow the safety instructions included with the acid.

2. GENTLY clean the glass bulb and platinum button by rubbing with a cotton swab soaked in the acid.

3. Rinse the sensor in clean water, wipe with a cotton swab saturated with clean water, and then re-rinse with clean water. To be certain that all traces of the acid are removed from the sensor crevices, soak the sensor in clean water for about an hour with occasional stirring.

   NOTE: If biological contamination of the reference junction is suspected or if good response is not restored by the above procedures, perform the following additional cleaning step:

1. Soak the sensor for approximately 1 hour in a 1 to 1 dilution of commercially available chlorine bleach.

2. Rinse the sensor with clean water and then soak for at least 1 hour in clean water with occasional stirring to remove residual bleach from the junction. (If possible, soak the sensor for period of time longer than 1 hour in order to be certain that all traces of chlorine bleach are removed.) Then re-rinse the sensor with clean water and retest.`

11.1.4 Temperature/Conductivity Sensor Cleaning

The single most important requirement for accurate and reproducible results in conductivity measurement is a clean cell. A dirty cell will change the conductivity of a solution by contaminating it. The small cleaning brush included in the YSI 5511 Maintenance Kit is ideal for this purpose.

To clean the conductivity cell:

1. Dip the brush in clean water and insert it into each hole 1520 times.

2. Rinse the cell thoroughly in deionized or clean tap water.

NOTE: In the event that deposits have formed on the electrodes, perform the following additional procedure:

1. Use a mild detergent solution in combination with the brush. Dip the brush in the solution and insert it into each hole 1520 times.

2. Rinse the cell thoroughly in deionized or clean tap water.

   NOTE: After cleaning, check the response and accuracy of the conductivity cell with a calibration standard.

   NOTE: If this procedure is unsuccessful, or if sensor performance is impaired, it may be necessary to return the sensor to a YSI authorized service center for service, Refer to *Appendix E Customer Service*.

   The temperature portion of the sensor requires no maintenance.

11.2 Upgrading YSI 556 MPS Software

1. Access the YSI Environmental Software Downloads page as described in *Appendix G EcoWatch* Step 1 through 3.

2. Click on the **YSI Instruments Software Updates** link (or scroll down until you see YSI 556 MPS).

3. Click on the file icon to the right of the **YSI 556 MPS** listing and save the file to a temporary directory on your computer.

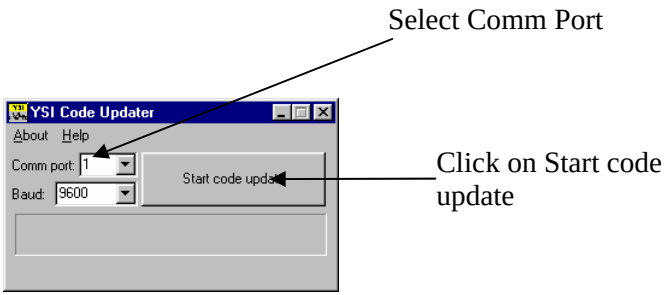
4. After the download is complete, run the file (that you just downloaded) and follow the on screen instructions to install the YSI Code Updater on your computer. If you encounter difficulties, contact YSI customer service for advice. Refer to *Appendix E Customer Service*.

5. If necessary, disconnect the YSI 5563 Probe Module from the YSI 556 MPS instrument.

6. Connect the YSI 556 MPS to a serial port of your computer via the 655173 PC interface cable. See Figure 8.6 Computer/Instrument Interface.

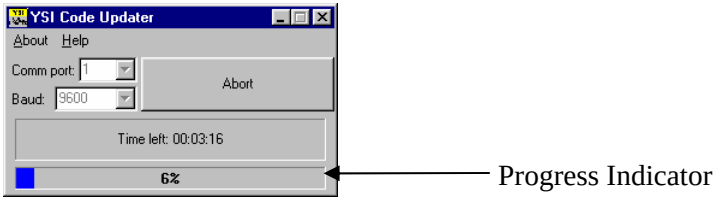
7. Press the **On/off** key on the YSI 556 MPS to display the run screen.

8. Run the YSI Code Updater software that you just installed on your computer. The following window will be displayed:



9. Set the Comm port number to match the port that you connected the 655173 PC Interface Cable to, then click on the Start Code Update button.

The YSI 556 MPS screen will blank out and a progress indicator will be displayed on the PC.



When the update is finished (indicated on the PC screen), the YSI 556 MPS will return to the Run screen. See Figure 7.1 Run Screen.

10. Close the YSI Code Updater window (on the PC) by clicking on the "X" in the upper right corner of the window.

11. Disconnect the YSI 556 MPS from the 655173 PC interface cable and reconnect it to the YSI 5563 Probe Module. Refer to Section *3.6 Instrument/Cable Connection*.

12. Storage

Proper storage between periods of usage will not only extend the life of the sensors, but will also ensure that the unit will be ready to use as quickly as possible in your next application.

12.1 General Recommendations for Short Term Storage

No matter what sensors are installed in the instrument, it is important to keep them moist without actually immersing them in liquid. Immersing them could cause some of them to drift or result in a shorter lifetime.

YSI recommends that short term storage of all multi-parameter instruments be done by placing approximately 1/2 inch of tap water in the transport/calibration cup that was supplied with the instrument, and by placing the probe module with all of the sensors installed into the cup. The use of a moist sponge instead of a 1/2 inch of tap water is also acceptable, as long as its presence does not compromise the attachment of the cup to the probe module. The transport/calibration cup should be sealed to prevent evaporation.

NOTE: Ensure that an o-ring is installed in the o-ring groove on the threaded end of the probe module body. See Figure 3.7 Transport/Calibration Cup Installation.

⚠ **CAUTION**: The water level has to be low enough so that none of the sensors are actually under water. Check the transport/calibration cup periodically to make certain that the water is still present or the sponge is still moist.

NOTE: If the storage water (tap water) is accidentally lost during field use, environmental water can be used.

12.2 General Recommendations for Long Term Storage

12.2.1 Probe Module Storage

1. Remove the pH or pH/ORP sensor from the probe module and store according to the individual sensor storage instructions found in Section *12.2.2 Sensor Storage*.

2. Seal the empty port with the provided port plug.

   NOTE: Leave the conductivity/temperature sensor and

dissolved oxygen sensor, with membrane cap still on, in the probe module.

3. Place 1/2″ of water, deionized, distilled or tap, in the transport/calibration cup.

⚠ **CAUTION:** The water level has to be low enough so that none of the sensors are actually under water. Check the transport/calibration cup periodically to make certain that the water is still present or the sponge is still moist.

4. Insert the probe module into the cup.

   NOTE: Ensure that an o-ring is installed in the o-ring groove on the threaded end of the probe module body. See Figure 3.7 Transport/Calibration Cup Installation.

12.2.2 Sensor Storage

Temperature/Conductivity Sensor

No special precautions are required. Sensor can be stored dry or wet, as long as solutions in contact with the thermistor and conductivity electrodes are not corrosive (for example, chlorine bleach). However, it is recommended that the sensor be cleaned with the provided brush prior to long term storage. Refer to Section *11.1.4 Temperature/Conductivity Sensor Cleaning.*

pH and Combination pH/ORP Sensor

The key to sensor storage is to make certain that the reference electrode junction does not dry out. Junctions which have been allowed to dry out due to improper storage procedures can usually be rehydrated by soaking the sensor for several hours (overnight is recommended) in a solution which is 2 molar in potassium chloride. If potassium chloride solution is not available, soaking the sensor in tap water or commercial pH buffers may restore sensor function. However in some cases the sensor may have been irreparably damaged by the dehydration and will require replacement.

⚠ **CAUTION:** Do not store the sensor in distilled or deionized water as the glass sensor may be damaged by exposure to this medium.

1. Remove the pH or pH/ORP sensor from the probe module.

2. Seal the empty port with the provided port plug.

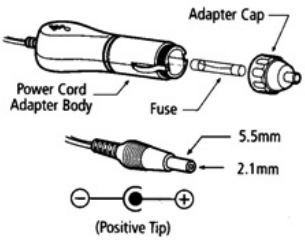
3. Place the sensor in the storage vessel (plastic boot or bottle) which was on the sensor at delivery. The vessel should contain a solution which is 2 molar in potassium chloride.

   NOTE: Make certain that the vessel is sealed to prevent evaporation of the storage solution.

13. Troubleshooting

The following sections describe problems you may encounter when using the YSI 556 MPS and provides suggestions to overcome the symptom.

PROBLEM	POSSIBLE SOLUTION
Display Problems	
No display is visible after pressing the on/off key.	If C cells are used, make certain that they are installed properly with regard to polarity and that good batteries are used. If a rechargeable battery pack is used, place the pack in the instrument and charge for 30 minutes.
Instrument software appears to be locked up as evidenced by no response to keypad entries or display not changing.	First, attempt to reset the instrument by simply turning off and then on again. If this fails, remove battery power from the instrument for 30 seconds and then reapply power. When using C cells, remove the battery lid and one of the batteries; when using the rechargeable battery pack, remove the pack completely from the instrument. After 30 seconds replace the battery or battery pack and check for instrument function.
The 556 display flashes and the instrument speaker makes a continuous clicking sound.	The battery voltage is low. Change to new C cells or recharge the 6117 battery pack.
Water Damage to Instrument	
Leakage detected in battery compartment when using C cells.	Dispose of batteries properly. Dry the battery compartment using compressed air if possible. If corrosion is present on battery terminals, contact YSI Customer Service.
Water has contacted rechargeable battery pack.	Remove battery pack immediately. Send battery pack to YSI Product Service for evaluation. **CAUTION**: DO NOT REUSE BATTERY PACK UNTIL YSI PRODUCT SERVICE HAS EVALUATED IT.
Leakage suspected into the main cavity of the instrument case.	Remove the batteries immediately. Return the instrument to YSI Product Service.

PROBLEM	POSSIBLE SOLUTIONS
Optional Cigarette Lighten Charger	
Power cord fuse blown. 	1. Unscrew adapter's cap, remove tip and pull out fuse. 2. Replace fuse with a new 2-amp fast-blow fuse from an electronics store such as Radio Shack. 3. Reassemble the adapter and securely screw the cap back onto the adapter body.
File Problems	
Upload of files from YSI 556 MPS to PC fails	1. Make sure that cable is connected properly to both 556 and PC. 2. Make certain that the proper Comm port is selected in EcoWatch for Windows.
Barometer data is not stored with sensor data file.	Make sure **Store barometer** is active in the 556 **Logging setup** menu.
Site Descriptions in the **Site List** are "grayed-out" and not available for appending files with additional data.	There is a parameter mismatch between the current 556 setup and that initially used. Change the current logging and sensor setup to match the setup that was initially used to create the file.
Sensor Problems	
Dissolved Oxygen reading is unstable or inaccurate. Out of Range message appears during calibration.	Sensor not properly calibrated. Follow DO cal procedures.
	Membrane not properly installed or may be punctured. Replace membrane cap.
	DO sensor electrodes require cleaning. Follow DO cleaning procedure. Use 5511 Maintenance kit.
	Water in sensor connector. Dry connector; reinstall sensor.
	Algae or other contaminant clinging to DO sensor. Rinse DO sensor with clean water.
	Barometric pressure entry is incorrect. Repeat DO cal procedure.
	Calibrated at extreme temperature. Recalibrate at (or near) sample temperature.
	DO sensor has been damaged. Replace sensor.
	Internal failure. Return probe module for service.

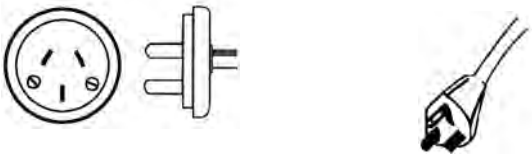
PROBLEM	POSSIBLE SOLUTIONS
Sensor Problems	
pH or ORP readings are unstable or inaccurate. Out of Range message appears during calibration.	Sensor requires cleaning. Follow sensor cleaning procedure.
	Sensor requires calibration. Follow cal procedures.
	pH sensor reference junction has dried out from improper storage. Soak sensor in tap water or buffer 4 until readings become stable.
	Water in sensor connector. Dry connector; reinstall sensor.
	Sensor has been damaged. Replace sensor.
	Calibration solutions out of spec or contaminated with other solution. Use new calibration solutions
	ORP fails Zobell check. Take into account temperature dependence of Zobell solution readings.
	Internal failure. Return probe module for service.
Conductivity unstable or inaccurate. Out of Range message appears during calibration	Conductivity improperly calibrated. Follow calibration procedure.
	Conductivity sensor requires cleaning. Follow cleaning procedure.
	Conductivity sensor damaged. Replace sensor.
	Calibration solution out of spec or contaminated. Use new calibration solution.
	Internal failure. Return probe module for service.
	Calibration solution or sample does not cover entire sensor. Immerse sensor fully.
Temperature, unstable or inaccurate	Water in connector. Dry connector; reinstall sensor.
	Sensor has been damaged. Replace the 5560 sensor.
Installed sensor has no reading	The sensor has been disabled. Enable sensor.
	Water in sensor connector. Dry connector; reinstall sensor.
	Sensor has been damaged. Replace sensor.
	Report output improperly set up. Set up report output.
	Internal failure. Return probe module for service.

If these guidelines and tips fail to correct your problem or if any other symptoms occur, contact YSI Customer Service for Advice. Refer to *Appendix E Customer Service*.

14. Appendix A YSI 556 MPS Specifications

For the most recent product specifications, please visit the YSI website: www.ysi.com

15. Appendix B Instrument Accessories

ITEM #	ACCESSORY
5563-4	4m Cable with DO/temp/conductivity
5563-10	10m Cable with DO/temp/conductivity
5563-20	20m Cable with DO/temp/conductivity
5564	pH Kit
5565	pH/ORP Kit
6118	Rechargeable Battery Pack Kit for use in US
5094	Rechargeable Battery Pack Kit with universal charger and three adapter cables for use in international applications
5095	Rechargeable Battery Pack Kit with universal charger and two adapter cables for use in international applications 
5083	Flow Cell – probe module is secured in the flow cell and groundwater is pumped through it. Displaced volume approx. 475 ml
3059	Flow Cell, low volume. Displaced volume approx. 200 ml
116505	Battery Lid
616	Charger, Cigarette Lighter – used to power up the instrument from a car's cigarette lighter
4654	Tripod
614	Ultra Clamp, C Clamp –used to clamp the instrument to a table top or car dashboard
6081	Large Carrying Case, Hard-sided
5085	Hands-free Harness
5065	Carrying Case, Form-fitted, for use in the field – has a clear vinyl window, shoulder strap, belt loop strap and hand strap

16. Appendix C Required Federal Communications Notice

The Federal Communications Commission defines this product as a computing device and requires the following notice.

This equipment generates and uses radio frequency energy and if not installed and used properly, may cause interference to radio and television reception. It has been type tested and found to comply with the limits for a Class A or Class B computing device in accordance with the specification in Subpart J of Part 15 of FCC Rules, which are designed to provide reasonable protection against such interference in a residential installation. However, there is no guarantee that interference will not occur in a particular installation. If this equipment does cause interference to radio or television reception, which can be determined by turning the equipment off and on, the user is encouraged to try to correct the interference by one or more of the following measures:

- Reorient the receiving antenna
- Relocate the computer with respect to the receiver
- Move the computer away from the receiver
- Plug the computer into a different outlet so that the computer and receiver are on different branch circuits.

If necessary, the user should consult the dealer or an experienced radio/television technician for additional suggestions. The user may find the following booklet, prepared by the Federal Communications Commission, helpful: "How to Identify and Resolve Radio-TV Interference Problems". This booklet is available from the U.S. Government Printing Office, Washington, D.C. 20402, Stock No.0004-000-00345-4.

17. Appendix D Health Safety

YSI Conductivity Solutions: 3161, 3163, 3165, 3167, 3168, 3169

INGREDIENTS:
- Iodine
- Potassium Chloride
- Water

WARNING: INHALATION MAY BE FATAL

⚠ ***CAUTION:*** **AVOID INHALATION, SKIN CONTACT, EYE CONTACT OR INGESTION. MAY EVOLVE TOXIC FUMES IN FIRE.**

Harmful if ingested or inhaled. Skin or eye contact may cause irritation. Has a corrosive effect on the gastro-intestinal tract, causing abdominal pain, vomiting, and diarrhea. Hyper-sensitivity may cause conjunctivitis, bronchitis, skin rashes etc. Evidence of reproductive effects.

FIRST AID:
INHALATION: Remove victim from exposure area. Keep warm and rest. In severe cases seek medical attention.
SKIN CONTACT: Remove contaminated cloth immediately. Wash affected area thoroughly with large amounts of water. In severe cases seek medical attention.
EYE CONTACT: Wash eyes immediately with large amounts of water, (approx. 10 minutes). Seek medical attention immediately.
INGESTION: Wash out mouth thoroughly with large amounts of water. Seek medical attention immediately.

YSI pH 4.00, 7.00, y 10.00: 3821, 3822, 3823

pH 4 INGREDIENTS:

- Potassium Hydrogen Phthalate
- Formaldehyde
- Water

pH 7 INGREDIENTS:

- Sodium Phosphate, Dibasic
- Potassium Phosphate, Monobasic
- Water

pH 10 INGREDIENTS:

- Potassium Borate, Tetra
- Potassium Carbonate
- Potassium Hydroxide
- Sodium (di) Ethylenediamine Tetraacetate
- Water

⚠ **CAUTION -AVOID INHALATION, SKIN CONTACT, EYE CONTACT OR INGESTION. MAY AFFECT MUCOUS MEMBRANES.**

Inhalation may cause severe irritation and be harmful. Skin contact may cause irritation; prolonged or repeated exposure may cause Dermatitis. Eye contact may cause irritation or conjunctivitis. Ingestion may cause nausea, vomiting and diarrhea.

FIRST AID:
INHALATION – Remove victim from exposure area to fresh air immediately. If breathing has stopped, give artificial respiration. Keep victim warm and at rest. Seek medical attention immediately.
SKIN CONTACT – Remove contaminated clothing immediately. Wash affected area with soap or mild detergent and large amounts of water (approx. 15-20 minutes). Seek medical attention immediately.
EYE CONTACT - Wash eyes immediately with large amounts of water (approx. 15-20 minutes), occasionally lifting upper and lower lids. Seek medical attention immediately.

INGESTION – If victim is conscious, immediately give 2 to 4 glasses of water and induce vomiting by touching finger to back of throat. Seek medical attention immediately.

YSI Zobell Solution: 3682

INGREDIENTS:

- Potassium Chloride
- Potassium Ferrocyanide Trihydrate
- Potassium Ferricyanide

⚠ **CAUTION -AVOID INHALATION, SKIN CONTACT, EYE CONTACT OR INGESTION. MAY AFFECT MUCOUS MEMBRANES.**

May be harmful by inhalation, ingestion, or skin absorption. Causes eye and skin irritation. Material is irritating to mucous membranes and upper respiratory tract. The chemical, physical, and toxicological properties have not been thoroughly investigated.

Ingestion of large quantities can cause weakness, gastrointestinal irritation and circulatory disturbances.

FIRST AID:
INHALATION – Remove victim from exposure area to fresh air immediately. If breathing has stopped, give artificial respiration. Keep victim warm and at rest. Seek medical attention immediately.
SKIN CONTACT – Remove contaminated clothing immediately. Wash affected area with soap or mild detergent and large amounts of water (approx. 15-20 minutes). Seek medical attention immediately.
EYE CONTACT - Wash eyes immediately with large amounts of water (approx. 15-20 minutes), occasionally lifting upper and lower lids. Seek medical attention immediately.
INGESTION – If victim is conscious, immediately give 2 to 4 glasses of water and induce vomiting by touching finger to back of throat. Seek medical attention immediately.

18. Appendix E Customer Service

18.1 Ordering and Technical Support

Telephone:	800 897 4151 (US)
	+1 937 767 7241 (Globally)
	Monday through Friday, 8:00 AM to 5:00 ET
Fax:	+1 937 767 9353 (orders)
	+1 937 767 1058 (technical support)
Email:	environmental@ysi.com or proseries@ysi.com
Mail:	YSI Incorporated
	1725 Brannum Lane
	Yellow Springs, OH 45387 USA
Website:	www.ysi.com

18.2 YSI Authorized Service Centers

YSI has authorized service centers throughout the United States and Internationally. For the nearest service center information, please visit www.ysi.com and click ‘Support’ or contact YSI Technical Support directly at 800-897-4151.

When returning a product for service, include the Product Return form with cleaning certification. The form must be completely filled out for a YSI Service Center to accept the instrument for service. The form may be downloaded from www.ysi.com by clicking on the ‘Support” tab, then the Product Return Form button.

18.3 Cleaning Instructions

Equipment exposed to biological, radioactive, or toxic materials must be cleaned and disinfected before being serviced. Biological contamination is presumed for any instrument, probe, or other device that has been used with body fluids or tissues, or with wastewater. Radioactive contamination is presumed for any instrument, probe or other device that has been used near any radioactive source.

If an instrument, probe, or other part is returned or presented for service without a Cleaning Certificate, and if in our opinion it represents a potential

biological or radioactive hazard, our service personnel reserve the right to withhold service until appropriate cleaning, decontamination, and certification has been completed. We will contact the sender for instructions as to the disposition of the equipment. Disposition costs will be the responsibility of the sender.

When service is required, either at the user's facility or at a YSI Service Center, the following steps must be taken to ensure the safety of service personnel.

- In a manner appropriate to each device, decontaminate all exposed surfaces, including any containers. 70% isopropyl alcohol or a solution of 1/4-cup bleach to 1-gallon tap water is suitable for most disinfecting. Instruments used with wastewater may be disinfected with .5% Lysol if this is more convenient to the user.
- The user shall take normal precautions to prevent radioactive contamination and must use appropriate decontamination procedures should exposure occur.
- If exposure has occurred, the customer must certify that decontamination has been accomplished and that no radioactivity is detectable by survey equipment.
- Any product being returned to the YSI Repair Center should be packed securely to prevent damage.
- Cleaning must be completed and certified on any product before returning it to YSI.

18.4 Packing Procedure

- Clean and decontaminate items to ensure the safety of the handler.
- Complete and include the Cleaning Certificate.
- Place the product in a plastic bag to keep out dirt and packing material.
- Use a large carton, preferably the original, and surround the product completely with packing material.
- Insure for the replacement value of the product.

18.5 Warranty

The instrument is warranted for three years against defects in workmanship and materials when used for its intended purposes and maintained according to instructions. The probe module and cables are warranted for one year. The dissolved oxygen, temperature/conductivity, pH, and pH/ORP combination sensors are warranted for one year. Damage due to accidents, misuse, tampering, or failure to perform prescribed maintenance is not covered. The warranty period for chemicals and reagents is determined by the expiration date printed on their labels. Within the warranty period, YSI will repair or replace, at its sole discretion, free of charge, any product that YSI determines to be covered by this warranty.

To exercise this warranty, write or call your local YSI representative, or contact YSI Customer Service in Yellow Springs, Ohio. Send the product and proof of purchase, transportation prepaid, to the Authorized Service Center selected by YSI. Repair or replacement will be made and the product returned transportation prepaid. Repaired or replaced products are warranted for the balance of the original warranty period, or at least 90 days from date of repair or replacement.

Limitation of Warranty

This Warranty does not apply to any YSI product damage or failure caused by (i) failure to install, operate or use the product in accordance with YSI's written instructions, (ii) abuse or misuse of the product, (iii) failure to maintain the product in accordance with YSI's written instructions or standard industry procedure, (iv) any improper repairs to the product, (v) use by you of defective or improper components or parts in servicing or repairing the product, or (vi) modification of the product in any way not expressly authorized by YSI.

THIS WARRANTY IS IN LIEU OF ALL OTHER WARRANTIES, EXPRESSED OR IMPLIED, INCLUDING ANY WARRANTY OF MERCHANTABILITY OR FITNESS FOR A PARTICULAR PURPOSE. YSI'S LIABILITY UNDER THIS WARRANTY IS LIMITED TO REPAIR OR REPLACEMENT OF THE PRODUCT, AND THIS SHALL BE YOUR SOLE AND EXCLUSIVE REMEDY FOR ANY DEFECTIVE PRODUCT COVERED BY THIS WARRANTY. IN NO EVENT SHALL YSI BE LIABLE FOR ANY SPECIAL, INDIRECT, INCIDENTAL OR CONSEQUENTIAL DAMAGES RESULTING FROM ANY DEFECTIVE PRODUCT COVERED BY THIS WARRANTY.

19. Appendix F Ferrite Bead Installation

⚠ **WARNING:** If you are using your YSI 556 in a European Community (CE) country or in Australia or New Zealand, you must attach a ferrite bead to the 655173 PC Interface Cable and the YSI 6117 Charger Adapter Cable in order to comply with the Residential, Commercial and Light Industrial Class B Limits for radio-frequency emissions specified in EN55011 (CISPR11) for Industrial, Scientific and Medical laboratory equipment. These ferrite assemblies are supplied as part of cable kits.

1. Make a small loop (approximately 5 cm in diameter) in the cable near the YSI 556 MS-19 connector.

2. Lay the open ferrite bead assembly under the loop with the cable cross-over position within the cylinder of the ferrite bead.

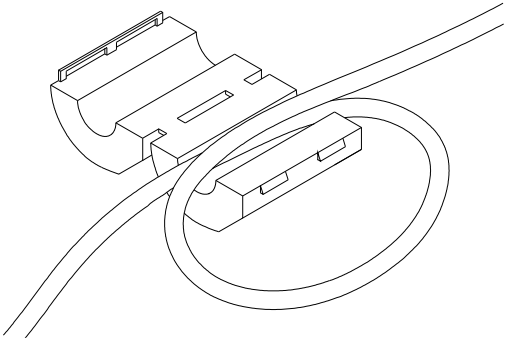


Figure 19.1 Ferrite Bead Installation

3. Snap the two pieces of the bead together making certain that the tabs lock securely.

4. When the installation is complete, the 655173 and 6117 cables should resemble the following drawings.

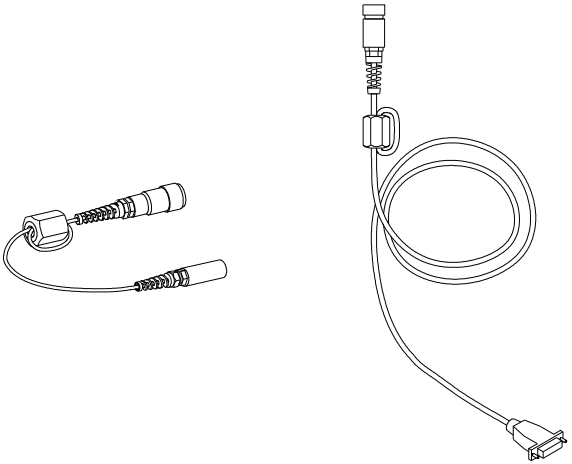


Figure 19.2 Cables with Ferrite Beads

20. Appendix G EcoWatch

EcoWatch™ for Windows™ must be used as the PC software interface to the YSI 556 MPS. EcoWatch is a powerful tool that can also be used with YSI 6-series sondes. Many features of the software will only be utilized by advanced users or are not relevant to the 556 MPS at all. This section is designed in tutorial format to familiarize you with the commonly used features of EcoWatch so that it will be possible to:

- Upload data from a 556 MPS to a PC
- Assemble plots and reports of your data
- Zoom in on certain segments of the plots of your data to facilitate analysis
- Show statistical data for your studies
- Export data in spreadsheet-compatible formats
- Print plots and reports

The advanced features of EcoWatch can be explored by downloading a 6-series manual from the YSI Web Site (www.ysi.com), purchasing a hard copy of the manual through YSI Customer Service (Item # 069300), or utilizing the on-line help feature of the software.

20.1 Installing EcoWatch for Windows

EcoWatch for Windows is available at no cost via a download from the YSI Web Site – www.ysi.com

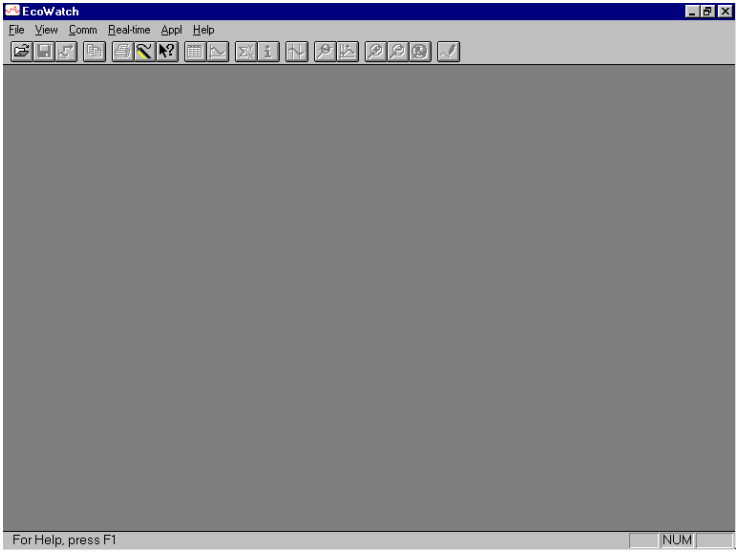
20.2 EcoWatch Tutorial

This EcoWatch tutorial is designed to teach you the commonly used operations associated with the software when used with your 556 MPS.

After you have uploaded a file, Refer to Section 8.4 Upload to PC, you will see two files in the C:\ECOWWIN\DATA directory; the file you transferred and a file supplied by YSI designated SAMPLE.DAT. This SAMPLE.DAT file is referred to in the remainder of this tutorial section. After following the instructions below for the analysis of SAMPLE.DAT, you apply the same analysis to the data file which was uploaded from your 556 MPS to assure that you are familiar with the basic features and capabilities of EcoWatch for Windows.

To start the analysis of the SAMPLE.DAT file, note that a shortened menu

bar is visible and many of the tools in the toolbar appear dimmed or “grayed out” before any file is opened (see below).

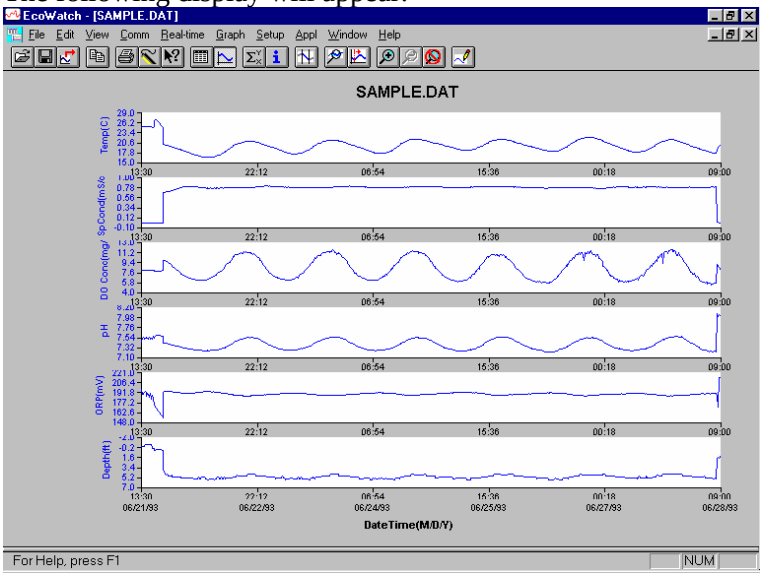


Full activation of EcoWatch features will be realized after a file is opened.

To open the sample data file:

1. Click the **File** menu button in the toolbar.
2. Select the **SAMPLE.DAT** file.
3. Click **OK** to open the file.

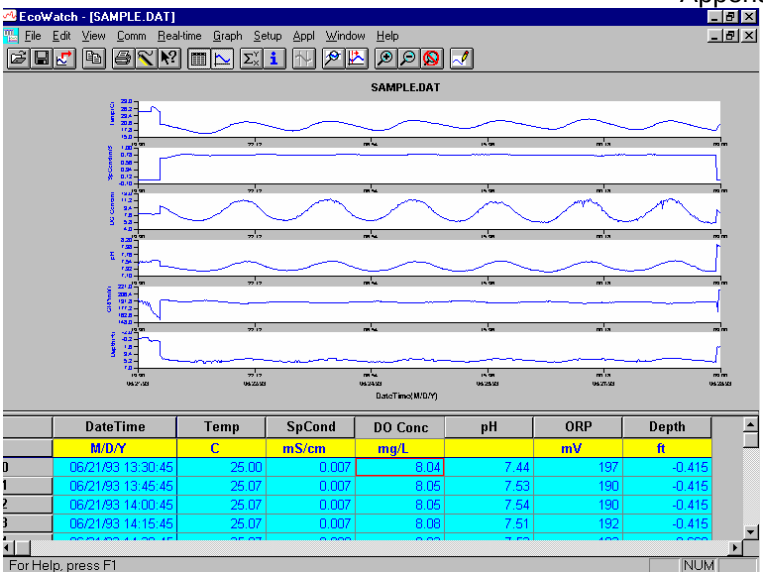
The following display will appear:



Note that the data in this file appears as a graph of temperature, specific conductance, dissolved oxygen, pH, ORP, and depth, all versus time. The graphs are scaled automatically so that all data fits comfortably on the computer screen. Note also that this data file was obtained with a 6-series sonde for which a depth sensor is available. Depth is NOT a current parameter for the 556 MPS.

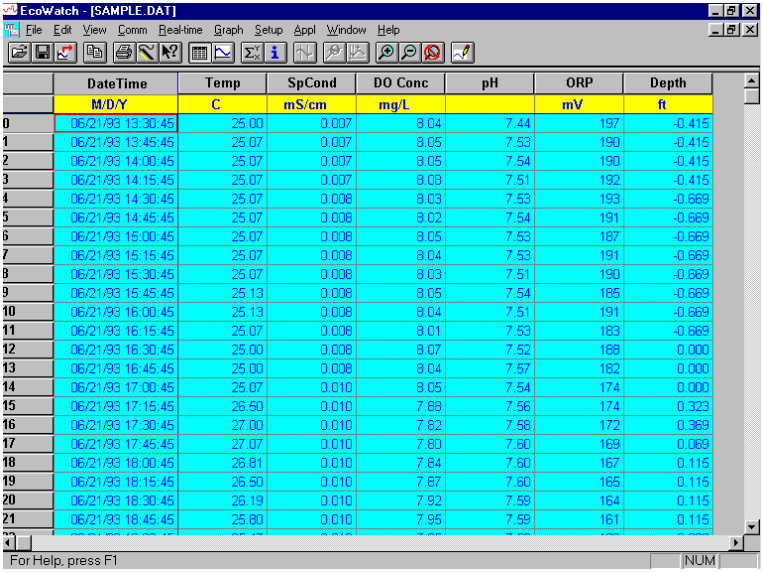
The **Table** and **Graph** buttons on the toolbar are on/off switches that are used to display or hide the graph and table pages respectively. When displaying a graph and a table at the same time, you can control the relative size of the two pages by placing the cursor over the small bar that separates them and

then dragging it to the desired location. Click the **Table** button to generate the following dual display of data.



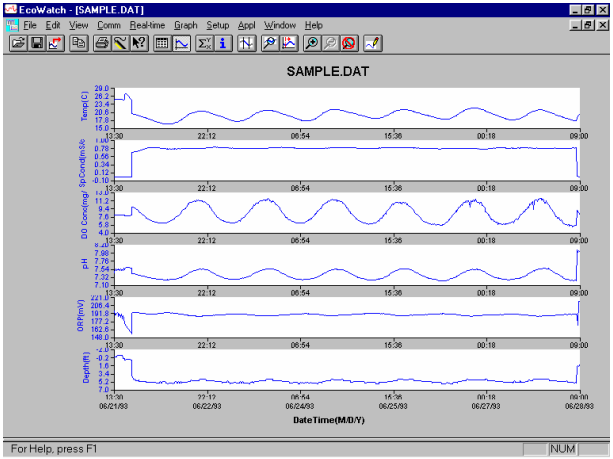
	DateTime	Temp	SpCond	DO Conc	pH	ORP	Depth
	M/D/Y	C	mS/cm	mg/L		mV	ft
0	06/21/93 13:30:45	25.00	0.007	8.04	7.44	197	-0.415
1	06/21/93 13:45:45	25.07	0.007	8.05	7.53	190	-0.415
2	06/21/93 14:00:45	25.07	0.007	8.05	7.54	190	-0.415
3	06/21/93 14:15:45	25.07	0.007	8.08	7.51	192	-0.415

Now click the **Graph** button (turn it off) to display only a report of your data as shown below. Note that the size of the report can be varied by clicking on the and buttons in the Toolbar.

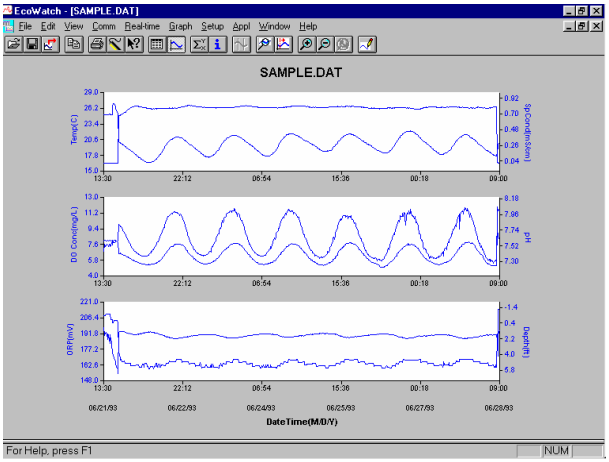


	DateTime	Temp	SpCond	DO Conc	pH	ORP	Depth
	M/D/Y	C	mS/cm	mg/L		mV	ft
0	06/21/93 13:30:45	25.00	0.007	8.04	7.44	197	-0.415
1	06/21/93 13:45:45	25.07	0.007	8.05	7.53	190	-0.415
2	06/21/93 14:00:45	25.07	0.007	8.05	7.54	190	-0.415
3	06/21/93 14:15:45	25.07	0.007	8.08	7.51	192	-0.415
4	06/21/93 14:30:45	25.07	0.008	8.03	7.53	193	-0.669
5	06/21/93 14:45:45	25.07	0.008	8.02	7.54	191	-0.669
6	06/21/93 15:00:45	25.07	0.008	8.05	7.53	187	-0.669
7	06/21/93 15:15:45	25.07	0.008	8.04	7.53	191	-0.669
8	06/21/93 15:30:45	25.07	0.008	8.03	7.51	190	-0.669
9	06/21/93 15:45:45	25.13	0.008	8.05	7.54	185	-0.669
10	06/21/93 16:00:45	25.13	0.008	8.04	7.51	191	-0.669
11	06/21/93 16:15:45	25.07	0.008	8.01	7.53	183	-0.669
12	06/21/93 16:30:45	25.00	0.008	8.07	7.52	188	0.000
13	06/21/93 16:45:45	25.00	0.008	8.04	7.57	182	0.000
14	06/21/93 17:00:45	25.07	0.010	8.05	7.54	174	0.000
15	06/21/93 17:15:45	26.50	0.010	7.88	7.56	174	0.323
16	06/21/93 17:30:45	27.00	0.010	7.82	7.58	172	0.369
17	06/21/93 17:45:45	27.07	0.010	7.80	7.60	169	0.069
18	06/21/93 18:00:45	26.81	0.010	7.84	7.60	167	0.115
19	06/21/93 18:15:45	26.50	0.010	7.87	7.60	165	0.115
20	06/21/93 18:30:45	26.19	0.010	7.92	7.59	164	0.115
21	06/21/93 18:45:45	25.80	0.010	7.95	7.59	161	0.115

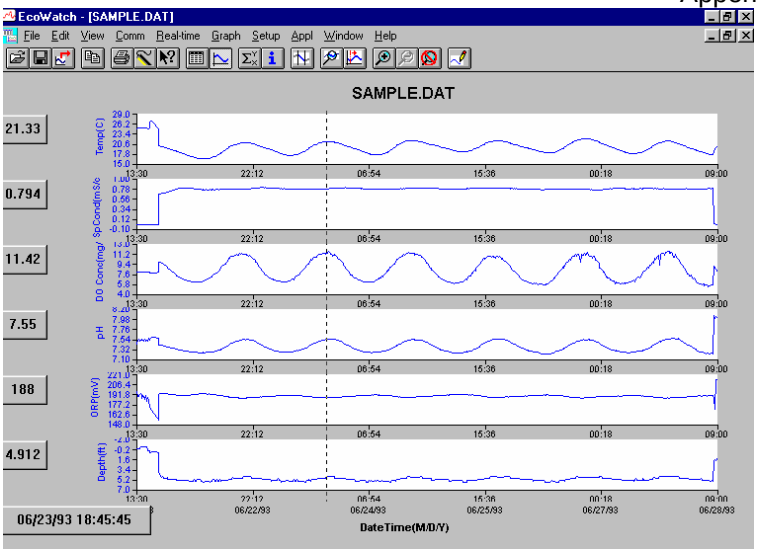
Now return to the original graphic display by toggling the **Table** button “off” and **Graph** button “on”.



From the **Setup** menu, click **Graph**. Click **2 Traces per Graph** and notice that the parameters are now graphed in pairs for easy comparison of parameters.

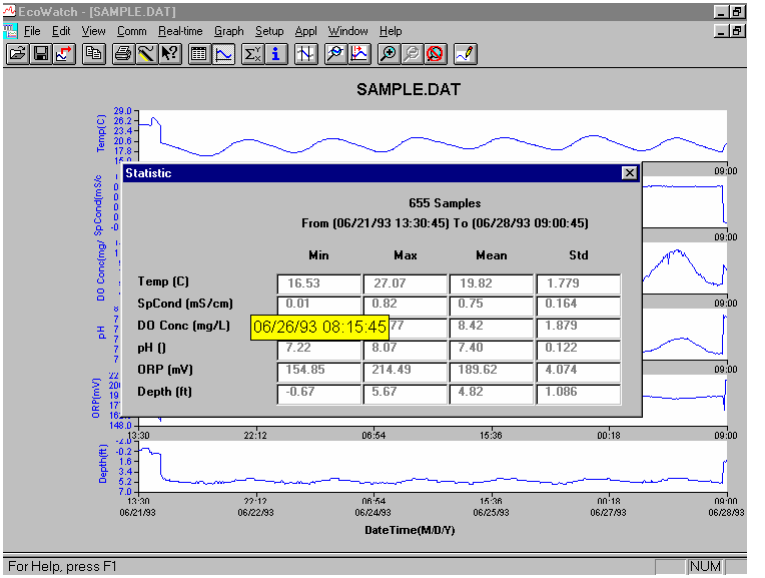


Click **1 Trace per Graph** to return the display to the original setting. Move the cursor to any position in the graph, then click and hold the right mouse button.



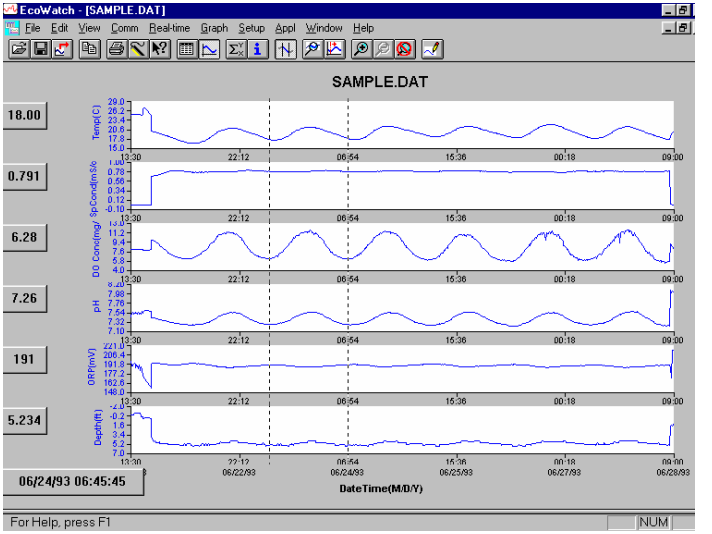
Note that the exact measurements for this point in time are displayed to the left of the graph. While holding down the right mouse button, move to another area on the graph. Notice how the measurements change as you move. When you release the mouse button, the display returns to normal.

To view statistical information for the study, click the **Statistics** button on the toolbar. On the statistics window, click on any min or max value to display the time when it occurred.

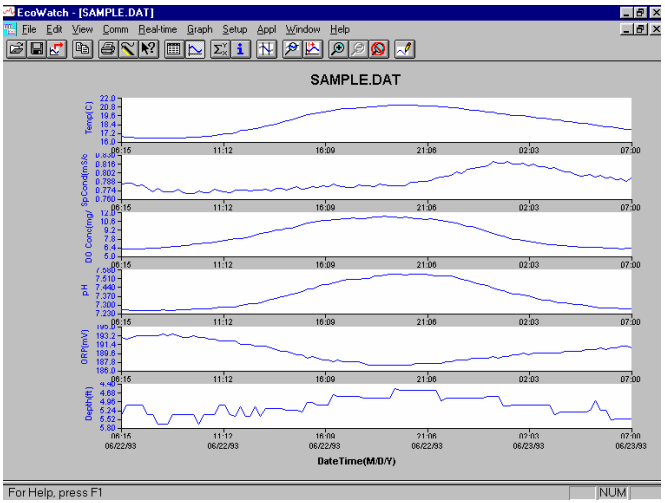


After viewing statistics, click the “x” at the upper right to close the window and return to the normal display.

Now click on the delimiter icon in the toolbar and then move the displayed icon to the graph. Click at the two points shown by dotted lines in the display below, being sure that the first click is to the left of the second.

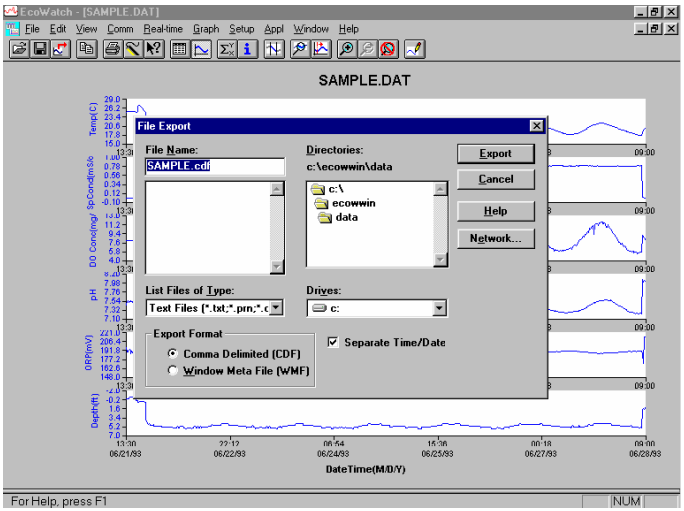


The data between the two selected points will then be graphed in higher resolution as shown below.

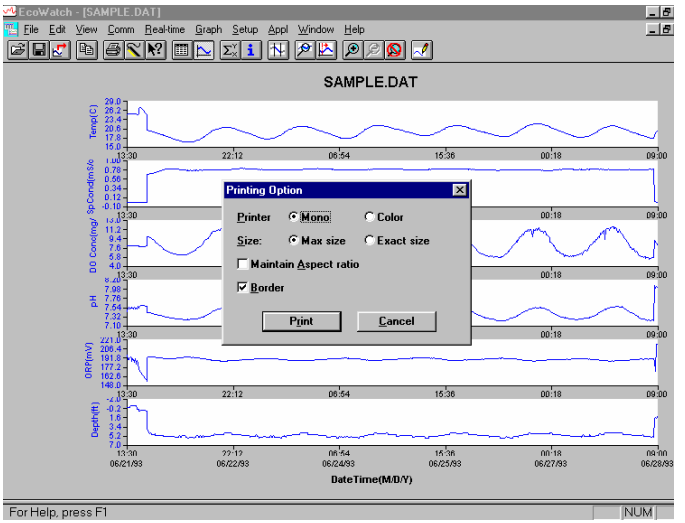


To return to the complete data set, select **Graph** from the toolbar and then click **Cancel Limits**.

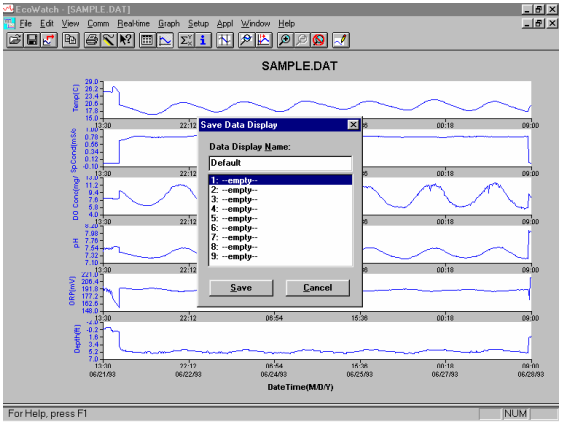
Now select the icon from the Toolbar to create a new data file which will allow your data to be imported into spreadsheets. Select the default export settings for a Comma Delimited File (.CDF) and click OK. A new spreadsheet-importable file (SAMPLE.CDF) is now present in the same folder as the SAMPLE.DAT file.



Now select the icon from the toolbar to print the plot. Accept the default settings and click OK to complete the printing operation.



Finally, end the tutorial by saving the **Data Display** in the format shown. From the File menu, click **Save Data Display**.



Then type “Default” for the file name and click **Save**. The parameters, colors, format, and x-axis time interval associated with the current display are now saved and can be accessed any time in the future. Nine different data displays may be saved for any data file. You can easily switch between various displays of the data. The data files can be accessed by clicking **Load Data Display** from the file menu and then selecting the desired presentation.

20.2.1 Summary of Toolbar Capability

The EcoWatch toolbar includes buttons for some of the most common commands in EcoWatch, such as **File Open**. To display or hide the toolbar,

open the **View** menu and click on the **Toolbar** command. A check mark appears next to the menu item when the toolbar is displayed.

The toolbar is displayed across the top of the application window, below the menu bar.

Click to:

Open an existing data file (.DAT). EcoWatch displays the **Open** dialog box, in which you can locate and open the desired file.

Save the working Data Display of the active data file. EcoWatch displays the **Save Data Display** dialog box in which you can overwrite existing Data Display or save to a new one.

Export data as a graph in Window Meta File (.WMF) format or as data in Comma Delimited (.CDF) format.

Copy the whole graph page or data from the selection on the table to the clipboard.

Print the active graph page or table page depending on which one is currently active.

Open a new terminal window to communicate with the sonde.

Access context sensitive help (Shift+F1).

Toggle table window during file processing.

Toggle graph window during file processing.

Display study statistics.

Display study info.

Limit the data to be processed.

Enlarge a selective portion of graph.

Center the graph under the cursor.

Enlarge graph of table 20%.

Reduce graph of table 20%.

Return graph or table to its normal size (unzoom).

Redraw the graph.

20.2.2 Other capabilities

The above tutorial and function list for the toolbar provide basic information to allow you to view and analyze the field data which was stored in your 556 MPS. Some of the other commonly used capabilities of EcoWatch which the user may want to explore are listed below:

- Customize the units for each parameter, e.g., report uS/cm instead of mS/cm for conductivity.
- Customize the order of parameters in each plot or report.
- Customize the colors and fonts of each data display.
- Manually scale the y-axis sensitivity for each parameter.
- Merging of two or more data files with compatible parameter formats
- View information about the study such as number of points, instrument serial number, etc. which was stored in the 556 with the data.
- Print data reports in different statistical formats.
- Create plots of parameter vs. parameter rather than parameter vs. time.

These additional features of EcoWatch for Windows are explained in detail in the YSI 6-series manual (which can be downloaded at no cost from the YSI Web Site as described above) and the Help selection in the EcoWatch menu bar. To purchase a hard copy of the 6-series manual, contact YSI Customer Service using the contact information in *Appendix E Customer Service*.

21. Appendix H Calibration Record Information

When your YSI 556 MPS sensors are initially calibrated, relevant information about the sensors will be stored in a separate file in the YSI 556 MPS memory.

NOTE: This file, by default, will have the name "556 Circuit Board Serial Number.glp." The circuit board serial number is assigned at the factory and has a hexadecimal format such as 000080A4. Thus the default calibration record file would be designated 00080A4.glp. Refer to Section *10.7 GLP Filename* to change the filename.

The information in the calibration record will track the sensor performance of your instrument and should be particularly useful for programs operating under Good Laboratory Practices (GLP) protocols.

21.1 Viewing the Calibration Record (.glp) File

NOTE: Make certain that you have performed a calibration on at least one of the sensors associated with your YSI 556 MPS.

Follow the procedures outlined in Section *8.3 View File*.

21.2 Uploading the Calibration Record (.glp) File

NOTE: Make certain that you have performed a calibration on at least one of the sensors associated with your YSI 556 MPS.

Follow the procedures outlined in Section *8.4 Upload to PC*.

21.3 Understanding the Calibration Record (.glp) File

1. Open a calibration record file. Refer to Section 8.3 View File.
2. Use the arrow keys to scroll horizontally and/or vertically to view all the data.

00008003.glp

m/d/y	hh:mm:ss	S/N
01/24/2001	08:17:51	00008003
01/24/2001	08:17:51	00008003
01/24/2001	08:17:51	00008003
01/24/2001	08:17:51	00008003
01/24/2001	08:17:51	00008003
01/24/2001	08:17:51	00008003
01/24/2001	08:17:51	00008003
01/24/2001	08:17:51	00008003
01/24/2001	08:25:40	00008003
01/24/2001	08:25:40	00008003

735.9mmHg

01/24/2001 08:39:53

Figure 21.1Calibration Record Screen 1

00008003.glp

Type	Value
Conductivity gain	1.000000
DO gain	1.000000
pH gain (pH-7)*K/mV	-5.05833
pH offest (pH-7)*K	0.000000
ORP offset mV	0.000000
TDS constant	0.650000
Barometer offset PSI	0.000000
DO gain	1.110250
pH gain (pH-7)*K/mV	-5.05833
pH offest (pH-7)*K	-12.2899

735.9mmHg

01/24/2001 08:39:19

Figure 21.2 Calibration Record Screen 2

NOTE: Each sensor (not parameter) is characterized by either 1 line (Conductivity, Dissolved Oxygen, ORP, TDS, or Barometer (Optional)) or 2 lines (pH) of calibration documentation.

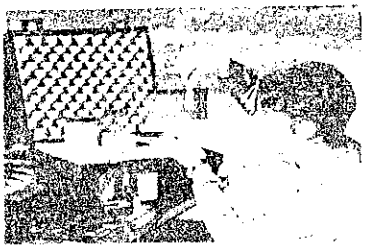
The left hand portion of each calibration entry shows the date and time that a calibration of a particular sensor was performed. In addition, each calibration entry is characterized by the instrument serial number, as defined by YSI. See Figure 21.1 Calibration Record Screen 1. The right hand portion shows the YSI designation of the calibration constants and their values after their calibration has been performed. A more detailed description of the calibration constants is provided below:

- **Conductivity Gain** – A relative number which describes the sensitivity of the sensor. Basically, the value represents the calculated cell constant divided by the typical value of the cell constant (5 cm^{-1}).
- **DO Gain** – A relative number which describes the sensitivity of the sensor. Basically, the value represents the sensor current at the time of calibration divided by the typical value of the sensor current (15 uA).
- **pH Gain** – A number which basically represents the sensitivity of the pH sensor. To remove the effect of temperature on the slope of the relationship of probe output in mv versus pH, the value of pH/mv is multiplied by the temperature in degrees Kelvin (K).
- **pH Offset** – A number which basically represents the offset (or intercept) of the relationship of probe output in mv versus pH, the value of pH is multiplied by the temperature in degrees Kelvin (K).

Anytime you perform a calibration, information concerning the calibration constants will be logged to the Calibration Record file (.glp file). However, if the **Delete All Files** command is used, Refer to Section *8.6 Delete All Files*, the Calibration Record file will also be lost. It is critical that this file should be uploaded to your PC prior to issuing a **Delete All Files** command. Refer to Section *8.4 Upload to PC.*

YSI Environmental
1700/1725 Brannum Lane
Yellow Springs, OH
45387 USA
937.767.7241
937.767.9353 fax
environmental@YSI.com
www.YSI.com

Item # 655279
Rev D
Drawing # A655279
August 2009
©2009 YSI Incorporated



HANBY
ENVIRONMENTAL
LABORATORY
PROCEDURES, INC. (H.E.L.P.)

Hanby Soil Test Kit Instructions

6817 HWY BOULEVARD
KATY, TX 77494
Mail to: PO Box 616, Katy, Tx 77492

281-391-4257
fax: 281-391-5002

email: jdhanby@msn.com
www.hanbytest.com
(U.S. EPA) www.epareachit.org

HANBY
ENVIRONMENTAL
LABORATORY
PROCEDURES, INC. (H.E.L.P.)

The Hanby Kit Method for Soils

Introduction:

The accurate determination of petroleum substances in soils, particularly when the contaminant is volatile, requires that analysis be performed as rapidly and efficiently as possible. Hanby Kits are well-designed to meet this first important objective. Please familiarize yourself with the Field Test Kit and its procedures. Watch the video instructions, observe all cautions concerning the chemical reagents, and set up your kit on a stable surface in an open, well ventilated area.

CAUTION: WEAR SAFETY GLASSES AND GLOES, AVOID FUMES. KEEP WORK AREA CLEAN AND WELL VENTILATED. SAFETY FIRST!

1. Place one of the beakers on the balance. Turn the balance on. It will automatically zero.
2. Add 5 grams of the soil sample to the beaker.
3. Remove one of the solvent ampoules from the foam block, hold it firmly on a flat surface, and snap off the top. **Avoid the sharp glass edges!** Pour the ampoule into the beaker. **Avoid Solvent Fumes!**
4. Chop the sample with the spatula and stir it in the solvent for one minute. If the soil is a clay it will be necessary to 'smear' the soil under the solvent with the spatula to insure good contact between the sample and the solvent.
5. Remove one of the capped test tubes from the foam, remove the cap, and carefully pour solvent from the beaker up to the mark on the test tube. **Avoid Solvent Fumes!**
6. Take one of the Color Development Vials out of the plastic jar, remove the cap, and carefully empty all the white powder into the test tube. **Avoid contacting the powder with skin, eyes and water!**
7. Firmly screw the test tube cap on and vigorously shake the tube for 15 seconds. Over the next three minutes, periodically shake the tube for about five seconds.
8. Compare the color of the solid material in the bottom of the tube to the appropriate calibration photograph in the photo booklet, e.g., Gasoline in Soil, Diesel in Soil, etc.
9. Dilutions of the extract remaining in the beaker can be prepared if the color is too dark. (Use the 10 mL Graduated Cylinder and another solvent ampoule to prepare dilutions.)

6817 HWY BOULEVARD
KATY, TX 77494
Mail to: PO Box 616, Katy, Tx 77492

281-391-4257
fax: 281-391-5002

email: jdhanby@msn.com
www.hanbytest.com
(U.S. EPA) www.epareachit.org

WASTE DISPOSAL

10. **IMPORTANT!** As soon as the analysis is completed and the concentration is recorded for the sample, carefully pour the excess solvent from the beaker and from the test tube into the waste bottle. It is very important not to leave the solvent in the test tubes for more than about thirty minutes. **Acid fumes will build up in the used test tubes! Put the used test tubes back in the foam block for later disposal! Do not Reopen!**

* H.E.L.P., Inc. will prepare special calibration photos for your specific needs. We also have a library of water and soil calibration photos, which can be ordered.

If necessary, H.E.L.P., Inc. will accept the waste solvent and test tubes (only) by ground shipment. Be certain that only one set (15 test tubes in the foam block and one waste bottle in its return carton) is contained in each return package. Follow appropriate packaging and shipping instructions for waste return (49CFR 173.4).

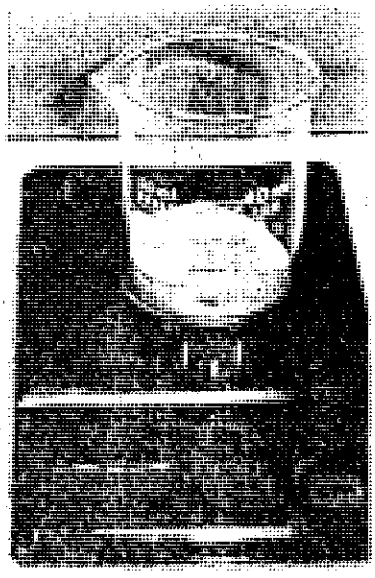
6817 HWY BOULEVARD
KATY, TX 77494
Mail to: PO Box 616, Katy, Tx 77492

281-391-4257
fax: 281-391-5002

email: jdhanby@msn.com
www.hanbytest.com
(U.S. EPA) www.epareachit.org

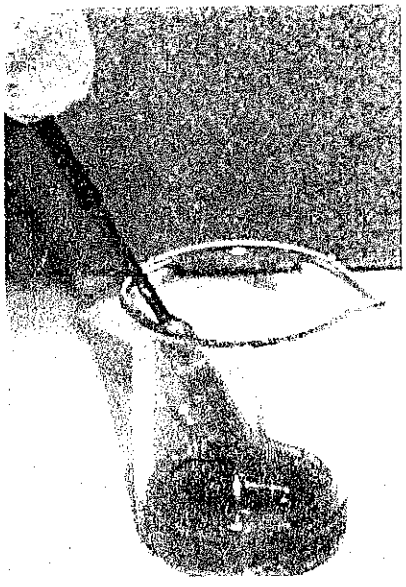
Six easy steps to Screening TPH's in Soil

Rapid, sensitive, positive detection of petroleum in solid samples



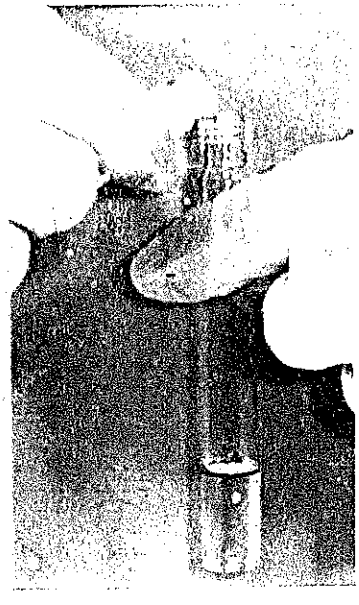
1. Weigh sample into tared beaker.

2. Snap ampoule, add solvent.

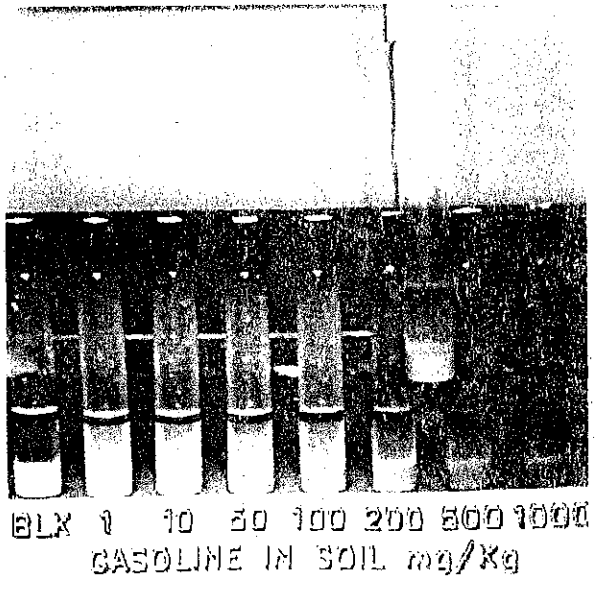


3. Stir, mix well.

4. Pour solvent into test tube.



5. Add catalyst, cap, shake.



6. Compare with Standard Photo.

MiniRAE Lite
User's Guide

Rev. A
December 2007
P/N 059-4022-000

© Copyright 2007 RAE Systems, Inc.

Contents

Read Before Operating 4
Special Notes 5
Warnings 6
Standard Contents 7
General Information 7
Physical Description 8
Specifications 8
Replacing Alkaline Batteries 11
Charging A Rechargeable Lithium Ion Battery 13
  Charging A Spare Rechargeable Lithium Ion Battery 14
  Low Voltage Warning 15
  Clock Battery 15
User Interface 16
Display 18
Operating The Instrument 19
Turning The Instrument On 19
Turning The Instrument Off 20
Operating The Built-In Flashlight 20
Pump Status 20
Calibration Status 21
Basic Operation 22
Alarm Signals 23
  Alarm Signal Summary 23
  Preset Alarm Limits & Calibration 24
Testing The Alarm 24
Integrated Sampling Pump 24
Backlight 24
Accessories 25
Standard Kit & Accessories 25
  Alkaline Battery Adapter 25
  External Filter 26
Optional Accessories 26
  Calibration Adapter 26
  Calibration Regulator 26

Organic Vapor Zeroing Kit 27
Travel Charger 27
Standard Two-Point Calibration (Zero & Span) 28
Entering Calibration 28
Zero (Fresh Air) Calibration 30
Span Calibration 32
Exiting Two-Point Calibration 34
Programming Mode 34
Entering Programming Mode 34
Programming Mode Menus 36
Zero Calibration 36
High Alarm 36
Date 36
Span Calibration 36
Low Alarm 36
Time 36
Alarm Type 36
Language 36
Buzzer & Light 36
Power On Zero 36
LCD Contrast 36
Exiting Programming Mode 37
Navigating Programming Mode Menus 37
Calibration 38
Zero Calibration 38
Span Calibration 38
Alarm Setting 39
High Alarm 40
Low Alarm 40
Alarm Type 41
Buzzer & Light 42
Date 42
Time 43
Language 43
Power On Zero 44
LCD Contrast 44
Diagnostic Mode 45
Entering Diagnostic Mode 45
Adjusting The Pump Stall Threshold 46

Pump High 46
Pump Low 46
Exiting Diagnostic Mode 47
Updating Firmware From A Computer 48
Maintenance 49
Battery Charging & Replacement 49
Replacing The Li-ion Battery Or Alkaline Adapter 50
PID Sensor & Lamp Cleaning/Replacement 51
Sensor Components 51
Cleaning The Lamp Housing Or Changing The Lamp 52
Sampling Pump 53
Cleaning The Instrument 53
Ordering Replacement Parts 53
Special Servicing Note 54
Troubleshooting 55
Technical Support 56
RAE Systems Contacts 57

Read Before Operating

This manual must be carefully read by all individuals who have or will have the responsibility of using, maintaining, or servicing this product. The product will perform as designed only if it is used, maintained, and serviced in accordance with the manufacturer's instructions. The user should understand how to set the correct parameters and interpret the obtained results.

CAUTION!

To reduce the risk of electric shock, turn the power off before removing the instrument cover. Disconnect the battery before removing sensor module for service. Never operate the instrument when the cover is removed. Remove instrument cover and sensor module only in an area known to be non-hazardous.

The instrument is NOT classified as to intrinsic safety for use in any hazardous area. This instrument has not been tested in an explosive gas/air atmosphere having and oxygen concentration greater than 21%. The instrument can only be used in non-hazardous locations.

Special Notes

When the instrument is taken out of the transport case and turned on for the first time, there may be some residual organic or inorganic vapor trapped inside the detector chamber. The initial PID sensor reading may indicate a few ppm. Enter an area known to be free of any organic vapor and turn on the instrument. After running for several minutes, the residual vapor in the detector chamber will be cleared and the reading should return to zero.

The battery of the instrument discharges slowly even if it is turned off. If the instrument has not been charged for 5 to 7 days, the battery voltage will be low. Therefore, it is a good practice to always charge the instrument before using it. It is also recommended to fully charge the instrument for *at least 10 hours* before first use. Refer to this User Guide's section on battery charging for more information on battery charging and replacement.

WARNINGS

STATIC HAZARD: Clean only with damp cloth.

For safety reasons, this equipment must be operated and serviced by qualified personnel only. Read and understand instruction manual completely before operating or servicing.

The calibration of all newly purchased RAE Systems instruments should be tested by exposing the sensor(s) to known concentration calibration gas before the instrument is put into service.

For maximum safety, the accuracy of the instrument should be checked by exposing it to a known concentration calibration gas before each day's use.

Do not use USB/PC communication in hazardous locations.

AVERTISSEMENT

DANGER RISQUE D'ORIGINE ELECTROSTATIQUE: Nettoyer uniquement avec un chiffon humide.

Pour des raisons de sécurité, cet équipment doit être utilisé, entretenu et réparé uniquement par un personnel qualifié. Étudier le manuel d'instructions en entier avant d'utiliser, d'entretenir ou de réparer l'équipement.

L'étalamage de tout instruments de RAE Systems doivent être testé en exposant l'instrument a une concentration de gaz connue par une procédure diétalonnage avant de mettre en service l'instrument pour la première fois.

Pour une securite maximale, la sensibilité du l'instrument doit être verifier en exposant l'instrument a une concentration de gaz connue par une procédure d'étalonnage avant chaque utilisation journalière.

Ne pas utiliser de connection USB/PC en zone dangereuse.

Standard Contents

Instrument
Calibration Kit
Travel Charger
AC/DC Adapter
Alkaline Battery Adapter
User's Guide

General Information

The compact instrument is designed as a broadband VOC gas monitor to work in non-hazardous environments. It monitors Volatile Organic Compounds (VOC) using a photoionization detector (PID) with a 10.6 eV gas-discharge lamp. Features are:

Lightweight and Compact

- Compact, lightweight, rugged design
- Built-in sample draw pump

Dependable and Accurate

- Up to 12 hours of continuous monitoring with rechargeable battery pack
- Designed to continuously monitor VOC vapor at parts-per-million (ppm) levels

User-friendly

- Preset alarm thresholds for low- and high-level peak values.
- Audio buzzer and flashing LED display are activated when the limits are exceeded.

The instrument consists of a PID with associated microcomputer and electronic circuit. The unit is housed in a rugged case with a backlit LCD and 3 keys to provide easy user interface. It also has a built-in flashlight for operational ease in dark locations.

Physical Description

The main components of the portable VOC monitoring instrument include:

- Three keys for user to interact with the instrument: 3 operation/programming keys for normal operation or programming
- LCD display with back light for direct readout and calculated measurements
- Built-in flashlight for illuminating testing points in dark environments
- Buzzer and red LEDs for alarm signaling whenever exposures exceed preset limits
- Charge contacts for plugging directly to its charging station
- Gas entry and exit ports
- USB communication port for PC interface
- Protective rubber cover

Specifications

Size:	9.25" L x 3.6" W x 2.9" H
Weight:	28 oz with battery pack
Detector:	Photoionization sensor with 10.6 eV UV lamp
Battery:	Rechargeable Lithium-Ion battery pack (snap in, field replaceable)
	Alkaline battery holder (for 4 AA batteries)
Battery Charging:	Less than 8 hours to full charge
Operating Hours:	Up to 12 hours continuous operation
Display:	Large dot matrix screen with backlight

Measurement range & resolution

Lamp	Range	Resolution
10.6 eV	0.1 ppm to 5,000 ppm	0.1 ppm

Response time (T_{90}):	2 seconds
Accuracy (Isobutylene):	10 to 2000 ppm: ±5% at calibration point.
PID Detector:	Easy access to lamp and sensor for cleaning and replacement
Calibration:	Two-point field calibration of zero and standard reference gases
Inlet Probe:	Flexible 5" tubing
Keypad:	1 operation key and 2 programming keys; 1 flashlight switch
Direct Readout:	Instantaneous, peak value, and battery voltage
EM Interference:	Highly resistant to EMI/RFI.
Alarm Setting:	Separate alarm limit settings for Low Alarm and High Alarm
Alarm:	Buzzer 95dB at 30cm and flashing red LEDs to indicate exceeded preset limits, low battery voltage, or sensor failure
Alarm Type:	Latching or automatic reset
Real-time Clock:	Automatic date and time
Communication:	Download instrument setup from PC via RS-232 with Travel Charger, or via USB with optional Charger Stand
Sampling Pump:	Internally integrated. Flow rate: 450 to 550 cc/min.
Temperature:	-20º C to 50º C (-4º to 122º F)

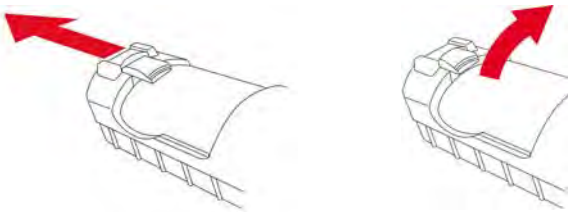
Humidity:	0% to 95% relative humidity (non-condensing)
Housing (including rubber boot):	Polycarbonate, splashproof and dustproof Battery can be changed without removing rubber boot.

Replacing Alkaline Batteries

An alkaline battery adapter is supplied with each instrument. The adapter (part number 059-3052-000) accepts four AA alkaline batteries (use only Duracell MN1500 or Energizer E91) and provides approximately 12 hours of operation. (An optional rechargeable lithium-ion battery pack, part number 059-3053-000, is also available.)

To install the adapter in the instrument:

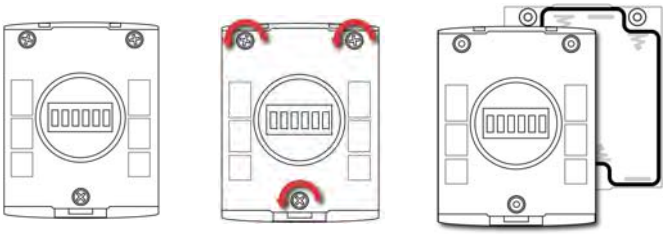
1. Remove the alkaline battery adapter from the instrument by sliding the tab and tilting out the adapter.



2. Replace the batteries (follow the procedure below).
3. Tilt the alkaline battery adapter and put it into the instrument.
4. Slide the tab back into place to secure the battery adapter.

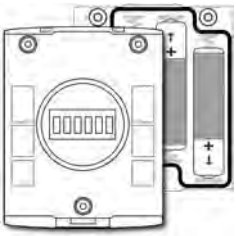
To insert batteries into the adapter:

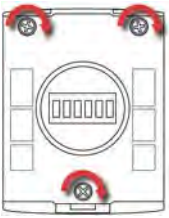
1. Remove the three Philips-head screws to open the compartment in the adapter.



2. Insert four fresh AA batteries as indicated by the polarity (+/-) markings.

3. Replace the cover. Replace the three screws.





IMPORTANT!

Alkaline batteries cannot be recharged. The instrument's internal circuit detects alkaline batteries and will not allow recharging. If you place the instrument in its Travel Charger or Charger Stand, the alkaline battery will not be recharged. The internal charging circuit is designed to prevent damage to alkaline batteries and the charging circuit when alkaline batteries are installed inside the instrument. If you try to charge an alkaline batteries installed in the instrument, the instrument's display will say, "Alkaline Battery," indicating that it will not charge the alkaline batteries.

Note: When replacing alkaline batteries, dispose of old ones properly.

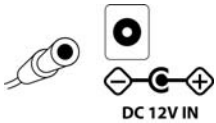
Charging A Lithium Ion Battery

Always fully charge the battery before using the instrument. The instrument's Li-ion battery is charged by attaching the instrument to the Travel Charger (or by placing the instrument in the optional Charger Stand). Contacts on the bottom of the instrument meet the Travel Charger's (or Charger Stand's) contacts, transferring power without other connections.

Note: Before connecting the mini charger to the instrument, visually inspect the contacts to make sure they are clean. If they are not, wipe them with a soft cloth. Do not use solvents or cleaners.

Follow this procedure to charge the instrument:

1. Plug the AC/DC adapter's barrel connector into the instrument's Charger Stand or Travel Charger.



2. Plug the AC/DC adapter into the wall outlet.
3. Connect the AC/DC adapter to the Travel Charger (or Charger Stand).
3. Place the instrument into the Travel Charger or Charger Stand. The LED in the Travel Charger (or Charger Stand) should glow.

The instrument begins charging automatically. (If the optional Charger Stand is used, the "Primary" LED blinks green to indicate charging.) During charging, the diagonal lines in the battery icon on the instrument's display are animated and you see the message "Charging..."

When the instrument's battery is fully charged, the battery icon is no longer animated and shows a full battery. The message "Fully charged!" is shown. (If the Charger Stand or Travel

Charger is used, its LED glows continuously green.)

Note: If you see the "Battery Charging Error" icon (a battery outline with an exclamation mark inside), check that the instrument or rechargeable battery has been properly set into the Travel Charger (or Charger Stand). If you still receive the message, check the Troubleshooting section of this guide.

Note: If the instrument or battery has been charging for more than 10 hours and you see the "Battery Charging Error" icon and a message that says, "Charging Too Long," this indicates that the battery is not reaching a full charge. Try changing the battery and make sure the contacts on the instrument are meeting the Travel Charger's (or Charger Stand's) contacts. If the message is still shown, consult your distributor or RAE Systems Technical Services.

Charging A Spare Rechargeable Battery (Optional Charger Stand Only)

A rechargeable Li-ion battery can be charged when it is not inside the monitor. The Charger Stand is designed to accommodate both types of charging. Contacts on the bottom of the battery meet the contacts on the Charger Stand, transferring power without other connections, and a spring-loaded capture holds the battery in place during charging.

1. Plug the AC/DC adapter into the Charger Stand.
2. Place the battery into the Charger Stand, with the gold-plated contacts on top of the six matching charging pins.
3. Plug the AC/DC adapter into the wall outlet.

The battery begins charging automatically. During charging, the Secondary LED in the Charger Stand blinks green. When charging is complete, it glows steady green.

Release the battery from the Charger Stand by pulling it back toward the rear of the Charger Stand and tilting it out of its slot.

Note: If you need to replace the Li-ion battery pack, replacements are available from RAE Systems. The part number is 059-3053-000.

Low Voltage Warning

When the battery's charge falls below a preset voltage, the instrument warns you by beeping once and flashing once every minute, and the "empty battery" icon blinks on and off once per second. You should turn off the instrument within 10 minutes and either recharge the battery by placing the instrument in its Travel Charger or Charger Stand, or replace the battery with a fresh one with a full charge.

Clock Battery

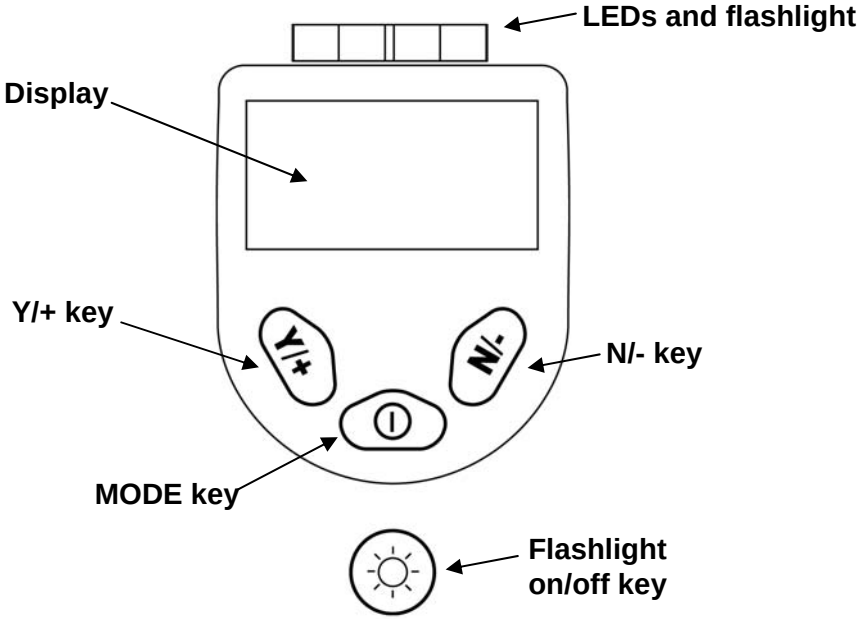
An internal clock battery is mounted on one of the instrument's printed circuit boards. This long-life battery keeps settings in memory from being lost whenever the Li-ion battery or alkaline batteries are removed. This backup battery should last approximately five years, and must be replaced by an authorized RAE Systems service technician. It is not user-replaceable.

User Interface

The instrument's user interface consists of the display, LEDs, an alarm transducer, and four keys. The keys are:

Y/+
MODE
N/-
Flashlight on/off

The LCD display provides visual feedback that includes the reading, time, battery condition, and other functions.

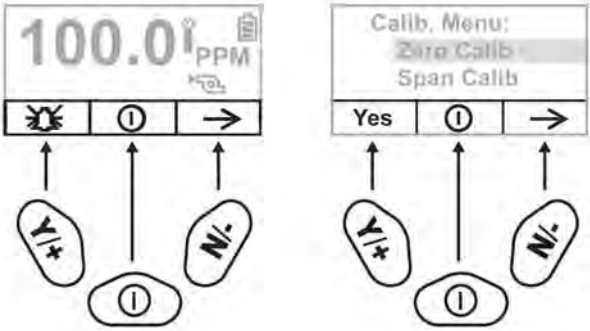


In addition to their labeled functions, the keys labeled Y/+, MODE, and N/- act as "soft keys" that control different parameters and make different selections within the instrument's menus. From menu to

menu, each key controls a different parameter or makes a different selection.

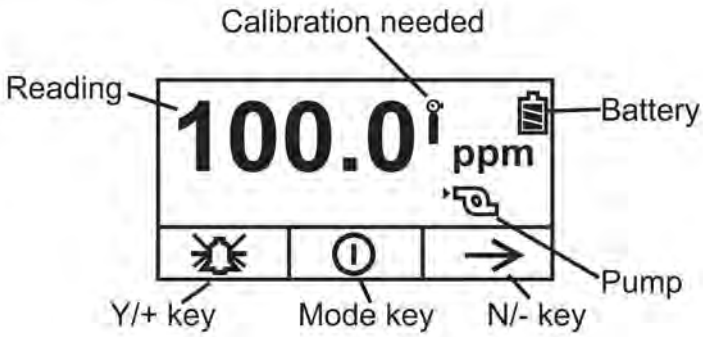
Three panes along the bottom of the display are "mapped" to the keys. These change as menus change, but at all times the left pane corresponds to the [Y/+] key, the center pane corresponds to the [MODE] key, and the right pane corresponds to the [N/-] key. Here are three examples of different menus with the relationships of the keys clearly shown:

RELATIONSHIP OF BUTTONS TO CONTROL FUNCTIONS



Display

The display shows the following information:



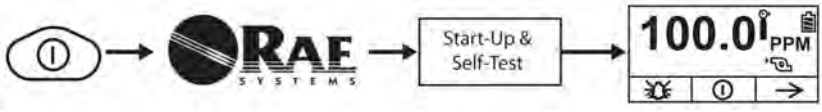
Reading	Concentration of gas as measured by the instrument
Calibration needed	Indicates that calibration should be performed
Battery	Indicates battery level in 3 bars
Pump	Indicates that pump is working
Y/+	Y/+ key's function for this screen
MODE	MODE key's function for this screen
N/-	N/- key's function for this screen

Operating The Instrument

The instrument is designed as a broadband VOC gas monitor for work in non-hazardous environments. It gives real-time measurements and activates alarm signals whenever the exposure exceeds preset limits. Prior to factory shipment, the instrument is preset with default alarm limits and the sensor is pre-calibrated with standard calibration gas. However, you should test the instrument and verify the calibration before the first use. After the instrument is fully charged and calibrated, it is ready for immediate operation.

Turning The Instrument On

1. With the instrument turned off, press and hold [MODE].
2. When the display turns on, release the [MODE] key.



The RAE Systems logo should appear first. (If the logo does not appear, there is likely a problem and you should contact your distributor or RAE Systems Technical Support.) The instrument is now operating and performs self tests. If any tests (including sensor and memory tests fail), refer to the Troubleshooting section of this guide.

Once the startup procedure is complete, the instrument shows a numerical reading screen with icons. This indicates that the instrument is fully functional and ready to use.

Turning The Instrument Off

1. Press and hold the Mode key for 3 seconds. A 5-second countdown to shutoff begins.
2. Once the countdown stops, the instrument is off. Release the Mode key.
3. When you see "Unit off..." release your finger from the [MODE] key. The instrument is now off.

Note: You must hold your finger on the key for the entire shutoff process. If you remove your finger from the key during the countdown, the shutoff operation is canceled and the instrument continues normal operation.

Operating The Built-In Flashlight

The instrument has a built-in flashlight that helps you point the probe in dark places. Press the flashlight key to turn it on. Press it again to turn it off.

Note: Using the flashlight for extended periods shortens the battery's operating time before it needs recharging.

Pump Status

IMPORTANT!

During operation, make sure the probe inlet and the gas outlet are free of obstructions. Obstructions can cause premature wear on the pump, false readings, or pump stalling. During normal operation, the pump icon alternately shows inflow and outflow as shown here:

During duty cycling (PID lamp cleaning), the display shows these icons in alternation:

If there is a pump failure or obstruction that disrupts the pump, you will see this icon blinking on and off:

If you see this blinking icon, consult the Troubleshooting section of this guide.

Calibration Status

The instrument displays this icon if it requires calibration:

Calibration is required (and indicated by this icon) if:

- The sensor has been replaced.
- It has been 30 days or more since the instrument was last calibrated.
- If you have changed the calibration gas type without recalibrating the instrument.

Basic Operation

The instrument is programmed to give you the most commonly needed information quickly.

Pressing [N/-] steps you from one screen to the next, and eventually return to the main display. If you do not press a key within 60 seconds after entering a display, the instrument reverts to its main display.

Note: While viewing any of these screens, you can shut off your instrument by pressing [MODE].

After the instrument is turned on, it runs through the start-up menu. Then the message "Please apply zero gas…" is displayed.

At this point, you can perform a zero air (fresh air) calibration. If the ambient air is clean, you can use that. Otherwise, use a cylinder of zero air. Refer to Zero Calibration on page 30 for a more detailed description of zero calibration.

Start zero calibration by pressing Start. You see the message "Zeroing…" followed by a 30-second countdown.

Note: You can press [MODE] to quit, bypassing the zero air calibration.

When zero calibration is complete, you see the message:

Zeroing is done!

Reading = 0.0 ppm

The instrument is now sampling and collecting data.

Note: At the Average & Peak, Date & Time & Temperature, and PC Communications screens, the instrument automatically goes to the main display after 60 seconds if you do not push a key to make a selection.

Alarm Signals

During each measurement period, the gas concentration is compared with the programmed alarm limits (gas concentration alarm limit settings). If the concentration exceeds any of the preset limits, the loud buzzer and red flashing LED are activated immediately to warn you of the alarm condition.

In addition, the instrument alarms if one of the following conditions occurs: battery voltage falls below a preset voltage level, failure of the UV lamp, or pump stall.

Alarm Signal Summary

Message	Condition	Alarm Signal
HIGH	Gas exceeds "High Alarm" limit	3 beeps/flashes per second
OVR	Gas exceeds measurement range	3 beeps/flashes per second
MAX	Gas exceeds electronics' maximum range	3 beeps/flashes per second
LOW	Gas exceeds "Low Alarm" limit	2 beeps/flashes per second
Pump icon flashes	Pump failure	3 beeps/flashes per second
Lamp	PID lamp failure	3 beeps/flashes per second plus "Lamp" message on display
Battery icon flashes	Low battery	1 flash, 1 beep per minute plus battery icon flashes on display
CAL	Calibration failed, or needs calibration	1 beep/flash per second
NEG	Gas reading measures less than number stored in calibration	1 beep/flash per second

Preset Alarm Limits & Calibration

The instrument is factory calibrated with standard calibration gas, and is programmed with default alarm limits.

Cal Gas (Isobutylene)	Cal Span	unit	Low	High
MiniRAE Lite	100	ppm	50	100

Testing The Alarm

You can test the alarm whenever the main (Reading) display is shown. Press [Y/+], and the audible and visible alarms are tested.

Integrated Sampling Pump

The instrument includes an integrated sampling pump. This diaphragm-type pump that provides a 450 to 550 cc per minute flow rate. Connecting a Teflon or metal tubing with 1/8" inside diameter to the gas inlet port of the instrument, this pump can pull in air samples from 200' (61 m) away horizontally, or 90' (27.5 m) vertically, at about 3' (0.9 m) per second flow speed.

Note: In Search Mode, the pump turns on when a sample measurement is started, and turns off when the sample is manually stopped.

If liquid or other objects are pulled into the inlet port filter, the instrument detects the obstruction and immediately shuts down the pump. The alarm is activated and a flashing pump icon is displayed.

You should acknowledge the pump shutoff condition by clearing the obstruction and pressing the [Y/+] key while in the main reading display to restart the pump.

Backlight

The LCD display is equipped with an LED backlight to assist in reading the display under poor lighting conditions.

Standard Accessories

The following accessories are included with the instrument:

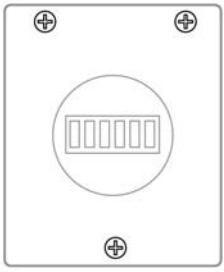
• 10.6 eV lamp
• Flex-I-Probe
• External filter
• Green rubber boot
• Alkaline battery adapter
• Lamp cleaning kit
• Tool Kit
• Lithium-Ion (Li-Ion) battery, if specified
• Travel Charger, if specified
• Universal wall adapter, if specified
• Operation & Maintenance manual
• Soft leather carrying case

Standard Kit & Accessories

Alkaline Battery Adapter

An alkaline battery adapter is supplied with each instrument. The adapter (part number 059-3052-000) accepts four AA alkaline batteries (use only Duracell MN1500 or Energizer E91) and provides approximately 12 hours of operation. (An optional rechargeable lithium-ion battery pack is also available.)

To insert batteries into the adapter:



4. Remove the three Philips-head screws to open the compartment in the adapter.
5. Insert four fresh AA batteries as indicated by the polarity (+/-) markings.
6. Replace the cover. Replace the three screws.

To install the adapter in the instrument:

5. Remove the Li-ion battery pack from the instrument by sliding the tab and tilting out the battery.

6. Replace it with the alkaline battery adapter
7. Slide the tab back into place to secure the battery adapter.

IMPORTANT!

Alkaline batteries cannot be recharged. The instrument's internal circuit detects alkaline batteries and will not allow recharging. If you place the instrument in its Travel Charger or Charger Stand, the alkaline battery will not be recharged. The internal charging circuit is designed to prevent damage to alkaline batteries and the charging circuit when alkaline batteries are installed inside the instrument. If you try to charge an alkaline batteries installed in the instrument, the instrument's display will say, "Alkaline Battery," indicating that it will not charge the alkaline batteries.

Note: When replacing alkaline batteries, dispose of old ones properly.

External Filter

The external filter is made of PTFE (Teflon®) membrane with a 0.45 micron pore size to prevent dust or other particles from being sucked into the sensor manifold, which would cause extensive damage to the instrument. It prolongs the operating life of the sensor. To install the external filter, simply connect it to the instrument's inlet tube.

Optional Accessories

Calibration Adapter

The calibration adapter for the instrument is a simple 6-inch Tygon tubing with a metal adapter on one end. During calibration, simply insert the metal adapter into the regular gas inlet probe of the instrument and the tubing to the gas regulator on the gas bottle.

Calibration Regulator

The Calibration Regulator is used in the calibration process. It regulates the gas flow rate from the Span gas cylinder into the gas inlet of the instrument during calibration process. The maximum flow rate allowed by the flow controller is about 0.5L/min (500 cc per

min.). Alternatively, a demand-flow regulator or a Tedlar gas bag may be used to match the pump flow precisely.

Organic Vapor Zeroing Kit

The Organic Vapor Zeroing Kit is used for filtering organic air contaminants that may affect the zero calibration reading. To use the Organic Vapor Zeroing Kit, simply connect the filter to the inlet port of the instrument.

Travel Charger

WARNING

A battery charging circuit is built into the mini charger. It only needs a regular AC to 12 VDC adapter (wall-mount transformer, part number 500-0114-000) to charge the instrument.

To charge the battery:

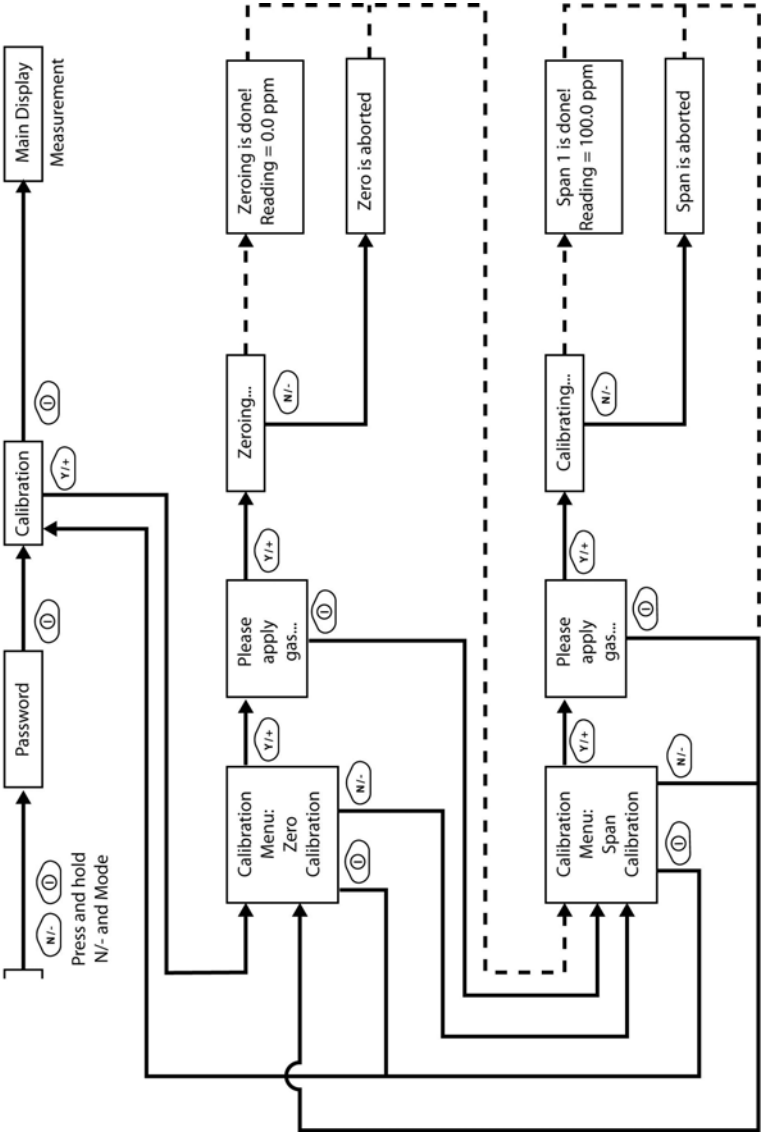
1. Power off the instrument.
2. Connect the AC adapter to the DC jack on the instrument's Travel Charger or Charger Stand. If the instrument is off, it automatically turns on.
3. While charging, the display message shows "Charging."
4. When the battery is fully charged, the message "Fully charged" appears on the display.

A completely discharged instrument can be charged to full capacity within 8 hours. Batteries drain slowly even if an instrument is off. Therefore, if the instrument has been in storage or has not been charged for several days or longer, check the charge before using it.

The factory-supplied battery is designed to last for 12 hours of normal operation (no alarm), for a new battery under the optimum circumstances. As the battery becomes older or is subject to adverse conditions (such as cold ambient temperature), its capacity will be significantly reduced.

Standard Two-Point Calibration (Zero & Span)

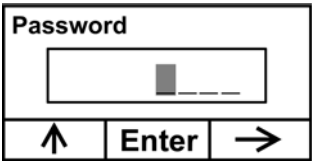
The following diagram shows the instrument's calibrations.



Note: Dashed line indicates automatic progression.

Entering Calibration

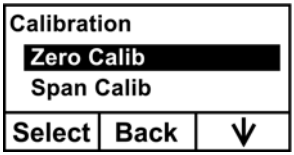
1. Press and hold [MODE] and [N/-] until you see the Password screen.



2. You do not need a password to perform calibrations. Instead of inputting a password, enter calibration by pressing [MODE].

   Note: If you inadvertently press [Y/+] and change any of the numbers, simply press [MODE] and you will be directed to the calibration menu.

The Calibration screen is now visible with Zero Calibration highlighted.



These are your options:

- Press [Y/+] to select the highlighted calibration (Zero Calib or Span Calib).
- Press [MODE] to exit calibration and return to the main display and resume measurement.
- Press [N/-] to toggle the highlighted calibration type.

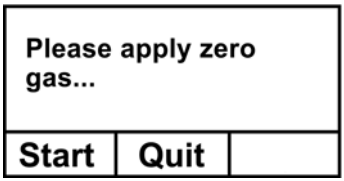
Zero (Fresh Air) Calibration

This procedure determines the zero point of the sensor calibration curve. To perform a fresh air calibration, use the calibration adapter to connect the instrument to a "fresh" air source such as from a cylinder or Tedlar bag (optional accessory). The "fresh" air is clean, dry air without organic impurities and an oxygen value of 20.9%. If such an air cylinder is not available, any clean ambient air without detectable contaminants or a charcoal filter can be used.

At the Zero Calibration menu, you can proceed to perform a Zero calibration or bypass Zero calibration and perform a Span calibration. You may also go back to the initial Calibration menu if you want to exit calibration.

- Press [Y/+] to start calibration.
- Press [MODE] to quit and return to the main calibration display.

If you have pressed [Y/+] to enter Zero calibration, then you will see this message:



1. Turn on your Zero calibration gas.
2. Press [Y/+] to start calibration.

   Note: At this point, you may press [MODE] if you decide that you do not want to initiate calibration. This will take you directly to the Calibration menu, highlighted for Span calibration.

3. Zero calibration starts a 30-second countdown and displays this message:

   Zeroing...

During the zeroing process, the instrument performs the Zero calibration automatically and does not require any action on your part.

Note: To abort the zeroing process at any time and proceed to Span calibration, press [N/-] at any time while zeroing is being performed. You will see a confirmation message that says "Zero aborted!" and then the Span calibration menu appears.

When Zero calibration is complete, you see this message:

Zeroing is done!
Reading = 0.0 ppm

The instrument will then show the Calibration menu on its display, with Span Calib highlighted.

Span Calibration

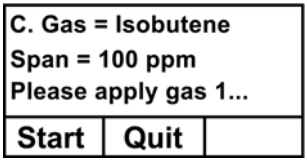
This procedure determines the second point of the sensor calibration curve for the sensor. A cylinder of standard reference gas (span gas) fitted with a 500 cc/min. flow-limiting regulator or a flow-matching regulator is the simplest way to perform this procedure. Choose the 500 cc/min. regulator only if the flow rate matches or slightly exceeds the flow rate of the instrument pump. Alternatively, the span gas can first be filled into a Tedlar bag or delivered through a demand-flow regulator. Connect the calibration adapter to the inlet port of the instrument, and connect the tubing to the regulator or Tedlar bag.

Another alternative is to use a regulator with >500 cc/min flow but allow the excess flow to escape through a T or an open tube. In the latter method, the span gas flows out through an open tube slightly wider than the probe, and the probe is inserted into the calibration tube.

At the Span Calibration menu, you perform a Span calibration. You may also go back to the Zero calibration menu or to the initial Calibration menu if you want to exit calibration.

- Press [Y/+] to enter Span calibration.
- Press [N/-] to skip Span calibration and return to Zero calibration.
- Press [MODE] to exit Span calibration and return to the top calibration menu.

If you have pressed [Y/+] to enter Span calibration, then you will see the name of your Span gas (the default is isobutylene) and the span value in parts per million (ppm). You will also see this message that prompts you:



1. Turn on your span calibration gas.

2. Press [Y/+] to initiate calibration.

   Note: You may press [MODE] if you decide that you do not want to initiate calibration. This will abort the span calibration and take you directly to the Calibration menu for Zero calibration.

3. Span calibration starts and displays this message:

   Calibrating...

During the Span calibration process, there is a 30-second countdown and the instrument performs the Span calibration automatically. It requires no actions on your part.

Note: If you want to abort the Span calibration process, press [N/-] at any time during the process. You will see a confirmation message that says "Span is aborted!" and then the Zero calibration menu appears. You can then proceed to perform a Zero calibration, perform a Span calibration, or exit to the topmost Calibration menu.

When Span calibration is complete, you see a message similar to this (the value is an example only):

Span 1 is done!
Reading = 100.0 ppm

The instrument then exits Span calibration and shows the Zero calibration menu on its display.

Note: The reading should be very close to the span gas value.

Exiting Two-Point Calibration

When you are done performing calibrations, press [MODE], which corresponds with "Back" on the display. You will see the following message:

Updating settings…

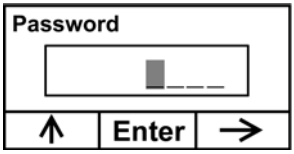
The instrument updates its settings and then returns to the main display. It begins or resumes monitoring.

Programming Mode

You must must provide a 4-digit password to enter Programming Mode.

Entering Programming Mode

1. Press and hold [MODE] and [N/-] until you see the Password screen.



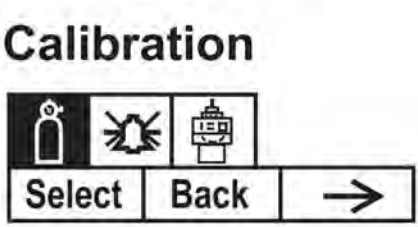
2. Input the 4-digit password:

- Increase the number from 0 through 9 by pressing [Y/+].
- Step from digit to digit using [N/-].
- Press [MODE] when you are done.

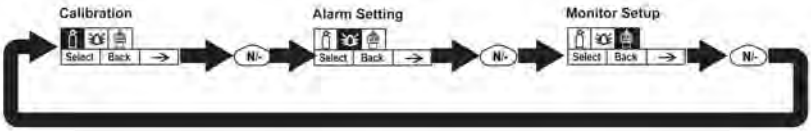
If you make a mistake, you can cycle through the digits by pressing [N/-] and then using [Y/+] to change the number in each position.

Note: The default password is 0000.

When you have successfully entered Programming Mode, you see this screen:



Note: The password can only be changed by connecting the instrument to a PC running ProRAE Studio software. Follow the instructions in ProRAE Studio to change it.The Calibration label is shown and its icon is highlighted, but you can press [N/-] to step from one programming menu to the next, with the name of the menu shown at the top of the display and the corresponding icon highlighted. As you repeatedly press [N/-], the selection moves from left to right, and you see these screens:



Note: When you reach Monitor Setup and press [N/-], the menu cycles back to Calibration.

Programming Mode Menus

The Programming Mode allows anyone with the password to change the instrument's settings, calibrate the instrument, enter user information, etc. Programming Mode has three menus. Each menu includes several sub-menus to perform additional programming functions.

This table shows the menus and sub-menus:

Calibration	**Alarm Setting**	**Monitor Setup**
Zero Calibration	High Alarm	Date
Span Calibration	Low Alarm	Time
	Alarm Type	Language
	Buzzer & Light	Power On Zero
		LCD Contrast

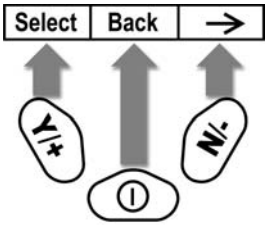
Once you enter Programming Mode, the LCD displays the first menu, Calibration. Each subsequent menu is accessed by pressing [N/-] repeatedly until the desired menu is displayed. To enter a sub-menu of a menu, press [Y/+].

Exiting Programming Mode

To exit Programming Mode and return to normal operation, press [MODE] once at any of the programming menu displays. You will see "Updating Settings…" as changes are registered and the mode changes.

Navigating Programming Mode Menus

Navigating through the Programming Mode menus is easy and consistent, using a single interface format of "Select," "Back" and "Next" at the top level. The three control buttons correspond to these choices as shown:



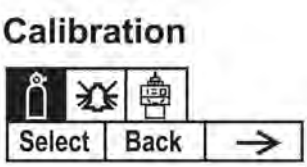
Note: Pressing [MODE] in the Programming Mode's top level causes the instrument to exit Programming Mode and return to monitoring.

The three keys perform the following functions in Programming Mode:

Key	Function in Programming Mode
[MODE]:	Exit menu when pressed momentarily or exit data entry mode
[Y/+]:	Increase alphanumerical value for data entry or confirm (yes) for a question
[N/-]:	Provides a "no" response to a question

Calibration

Two types of calibration are available: Zero (fresh air) and Span.



Select Zero or Span Calibration by pressing [N/+]. Once your choice is highlighted, press [Y/+].

Zero Calibration

The procedure for performing a zero calibration is covered on page 28.

Span Calibration

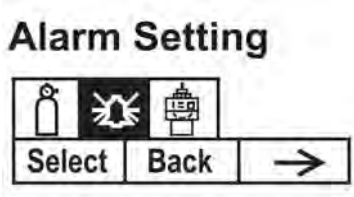
The procedure for performing a basic span calibration is covered on page 28.

Alarm Setting

During each measurement period, the gas concentration is compared with the programmed alarm limits (gas concentration alarm limit settings: Low and High). If the concentration exceeds any of the preset limits, the loud buzzer and red flashing LED are activated immediately to warn of the alarm condition.

An alarm signal summary is shown on page 23.

In this menu, you can change the High and Low alarm limits. Press [Y/+] to to enter the Alarm Setting menu. **Note:** All settings are shown in ppb (parts per billion), or µg/m^3 (micrograms per cubic meter), depending on your setting.



1. Scroll through the Alarm Limit sub-menu using the [N/-] key until the display shows the desired limit to be changed (High Alarm and Low Alarm).
2. Press [Y/+] to select one of the alarm types. The display shows a flashing cursor on the left-most digit of the previously stored alarm limit.
3. Press [Y/+] to increase each digit's value.
4. Press [N/-] to advance to the next digit.
5. Again, use [Y/+] to increase the number.

Repeat this process until all numbers are entered.

Press [MODE] when you are done.

- Press [Y/+] to save the changes.
- Press [N/-] to undo the changes and revert to the previous settings.

When all alarm types have been changed or bypassed, press [MODE] to exit to the Programming Menu.

High Alarm

You can change the High Alarm limit value. The value is typically set by the instrument to match the value for the current calibration gas. It is expressed in parts per billion (ppb). **Note:** The default value depends on the measurement gas.

To change the High Alarm value:

1. Press [Y/+] to increase each digit's value.
2. Press [N/-] to advance to the next digit.
3. Again, use [Y/+] to increase the number.

Repeat this process until all numbers are entered.
When you have completed your selections, press [MODE]. You will see two choices: Save and Undo. You have the opportunity to register the new settings or to change your mind and revert to your previous settings.

Press [Y/+] to save the changes.
Press [N/-] to undo the changes and revert to the previous settings.

Low Alarm

You can change the Low Alarm limit value. The value is typically set by the instrument to match the value for the current calibration gas. It is expressed in parts per billion (ppb). **Note:** The default value depends on the measurement gas.

To change the Low Alarm value:

1. Press [Y/+] to increase each digit's value.
2. Press [N/-] to advance to the next digit.
3. Again, use [Y/+] to increase the number.

Repeat this process until all numbers are entered.

When you have completed your selections, press [MODE]. You will see two choices: Save and Undo. You have the opportunity to register the new settings or to change your mind and revert to your previous settings.

- Press [Y/+] to save the changes.
- Press [N/-] to undo the changes and revert to the previous settings.

Alarm Type

There are two selectable alarm types:

Latched	When the alarm is triggered, you can manually stop the alarm. The latched setting only controls alarms for High Alarm and Low Alarm. **Note:** To clear an alarm when the instrument is set to "Latched," press [Y/+] when the main (Reading) display is shown.
Automatic Reset	When the alarm condition is no longer present, the alarm stops and resets itself.

1. Press [N/-] to step from one alarm type to the other.
2. Press [Y/+] to select an alarm type.

When you have completed your selections, press [MODE].

You will see two choices: Save and Undo. You have the opportunity to register the new settings or to change your mind and revert to your previous settings.

- Press [Y/+] to save the changes.
- Press [N/-] to undo the changes and revert to the previous settings.

Buzzer & Light

The buzzer and light alarms can be programmed to be on or off individually or in combination. Your choices are:

- Both on
- Light only
- Buzzer only
- Both off

1. Press [N/-] to step from one option to the next.
2. Press [Y/+] to make your selection (the dark circle in the "radio button" indicates your selection).
3. When you have completed your selections, press [MODE].

   You will see two choices: Save and Undo. You have the opportunity to register the new settings or to change your mind and revert to your previous settings.

   - Press [Y/+] to save the changes.
   - Press [N/-] to undo the changes and revert to the previous settings.

Date

The Date is expressed as Month/Day/Year, with two digits for each.

1. Press [Y/+] and the display shows the current date. Note that the left-most digit flashes to indicate it is selected.
2. Press [Y/+] to step through all 10 numerals (0 to 9).
3. Press [N/-] to advance to the next digit. The next digit to the right flashes.

Repeat this process until all six digits of the new date are entered.

Press [MODE] to exit.

- Press [Y/+] to save the new date.
- Press [N/-] to undo the change and move to the next sub-menu.

Time

The Time is expressed as Hours/Minutes/Seconds, with two digits for each. The time is in 24-hour (military) format.

1. Press [Y/+] and the display shows the current time. Note that the left-most digit flashes to indicate it is selected.
2. Press [Y/+] to step through all 10 numerals (0 to 9).
3. Press [N/-] to advance to the next digit. The next digit to the right flashes.

Repeat this process until all six digits of the new time are entered.

Press [MODE] to exit.

- Press [Y/+] to save the new date.
- Press [N/-] to undo the change and move to the next sub-menu.

Language

English is the default language, but other languages can be selected for the instrument.

1. Press [N/-] to step from one option to the next.
2. Press [Y/+] to make your selection (the dark circle in the "radio button" indicates "on").
3. When you have completed your selection, press [MODE].
   - Press [Y/+] to save your new language choice.
   - Press [N/-] to undo it and return to the previous language selection.

Power On Zero

When Power On Zero is on, the instrument performs a zero calibration when it is turned on.

1. Press [N/-] to step from one option to the next.
2. Press [Y/+] to make your selection (the dark circle in the "radio button" indicates your selection).
3. When you have completed your selection, press [MODE].
   - Press [Y/+] to save the change.
   - Press [N/-] to discard the change and move to the next sub-menu.

LCD Contrast

The display's contrast can be increased or decreased from its default setting. You may not need to ever change the default setting, but sometimes you can optimize the display to suit extreme temperature and ambient brightness/darkness conditions.

- The minimum value is 20.
- The maximum value is 60.

1. Press [Y/+] to increase the value or [N/-] to decrease the value.
2. Press [MODE] to save your selection.
   - Press [Y/+] to save your new contrast value.
   - Press [N/-] to undo it and return to the previous value.

Diagnostic Mode

IMPORTANT! Diagnostic Mode is designed for servicing and manufacturing, and therefore is not intended for everyday use, even by advanced users. It provides raw data from sensors and about settings, but only allows adjustment of pump stall parameters, which should only be changed by qualified personnel.

Note: If the instrument is turned on in Diagnostic Mode and you switch to User Mode, data remains in raw count form. To change to standard readings, you must restart the instrument.

Entering Diagnostic Mode

Note: To enter Diagnostic Mode, you must begin with the instrument turned off.

Press and hold [Y/+] and [MODE] until the instrument starts.

The instrument goes through a brief startup, and then displays raw data for the PID sensor. These numbers are raw sensor readings without calibration. The instrument is now in Diagnostic Mode.

Note: In Diagnostic Mode, the pump and lamp are normally on.

You can enter Programming Mode and calibrate the instrument as usual by pressing both [MODE] and [N/-] for three seconds.

You can enter Monitoring Mode by pressing [MODE] and [Y/+] together for three seconds.

Once the instrument is started up in Diagnostic Mode, you can switch between Diagnostic Mode and Monitoring Mode by pressing and holding [MODE] and [Y/+] simultaneously for two seconds.

In Diagnostic mode, you can step through parameter screens by pressing [MODE].

Adjusting The Pump Stall Threshold

If the gas inlet is blocked but the pump does not shut down, or the pump shuts down too easily with a slight blockage, the pump stall threshold value may be set too high or too low.

Use the following steps to adjust the pump stall threshold:

Pump High

In Diagnostic Mode, press the [MODE] key until "Pump High" is displayed. The pump shows the maximum, minimum, and stall values for the pump at its high speed.

Block the gas inlet and watch the pump current reading (labeled "I") increase. Write down the blocked reading. If the pump current reading does not increase significantly (for example, more than 10 counts), then there may be a leak in the gas inlet or the pump is weak or defective.

Use the [Y/+] or [N/-] key to increase or decrease the stall value until it is the average of the maximum block count and the maximum idle count.

Press the [MODE] key to exit this display.

Pump Low

In Diagnostic Mode, press the [MODE] key until "Pump Low" is displayed. The pump shows the maximum, minimum, and stall values for the pump at its low speed.

Block the gas inlet and watch the pump current reading (labeled "I") increase. Write down the blocked reading. If the pump current reading does not increase significantly (for example, more than 10 counts), then there may be a leak in the gas inlet or the pump is weak or defective.

Use the [Y/+] or [N/-] key to increase or decrease the stall value until it is the average of the maximum block count and the maximum idle count.

Press the [MODE] key to exit this display.

Exiting Diagnostic Mode

You can exit Diagnostic Mode and go directly to Programming Mode or Monitor Mode as outlined above, or you can exit Diagnostic Mode completely.

To exit Diagnostic Mode so that it cannot be re-entered without a restart:

Shut down the instrument. When it is off, restart it by holding the [MODE] key. Diagnostic Mode cannot be entered until the instrument is restarted as outlined in "Entering Diagnostic Mode."

Updating Firmware From A Computer

You can connect the Travel Charger or optional Charger Stand to a PC and transfer updated firmware to the instrument (should this ever be necessary).

Uploading Firmware To The instrument From A PC

Uploading new firmware to your instrument requires connecting the instrument and PC via the Travel Charger or optional Charger Stand. It also requires RAE Programmer 7000 software, which is available by downloading ProRAE Studio version 1.12 from the RAE Systems website (www.raesystems.com). Follow these steps to make the connection:

1. Connect the data cable to the PC and the charger/cradle.
2. Place the instrument into the charger/cradle. The charging LED should be illuminated.
3. Start RAE Programmer 7000 on your PC.
4. Select "Operation" and select Setup Connection.
5. Select the COM port to establish a communication link between the PC and the instrument.
6. Select Operation → Download Firmware.

Once communication is established, follow the software's instructions to upload the new firmware to your instrument.

Note: Check for the latest updates to ProRAE Studio at www.raesystems.com.

Maintenance

The major maintenance items of the instrument are:

- Battery pack
- Sensor module
- PID lamp
- Sampling pump
- Inlet connectors and filters

Note: Maintenance should be performed by qualified personnel only.

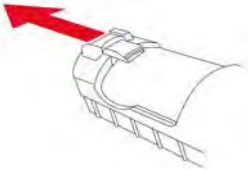
NOTE: The printed circuit board of the instrument is connected to the battery pack even if the power is turned off. **Therefore, it is very important to disconnect the battery pack before servicing or replacing any components inside the instrument**. **Severe damage to the printed circuit board or battery may occur if the battery pack is not disconnected before servicing the unit.**

Battery Charging & Replacement

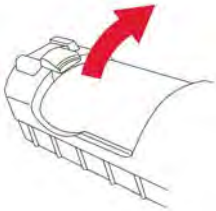
When the display shows a flashing empty battery icon, the battery requires replacement or recharging. It is recommended to do this upon returning from fieldwork. The charging time for a lithium ion battery is less than 8 hours if it is fully discharged. The Alkaline Battery Adapter or lithium ion battery may be replaced in the field (in areas known to be non-hazardous), if required.

Replacing The Li-ion Battery Or Alkaline Adapter

1. Turn off the instrument.
2. Located on the rear of the instrument is a battery tab. Slide it down to unlock the battery.



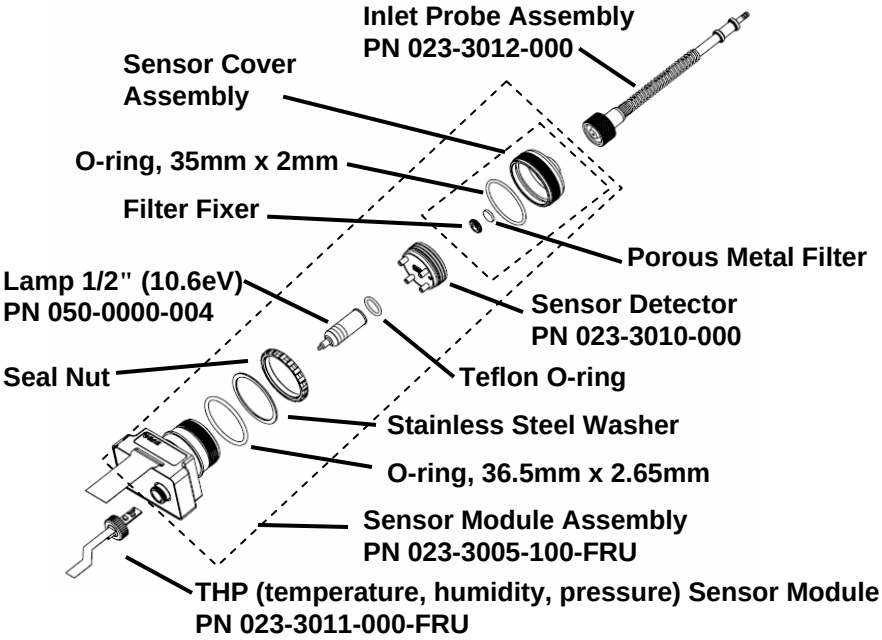
3. Remove the battery pack from the battery compartment by tilting it out.



4. Replace a fully charged spare battery pack inside the battery compartment. Make sure the battery pack is oriented properly inside the compartment.
5. Slide the capture tab back up to its locked position.

PID Sensor & Lamp Cleaning/Replacement

The sensor module is made of several components and is attached to the lamp-housing unit as shown below.



Sensor Components

Note: The cleaning procedure is not normally needed. Clean the PID sensor module, the lamp and the lamp housing only if:

1. The reading is inaccurate even after calibration.
2. The reading is very sensitive to air moisture.
3. A liquid has been sucked into the unit and damaged the unit.

Use of the external filter helps to prevent contamination of the sensor.

To access the sensor components and lamp, gently unscrew the lamp-housing cap, remove the sensor adapter with the gas inlet probe and the metal filter all together. Then hold the PID sensor and pull it straight out. A slight, gentle rocking motion helps release the sensor.

Cleaning The PID Sensor

Place the entire PID sensor module into GC grade methanol. It is highly recommended that an ultrasound bath to be used to clean the sensor for at least 15 minutes. Then dry the sensor thoroughly. Never touch the electrodes of the sensor by hand.
Also use a methanol-soaked cotton swab to wipe off the lamp housing where it contacts the sensor when the sensor is installed.

Turn over the sensor so that the pins point up and the sensor cavity is visible. Examine the sensor electrodes for any corrosion, damage, or bending out of alignment. The metal sensor electrode "fingers" should be flat and straight. If necessary, carefully bend the sensor fingers to ensure that they do not touch the Teflon portions and that they are parallel to each other. Make sure that the nuts on the sensor pins are snug but not overtight. If the sensor is corroded or otherwise damaged, it should be replaced.

Cleaning The Lamp Housing Or Changing The Lamp

If the lamp does not turn on, the instrument will display an error message to indicate replacement of the lamp may be required.

1. If the lamp is operational, clean the lamp window surface and the lamp housing by wiping it with GC grade methanol using a cotton swab using moderate pressure. After cleaning, hold the lamp up to the light at an angle to detect any remaining film. Repeat the process until the lamp window is clean. Never use water solutions to clean the lamp. Dry the lamp and the lamp housing thoroughly after cleaning.

   CAUTION: Never touch the window surface with the fingers or anything else that may leave a film. Never use acetone or aqueous solutions.

2. If the lamp does not turn on, remove the lamp from the lamp housing. Place the lamp O-ring onto the new lamp. Insert the new lamp, avoiding contact with the flat window surface.
3. Reinstall the PID sensor module.
4. Tighten the Lamp Housing Cap.

Sampling Pump

When approaching the end of the specified lifetime of the pump, it will consume higher amount of energy and reduce its sample draw capability significantly. When this occurs, it is necessary to replace or rebuild the pump. When checking the pump flow, make sure that the inlet connector is tight and the inlet tubing is in good condition. Connect a flow meter to the gas inlet probe. The flow rate should be above 450 cc/min when there is no air leakage.

If the pump is not working properly, refer the instrument to qualified service personnel for further testing and, if necessary, pump repair or replacement.

Cleaning The Instrument

Occasional cleaning with a soft cloth is recommended. Do not use detergents or chemicals.

Visually inspect the contacts at the base of the instrument, on the battery, and on the Travel Charger or Charger Stand to make sure they are clean. If they are not, wipe them with a soft, dry cloth. Never use solvents or cleaners.

Ordering Replacement Parts

If you need replacement parts, contact your local RAE Systems distributor. A list is available online:

http://www.raesystems.com

In the U.S., you can order sensors, replacement batteries, and other accessories online at:

http://istore.raesystems.com/

Special Servicing Note

If the instrument needs to be serviced, contact either:

1. The RAE Systems distributor from whom the instrument was purchased; they will return the instrument on your behalf.

   or

2. The RAE Systems Technical Service Department. Before returning the instrument for service or repair, obtain a Returned Material Authorization (RMA) number for proper tracking of your equipment. This number needs to be on all documentation and posted on the outside of the box in which the instrument is returned for service or upgrade. Packages without RMA Numbers will be refused at the factory.

Troubleshooting

Problem	Possible Reasons & Solutions
Cannot turn on power after charging the battery	**Reasons:** Discharged battery. Defective battery. **Solutions:** Charge or replace battery.
Lost password	**Solutions:** Call Technical Support at +1 408-752-0723 or toll-free at +1 888-723-4800
Reading abnormally High	**Reasons:** Dirty filter. Dirty sensor module. Excessive moisture and water condensation. Incorrect calibration. **Solutions:** Replace filter. Blow-dry the sensor module Calibrate the unit.
Reading abnormally Low	**Reasons:** Dirty filter. Dirty sensor module. Weak or dirty lamp. Incorrect calibration. **Solutions:** Replace filter. Remove Calibration Adapter. Calibrate the unit. Check for air leakage.
Buzzer Inoperative	**Reasons:** Bad buzzer. **Solutions:** Check that buzzer is not turned off. Call authorized service center.

Inlet flow too low	**Reasons:**	Pump diaphragm damaged or has debris. Flow path leaks.
	Solutions:	Check flow path for leaks; sensor module O-ring, tube connectors, Teflon tube compression fitting. Call Technical Support at +1 408-752-0723 or toll-free at +1 888-723-4800.
"Lamp" message during operation	**Reasons:**	Lamp drive circuit. Weak or defective PID lamp, defective.
	Solutions:	Turn the unit off and back on. Replace UV lamp

Technical Support

To contact RAE Systems Technical Support Team:

Monday through Friday, 7:00AM to 5:00PM Pacific (US) Time
Phone (toll-free): +1 888-723-4800
Phone: +1 408-952-8461
Email: tech@raesystems.com

Life-critical after-hours support is available:

+1 408-952-8200 select option 9

RAE Systems Contacts

RAE Systems
World Headquarters
3775 N. First St.
San Jose, CA 95134-1708 USA
Phone: +1 408.952.8200
Fax: +1 408.952.8480

E-mail: customerserv@raesystems.com
Web Site: www.raesystems.com

RAE Systems Technical Support
Monday through Friday, 7:00AM to 5:00PM Pacific Time
Phone: +1.408.952.8461
Email: tech@raesystems.com

Life-critical after-hours support is available:
+1.408.952.8200 select option 9

RAE Systems Europe ApS
Kirstinehøj 23 A
DK-2770 Kastrup
Denmark
Phone: +45 86 52 51 55
Fax: +45 86 52 51 77
orders@raeeurope.com
sales@raeeurope.com
service@raesystems.com
Web: www.raesystems.dk

RAE Systems UK Ltd
D5 Culham Innovation Centre
Culham Science Centre
Abingdon, Oxon OX14 3DB
United Kingdom
Phone: +44 1865408368
Fax: +44 1235531119
Mobile: +44 7841362693
Email: raeuk@raeeurope.com

RAE Systems France
336, rue de la fée des eaux
69390 Vernaison
France
Phone: +33 4 78 46 16 65
Fax: +33 4 78 46 25 98
Email: info-france@raeeurope.com
Web: www.raesystems.fr

RAE BeNeLux BV
Rijndal 20
2904 DC Capelle a/d IJssel
Phone: +31 10 4426149
Fax: +31 10 4426148
Email: info@rae.nl
Web: www.rae.nl

RAE Systems Spain, s.l.
Av. Remolar, 31
08820 El Prat de Llobregat
Spain
Phone: +34 933 788 352
Fax: +34 933 788 353
Mobile: +34 687 491 106
Email: mdelgado@raespain.com
Web: www.raespain.com

RAE Systems Middle East
Mobile: +45 2674 9791
or +97 50 429 1385
Email: mjorgensen@raesystems.com

RAE Systems (Hong Kong) Ltd.
Room 8, 6/F, Hong Leong Plaza
33 Lok Yip Road
Fanling, N.T, Hong Kong
Phone: +852.2669.0828
Fax: +852.2669.0803
Email: hksales@raesystems.com

RAE Systems Japan
403 Plaza Ochanomizu Bldg. 2-1
Surugadai Kanda Chiyoda-Ku
Tokyo, Japan
Phone: 81-3-5283-3268
Fax: 81-3-5283-3275
Email: jpsales@raesystems.com

RAE Systems Korea
#1010, DaeMyungAnsVill First,
Sang-Dong 412-2, Wonmi-Gu, Bucheon,
Kyungki-Do, Korea
Phone: 82-32-328-7123
Fax: 82-32-328-7127
Email: krsales@raesystems.com

RAE Systems
World Headquarters
3775 N. First St.
San Jose, CA 95134-1708 USA
Phone: 408.952.8200
Fax: 408.952.8480

E-mail: customerserv@raesystems.com
Web Site: www.raesystems.com

Rev. A
December 2007
P/N 059-4022-000

QRAE II User's Guide

P/N 020-4100-000 Rev. A
October 2006

© Copyright 2006 RAE Systems, Inc.

Contents

Read Before Operating 3
General Information 6
Specifications 7
Charging The QRAE II Battery 8
Clock Battery 9
User Interface 10
User Interface Icons 10
Turning The QRAE II On 11
Inverting The Display 12
Testing The Alarms (Anytime) 12
Turning The QRAE II Off 12
Operating The QRAE II 13
Mode Overview 13
Normal Mode 14
Diagnostic Mode 15
Programming Mode 18
Enter Programming Mode 19
Navigating Programming Mode 20
Calibrate Monitor 21
Change Alarm Limits 23
Change Datalog 26
Change Monitor Setup 29
Change Sensor Configuration 34
Alarm Signal Summary 37
Calibrating The QRAE II 38
Connecting The Calibration Adapter 38
Disconnecting The Calibration Adapter 39
Zero (Fresh Air) Calibration 40
Multiple Sensor Calibration 41
Single Sensor Calibration 42
Connecting To A Computer 44
Transferring Data To & From A Computer 44
Downloading The Datalog To A PC 44
Uploading Firmware To The QRAE II From A PC 45
Maintenance 46
Replacing the QRAE II Li-Ion Battery 46
Installing The Alkaline Battery Adapter 47
Charging A Spare QRAE II Li-Ion Battery 48
Replacing Sensors 49
Replacing The Filter 50
Cleaning 51
Firmware Updates 51
Ordering Replacement Parts 51
Troubleshooting 52

Technical Support 53
RAE Systems Contacts 53
Appendix A: Regulatory Information.....66

WARNINGS

Read Before Operating

This manual must be carefully read by all individuals who have or will have the responsibility of using, maintaining, or servicing this product. The product will perform as designed only if it is used, maintained, and serviced in accordance with the manufacturer's instructions.

CAUTION!

To reduce the risk of electric shock, turn the power off before removing the monitor cover. Disconnect the battery before removing sensor module for service. Never operate the monitor when the cover is removed. Remove monitor cover and sensor module only in an area known to be non-hazardous.

Note: Users are recommended to refer to ISA -RP12.13, Part II-1987 for general information on installation, operation, and maintenance of combustible gas detection instruments.

⚠ WARNINGS ⚠

Use only RAE Systems rechargeable battery pack part number 020-3402-000, or alkaline battery pack part number 020-3403-000. Use only DURACELL MN1500 or ENERGIZER E91 batteries with alkaline battery pack. This instrument has not been tested in an explosive gas/air atmosphere having an oxygen concentration greater than 21%. Substitution of components may impair suitability for intrinsic safety. Recharge batteries only in non-hazardous locations. Do not connect the serial communication port in a hazardous location.

STATIC HAZARD: Clean only with a damp cloth.

For safety reasons this equipment must be operated and serviced by qualified personnel only. Read and understand instruction manual completely before operating or servicing.

Only the combustible gas detection portion of this instrument has been assessed for performance.

All newly purchased RAE Systems instruments should be bump tested by exposing the sensor(s) to known concentrations of calibration gas before the instrument is put into service, and later, prior to each use. A bump test is defined as a brief exposure of the monitor to the calibration gas and the sensors to show response and trigger the lowest alarm set point for each sensor.

The QRAEII Multi Gas detector must be calibrated if it does not pass a Bump Test, or at least once every 180 days, depending on use and sensor exposure to poisons and contaminants.

- Calibration intervals and bump test procedures may vary due to national legislation.
- RAE Systems recommends using RAE calibration gas cylinders with a 4 gas mix containing 10 ppm H2S, 50 ppm CO, 50% LEL Methane, and 18.5% Oxygen.
- Any rapid up-scale reading followed by a declining or erratic reading may indicate a gas concentration beyond upper scale limit which may be hazardous.

Note: Bump test and calibration can also be performed by using the RAE Systems AutoRAE™ docking station.

.

⚠ AVERTISSEMENT ⚠

Utiliser seulement l'ensemble de batterie RAE Systems, la référence 020-3403-000. Utiliser uniquement des piles alcalines modele DURACELL MN1500 ou ENERGIZER E91 avec l'adaptateur piles alcalines Cet instrument n'a pas été testé dans une atmosphère de gaz/air explosive ayant une concentration d'oxygène plus élevée que 21%. La substitution de composants peut compromettre la sécurité intrinsèque. Ne charger les batteries que dans un emplacement désigné non dangereux. Ne reliez pas le port de communication série dans un endroit dangereux.

RISQUE D'ORIGINE ELECTROSTATIQUE: Nettoyer uniquement avec un chiffon humide.

Pour des raisons de sécurité, cet équipement doit être utilisé, entretenu et réparé uniquement par un personnel qualifié. Étudier le manuel d'instructions en entier avant d'utiliser, d'entretenir ou de réparer l'équipement.

Uniquement, la portion pour détecter les gaz combustibles de cet instrument a été évaluée.

Tout appareil neuf de RAE Systems doit préalablement passer le test de vérification d'étalonnage qui consiste à exposer les capteurs a une concentration connue de gaz étalon, et ce avant que l'instrument soit mis en service puis avant chaque utilisation. Une vérification d'étalonnage est définie par une exposition courte du détecteur au gaz d'étalonnage, ainsi les capteurs doivent réagir et déclencher les seuils d'alarmes bas pour chacun des gaz détectables.

Le détecteur multi gaz QRAEII doit être impérativement étalonné si il ne passe pas le test de vérification d'étalonnage, ou bien au moins tous les 180 jours, selon l'utilisation et l' exposition des capteurs a des gaz poisons ou a des niveaux élevés de contaminants.

- Les intervalles d'étalonnage et les procédures de vérifications peuvent varier en fonction des législations nationales.
- RAE Systems recommande d'utiliser les bouteilles d'étalonnage RAE Systems avec un mélange des quatre gaz suivant 10 ppm H2S, 50 ppm CO, 50% LIE méthane, et 18.5% d'oxygène.
- Toute variation de la lecture rapide et positive, suivie d'une baisse subite ou erratique de la valeur, peut indiquer une concentration de gaz hors gamme de détection qui peut être dangereuse.

Note : les vérifications d'étalonnage ainsi que les étalonnages peuvent être réalisés sur la station d'auto-étalonnage RAE Systems AutoRAE™.

General Information

QRAE II is a programmable Multi-gas monitor designed to provide continuous exposure monitoring of oxygen, hydrogen sulfide, carbon monoxide and combustible gases for workers in hazardous environments. It monitors with the following types of sensors:

1. Combustible gases are monitored with catalytic bead sensors.
2. Hydrogen sulfide and carbon monoxide are monitored with electrochemical sensors.
3. Oxygen is monitored with a solid polymer electrolyte (SPE) sensor.

Key Features

Lightweight and Compact
12.3 oz (350 g), handheld size.
Dependable and Accurate
14 hours of monitoring with microcontroller.
User Friendly
Menu-driven, intuitive end-use operation.
Programmable Alarm Thresholds
Audio buzzer and flashing display alarm.

- Standard configuration includes CO, H_2S, LEL and O_2 sensors
- Replaceable, rechargeable Li-Ion battery
- Large, easy-to-read display
- Datalogging with large memory
- Visual alarm with bright red flashing LEDs
- Loud audible alarm (95dB at 30 cm)
- Vibration alarm
- Rugged weather-resistant composite case

Specifications

QRAE II Specifications

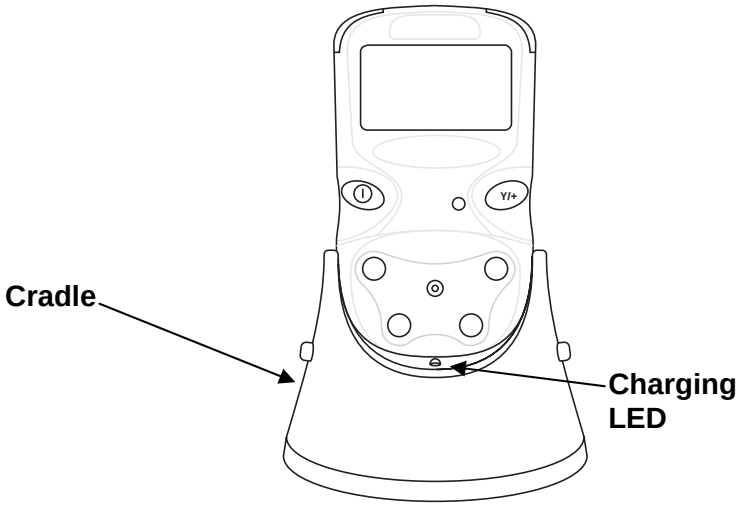
Configuration	Diffusion 4-gas with datalogging
Dimensions:	4.94" L x 2.76" W x 1.5" H (125.5 mm x 70 mm x 38 mm)
Weight:	12.3 oz (350 g) with battery
Detectors:	2 Electrochemical toxic gases sensors 1 Solid Polymer Electrolyte oxygen sensor 1 Catalytic sensor for combustible level organics
Battery:	Rechargeable 3.7V Li-ion battery pack (6-hour charge time) or a 3 AA alkaline battery adapter.
Operating Time:	Up to 10 hours continuous w/ Li-ion battery pack
Display:	4-line graphical LCD with automatic LED backlight for dim lighting conditions
Keypad:	2 programming/operation keys
Direct Readout:	Up to 4 simultaneous values with sensor name, battery charge, high and low values for all sensors, elapsed time, and datalogging on/off state
Sampling Method:	Diffusion
Range, Resolution & Response Time:	LEL 0-100% 1 % 15 sec O_2 0-30% 0.1 % 15 sec CO 0-1000 ppm 1 ppm 20 sec H_2S 0-100 ppm 1 ppm 30 sec
Alarm Settings:	Separate limits for TWA, STEL, Peak, Min
Alarms:	$\geq$95 dB @ 30 cm buzzer, flashing red LEDs, vibration alarm, LCD to indicate exceeded preset limits, low battery, or sensor failure
Calibration:	Two-point field calibration for fresh air and standard reference gas
Protection:	Password protected calibration settings, alarm limits, and data
Intrinsic Safety:	CSA Class 1, Division I, Group A, B, C, D, T4 (US & Canada), KEMA ATEX II 2G EEx d ia II C T4 (Europe)
EM Immunity:	No effect when exposed to 0.43mW/cm^2 RF interference (5-watt transmitter at 12"/10cm).
Data Storage:	64,000 readings (64 hours, 5 channels at 1 minute interval) in non-volatile memory.
Datalog Interval:	Programmable 1- to 3,600-second intervals
Alarm Settings:	Separate alarm limit settings for TWA, STEL, Low and High alarm.
Communication:	Download data to PC and upload monitor setup from PC through an RS-232 link to PC serial port
Temperature:	-20° C to 50° C (-4° F to 122° F)
Humidity:	0% to 95% relative humidity (non-condensing)

Caution:

Refer to RAE Systems Technical Note TN-114 for sensor cross-sensitivities.
Refer to RAE Systems Technical Note TN-144 for LEL sensor poisoning.

Charging The QRAE II Battery

Always fully charge the battery before using the QRAE II. The QRAE II's Li-ion battery is charged by placing the QRAE II in its cradle. Contacts on the bottom of the QRAE II meet the cradle's contacts, transferring power without other connections.



Note: Before setting the QRAE II into its charging cradle, visually inspect the contacts to make sure they are clean. If they are not, wipe them with a soft cloth. Do not use solvents or cleaners.

Follow this procedure to charge the QRAE II:

1. Plug the AC/DC adapter into the QRAE II's cradle.
2. Plug the AC/DC adapter into the wall outlet.
3. Place the QRAE II into the cradle and press down until the LED glows.

The QRAE II begins charging automatically. The LED in the cradle should glow red to indicate charging. During charging, the display shows this message:

Charging...

At the same time, the voltage is indicated in the QRAE II's display, as well as an electrical plug icon is shown next to the battery:

When the QRAE II's battery is fully charged, the message "Fully Charged!" is shown in the display, and the icons for the electrical plug and the battery are shown.

When charging is complete, the LED in the cradle glows green.

Note: A spare battery can be charged by placing it directly on the charging cradle. See "Charging A Spare QRAE II Battery" on page 48 for details.

Note: An Alkaline Battery Adapter (part number 020-3403-000), which uses three AA alkaline batteries, may be substituted for the Li-Ion battery. See page 47 for details.

WARNING!

To reduce the risk of ignition of hazardous atmospheres, recharge batteries only in areas known to be non-hazardous. **Remove and replace batteries only in areas known to be non-hazardous.**

Low Voltage Warning

When the battery's charge falls below the shutoff voltage, the display shows:

Battery low,
turning off. . .

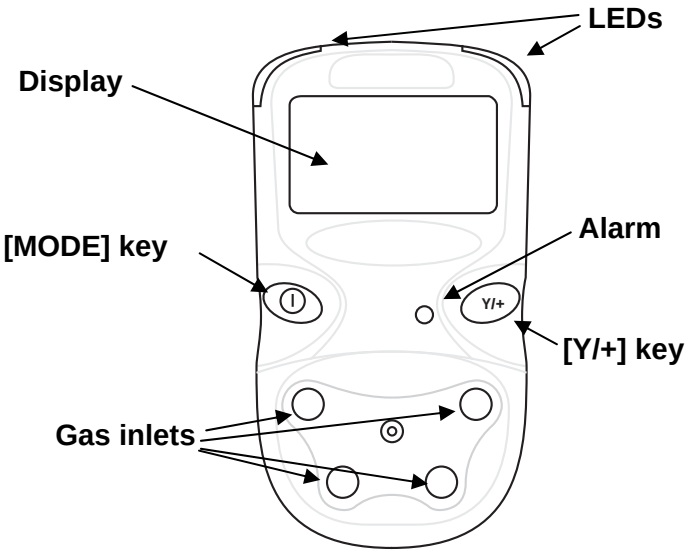
The QRAE II then turns itself off. Recharge the battery by placing the QRAE II in its cradle.

Clock Battery

An internal clock battery is mounted on one of the QRAE II's printed circuit boards. This long-life battery keeps settings in memory from being lost whenever the Li-ion battery or alkaline batteries are removed. This backup battery should last approximately five years, and must be replaced by an authorized RAE Systems service technician. It is not user-replaceable.

User Interface

The QRAE II's user interface consists of the display, LEDs, an alarm transducer, and two keys, labeled [MODE] and [Y/+]. The LCD display provides visual feedback that includes time, sensor mode, battery condition, and datalog enable/disable status.



User Interface Icons

Icon	Indication
	Battery Voltage low (flashing) Battery Low alarm triggered
	Battery fully charged
	Battery charging
	Alkaline Battery Adapter in use
	Alkaline Battery cannot be charged
	Datalogging active (flashing)
F	Datalog memory full

Turning The QRAE II On

To turn the QRAE II on, hold down [MODE] for 2 seconds.

Caution: The alarm is very loud. During startup, you can mute most of the sound by holding a finger over the alarm port.

Note: Do not put tape over the alarm port to permanently mute it.

When starting up, the QRAE II simultaneously turns the backlight on and off, beeps once, blinks once, and vibrates. The screen shows:

On...

RAE Systems Inc.
QRAE II
(Language)

This is followed by a progression of screens that tell you the QRAE II's current settings:

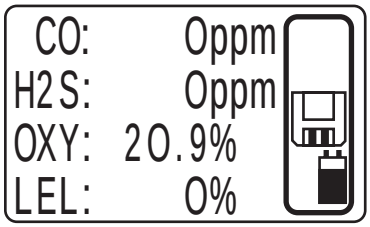
- Firmware version number and serial number
- List of installed sensors
- Last Calibration date and time
- Alarm Limits (High, Low, STEL, TWA)
- Today's date, the current time, and the current temperature
- Alarm Mode, battery voltage, shutoff voltage
- Datalog Mode,
- Datalog Period
- Datalog Time Left

The QRAE performs a final checkout and the screen shows a countdown to full operational functionality.

If Datalog is on, this message is displayed after the countdown:

Datalog Started

When the QRAE II is ready for use, it shows this screen:



Note: If datalogging is off, you will not see the datalogging icon (see icons, page 10).

Inverting The Display

The QRAE II is easy to read, whether held in the hand or clipped to a belt. To flip the screen, press the [Y/+] key and hold it down for 3 seconds. When the image inverts, release the key.

Testing The Alarms (Anytime)

Under normal non-alarm conditions, the buzzer, vibration alarm, LED, and backlight can be tested at any time by pressing [Y/+] once.

Turning The QRAE II Off

Press and hold [MODE]. In 2 seconds, a 5-second countdown to shutoff begins. You must hold your finger on the key for the entire shutoff process. If you remove your finger from the key during the countdown, the shutoff operation is canceled and the QRAE II continues normal operation.

The countdown proceeds as follows, accompanied at each step with an alarm beep and light flash. The display shows the countdown in sequence:

Unit off in 5 seconds...
Unit off in 4 seconds...
Unit off in 3 seconds...
Unit off in 2 seconds...
Unit off in 1 seconds...
Unit off in 0 seconds...
Unit off...

When you see "Unit off..." release your finger from the [MODE] key. The QRAE II is now off.

Caution: The alarm is very loud. During shutdown, you can mute most of the sound by holding a finger over the alarm port.

Operating The QRAE II

Mode Overview

The QRAE II has three operational modes:

- **Normal** — see page 14 for detailed instructions.
- **Diagnostic** — see page 15 for detailed instructions.
- **Programming** — see page 19 for detailed instructions.

The following is an overview of the three modes:

Normal Mode is the default mode. It is accessed when you turn on the QRAE II. There are no access restrictions (you do not need a password), and it provides the indications and data you need most for typical monitoring applications, including:

- Readings of carbon monoxide (CO), hydrogen sulfide (H_2S), oxygen, and lower explosive limit (LEL).
- Peak.
- Min (minimum).
- STEL (short-term exposure limit).
- TWA (time-weighted average).
- Battery level.
- Run time since the QRAE II was turned on.
- Time, date, and temperature.
- Name of the LEL span gas used for calibration and the measurement gas.

Normal Mode also allows you to turn datalogging on and off and communicate with a PC to download data.

Diagnostic Mode is primarily designed for technicians during troubleshooting, although it also offers access to a few changeable parameters that you may rarely (if ever) change. You can enter Diagnostic Mode without restriction. In Diagnostic Mode, QRAE II displays readings in raw counts (cts) instead of units such as percentage or parts per million.

- Battery type indicator (Li-ion or alkaline)
- Battery level and shutoff voltage, and run time
- COx1 and COx5 (dual-range CO sensing)
- Display Contrast*
- Date, time, and temperature
- Backlight level and threshold*

Parameters marked with an asterisk (*) can be adjusted by entering Programming Mode from Diagnostic Mode. See Page 15 for details.

Programming Mode is password-protected and is includes adjustable settings to accomplish the following:

- Calibrate the monitor (this includes changing span gases and values, and selecting single or multiple sensor calibrations).
- Change alarm limits.
- Change/enable/disable the datalog.
- Change the monitor setup.
- Change the sensor configuration.

Normal Mode

Normal Mode is the default mode of the QRAE II when it is turned on. By using the [MODE] key, you can step through the screens that provide you with information from the sensors, as well as the QRAE II's current settings.

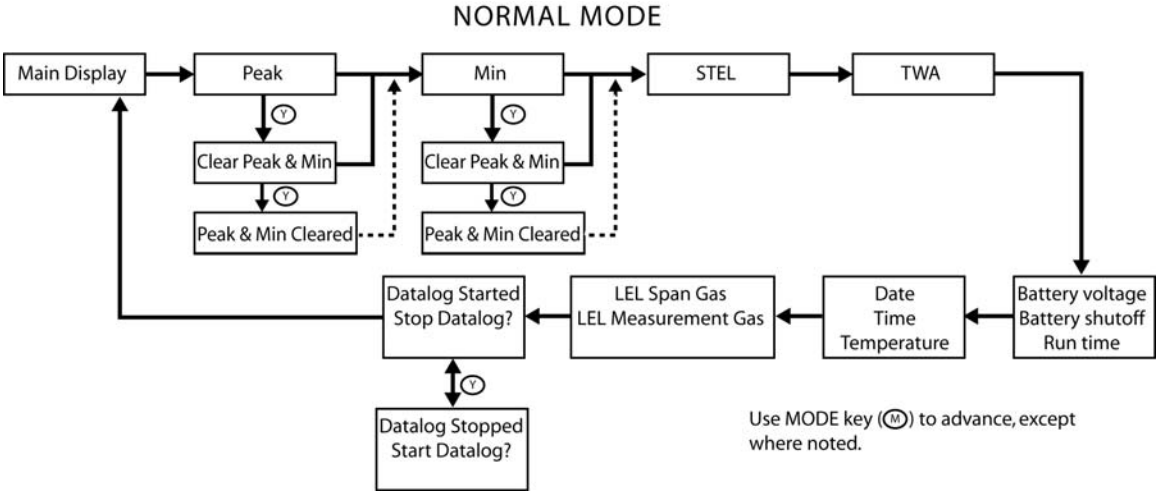
Enter Normal Mode:

With the QRAE II turned off, press and hold [MODE].
When the display turns on, release the keys. The QRAE II is now operating in Normal Mode.

To Exit Normal Mode:

Whenever you turn off the QRAE II, it will start up in Normal Mode, unless you intentionally place it in Diagnostic Mode. Follow the detailed instructions on entering Diagnostic Mode and Programming Mode for information on entering the other two modes.

After it is shut off, the QRAE II will automatically be in Normal Mode the next time you start it.



Peak. This tells you the highest reading for each sensor since the QRAE II was turned on. Press [Y/+] twice to clear the Peak and Min or [MODE] once to advance to Min.

Min. This tells you the lowest (minimum) reading for each sensor since the QRAE II was turned on. Press [Y/+] twice to clear the Peak and Min or [MODE] once to advance to STEL.

STEL. This provides Short Term Exposure Limit (STEL) data. These are based on 15-minute STEL values of H_2S and CO in ppm and 8-hour Time Weighted Average (TWA).

TWA. The TWA (time-weighted average) reading is the average reading of the gas concentration times the fraction of 8 hours that the monitor has been on.

Battery Voltage & Shutoff Voltage. This tells you the battery's voltage right now and the voltage at which the QRAE II will shut off. This varies, depending on whether a Li-ion battery or an alkaline battery adapter is used.

Run time. This tells you in hours and minutes how much time has elapsed since the QRAE II was turned on. Run time resets to 0:00 every time the QRAE II is turned on.

Date, Time, and Temperature. The full date of month, day, and year, as well as the 24-hour time in hours, minutes, and seconds, is shown. Temperature is shown in degrees Fahrenheit or Celsius, depending on the user configuration (see Set Temperature Unit, page 33).

LEL Span Gas, LEL Measurement Gas. These two indications tell you the gas selected for performing a span and for performing a full measurement on the LEL sensor.

Datalog Started. This informs you that the datalog has been started.

Communicate With PC. When the QRAE II is connected to a computer running ProRAE Studio software, it can download datalog data and upload revised firmware.

Note: If you access any screen in Normal Mode and leave that screen unattended for a few minutes, QRAE II automatically reverts to Normal Mode's main screen.

Diagnostic Mode

The QRAE II's Diagnostic Mode can only be accessed at startup time.

To Enter Diagnostic Mode:

1. With the QRAE II turned off, press and hold both [MODE] and [Y/+].
2. When the display turns on, release the keys.

To Exit Diagnostic Mode:

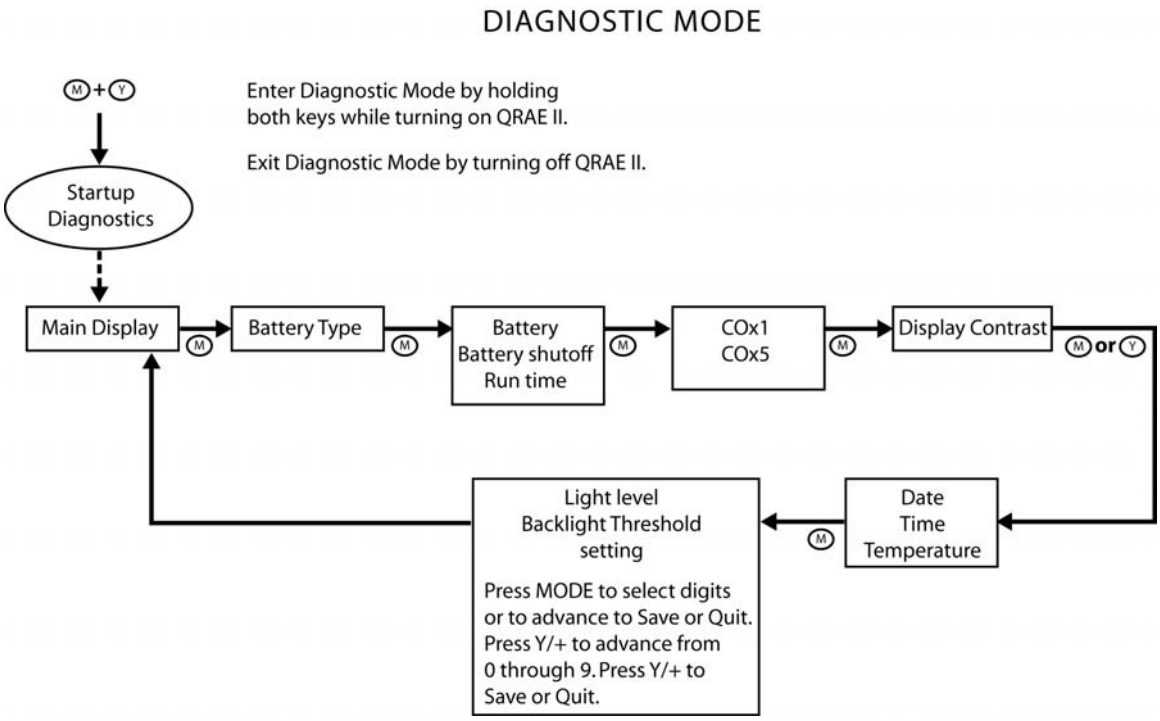
1. Turn off the QRAE II by pressing and holding [MODE]. There will be a standard shutoff countdown.
2. When it shuts off, you will be alerted. Release your finger.

Note: The next time you start QRAE II, hold only [MODE], and it will automatically be in Normal Mode.

When the QRAE II runs in Diagnostic Mode, the screen shows raw counts for sensors and battery levels (expressed as "cts," short for "counts"). You can step through other diagnostic information by pressing the [MODE] key. The items followed by an asterisk (*) are user-changeable.

- Battery type (Li-ion or alkaline)
- Battery level and shutoff voltage, and run time
- COx1 and COx5
- Display Contrast*
- Date, time, and temperature
- Backlight level and threshold*

Note: If the QRAE II is in its cradle, and the cradle is connected via its data cable to a PC, communication is possible in this mode.



Battery type. This indicates the type of battery that is currently in use (Li-ion or alkaline).

Battery level and shutoff voltage, and run time. This screen tells you the battery's voltage right now and the voltage at which the QRAE II will shut off. This varies, depending on whether a Li-ion battery or an alkaline battery adapter is used.

Run time. This tells you in hours and minutes how much time has elapsed since the QRAE II was turned on. Run time resets to 0:00 every time the QRAE II is turned on.

COx1 and COx5. The QRAE II's carbon monoxice (CO) sensing operates in two ranges, providing optimal sensing based on concentration. When the signal is in the low range, the COx5 reading is monitored, and when the signal is in the high range, the COx1 reading is monitored.

Display Contrast. You can set the display contrast for maximum sharpness under the lighting conditions in which you are operating.

1. Press [MODE] until the bar graph blinks, showing it is selected.
2. Repeatedly press the [Y/+] button to incrementally increase the darkness of the display.

   Note: Pressing [Y/+] several times causes the screen to appear fully dark. To return to a lighter screen, keep pressing [Y/+] until the screen suddenly becomes light again. This "wrap-around" feature ensures that you can correct for too much contrast setting.

3. Once the display contrast is set to your satisfaction, press [MODE], which will cause Save to blink. If you want to save the new setting, press [Y/+]. If you want to quit without saving the new setting, press [MODE] to advance to Quit. Then press [Y/+].

Date, Time, and Temperature. The full date of month, day, and year, as well as the 24-hour time in hours, minutes, and seconds, is shown. Temperature is shown in degrees Fahrenheit or Celsius, depending on the user configuration (see Set Temperature Unit, page 33).

Backlight level and threshold. This screen shows two values:

> **Light:** The amount of ambient light where the QRAE is in operation.
> **Backlight Thresh Setting:** The adjustable setting that sets the threshold at which the backlight illuminates when the QRAE II backlight is in automatic mode.

Whenever the QRAE II's backlight is in automatic mode and the amount of ambient light is lower than the Backlight Thresh Setting, the backlight illuminates.

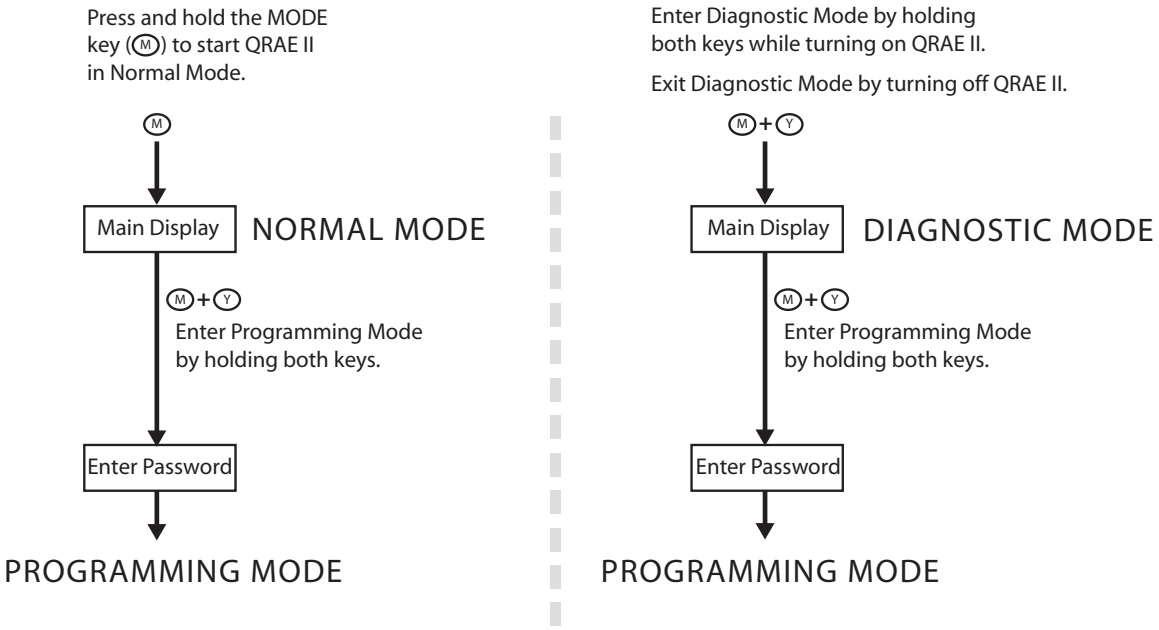
Set the threshold level for normal lighting conditions. However, if you are going to be in a dark location for extended periods and want to conserve battery power, you may want to set the QRAE's backlight to operate in manual mode. See page 32 for details.

Programming Mode

Programming Mode can be entered from Normal Mode or Diagnostic Mode. This mode contains most adjustable settings for the QRAE II. It is organized into five submenus:

- Calibrate Monitor
- Change Alarm Limits
- Change Datalog
- Change Monitor Setup
- Change Sensor Configuration

The following diagram shows how to enter Programming Mode from Normal Mode and from Diagnostic Mode:



Enter Programming Mode

To enter Programming Mode, hold down [MODE] and [Y/+] simultaneously for three seconds. To exit this mode, press [MODE] repeatedly until the main concentration display shows.

When you enter Programming mode, you see:

Enter Password?

0000

OK Cancel

Increase a number by pressing the [Y/+] key (1, 2, 3 ... etc.). **Note:** The numbers advance until the number 9 and then "wrap around" to 0 again.

Advance to the next digit by pressing the [MODE] key (**0**000, 0**0**00, etc.). **Note:** The display has a "wrap-around," so once you reach the last digit, pressing [MODE] advances to the first digit again.

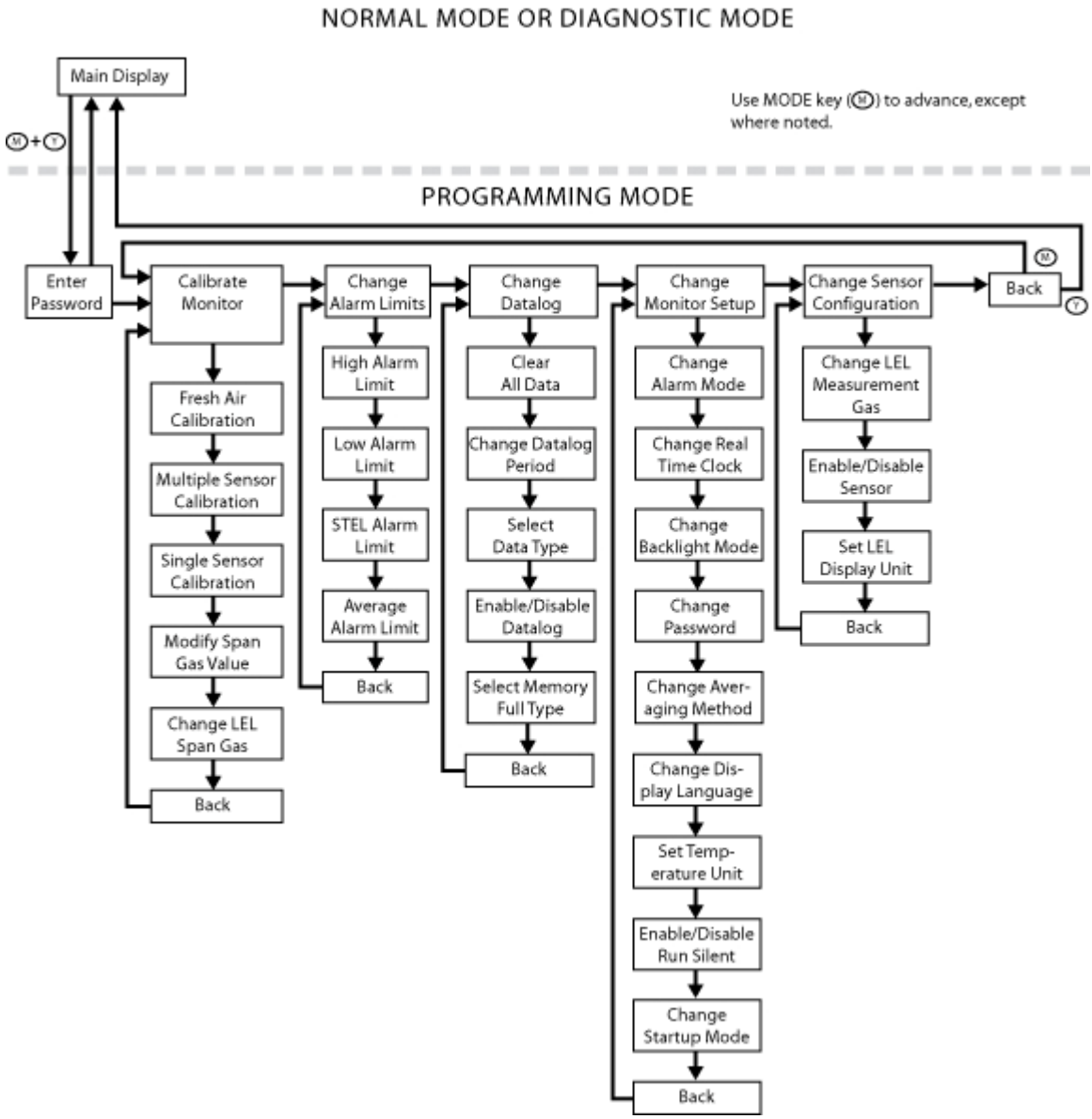
Once you are satisfied with the password, advance to OK, and press [MODE].
If you want to cancel, advance to Cancel and press the [Y/+] key.

Note: The default password is 0000. If you change the password, write it down and save it in a safe location. You do not have to change the password from 0000. You can simply continue using 0000, step through the digits and then press the [Y/+] key to enter Programming Mode.

Navigating Programming Mode

The following diagram shows Programming Mode's five submenus and how to navigate through them.

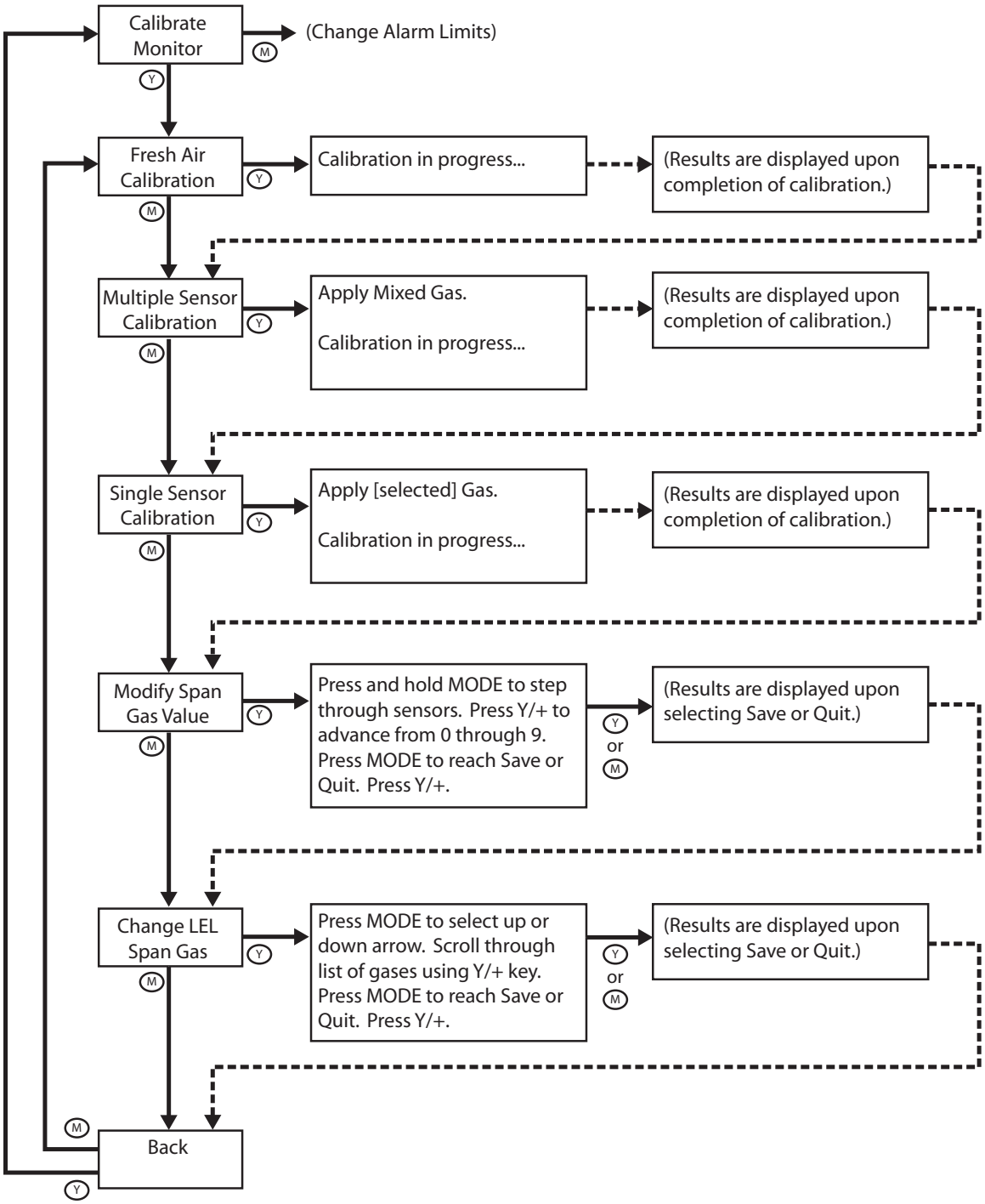
Note: Press [MODE] to navigate from one menu choice to the next and [Y/+] to make selections.



Calibrate Monitor

The QRAE II is designed to perform automated calibrations.

Note: The procedures for calibration are covered in a separate section, "Calibrating The QRAE II," on page 38.
The submenus and actions are shown in the following diagram.



Fresh Air Calibration. This sets the zero point of the sensor calibration curve for clean air. Expose the inlet to a clean air source with 20.9% oxygen and without any organic, toxic or combustible gas impurities. Follow the procedure outlined in "Zero (Fresh Air) Calibration" on page 39.

Multiple Sensor Calibration. This function simultaneously determines the second point of the calibration curve for multiple sensors in the monitor. To calibrate, follow the procedure outlined in "Calibrating The QRAE II," on page 38.

Single Sensor Calibration. This procedure determines the second point of the sensor calibration curve for a single sensor. To calibrate, follow the procedure outlined in "Calibrating The QRAE II," on page 38.

Modify Span Gas Value. This function allows selection of the gas concentration for each sensor.

Setting the span value separately for each sensor.

1. Select a sensor by pressing [MODE] until the sensor's name is highlighted.
2. Press [MODE] to advance through the digits on each sensor.
3. Press [Y/+] to increase the number (0 to 9).

   Note: Once the number reaches 9, pressing [Y/+] returns to 0 and starts counting up again each time [Y/+] is pressed.

4. Press and hold [MODE] for 3 seconds and release to advance to the next sensor.
5. After you have set all of the span values, hold [MODE] for 3 seconds and release. Save is highlighted.
6. Press [Y/+] to save your settings, or [MODE] to advance to Quit (without saving settings).

If you choose to quit without saving the changes, press [Y/+]. You will see this message:

Not Saved!

If you want to make further changes to the settings, press [MODE] to repeat stepping through the sensors.

Change LEL Span Gas. This function allows selection of the gas to be used for span calibration of the LEL sensor. The correction for the measurement gas is automatically divided by the correction factor the span gas, selected previously, to obtain a new factor for the combination of gases. The new factor is applied to the readings to obtain a true concentration.

1. Press [MODE] until the up or down arrow is highlighted.
2. Press [Y/+] to move through the list of LEL span gases.

3. Press [MODE] to select either the other arrow or to advance to Save or Quit.
4. With Save selected, press [Y/+] to save your settings, or [MODE] to advance to Quit (without saving settings).

If you choose to quit without saving the changes, press [Y/+]. You will see this message:

Not Saved!

If you want to make further changes to the settings, press [MODE] to repeat stepping through the choices.

Back. Press [MODE] to return to the top of the Calibrate Monitor menu, or press [Y/+] to return to the top of the Normal Mode menu.

Change Alarm Limits

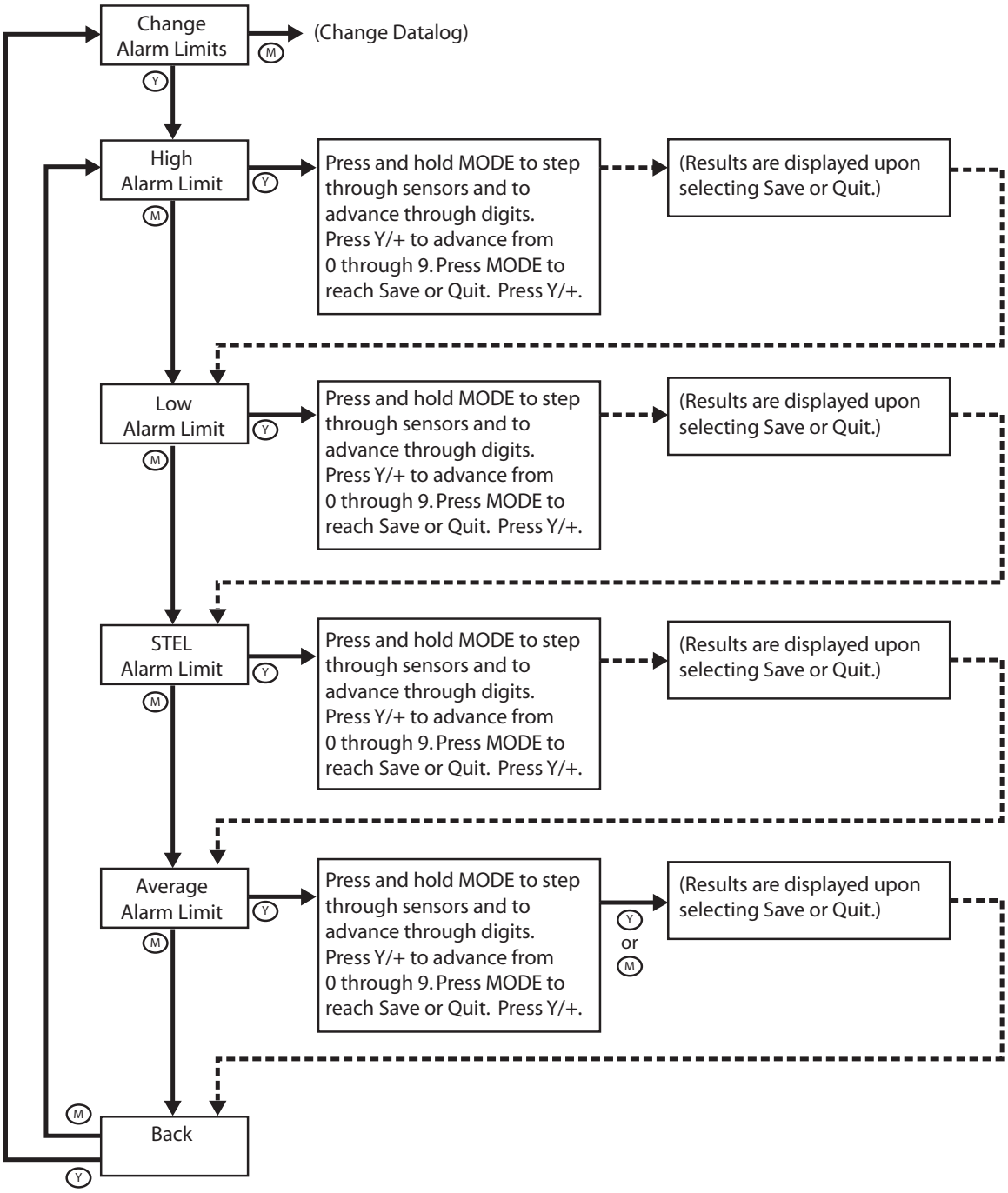
The high and low alarm limits, as well as the points at which the STEL and TWA alarms are triggered, can be modified in this set of menus. Each sensor's limits for each of these can be set separately, providing extremely precise alarm thresholds.

Alarm Signals. During each measurement period, the gas concentration is compared with the programmed alarm limits (gas concentration alarm limit settings: Low, High, TWA and STEL). If the concentration exceeds any of the preset limits, the loud buzzer, red flashing LED, and vibration alarm are activated immediately to warn of the alarm condition. In addition, the QRAE II alarms if one of the following conditions occurs: battery voltage falls below a preset voltage level (3.1 volts for Li-ion, 3.3 volts for alkaline batteries), or when the datalog memory is full. When the low battery alarm occurs, there will be approximately 20 to 30 minutes of operating time remaining. When the battery voltage falls below the low threshold, the QRAE II turns off automatically.

Submenus in this section are:

- High Alarm Limit
- Low Alarm Limit
- Stel Alarm Limit
- Average Alarm Limit

The submenus and actions are shown in the following diagram:



Change High Alarm Limit. This function allows you to selectively set the high limit of individual sensors in the QRAE II.

1. Select a sensor by pressing [MODE] until the sensor's name is highlighted.
2. Press [MODE] to step through the sensor's digits.
3. Press [Y/+] to increase the number from 0 through 9. Once the number 9 is reached, pressing [Y/+] causes the numbers to "wrap around" to 0 and count up again.

4. Press and hold [MODE] for 3 seconds to advance to the next sensor.
5. Follow the same procedure as steps 2 and 3 for each sensor.

After you have modified all necessary sensor settings:

6. Press [MODE] to advance to Save or Quit.
7. With Save selected, press [Y/+] to save your settings, or [MODE] to advance to Quit (without saving settings).

Change Low Alarm Limit. This function allows you to selectively set the low limit of individual sensors in the QRAE II.

1. Select a sensor by pressing [MODE] until the sensor's name is highlighted.
2. Press [MODE] to step through the sensor's digits.
3. Press [Y/+] to increase the number from 0 through 9. Once the number 9 is reached, pressing [Y/+] causes the numbers to "wrap around" to 0 and count up again.
4. Press and hold [MODE] for 3 seconds to advance to the next sensor.
5. Follow the same procedure as steps 2 and 3 for each sensor.

After you have modified all necessary sensor settings:

6. Press [MODE] to advance to Save or Quit.
7. With Save selected, press [Y/+] to save your settings, or [MODE] to advance to Quit (without saving settings).

Change STEL Alarm Limit. This function allows you to selectively set the STEL (short term exposure limit) of individual sensors in the QRAE II. Note: This function does not include LEL or oxygen sensors.

1. Select a sensor by pressing [MODE] until the sensor's name is highlighted.
2. Press [MODE] to step through the sensor's digits.
3. Press [Y/+] to increase the number from 0 through 9. Once the number 9 is reached, pressing [Y/+] causes the numbers to "wrap around" to 0 and count up again.
4. Press and hold [MODE] for 3 seconds to advance to the next sensor.
5. Follow the same procedure as steps 2 and 3.

After you have performed all necessary modifications:

6. Press [MODE] to advance to Save or Quit.
7. With Save selected, press [Y/+] to save your settings, or [MODE] to advance to Quit (without saving settings).

Change Average Alarm Limit. This function allows you to selectively set the STEL (short term exposure limit) of individual sensors in the QRAE II. Note: This function does not include LEL or oxygen sensors.

1. Select a sensor by pressing [MODE] until the sensor's name is highlighted.
2. Press [MODE] to step through the sensor's digits.
3. Press [Y/+] to increase the number from 0 through 9. Once the number 9 is reached, pressing [Y/+] causes the numbers to "wrap around" to 0 and count up again.
4. Press and hold [MODE] for 3 seconds to advance to the next sensor.
5. Follow the same procedure as steps 2 and 3.

After you have performed all necessary modifications:

6. Press [MODE] to advance to Save or Quit.
7. With Save selected, press [Y/+] to save your settings, or [MODE] to advance to Quit (without saving settings).

Back. Press [MODE] to return to the top of the Change Alarm Limits menu, or press [Y/+] to return to the top of the Normal Mode menu.

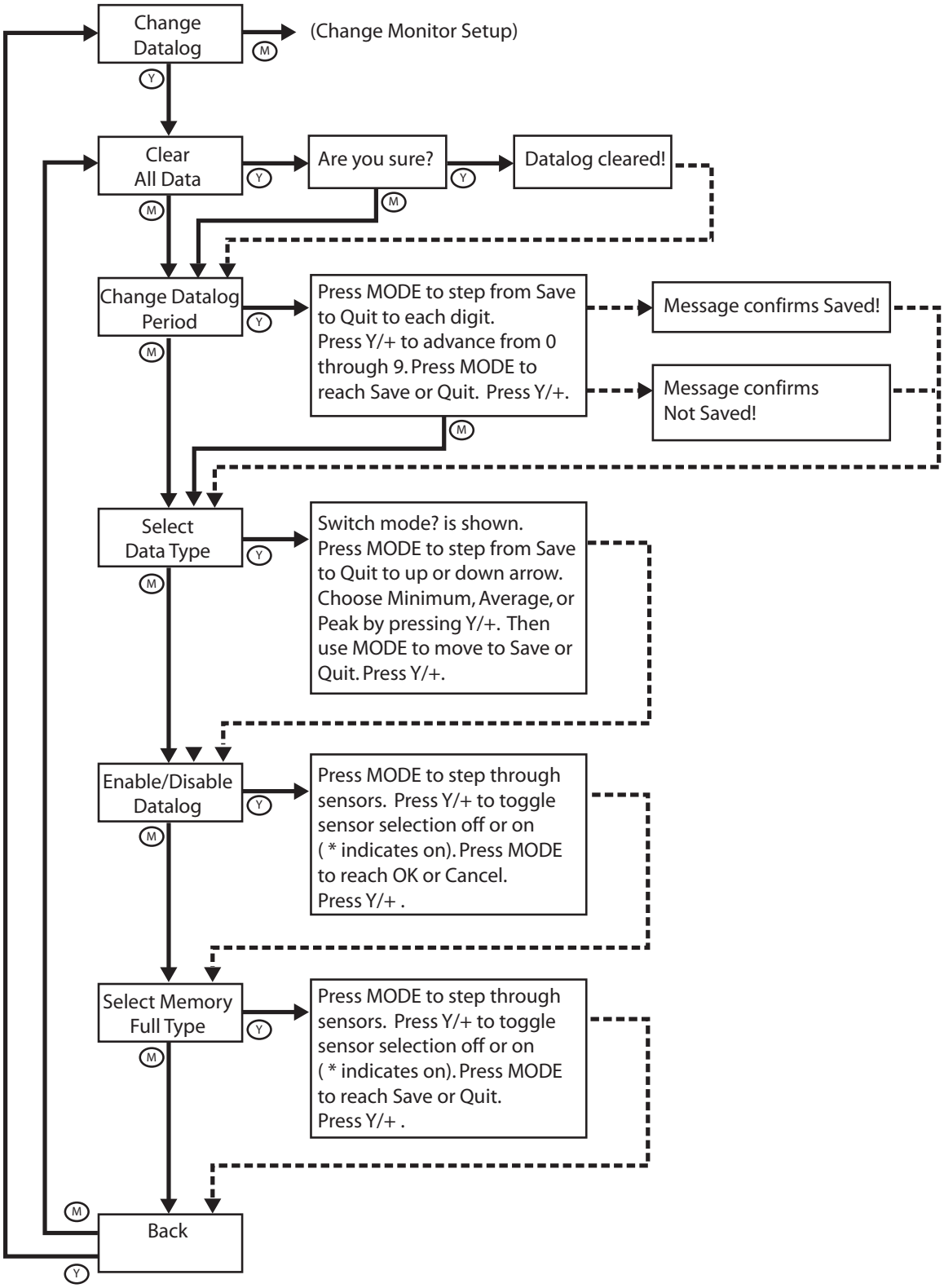
Change Datalog

The QRAE II calculates and stores the gas readings based on a user-specified datalogging period and the type of measurement. Average, peak and minimum values can be stored for each sensor during each datalogging interval. The datalogging interval can be programmed from one second to 3,600 seconds (60 minutes) in 1-second intervals. In addition, serial number, last calibration date, and alarm limits are stored. All data are retained in non-volatile memory for later download to a PC.

There are five submenus to Change Datalog:

- Clear All Data
- Change Datalog Period
- Select Data Type
- Enable/Disable Datalog
- Select Memory Full Type

The following diagram shows the navigation of this set of submenus:



Clear All Data. You can clear all data from the datalog. The QRAE II has a challenge that asks you to confirm that you want to clear the data.

1. Press [Y/+]. The display shows:

   Are you sure?

2. Press [Y/+] to clear the datalog or [MODE] if you do not want to clear the datalog. If you choose to clear it, the display shows:

   Datalog cleared!

   If you press [MODE], the display shows:

   Datalog not cleared.

Important! If you clear the datalog's data, it cannot be retrieved, or "unerased."

Change Datalog Period. The datalogging interval can be set from 1 second to 3,600 seconds (60 minutes). This is the time between data points. The QRAE II can store 64,000 datapoints.

1. Press [MODE] to step from Save and Quit and to advance through the digits.
2. Press [Y/+] to advance from 0 through 9. If you press [Y/+] when 9 is selected, it "wraps" around to 0 and begins counting up again each time you press [Y/+].
3. Press [MODE] to step to the next digit.
4. Press [Y/+] to advance from 0 through 9. Repeat steps 3 and 4 until you advance to Save or Quit.
5. Press [Y/+] to commit your change or to exit to the next menu item.

Select Data Type. You have three choices for the type of data collected in the QRAE II. You may select one. The screen shows:

Switch mode?

Your choices are:

- Average
- Peak
- Minimum

Press [MODE] to move from the up arrow to the down arrow to Save and to Quit. When the cursor is on the choice you want, press the [Y/+] key.

Enable/Disable Datalog? You can selectively enable or disable the data from each sensor to be stored in the datalog. This requires turning on or off each sensor in the menu.

1. Select a sensor to enable/disable by pressing [MODE] until the sensor's name is highlighted.
2. Press [Y/+] to toggle the selection on (indicated by an asterisk, *) or off (no asterisk).

   To select other sensors to datalog, press [MODE] until you reach the sensor you want to select. Then press [Y/+].

After you have selected all the sensors you want enabled or disabled:

3. Press [MODE] to advance to OK or Cancel.

With OK selected, press [Y/+] to save your settings, or press [MODE] to advance to Cancel (without saving settings).

Select Memory Full Type. You can set how the QRAE II deals with a full datalog memory. Once the datalog is full, it can either stop (retaining all data to that point) or wrap around, meaning that it begins overwriting the earliest data and proceeding to replace old data in an ongoing manner.

1. Press [MODE] to step from Save to Quit to the up or down arrow.
2. Choose Stop or Wrap Around by pressing [Y/+].
3. Press [MODE] to step to Save or Quit.
4. Press [Y/+] to commit your change or to exit to the next menu item.

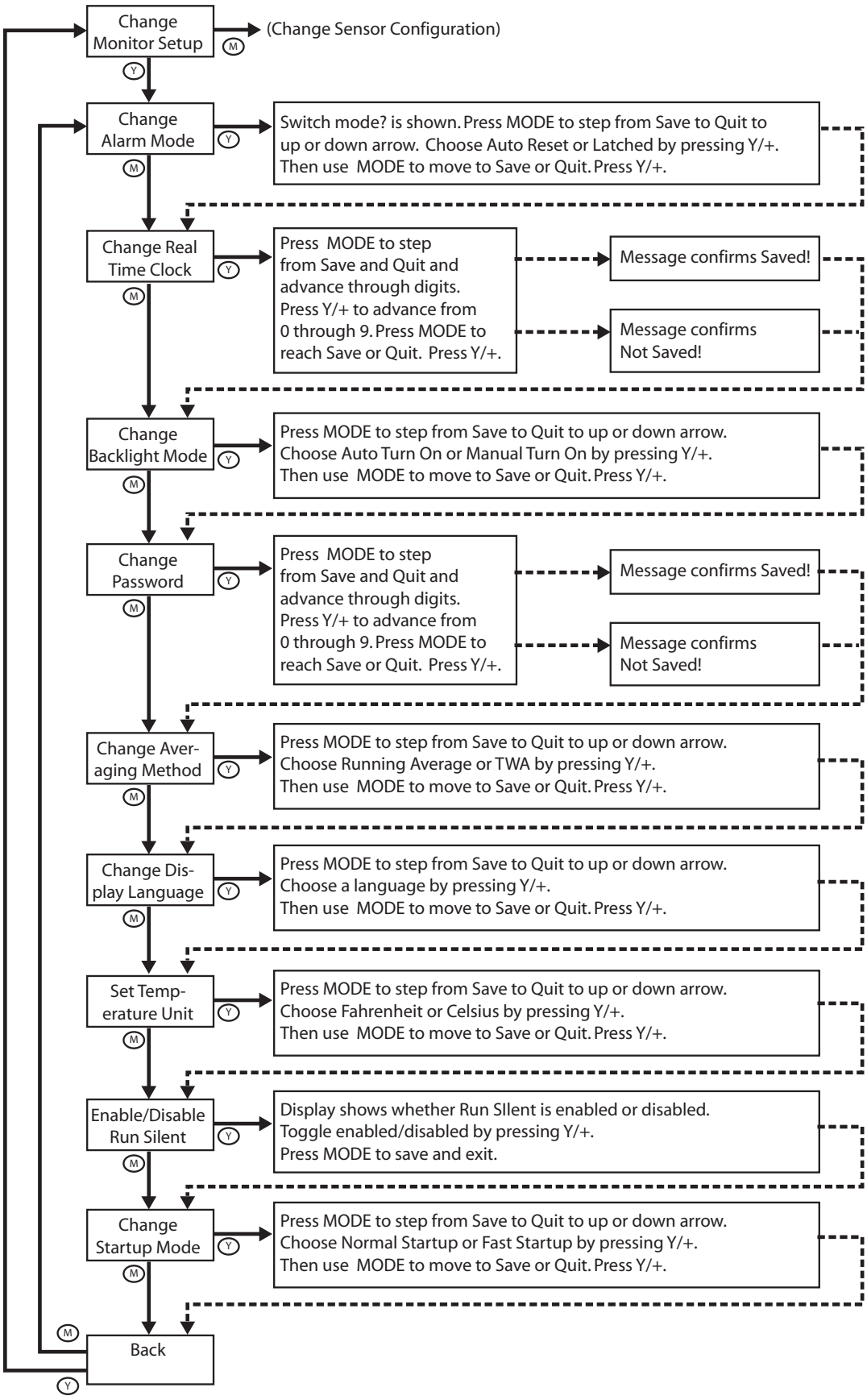
Back. Press [MODE] to return to the top of the Change Datalog menu, or press [Y/+] to return to the top of the Normal Mode menu.

Change Monitor Setup

This is the most extensive submenu set in the QRAE II. It includes:

- Change Alarm Mode
- Change Real Time Clock
- Change Backlight Mode
- Change Password
- Change Averaging Method
- Change Display Language
- Set Temperature Unit
- Enable/Disable Run Silent
- Change Startup Mode

The diagram on the next page shows how Change Monitor Setup is organized and how to navigate through it.


Change Monitor Setup
(Change Sensor Configuration)
Change Alarm Mode
Switch mode? is shown. Press MODE to step from Save to Quit to up or down arrow. Choose Auto Reset or Latched by pressing Y/+. Then use MODE to move to Save or Quit. Press Y/+.
Change Real Time Clock
Press MODE to step from Save and Quit and advance through digits. Press Y/+ to advance from 0 through 9. Press MODE to reach Save or Quit. Press Y/+.
Message confirms Saved!
Message confirms Not Saved!
Change Backlight Mode
Press MODE to step from Save to Quit to up or down arrow. Choose Auto Turn On or Manual Turn On by pressing Y/+. Then use MODE to move to Save or Quit. Press Y/+.
Change Password
Press MODE to step from Save and Quit and advance through digits. Press Y/+ to advance from 0 through 9. Press MODE to reach Save or Quit. Press Y/+.
Message confirms Saved!
Message confirms Not Saved!
Change Aver-aging Method
Press MODE to step from Save to Quit to up or down arrow. Choose Running Average or TWA by pressing Y/+. Then use MODE to move to Save or Quit. Press Y/+.
Change Dis-play Language
Press MODE to step from Save to Quit to up or down arrow. Choose a language by pressing Y/+. Then use MODE to move to Save or Quit. Press Y/+.
Set Temp-erature Unit
Press MODE to step from Save to Quit to up or down arrow. Choose Fahrenheit or Celsius by pressing Y/+. Then use MODE to move to Save or Quit. Press Y/+.
Enable/Disable Run Silent
Display shows whether Run SIlent is enabled or disabled. Toggle enabled/disabled by pressing Y/+. Press MODE to save and exit.
Change Startup Mode
Press MODE to step from Save to Quit to up or down arrow. Choose Normal Startup or Fast Startup by pressing Y/+. Then use MODE to move to Save or Quit. Press Y/+.
Back
Y
M

Change Alarm Mode. Your choices are Auto Reset and Latched. A latched alarm stays in alarm until you acknowledge the alarm by pressing a button. An auto-reset alarm turns off when the condition that set off the alarm is no longer present (for instance, a high H2S reading that exceeds the preset threshold and triggers an alarm, but then lowers below that threshold, turning the alarm off).

1. Press [MODE] to step from Save to Quit to the up or down arrow.
2. Choose Auto Reset or Latched by pressing [Y/+].
3. Press [MODE] to step to Save or Quit.
4. Press [Y/+] to commit your change or to exit to the next menu item.

Change Real Time Clock. Date (month, day, year) and time (hour, minute, second) are adjustable. Time is in 24-hour mode.

1. Press [MODE] to step from Save and Quit and to advance through the digits.
2. Press [Y/+] to advance from 0 through 9. If you press [Y/+] when 9 is selected, it "wraps" around to 0 and begins counting up again each time you press [Y/+].
3. Press [MODE] to step to Save or Quit.
4. Press [Y/+] to commit your change or to exit to the next menu item.

Change Backlight Mode. In **Manual Turn On**, the backlight can be toggled on and off manually by pressing and holding **[Y/-]** for one second. In **Auto Turn On**, the backlight switches on automatically when the ambient light level exceeds a threshold. See Setting Backlight Mode on page 17 for details on setting the turn-on/turn-off threshold.

1. Press [MODE] to step from Save to Quit to the up or down arrow.
2. Choose Manual Turn On or Auto Turn On by pressing [Y/+].
3. Press [MODE] to step to Save or Quit.
4. Press [Y/+] to commit your change or to exit to the next menu item.

Change Password. You can change the 4-digit password from its default of 0000.

1. Press [MODE] to step from Save and Quit and to advance through the digits.
2. Press [Y/+] to advance from 0 through 9. If you press [Y/+] when 9 is selected, it "wraps" around to 0 and begins counting up again each time you press [Y/+].
3. Press [MODE] to step to Save or Quit.
4. Press [Y/+] to commit your change or to exit to the next menu item.

Important! If you change the password, write down the new password and save it.

Change Averaging Method. Toggle between Running Average and TWA (time-weighted average).

1. Press [MODE] to step from Save to Quit to the up or down arrow.
2. Choose Running Average or TWA by pressing [Y/+].

3. Press [MODE] to step to Save or Quit.
4. Press [Y/+] to commit your change or to exit to the next menu item.

Change Display Language. Scroll from English to other languages (language options depend on firmware version).

1. Press [MODE] to step from Save to Quit to the up or down arrow.
2. Choose a language by pressing [Y/+].
3. Press [MODE] to step to Save or Quit.
4. Press [Y/+] to commit your change or to exit to the next menu item.

Set Temperature Unit. Select Fahrenheit or Celsius.

1. Press [MODE] to step from Save to Quit to the up or down arrow.
2. Choose Fahrenheit or Celsius by pressing [Y/+].
3. Press [MODE] to step to Save or Quit.
4. Press [Y/+] to commit your change or to exit to the next menu item.

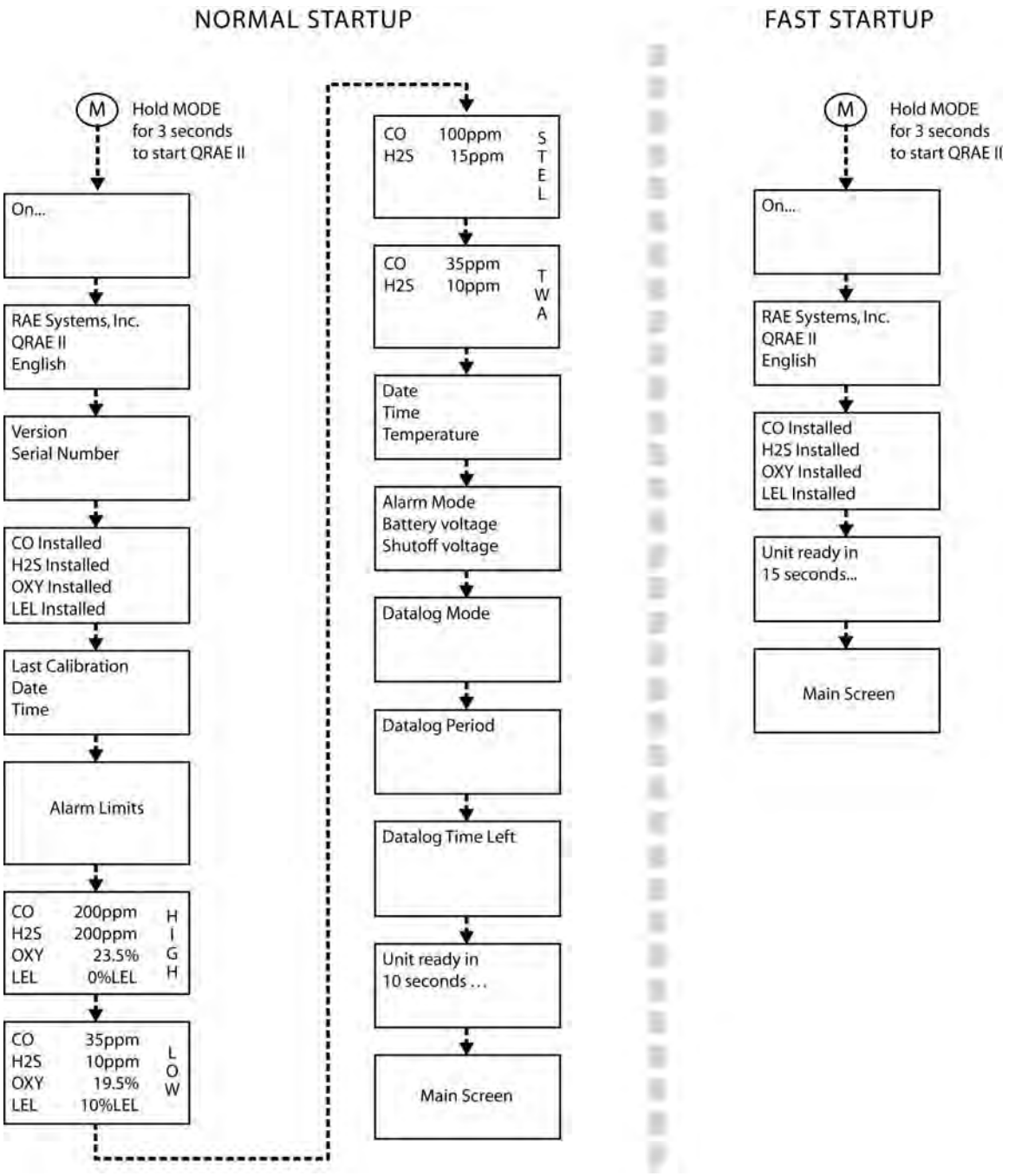
Enable/Disable Run Silent. You can select Run Silent Enabled or Run Silent Disabled modes.

1. Toggle enabled or disabled by pressing [Y/+].
2. Press [MODE] to save your choice and to exit to the next menu item.

Change Startup Mode. Options are Normal Startup and Fast Startup. Fast Startup skips showing you many settings and is best suited to environments where the QRAE II is turned on and off very often during a given day.

1. Press [MODE] to step from Save to Quit to the up or down arrow.
2. Choose Normal Startup or Fast Startup by pressing [Y/+].
3. Press [MODE] to step to Save or Quit.
4. Press [Y/+] to commit your change or to exit to the next menu item.

The diagram below shows the difference between the two startup modes:

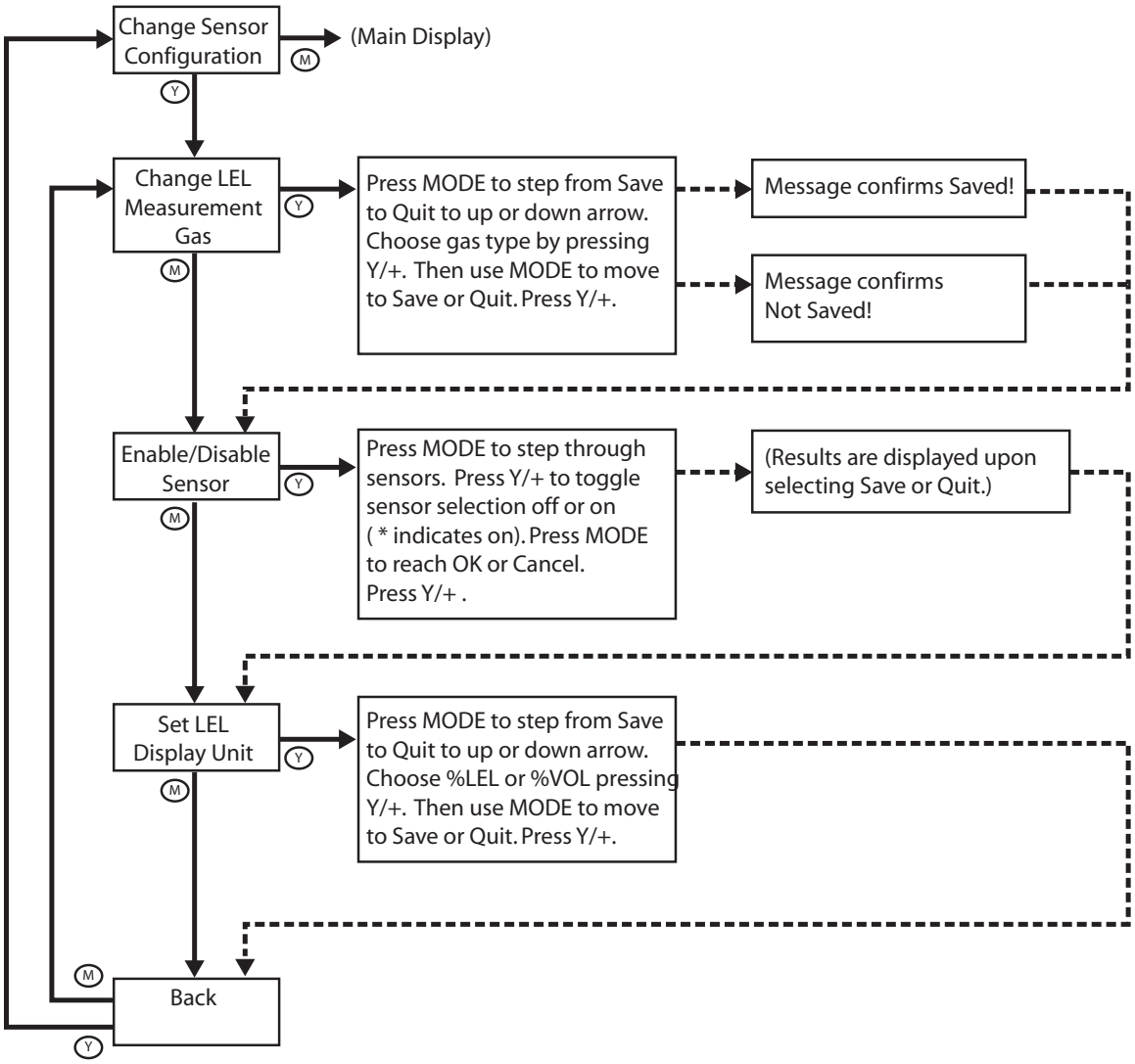


Back. Press [MODE] to return to the top of the Change Monitor Setup menu, or press [Y/+] to return to the top of the Normal Mode menu.

Change Sensor Configuration

You can select which sensors are enabled/disabled, and change the type of LEL measurement gas and unit of display for LEL in this set of submenus.

The following diagram shows how Change Sensor Configuration's menus are organized and how to navigate them:



Change LEL Measurement Gas. This function allows selection of the measurement gas for the LEL sensor. The correction for the measurement gas is automatically divided by the correction factor the span gas, selected previously, to obtain a new factor for the combination of gases. The new factor is applied to the readings to obtain a true concentration.

1. Press [MODE] until the up or down arrow is highlighted.
2. Press [Y+] to move through the list of LEL measurement gases.
3. Press [MODE] to select either the other arrow or to advance to Save or Quit.
4. With Save selected, press [Y/+] to save your settings, or [MODE] to advance to Quit (without saving settings).

If you choose to quit without saving the changes, press [Y/+]. You will see this message:

Not Saved!

If you want to make further changes to the settings, press [MODE] to repeat stepping through the choices.

Enable/Disable Sensor. This function allows you to selectively enable or disable individual sensors in the QRAE II. When a sensor is disabled, the unit does not datalog or display gas concentrations of that type.

8. Select a sensor to enable/disable by pressing [MODE] until the sensor's name is highlighted.
9. Press [Y/+] to toggle the selection on (indicated by an asterisk, *) or off (no asterisk).

   To select other sensors to calibrate, press [MODE] until you reach the sensor you want to select. Then press [Y/+].

After you have selected all the sensors you want enabled or disabled:

10. Press [MODE] to advance to Save or Cancel.
11. With Save selected, press [Y/+] to save your settings, or [MODE] to advance to Cancel (without saving settings).

Set LEL Display Unit. Choose from %LEL for percentage of lower explosive limit or %VOL for volume percent.

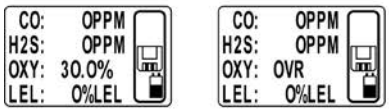
1. Press [MODE] to step from Save to Quit to the up or down arrow.
2. Choose %LEL or %VOL by pressing [Y/+].
3. Press [MODE] to step to Save or Quit.
4. Press [Y/+] to commit your change or to exit to the next menu item.

Back. Press [MODE] to return to the top of the Change Sensor Configuration menu, or [Y/+] to exit the Change Sensor Configuration menu and return to the Programming Mode menu.

Alarm Signal Summary

Note: Backlight automatically turns on when QRAE II alarms.

Over Range



Over Range:
Buzzer, LED: 3 times/second
Vibrator: 400ms/second

High Alarm

High Alarm:
Buzzer, LED: 3 times/second
Vibrator: 400ms/second

Low Alarm

Low Alarm:
Buzzer, LED: 2 times/second
Vibrator: 400ms/second

STEL Alarm

STEL Alarm:
Buzzer, LED: 1 time/second
Vibrator: 400ms/second

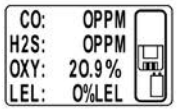
TWA Alarm

TWA Alarm:
Buzzer, LED: 1 time/second
Vibrator: 400ms/second

Negative Drift

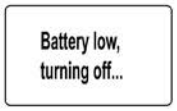
Negative Drift:
Buzzer, LED: 1 time/second
Vibrator: 400ms/second

Battery Low



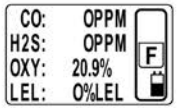
Battery Low:
Buzzer, LED: 1 time/minute
Vibrator: 400ms/minute

Battery Dead



Battery Dead:
LCD display "Battery low, turning off..."
Unit turns off after 15 seconds

Datalog Full



Datalog Full:
Buzzer, LED: 1 time/second
Vibrator: 400ms/second

Bump Testing & Calibrating The QRAE II

RAE Systems recommends that a bump test be performed on the QRAEII before the monitor is put into service, and later, prior to each use. A bump test is defined as a brief exposure of the monitor to the calibration gas and the sensors to show response and trigger the lowest alarm set point for each sensor.

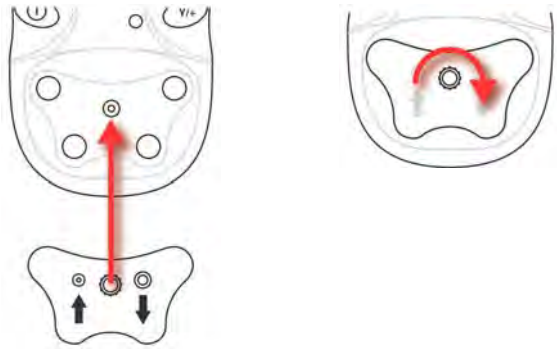
- The QRAEII Multi Gas detector must be calibrated if it does not pass a Bump Test, or at least once every 180 days, depending on use and sensor exposure to poisons and contaminants.
- Calibration intervals and bump test procedures may vary due to national legislation.
- RAE Systems recommends using RAE calibration gas cylinders with a 4 gas mix containing 10 ppm H2S, 50 ppm CO, 50% LEL Methane, and 18.5% Oxygen.

Note: Bump test and calibration can also be performed by using the RAE Systems AutoRAE™ docking station.

Connecting The Calibration Adapter

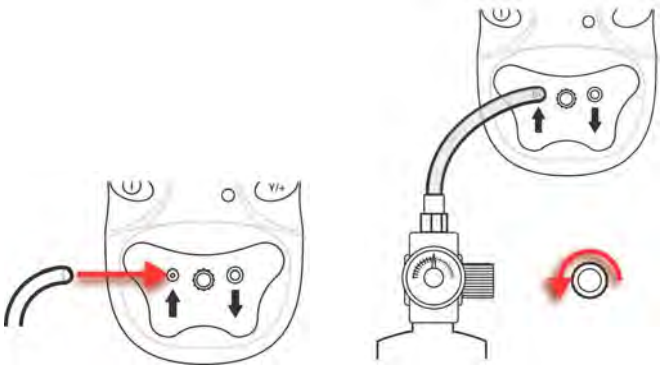
The QRAE II is a diffusion monitor and must be calibrated using a fixed-flow regulator with a flow rate between 0.5 and 1.0 liters per minute. The QRAE II is supplied with a special calibration adapter that covers the gas diffusion ports.

1. Place the Calibration Adapter over the filter area on the front of the QRAE II.
2. Finger-tighten the screw at the center, as shown:



Note: Finger-tighten this only. Do not use a pliers or other tools. The Calibration Adapter should be snug, but an air-tight seal is unnecessary.

3. Attach the hose from the regulator/calibration gas cylinder to the Calibration Adapter's inlet.



Caution! After calibration is complete, remove the Calibration Adapter. When monitoring, never operate the QRAE II with the Calibration Adapter attached. The QRAE II's sensors operate by diffusion. If the Calibration Adapter is attached during normal operation, inconsistent and lower-than-normal readings will occur because of decreased concentration of the gas being monitored.

Disconnecting The Calibration Adapter

After calibrating the QRAE II, you must remove the Calibration Adapter.

Sensor Calibration Time

The QRAE II is designed to automatically use a predetermined calibration time. Never operate the QRAE II with the Calibration Adapter attached

To disconnect the Calibration Adapter:

1. Loosen the finger-screw at the center of the Calibration Adapter.

2. Lift the Calibration Adapter from the QRAE II and store it.

WARNING!

The calibration of any newly purchased RAE Systems instrument should be tested by exposing the sensor(s) to known concentration calibration gas(es) before the instrument is put into service the first time. For maximum safety, the accuracy of the QRAE II should be checked by exposing the sensor(s) to known concentration calibration gas(es) before each day's use.

Zero (Fresh Air) Calibration

This procedure determines the zero point of the sensor calibration curve. Expose the inlet to a clean air source with 20.9% oxygen and without any organic, toxic or combustible gas impurities. This "Zero Air" can be from a cylinder, clean ambient air, or ambient air purified through a charcoal filter.

Using Fresh Air

Caution! Do not use the Calibration Adapter while performing zero calibration in fresh air.

Turn on the QRAE II and follow this procedure:

1. Enter Program Mode.
2. At Calibrate Monitor, press [Y/+].

You should see:

Fresh Air Calibration?

3. Press Y+ to start calibration. Calibration is performed automatically.

Using Zero Gas

1. Attach the Calibration Adapter to the QRAE II.
2. Attach the regulator to the Zero Gas cylinder.
3. Attach a hose to the Calibration Adapter and to the regulator.
4. Enter Password.
5. At Calibrate Monitor, press [Y/+].
6. When you see Fresh Air Calibration? turn on the regulator to start the Zero Gas flow.
7. Press [Y/+].

You should see a countdown from 30 seconds:

Calibration
in progress ... 30

When calibration is complete, you will see the screen change between these two:

CO:	**Zeroed**		**CO:**	**0ppm**
H2S:	**Zeroed**	**and**	**H2S:**	**0ppm**
OXY:	**Zeroed**		**OXY:**	**20.9%**
LEL:	**Zeroed**		**LEL:**	**0%LEL**

When the zero calibration is successful, the display should show a reading of "20.9" for the oxygen sensor and "0" for all other sensors.

Multiple Sensor Calibration

This function simultaneously determines the second point of the calibration curve for multiple sensors in the monitor. The display counts down from 60 to 0 seconds, and then shows the name of each sensor, whether it passed or failed calibration, and the calibrated value for each sensor.

Select a sensor to calibrate by pressing [MODE] until the sensor's name is highlighted. Then press [Y/+] to toggle the selection on (indicated by an asterisk, *) or off (no asterisk). To select other sensors to calibrate, press [MODE] until you reach the sensor you want to select. Then press [Y/+].

After you have selected all the sensors you want calibrated, it is time to perform the calibration.

1. Attach the Calibration Adapter to the QRAE II.
2. Attach the regulator to the Calibration Gas cylinder.
3. Attach a hose to the Calibration Adapter and to the regulator.
4. Turn on the regulator to start the Calibration Gas flow.
5. Press [MODE] until OK is highlighted.
6. Press [Y/+] to start calibration.

You should see a countdown from 60 seconds:

Calibration
in progress ... 60

Note: If you see the following message, check that the gas is flowing and the hose is attached:

No Gas Flow... Apply gas or hit any key to start.

When calibration is complete, you will see the screen change between these two:

Multiple Sensor Calibrated!

and

CO = Pass
H2S = Pass
O2 = Pass
LEL = Pass

A sensor calibration failure is indicated like this:

CO = Fail
H2S = Pass
O2 = Pass
LEL = Pass

If a sensor fails, try calibrating again. If calibration fails again, replace the sensor.

Single Sensor Calibration

This procedure determines the second point of the sensor calibration curve for a single sensor. The display counts down from 60 to 0 seconds, and then shows the name of each sensor, whether it passed or failed calibration, and the calibrated value for each sensor.

Select the sensor to calibrate by pressing [MODE] until the sensor's name is highlighted. Then press [Y/+] to toggle the selection on (indicated by an asterisk, *) or off (no asterisk).

After you have selected the sensor you want calibrated, it is time to perform the calibration.

1. Attach the Calibration Adapter to the QRAE II.
2. Attach the regulator to the Calibration Gas cylinder.

   Important! Make sure you have the correct calibration gas in the correct concentration. Also make sure that the calibration gas is not beyond its "Best when used by" date, which is stamped on the cylinder's label.

3. Attach a hose to the Calibration Adapter and to the regulator.
4. Turn on the regulator to start the Calibration Gas flow.
5. Press [MODE] until OK is highlighted.
6. Press [Y/+] to start calibration.

You should see a countdown from 60 seconds:

Calibration
in progress ... 60

Note: If you see the following message, check that the gas is flowing and the hose is attached:

No Gas Flow... Apply gas or hit any key to start.

When calibration is complete, you will see the screen change between these two:

Multiple Sensor Calibrated!

and

CO = Pass
H2S = Pass
O2 = Pass
LEL = Pass

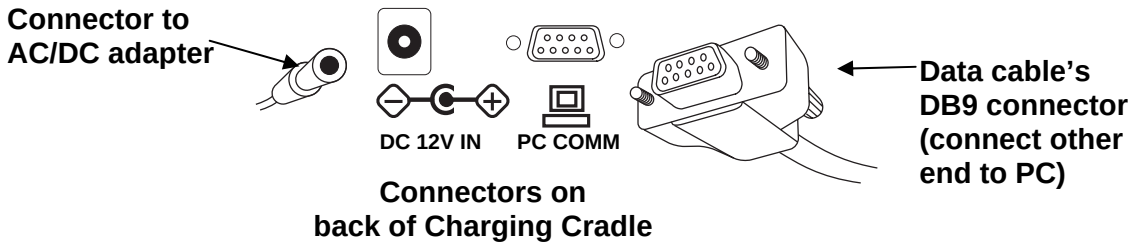
If the sensor calibration passes, then you should see this message (shown here for carbon monoxide, CO):

CO calibration pass!

If the sensor calibration fails, try calibrating again. If calibration fails again, replace the sensor.

Connecting To A Computer

Downloading datalog data to computer requires placing the QRAE II in its cradle and connecting the cradle to a computer.



The QRAE II's cradle connects via a DB9 (9-pin) connector to a computer running ProRAE Remote software.

Press the DB9 connector into the DB9 serial port on the cradle, and then finger-tighten the two screws on the connector. These keep the connector from coming loose and ensure the best electrical connection. Make sure the AC/DC adapter is connected to the cradle, as well, to power the QRAE II and keep its battery from draining.

Note: If your computer has a USB port instead of a DB9 connector, use a USB to 9-pin serial adapter (part number 410-0210-000).

Transferring Data To & From A Computer

Once you have connected your QRAE II cradle to the PC, you can can transfer data, including a download of the datalog to the computer and updates of firmware to the QRAE II (should this ever be necessary).

Downloading The Datalog To A PC

1. Connect the data cable to the PC and the cradle.
2. Place the QRAE II into its cradle. The charging LED should be illuminated.
3. Start ProRAE Studio on your PC.
4. From ProRAE Studio, select "Operation" and select Setup Connection.
5. Select the COM port to establish a communication link between the PC and the QRAE II.
6. To receive the datalog in the PC, select "Downlog Datalog."
7. When you see "Unit Information," click OK.

The download process typically takes less than 10 seconds. During the data transfer, the display shows a progress bar.

When the transfer is done, you will see a screen with the datalog information. You can now export this datalog for other use or printing.

Uploading Firmware To The QRAE II From A PC

Uploading new firmware to your QRAE II requires connecting the QRAE II and PC. Follow these steps to make the connection:

1. Connect the data cable to the PC and the cradle.
2. Place the QRAE II into its cradle. The charging LED should be illuminated.
3. Start ProRAE Studio on your PC.
4. From ProRAE Studio, select "Operation" and select Setup Connection.
5. Select the COM port to establish a communication link between the PC and the QRAE II.

Once communication is established, follow the instructions that accompany ProRAE Studio and the firmware to upload the new firmware to your QRAE II.

Maintenance

⚠ WARNING! ⚠

Maintenance should be performed only by a qualified person who has proper training and fully understands the contents of this manual.

The following guidelines should be followed when changing components:

1. Turn off the unit and unplug the charger before changing a battery.
2. When changing a sensor, detach the battery first. Identify the location of a specific sensor and pay attention to the sensor pin orientation when removing and plugging in sensors.

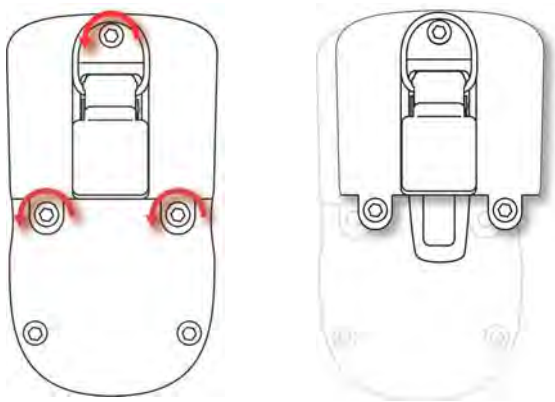
WARNING

To reduce the risk of ignition of hazardous atmospheres, recharge battery only in area known to be non-hazardous. Remove and replace battery only in an area known to be non-hazardous.

Replacing the QRAE II Li-Ion Battery

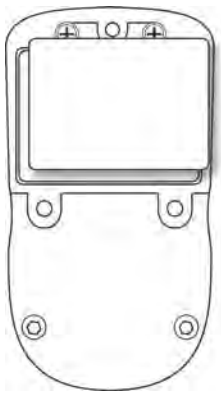
Caution: Turn off the QRAE II before removing or replacing the battery.

1. Place the QRAE II face-down on a soft surface.
2. Use the hex wrench to loosen each of the three screws by turning them counterclockwise.

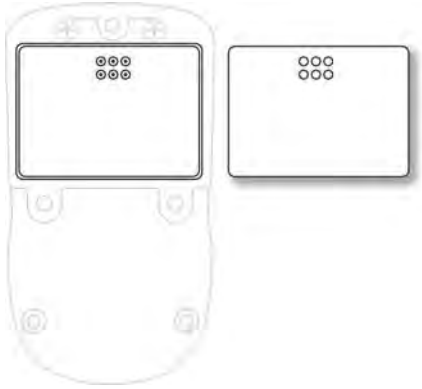


3. Hold the QRAE II down and lift the compartment cover by pulling on the belt clip.

4. Turn the QRAE over and slide the battery out of its compartment.



5. Place the new battery into the compartment with its six gold-plated contacts aligned over the six pins.



Note: Before installing the new battery, visually inspect the contacts to make sure they are clean. If they are not, wipe them with a soft cloth. Do not use solvents or cleaners.

6. Place the cover over the compartment.
7. Tighten all three screws by turning them clockwise with the hex wrench.

Installing The Alkaline Battery Adapter

The Alkaline Battery Adapter substitutes for the Li-Ion battery. The adapter (part number 020-3403-000) accepts three AA alkaline batteries (use only Duracell MN1500 or Energizer E91) to provide approximately 12 hours of operation.

2. Place the QRAE II face-down on a soft surface.
3. Use the hex wrench to loosen each of the three screws by turning them counterclockwise.
4. Hold the QRAE II down and lift the compartment cover by pulling on the belt clip.
5. Turn the QRAE over and slide the Li-ion battery out of its compartment.

6. Install three AA alkaline batteries into the Alkaline Battery Adapter, making sure the battery polarity is correct.
7. Place the Alkaline Battery Adapter into the compartment with its six gold-plated contacts aligned over the six pins.
8. Place the cover over the compartment.
9. Tighten all three screws by turning them clockwise with the hex wrench.
10. The monitor automatically detects the alkaline batteries.

IMPORTANT!

Alkaline batteries cannot be recharged. The QRAE II's internal circuit detects alkaline batteries and will not allow recharging. If you place the QRAE II in its cradle, the alkaline battery will not be recharged, and you will see this icon:

Note: When replacing alkaline batteries, dispose of old ones properly.

WARNING!

To reduce the risk of ignition of hazardous atmospheres, recharge the battery only in areas known to be non-hazardous. Remove and replace the battery only in areas known to be non-hazardous.

Charging A Spare QRAE II Li-Ion Battery

A QRAE II Li-ion battery can be charged when it is not inside the QRAE II. The charging cradle is designed to accommodate both types of charging. Contacts on the bottom of the QRAE II battery meet the contacts on the cradle, transferring power without other connections, and two clips hold the battery in place during charging.

1. Plug the AC/DC adapter into the QRAE II's cradle.
2. Place the battery into the cradle, with the six gold-plated contacts on top of the six matching charging pins.
3. Plug the AC/DC adapter into the wall outlet.

The QRAE II battery begins charging automatically. During charging, the LED in the cradle glows red. When charging is complete, it glows green.

Release the battery from the cradle by pressing the keys on both sides of the cradle and lifting the battery out of its slot.

Note: If you need to replace the 3.7V Li-ion battery pack, replacements are available from RAE Systems. The part number is 020-3402-000.

Replacing Sensors

Under normal operating conditions, sensors lose their original sensitivity after the expected operating life and need to be replaced.

Warranties: The oxygen (O_2), combustible gas (LEL), hydrogen sulfide (H_2S) and carbon monoxide (CO) sensors all have a 2-year warranty.

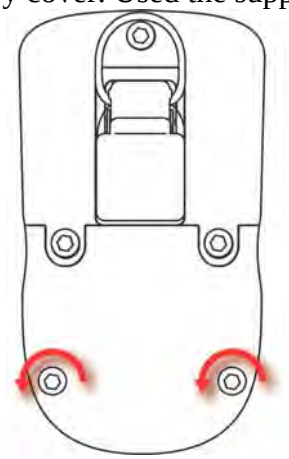
Replace a sensor when it fails to calibrate. If it fails to calibrate, retry calibrating it. Check the QRAE II's parameter settings, and check that the calibration gas is not beyond its "Use by" date. For further details, refer to RAE Systems Technical Note TN-123, "Special Diagnostic Modes For RAE Systems Instruments," available at www.raesystems.com.

The sensors are located inside the front of the monitor.

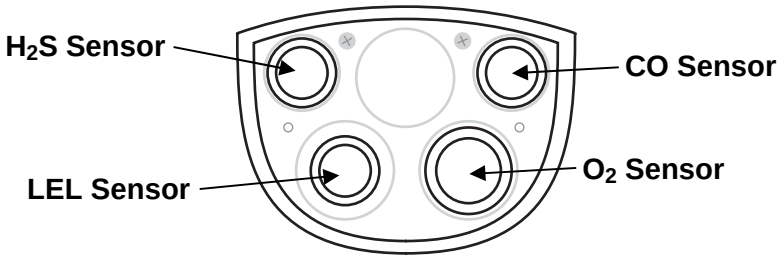
See RAE Systems Technical Note TN-114, Sensor Specifications And Cross-Sensitivities, for additional information, available at www.raesystems.com.

To replace a sensor:

1. Turn off the QRAE II.
2. Place the QRAE II face-down on a soft surface.
3. Remove the front sensor cover by loosening the two screws on the back of the monitor below the battery cover. Used the supplied hex wrench.



4. Push the screws from the back of the monitor to pop the cover off the front.
5. Locate the sensor you need to replace.



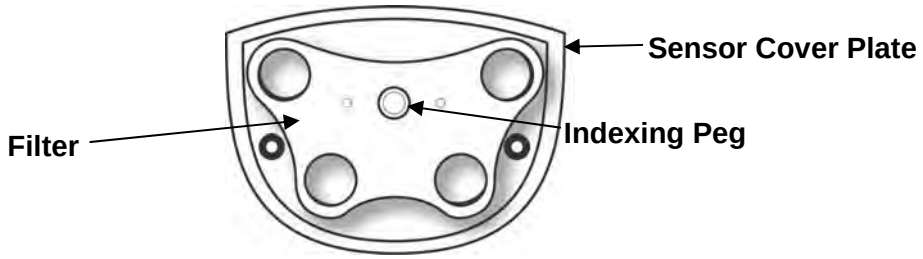
6. Using the sensor puller, carefully remove the sensor by pulling it straight out.
7. Replace the sensor with a new one. Make sure the pins are not bent or corroded. Align the pins to the corresponding holes and push straight in. Do not push on the middle of the sensor with your finger, as this may damage it. The sensor should fit flush against the printed circuit board.

   Note: Each sensor socket is labeled to guide you in placing the correct sensor into it. If a sensor does not seem to fit, examine it to ensure that it is the correct sensor, the pins are not bent, and that the pins are aligned to the holes.

8. Press the sensors all the way into the socket.

   Important! Sensors are not interchangeable. Use only RAE Systems sensors, and use only the sensor type specified for your QRAE II monitor. Use of non-RAE Systems components will void the warranty and can compromise the safe performance of this product.

9. Inspect the filter. If it is dirty, discolored, or wet, replace it. See Replacing The Filter, below.



10. Place the filter onto the inside of the sensor cover. Make sure it is aligned properly by matching its center hole with the indexing peg at the middle of the cover.
11. Replace the sensor cover. Hold the cover against the QRAE II and place the QRAE II on a soft surface. Use the hex wrench to tighten the two screws. Do not overtighten them.
12. Turn the monitor on and the newly installed sensors should be properly identified by the EntryRAE in the start-up screen. Let the monitor run for 15 minutes before calibration.
13. Calibrate all sensors prior to using the QRAE II.

Replacing The Filter

The filter is made of PTFE (Teflon®) membrane designed to prevent water and dust from entering the sensors. The filter should be replaced if it changes color, traps dust or other particulates, or if it has been saturated with water or other liquids. Use only the filter with part number 020-2419-000.

Cleaning

Occasional cleaning with a soft cloth is recommended. Do not use detergents or chemicals. Do not submerge the QRAE II or place it under running water. If necessary, you can use a damp cloth (water only). It is a good idea to install the Calibration Adapter before cleaning the QRAE II's housing, to keep dirt, dust, or moisture away from the sensor openings and to keep the filter clean.

Visually inspect the contacts at the base of the QRAE II, on the battery, and on the Charging cradle to make sure they are clean. If they are not, wipe them with a soft, dry cloth. Never use solvents or cleaners.

Firmware Updates

Firmware is occasionally updated for RAE Systems products. Check the RAE Systems website for updates:

http://www.raesystems.com

Ordering Replacement Parts

If you need replacement parts, contact your local RAE Systems distributor. A list is available online:

http://www.raesystems.com

In the U.S., you can order sensors, replacement batteries, and other accessories online at:

http://istore.raesystems.com/

Troubleshooting

Problem	Possible Reasons & Solutions	
Cannot turn on power after charging the battery	**Reasons:**	Discharged battery. Defective battery.
	Solutions:	Charge or replace battery.
No LCD back light	**Reasons:**	Trigger level too low, the current mode is not user mode, and the mode does not support automatic turn on back light.
	Solutions:	Adjust trigger level. Verify the back light can be turned on. Verify that the backlight is not in manual mode. Call authorized service center.
Lost password	**Solutions:**	Call Technical Support at +1 408-752-0723 or toll-free at +1 888-723-4800
Reading abnormally Low	**Reasons:**	Dirty filter. Calibration Adapter is attached. Incorrect calibration.
	Solutions:	Replace filter. Remove Calibration Adapter. Calibrate the QRAE II.
Buzzer Inoperative	**Reasons:**	Bad buzzer.
	Solutions:	Check that buzzer is not turned off. Call authorized service center.

Technical Support

To contact RAE Systems Technical Support Team:

Monday through Friday, 7:00AM to 5:00PM Pacific (US) Time
Phone (toll-free): +1 888-723-4800
Phone: +1 408-952-8461
Email: tech@raesystems.com

Life-critical after-hours support is available:

+1 408-952-8200 select option 8

RAE Systems Contacts

RAE Systems
World Headquarters
3775 N. First St.
San Jose, CA 95134-1708 USA
Phone: +1 408.952.8200
Fax: +1 408.952.8480

E-mail: customerserv@raesystems.com
Web Site: www.raesystems.com

RAE Systems Technical Support
Monday through Friday, 7:00AM to 5:00PM Pacific Time
Phone: +1.408.952.8461
Email: tech@raesystems.com

Life-critical after-hours support is available
+1.408.952.8200 select option 9

RAE Systems Europe ApS
Ørestads Boulevards 69
2300 Copenhagen S
Denmark
Phone: +45 86525155
Fax: +45 86525177
orders@raeeurope.com
sales@raeeurope.com
service@raesystems.com
Web: www.raesystems.dk

RAE Systems UK Ltd
D5 Culham Innovation Centre
Culham Science Centre
Abingdon, Oxon OX14 3DB
United Kingdom
Phone: +44 1865408368
Fax: +44 1235531119
Mobile: +44 7841362693
Email: raeuk@raeeurope.com

RAE Systems France
336, rue de la fée des eaux
69390 Vernaison
France
Phone: +33 4 78 46 16 65
Fax: +33 4 78 46 25 98
Email: info-france@raeeurope.com
Web: www.raesystems.fr

RAE BeNeLux BV
Rijndal 20
2904 DC Capelle a/d IJssel
Phone: +31 10 4426149
Fax: +31 10 4426148
Email: info@rae.nl
Web: www.rae.nl

RAE Systems Spain, s.l.
Av. Remolar, 31
08820 El Prat de Llobregat
Spain
Phone: +34 933 788 352
Fax: +34 933 788 353
Mobile: +34 687 491 106
Email: mdelgado@raespain.com
Web: www.raespain.com

RAE Systems Middle East
Mobile: +45 2674 9791
or +97 50 429 1385
Email: mjorgensen@raesystems.com

RAE Systems (Hong Kong) Ltd.
Room 8, 6/F, Hong Leong Plaza
33 Lok Yip Road

Fanling, N.T, Hong Kong
Phone: +852.2669.0828
Fax: +852.2669.0803
Email: hksales@raesystems.com

RAE Systems Japan
403 Plaza Ochanomizu Bldg. 2-1
Surugadai Kanda Chiyoda-Ku
Tokyo, Japan
Phone: 81-3-5283-3268
Fax: 81-3-5283-3275
Email: jpsales@raesystems.com

RAE Systems Korea
#1010, DaeMyungAnsVill First,
Sang-Dong 412-2, Wonmi-Gu, Bucheon,
Kyungki-Do, Korea
Phone: 82-32-328-7123
Fax: 82-32-328-7127
Email: krsales@raesystems.com

Appendix A: Regulatory Information

Intrinsic Safety:	CSA Class 1, Division I, Group A, B, C, D, T4 (US & Canada), KEMA ATEX II 2G EEx d ia II C T4 (Europe)
EM Immunity:	No effect when exposed to 0.43mW/cm^2 RF interference (5-watt transmitter at 12"/10cm).
Temperature:	-20° C to 50° C (-4° F to 122° F)
Humidity:	0% to 95% relative humidity (non-condensing)

Caution:

Refer to RAE Systems Technical Note TN-114 for sensor cross-sensitivities.
Refer to RAE Systems Technical Note TN-144 for LEL sensor poisoning.

Basic Operation

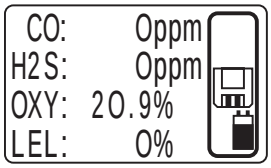
Turning The QRAE II On

To turn the QRAE II on, hold down [MODE] for 2 seconds.

The QRAE performs self-testing and the screen shows a countdown to full operational functionality. If Datalog is on, this message is displayed after the countdown:

Datalog Started

When the QRAE II is ready for use, it shows this screen:



Note: If datalogging is off, you will not see the datalogging icon (see icons, page 10).

Turning The QRAE II Off

Press and hold [MODE]. In 2 seconds, a 5-second countdown to shutoff begins. You must hold your finger on the key for the entire shutoff process. If you remove your finger from the key during the countdown, the shutoff operation is canceled and the QRAE II continues normal operation.

The countdown proceeds as follows, accompanied at each step with an alarm beep and light flash.

When you see "Unit off..." release your finger from the [MODE] key. The QRAE II is now off.

Inverting The Display

To flip the screen, press the [Y/+] key and hold it down for 3 seconds. When the image inverts, release the key.

Testing The Alarms (Anytime)

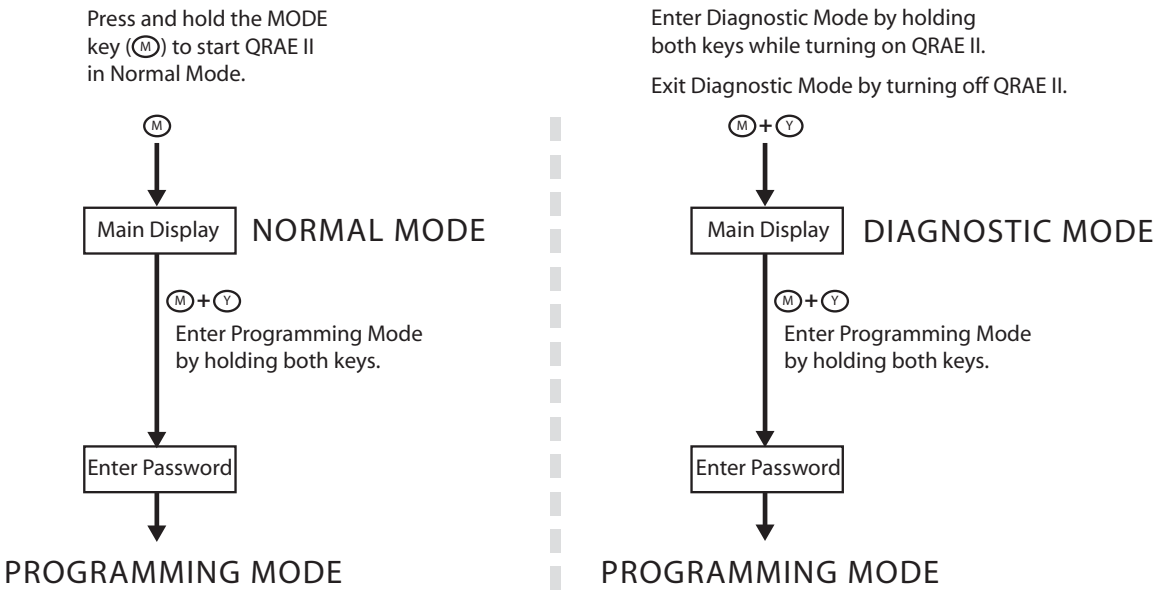
Under normal non-alarm conditions, the buzzer, vibration alarm, LED, and backlight can be tested at any time by pressing [Y/+] once.

Programming Mode

Enter Programming Mode from Normal Mode or Diagnostic Mode. This mode contains most adjustable settings for the QRAE II. It is organized into five submenus:

- Calibrate Monitor
- Change Alarm Limits
- Change Datalog
- Change Monitor Setup
- Change Sensor Configuration

The following diagram shows how to enter Programming Mode from Normal Mode and from Diagnostic Mode:



Change Alarm Mode. Your choices are Auto Reset and Latched. A latched alarm stays in alarm until you acknowledge the alarm by pressing a button. An auto-reset alarm turns off when the condition that set off the alarm is no longer present (for instance, a high H2S reading that exceeds the preset threshold and triggers an alarm, but then lowers below that threshold, turning the alarm off).

5. Press [MODE] to step from Save to Quit to the up or down arrow.
6. Choose Auto Reset or Latched by pressing [Y/+].
7. Press [MODE] to step to Save or Quit.
8. Press [Y/+] to commit your change or to exit to the next menu item.

Alarm Signal Summary

Note: Backlight automatically turns on when QRAE II alarms.

Over Range

Over Range:
Buzzer, LED: 3 times/second
Vibrator: 400ms/second

High Alarm

High Alarm:
Buzzer, LED: 3 times/second
Vibrator: 400ms/second

Low Alarm

Low Alarm:
Buzzer, LED: 2 times/second
Vibrator: 400ms/second

STEL Alarm

STEL Alarm:
Buzzer, LED: 1 time/second
Vibrator: 400ms/second

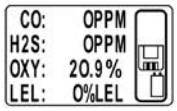
TWA Alarm

TWA Alarm:
Buzzer, LED: 1 time/second
Vibrator: 400ms/second

Negative Drift

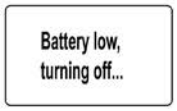
Negative Drift:
Buzzer, LED: 1 time/second
Vibrator: 400ms/second

Battery Low



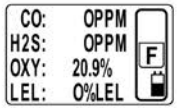
Battery Low:
Buzzer, LED: 1 time/minute
Vibrator: 400ms/minute

Battery Dead



Battery Dead:
LCD display "Battery low, turning off..."
Unit turns off after 15 seconds

Datalog Full



Datalog Full:
Buzzer, LED: 1 time/second
Vibrator: 400ms/second

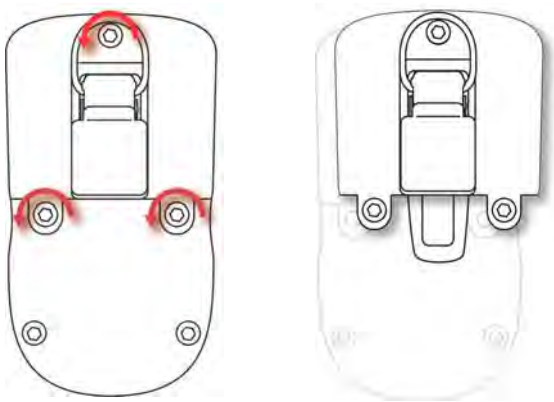
WARNING

To reduce the risk of ignition of hazardous atmospheres, recharge battery only in area known to be non-hazardous. Remove and replace battery only in an area known to be non-hazardous.

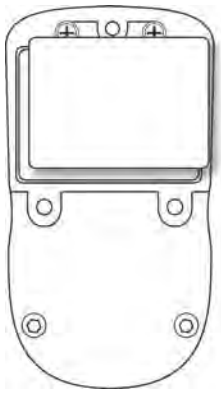
Replacing the QRAE II Li-Ion Battery

Caution: Turn off the QRAE II before removing or replacing the battery.

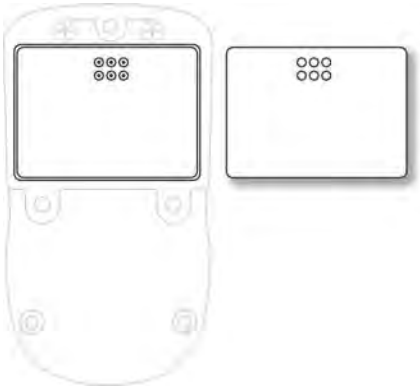
1. Place the QRAE II face-down on a soft surface.
2. Use the hex wrench to loosen each of the three screws by turning them counterclockwise.



3. Hold the QRAE II down and lift the compartment cover by pulling on the belt clip.
4. Turn the QRAE over and slide the battery out of its compartment.



5. Place the new battery into the compartment with its six gold-plated contacts aligned over the six pins.



Note: Before installing the new battery, visually inspect the contacts to make sure they are clean. If they are not, wipe them with a soft cloth. Do not use solvents or cleaners.

6. Place the cover over the compartment.
7. Tighten all three screws by turning them clockwise with the hex wrench.

Installing The Alkaline Battery Adapter

The Alkaline Battery Adapter substitutes for the Li-Ion battery. The adapter (part number 020-3403-000) accepts three AA alkaline batteries (use only Duracell MN1500 or Energizer E91) to provide approximately 12 hours of operation.

Place the QRAE II face-down on a soft surface.

1. Use the hex wrench to loosen each of the three screws by turning them counterclockwise.
2. Hold the QRAE II down and lift the compartment cover by pulling on the belt clip.
3. Turn the QRAE over and slide the Li-ion battery out of its compartment.
4. Install three AA alkaline batteries into the Alkaline Battery Adapter, making sure the battery polarity is correct.
5. Place the Alkaline Battery Adapter into the compartment with its six gold-plated contacts aligned over the six pins.
6. Place the cover over the compartment.
7. Tighten all three screws by turning them clockwise with the hex wrench.
8. The monitor automatically detects the alkaline batteries.

Replacing The Filter

The filter is made of PTFE (Teflon®) membrane designed to prevent water and dust from entering the sensors. The filter should be replaced if it changes color, traps dust or other particulates, or if it has been saturated with water or other liquids. Use only the filter with part number 020-2419-000.

Troubleshooting

Problem	Possible Reasons & Solutions
Cannot turn on power after charging the battery	**Reasons:** Discharged battery. Defective battery. **Solutions:** Charge or replace battery.
No LCD back light	**Reasons:** Trigger level too low, the current mode is not user mode, and the mode does not support automatic turn on back light. **Solutions:** Adjust trigger level. Verify the back light can be turned on. Verify that the backlight is not in manual mode. Call authorized service center.
Lost password	**Solutions:** Call Technical Support at +1 408-752-0723 or toll-free at +1 888-723-4800
Reading abnormally Low	**Reasons:** Dirty filter. Calibration Adapter is attached. Incorrect calibration. **Solutions:** Replace filter. Remove Calibration Adapter. Calibrate the QRAE II.
Buzzer Inoperative	**Reasons:** Bad buzzer. **Solutions:** Check that buzzer is not turned off. Call authorized service center.

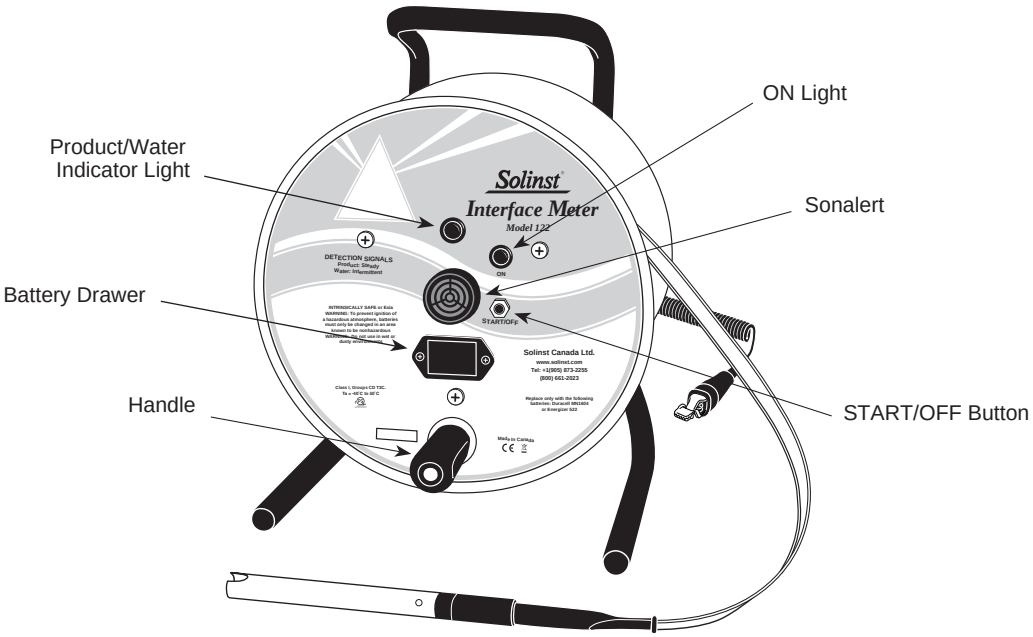
RAE Systems
World Headquarters
3775 N. First St.
San Jose, CA 95134-1708 USA
Phone: 408.952.8200
Fax: 408.952.8480

E-mail: customerserv@raesystems.com
Web Site: www.raesystems.com

P/N 020-4100-000 Rev. A
October 2006

Interface Meter Operating Instructions

Model 122



Operating Principle

The Solinst Model 122 Interface Meter has a narrow 5/8" (16 mm) diameter probe and uses laser-marked PVDF flat tape. It is certified to CSA Standards, for use in hazardous locations Class 1, Groups C & D. It has an infra-red circuit which detects the presence of a liquid. A conductivity circuit differentiates between conductive liquid (water) and non-conductive liquid (LNAPL or DNAPL product).

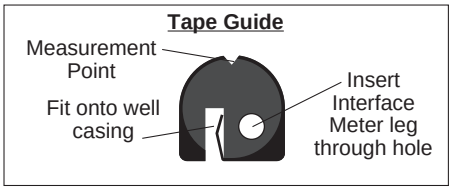
Equipment Check

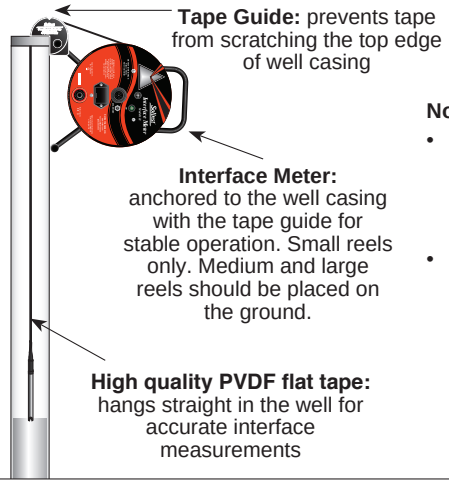
Before beginning any measurements, check the electronics and battery condition by pushing the 'START/OFF' button. A brief tone and red light indicate that the meter is functional.

A flashing green light indicates the meter is on. It will automatically turn off after 10 minutes to preserve battery life.

Using the Tape Guide

1. The tape guide has been designed to: improve accuracy when reading interface measurements; to prevent the laser-marked PVDF flat tape being cut by well casing; and to allow the tape and probe to hang straight from the side of the well.
2. If interface measurements are being taken in a 2" dia well then simply fit the small end of the tape guide onto the edge of the well casing.
3. The small end must be inserted if the Interface Meter is to be suspended from the tape guide.
4. Insert the leg of the Interface Meter into the hole on the Tape Guide (small reels only).
5. Once inserted, rest the Interface Meter on the side of the well casing.





Notes:

- When using the tape guide, the measuring point is offset from the top of casing
- To adjust your measurements, simply subtract the amount indicated on the front of the tape guide (eg. 6 cm or 2/10 ft.)

Field Measurements

IMPORTANT

1. To meet safety requirements and to protect the electronics from damage, always ground the meter by attaching the grounding clip to the metal well casing or to a suitable ground rod.

Continued overleaf...

2. Push the 'START/OFF' button. A brief tone and light indicate that the meter is functional and the flashing green light indicates that the meter is on. The meter automatically turns off after 10 minutes. Press the 'START/OFF' button as necessary during operation to turn the meter back on.
3. Place the slotted part of the tape guide onto the edge of the well casing. Lay the Interface Meter laser-marked PVDF flat tape into the groove on the top of the tape guide. Measurements will be read at the apex of the V-notch on the tape guide.

> **Note:** When using the tape guide, remember to subtract the compensation factor stamped onto the side of the guide from each measurement.

4. A steady tone and light indicates a non-conductive liquid (e.g. product). An intermittent tone and light indicates a conductive liquid (e.g. water).
5. For floating product (LNAPL), take the air/product interface measurement on the way into the liquid, and the water/product interface on the way up. When passing through product into water, some product may adhere to the probe sensors due to surface tension. Therefore, when water is detected below product, the probe should be raised and lowered rapidly in a short vertical motion to remove any product that may have been carried down with the probe.
6. The water/product interface should then be measured as the probe is raised very slowly back up. Once the interface is detected the probe can be raised and lowered in small increments to precisely determine the interface.
7. Repeat measurements to confirm water/product interface.
8. To determine the thickness of product, subtract the water/product interface from the product/air interface.
9. To determine if there is any sinking product (DNAPL) in the well, continue lowering the probe slowly. If steady signals activate, determine the top of the sinking layer by reading directly from the PVDF flat tape. Continue lowering the probe slowly until the tape slackens when the well bottom is reached. Read the level directly from the PVDF flat tape and subtract one from the other to determine thickness.
10. Upon completion of readings clean the tape and probe; as described in the Cleaning and Maintenance section.

Cleaning and Maintenance

After each use, the laster-marked PVDF tape should be wiped clean and carefully rewound onto the reel.

The probe should be cleaned as follows:

- Wash probe thoroughly with a non-abrasive mild detergent. **DO NOT USE ANY SOLVENTS**. Use a soft cloth around the pins on the end of the probe to remove all product. Use the brush provided to remove all product from inner part of the probe.

  USE LUKE-WARM, NOT HOT WATER. DAMAGE TO THE PROBE MAY RESULT.
- Rinse probe thoroughly with distilled water, wipe dry.
- Return the probe to the holder.

Other suitable cleaning method:

- Steam clean the PVDF flat tape only.

Battery Replacement

Push the battery drawer in and up and then release. The battery drawer should eject slightly, allowing it to be pulled out. Replace the 9V alkaline battery.

Other General Tips:

1. The probe should be cleaned after each use.
2. Always use the grounding cable.
3. Do not drop probe: damage to probe tip may result.
4. If battery is weak, the start tone will not sound, and flashing "green" light will be off. Replace the 9V alkaline battery.
5. Before storage, make sure the meter is turned off. If the Interface Meter is going to be stored for longer than two months, the 9V alkaline battery should be removed to avoid potential leakage.
6. The meter can be checked by placing the probe in distilled (non-conductive) water or pure phase product, for example lamp oil (**avoid bright sunlight during testing and resting the probe on the bottom of the container**). A steady tone and light should be observed.

®*Solinst is a registered trademark of Solinst Canada Ltd.*

For further information contact: Solinst Canada Ltd.
Fax: +1 (905) 873-1992; (800) 516-9081 Tel: +1 (905) 873-2255; (800) 661-2023
35 Todd Road, Georgetown, Ontario Canada L7G 4R8
Web Site: www.solinst.com E-mail: instruments@solinst.com

Solinst®

CAT. NO. 46500-88

PORTABLE TURBIDIMETER

Model 2100P

Instrument and Procedure Manual

© Hach Company, 1991-2004, 2008.
All rights reserved. Printed in China.

4/08 9ed

TABLE OF CONTENTS

TABLE OF CONTENTS 3
CERTIFICATION 5
SAFETY PRECAUTIONS 7
SPECIFICATIONS 9

OPERATION 11

SECTION 1 DESCRIPTION 13
1.1 General Description 13
1.2 Accessories 14
1.3 Principle of Operation 14
1.4 Preparation for Use 15
1.4.1 Unpacking 15
1.4.2 Battery Installation 16
1.4.3 Using the Battery Eliminator and Rechargeable Batteries... 16
1.4.4 Calibration 16

SECTION 2 TURBIDITY MEASUREMENT 19
2.1 Operating Controls and Indicators 19
2.2 Turbidity Measurement 19
2.2.1 Turbidity Measurement Procedure 20
2.2.2 Measurement Notes 22
2.3 Measurement Techniques 22
2.3.1 Cleaning Sample Cells 23
2.3.2 Oiling the Sample Cell 23
2.3.3 Orienting Sample Cells 24
2.3.4 Matching multiple sample cells 26
2.3.5 Removing Bubbles (Degassing) 28
2.3.6 Measuring Overrange Samples 31
2.3.7 Condensation (fogging) 31
2.3.8 Calibration 31
2.3.9 Representative Sampling 32

TABLE OF CONTENTS, continued

SECTION 3 OPERATION 33
3.1 Operational Controls and Indicators 33
3.2 Using the Read Key 35
3.2.1 Continuous Reading 35
3.3 Using the Signal Averaging Key 35
3.4 Using the Range Selection Key 36
3.5 Restoring the Default Calibration 36
3.6 Calibration 37
3.6.1 StablCal Stabilized Formazin Standards 37
3.6.2 Formazin Primary Standards 40
3.6.3 Calibrating the Turbidimeter 44
3.6.4 Using Gelex® Secondary Turbidity Standards 54

MAINTENANCE 57

SECTION 4 MAINTENANCE 59
4.1 Cleaning 59
4.2 Battery Replacement 59
4.3 Lamp Replacement 59

SECTION 5 TROUBLESHOOTING 67
5.1 Using the Diagnostic Functions Key 67
5.1.1 Basic Diagnostic Codes 67
5.2 The Diagnostic Procedure 68
5.3 Other Instrument Diagnostics 70
5.3.1 Display Test 70
5.4 Error Messages 70
5.4.1 Flashing Numeric Display 70
5.4.2 E Messages 70
5.4.3 CAL? 70

GENERAL INFORMATION 73
Replacement Parts & Accessories 74
HOW TO ORDER 76
REPAIR SERVICE 77
WARRANTY 78

CERTIFICATION

Hach Company certifies this instrument was tested thoroughly, inspected and found to meet its published specifications when it was shipped from the factory.

The Model 2100P Portable Turbidimeter has been tested and is certified as indicated to the following instrumentation standards:

Product Safety

Battery/Eliminator Power Supply Only:
120 Vac, 60 Hz, UL Listed & CSA Certified, Class 2
230 Vac, 50 Hz, VDE Approved, GS & CE marked

Immunity

2100P Turbidimeter Tested with external Battery/Eliminator Power Supply:
EN 50082-1 (European Generic Immunity Standard) **per 89/336/EEC EMC:** Supporting test records with Dash Straus and Goodhue, Inc. (now Intertek Testing Services), certified compliance by Hach Company.

Standards include:
IEC 801-2 Electro-Static Discharge
IEC 801-3 Radiated RF Electro-Magnetic Fields
IEC 801-4 Electrical Fast Transients/Burst

Emissions

2100P Turbidimeter Tested with external Battery/Eliminator Power Supply:
EN 50081-1 (Emissions) **per 89/336/EEC EMC:** Supporting test records by Amador Corp. (now TUV Product Services), certified compliance by Hach Company

Standards include:
EN 55022 (CISPR 22) Emissions, Class B Limits

Canadian Radio Interference-Causing Regulation, Chapter 1374, Class A: Supporting test records by Amador Corp. (now TUV Product Services), certified compliance by Hach Company

This Class A digital apparatus meets all requirements of the Canadian Interference-Causing Equipment Regulations.

Cet appareil numérique de la classe A respecte toutes les exigences du Règlement sur le matériel brouilleur du Canada.

FCC Part 15, Class "A" Limits: Supporting test records by Amador Corp. (now TUV Product Services), certified compliance by Hach Company.

This device complies with Part 15 of the FCC Rules. Operation is subject to the following two conditions:

1. this device may not cause harmful interference, and
2. this device must accept any interference received, including interference that may cause undesired operation.

Changes or modifications to this unit not expressly approved by the party responsible for compliance could void the user's authority to operate the equipment.

This equipment has been tested and found to comply with the limits for a Class A digital device, pursuant to Part 15 of the FCC Rules. These limits are designed to provide reasonable protection against harmful interference when the equipment is operated in a commercial environment. This equipment generates, uses, and can radiate radio frequency energy and, if not installed and used in accordance with the instruction manual, may cause harmful interference to radio communications. Operation of this equipment in a residential area may cause harmful interference in which case the user will be required to correct the interference at his own expense.

The following techniques of reducing interference problems are applied easily:

1. Disconnect the battery eliminator from it's power source and from the 2100P Portable Turbidimeter to verify if it is the source of the interference
2. If the battery eliminator for the 2100P Portable Turbidimeter is plugged into the same outlet as the device with which it is interfering, try another outlet.
3. Move the 2100P Portable Turbidimeter away from the device receiving the interference.
4. Reposition the receiving antenna for the device receiving the interference.
5. Try combinations of the above.

SAFETY PRECAUTIONS

Please read this entire manual before unpacking, setting up, or operating this instrument. Pay particular attention to all danger and caution statements. Failure to do so could result in serious injury to the operator or damage to the equipment.

To ensure the protection provided by this equipment is not impaired, do not use or install this equipment in any manner other than that which is specified in this manual.

Use of Hazard Information

If multiple hazards exist, this manual will use the signal word (Danger, Caution, Note) corresponding to the greatest hazard.

DANGER
Indicates a potentially or imminently hazardous situation which, if not avoided, could result in death or serious injury.

CAUTION
Indicates a potentially hazardous situation that may result in minor or moderate injury.

NOTE
Information that requires special emphasis.

Precautionary Labels

Read all labels and tags attached to the instrument. Personal injury or damage to the instrument could occur if not observed.

⚠ This symbol, if noted on the instrument, references the instruction manual for operational and/or safety information.

SPECIFICATIONS

Specifications subject to change without notice.
Operating specifications applicable at 25 °C unless noted.
Program software copyrighted by Hach Company, 1991.

Measurement Method: Ratio Nephelometric signal (90°) scatter light ratio to transmitted light

Range: 0-1000 NTU with automatic decimal point placement or manual range selection of 0-9.99, 0-99.9 and 0-1000 NTU

Accuracy: ± 2% of reading plus stray light from 0-1000 NTU

Resolution: 0.01 NTU on lowest range

Repeatability: ±1% of reading or 0.01 NTU, whichever is greater (with Gelex standards)

Response Time: 6 seconds for full step change without signal averaging in constant reading mode

Stray Light: <0.02 NTU

Standardization: StablCal® Stabilized Formazin primary standards or Formazin primary standards

Secondary Standards: Gelex® Secondary Standards

Display: Four-digit liquid crystal; 10.16 mm (0.4 in) high digits with custom icons

Light Source: Tungsten filament lamp; lamp life typically greater than 100,000 readings

Detectors: Silicon photovoltaic

Signal Averaging: Operator selectable on or off

Sample Cells: (Height X width) 60.0 X 25 mm (2.36 X 1 in) Borosilicate glass with screw caps, marking band and fill line

Sample Required: 15 mL (0.5 oz.)

Storage Temperature: -40 to 60 °C (-40 to 140 °F) (instrument only)

Operating Temperature: 0 to 50 °C (32 to 122 °F) (instrument only)

Operating Humidity Range: 0 to 90% RH noncondensing at 30 °C;
0 to 80% RH noncondensing at 40 °C;
0 to 70% RH noncondensing at 50 °C

Power Requirements: Four AA Alkaline cells or optional battery eliminator

Battery Life: Typically 300 tests with signal average mode off;
180 tests with signal average mode on

Battery Eliminator (optional):

For 120 V eliminator: CSA and UL approved for 120 VAC ±10%, 60 Hz, 6 V at 800 mA DC output

For 230 V eliminator: CE (VDE) approval pending for 230 VAC ±10%, 50 Hz, 6 V at 900 mA DC output

Enclosure: High impact ABS plastic

Dimensions: 22.2 X 9.5 X 7.9 cm (8.75 X 3.75 X 3.12 in)

Instrument Weight: 520 kg (1 lb 2.5 oz)

Shipping Weight: 3.1 kg (6 lbs 8.5 oz)

OPERATION

DANGER

Handling chemical samples, standards, and reagents can be dangerous. Review the necessary Material Safety Data Sheets and become familiar with all safety procedures before handling any chemicals.

DANGER

La manipulation des échantillons chimiques, étalons et réactifs peut être dangereuse. Lire les Fiches de Données de Sécurité des Produits (FDSP) et se familiariser avec toutes les procédures de sécurité avant de manipuler tous les produits chimiques.

PELIGRO

La manipulación de muestras químicas, estándares y reactivos puede ser peligrosa. Revise las fichas de seguridad de materiales y familiarícese con los procedimientos de seguridad antes de manipular productos químicos.

GEFAHR

Das Arbeiten mit chemischen Proben, Standards und Reagenzien ist mit Gefahren verbunden. Es wird dem Benutzer dieser Produkte empfohlen, sich vor der Arbeit mit sicheren Verfahrensweisen und dem richtigen Gebrauch der Chemikalien vertraut zu machen und alle entsprechenden Materialsicherheitsdatenblätter aufmerksam zu lesen.

PERIGO

A manipulação de amostras, padrões e reagentes químicos pode ser perigosa. Reveja a folha dos dados de segurança do material e familiarize-se com todos os procedimentos de segurança antes de manipular quaisquer produtos químicos.

PERICOLO

La manipolazione di campioni, standard e reattivi chimici può essere pericolosa. La preghiamo di prendere conoscenza delle Schede Techniche necessarie legate alla Sicurezza dei Materiali e di abituarsi con tutte le procedure di sicurezza prima di manipolare ogni prodotto chimico.

SECTION 1 DESCRIPTION

1.1 General Description

The Hach Model 2100P Portable Turbidimeter (*Figure 1*) measures turbidity from 0.01 to 1000 NTU in automatic range mode with automatic decimal point placement. The manual range mode measures turbidity in three ranges: 0.01 to 9.99, 10 to 99.9 and 100 to 1000 NTU. Designed primarily for field use, the microprocessor-based Model 2100P has the range, accuracy, and resolution of many laboratory instruments. The instrument operates on four AA batteries or with an optional battery eliminator. Rechargeable nickel-cadmium cells may be used, but cannot be recharged in the instrument. The instrument automatically shuts off after 5.5 minutes if no keystrokes occur (does not influence operation). If this occurs, simply turn the instrument on – the 2100P will resume operation as if the power had not been interrupted. The instrument, all standard accessories, and the optional battery eliminator may be conveniently stored in the carrying case.

Figure 1 2100P Turbidimeter and Accessories

Note: *Avoid prolonged exposure to ultraviolet light and sunlight.*

Note: *Do not hold the instrument during measurements; place the instrument on a flat, steady surface.*

1.2 Accessories

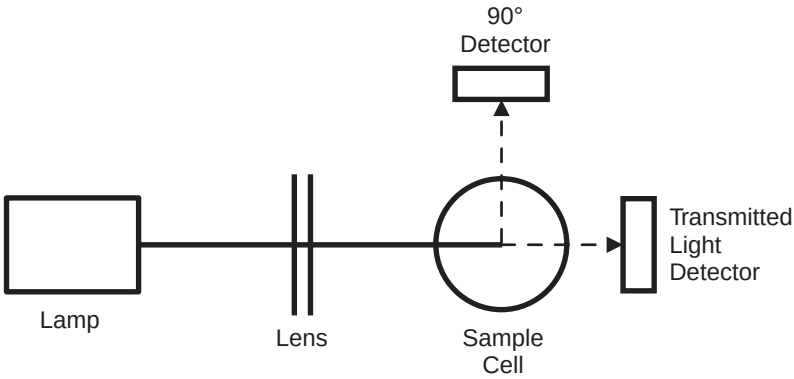
Accessories supplied with the turbidimeter include nine sample cells; three Gelex® Secondary Standards (included with 4650000 only); one sealed vial each of:
<0.1-NTU, 20-NTU, 100-NTU, and 800-NTU StablCal® Stabilized Formazin Standards; 4 AA alkaline batteries; 15 mL of silicone oil; oiling cloth; carrying case; instrument manual; and quick reference card.

1.3 Principle of Operation

The Model 2100P Portable Turbidimeter operates on the nephelometric principle of turbidity measurement. This instrument meets the design criteria specified by the United States Environmental Protection Agency, Method 180.1.

The optical system* (*Figure 2*) includes a tungsten-filament lamp, a 90° detector to monitor scattered light and a transmitted light detector. The instrument's microprocessor calculates the ratio of the signals from the 90° and transmitted light detectors. This ratio technique corrects for interferences from color and/or light absorbing materials (such as activated carbon) and compensates for fluctuations in lamp intensity, providing long-term calibration stability. The optical design also minimizes stray light, increasing measurement accuracy.

Figure 2 Ratio Optical System



* Patent number 4,198,161; other patents pending.

1.4 Preparation for Use

1.4.1 Unpacking

Remove the instrument and accessories from the shipping box and inspect them for damage that may have occurred due to rough handling or extreme weather conditions. Verify the following are present:

- Model 2100P Portable Turbidimeter
- Instrument Manual (with quick reference card)
- Set of StablCal Primary Standards in sealed vials, one each of:
  <0.1 NTU*
  20 NTU
  100 NTU
  800 NTU
- Standardization Kit containing Gelex Secondary Standards (0-10, 0-100 and 0-1000 ranges) (included with 4650000 only) plus nine sample cells with caps.
- Silicone Oil, 15-mL (0.5 oz) dropping bottle
- Oiling Cloth
- Carrying Case
- Four AA alkaline batteries

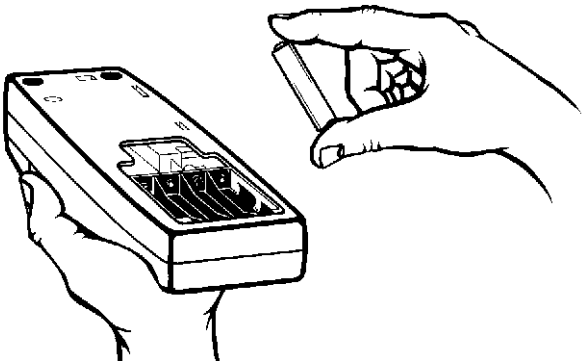
If any of the items are missing or damaged, please contact the Customer Service Department, Hach Company, Loveland, Colorado. The toll-free number in the United States is 800-227-4224. International customers should contact the Hach office or authorized distributor serving your area. Refer to *REPAIR SERVICE* on page *77*. **Please do not return the instrument without prior authorization from Hach.**

* Used in place of the dilution water standard when performing a calibration.

1.4.2 Battery Installation

The instrument is shipped completely assembled without the batteries installed. Before use, install the four AA alkaline batteries or connect the battery eliminator (*Figure 3*). For battery operation, remove the battery compartment cover on the instrument bottom and install the batteries. Correct battery polarity is shown on the battery holder. The instrument will not function if the batteries are not installed correctly. Reinstall the battery compartment cover.

Figure 3 Battery Installation



1.4.3 Using the Battery Eliminator and Rechargeable Batteries

For operation with the optional battery eliminator, plug the eliminator jack into the connector on the turbidimeter side. The battery eliminator may be used with or without the batteries installed. **The eliminator will not charge batteries.** Rechargeable batteries may be used in the instrument, but must be removed for recharging. See *HOW TO ORDER* on page *76* for ordering information. To prolong battery life, the instrument lamp turns on temporarily when the **READ** key is depressed. Batteries are not necessary for battery eliminator operation.

1.4.4 Calibration

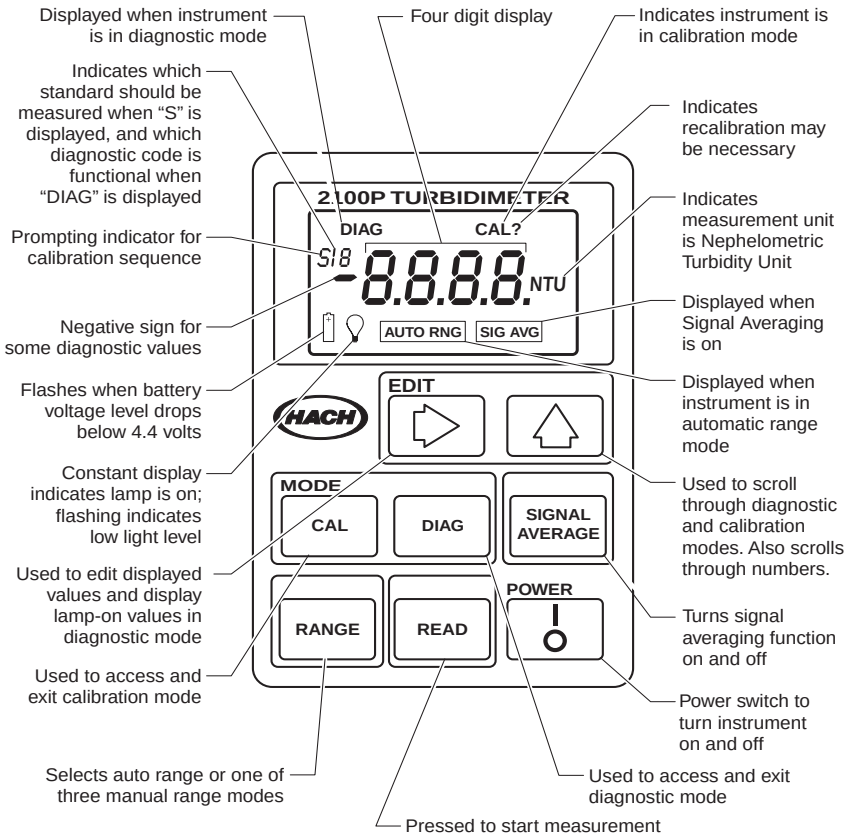
The 2100P Portable Turbidimeter is calibrated with Formazin Primary Standard at the factory**.** However, the instrument should be calibrated upon receipt for best results. Hach recommends recalibration with

formazin once every three months, or more often as experience dictates. The Gelex Secondary Standards supplied with the instrument (included with 4650000 only) are labelled with general ranges for application, but must be assigned values before use from formazin calibration. See *Section 3.6* on page *37* for calibration instructions.

2.1 Operating Controls and Indicators

Figure 4 shows the 2100P controls and indicators. Refer to *SECTION 3* for a detailed description of each control and indicator.

Figure 4 Keyboard and Display with Descriptions



2.2 Turbidity Measurement

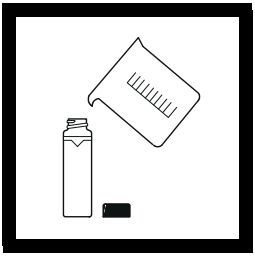
Measurements may be made with the signal average mode on or off and in manual or automatic range selection mode. Using automatic range selection is recommended. Signal averaging uses more power and should be used only when the sample causes an unstable reading. Signal averaging measures and averages ten measurements while displaying

intermediate results. The initial value is displayed after about 11 seconds and the display is updated every 1.2 seconds until all ten measurements are taken (about 20 seconds). After this, the lamp turns off, but the final measured turbidity value continues to be displayed until another key is pressed.

When not in signal average mode, the final value is displayed after about 13 seconds.

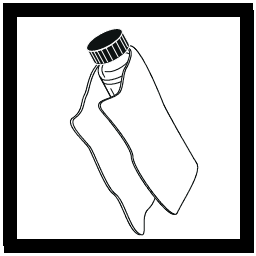
Accurate turbidity measurement depends on good measurement technique by the analyst, such as using clean sample cells in good condition and removing air bubbles (degassing). Refer to *Section 2.3* on page *22* for a detailed discussion of measurement techniques.

2.2.1 Turbidity Measurement Procedure

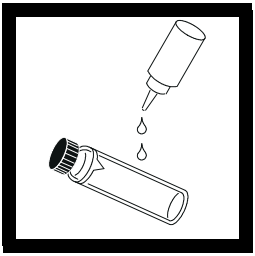


1. Collect a representative sample in a clean container. Fill a sample cell to the line (about 15 mL), taking care to handle the sample cell by the top. Cap the cell. (See *Section 2.3* on page *22* for more information about collecting a representative sample).

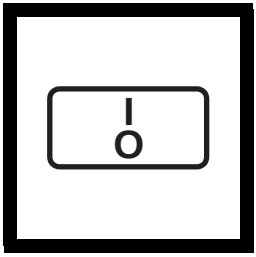
Note: *The instrument automatically shuts off after 5.5 minutes if no keystrokes occur. To resume operation, press* **I/O**.



2. Wipe the cell with a soft, lint-free cloth to remove water spots and fingerprints.

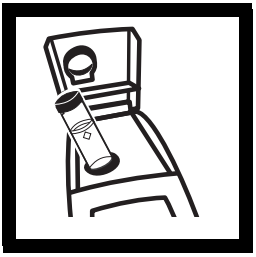


3. Apply a thin film of silicone oil. Wipe with a soft cloth to obtain an even film over the entire surface.



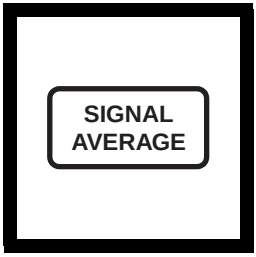
4. Press: **I/O**.

The instrument will turn on. Place the instrument on a flat, sturdy surface. Do not hold the instrument while making measurements.

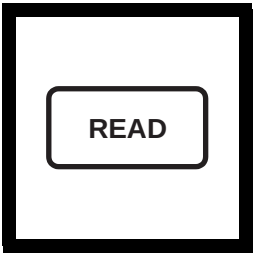


5. Insert the sample cell in the instrument cell compartment so the diamond or orientation mark aligns with the raised orientation mark in front of the cell compartment.
Close the lid.

6. Select manual or automatic range selection by pressing the **RANGE** key. The display will show **AUTO RNG** when the instrument is in automatic range selection.



7. Select signal averaging mode by pressing the **SIGNAL AVERAGE** key. The display will show **SIG AVG** when the instrument is using signal averaging. Use signal average mode if the sample causes a noisy signal (display changes constantly).



8. Press: **READ**

The display will show **- - - - NTU**, then the turbidity in NTU. Record the turbidity after the lamp symbol turns off.

Note: *The instrument defaults to the last operating mode selected. If automatic range mode and signal averaging were used on the previous measurements, these options will automatically be selected for subsequent samples.*

2.2.2 Measurement Notes

- Always cap the sample cell to prevent spillage of sample into the instrument.
- When taking a reading, place the instrument on a level, stationary surface. It should not be held in the hand during measurement.
- Always close the sample compartment lid during measurement and storage.
- Always use clean sample cells in good condition. Dirty, scratched, or damaged cells can cause inaccurate readings.
- Do not leave a sample cell in the cell compartment for extended periods of time. This may compress the spring in the cell holder.
- Remove sample cell and batteries from instrument if the instrument is stored for extended time period (more than a month).
- Avoid operating in direct sunlight.
- Make certain cold samples do not “fog” the sample cell.
- Avoid settling of sample prior to measurement.
- Keep sample compartment lid closed to prevent dust and dirt from entering.

2.3 Measurement Techniques

Proper measurement techniques are important in minimizing the effects of instrument variation, stray light and air bubbles. Regardless of the instrument used, measurements are more accurate, precise and repeatable if the analyst pays close attention to proper measurement techniques.

Measure samples immediately to prevent temperature changes and settling. Avoid sample dilution when possible. Particles suspended in the original sample may dissolve or otherwise change characteristics when the sample temperature changes or when the sample is diluted, resulting in a non-representative sample measurement.

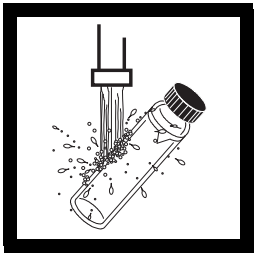
2.3.1 Cleaning Sample Cells

Cells must be extremely clean and free from significant scratches. The glass used to make cells is easily scratched – manufacturing cells free of minor scratches and other imperfections is difficult. However, minor imperfections are effectively masked by applying silicone oil as outlined in *Section 2.3.2*.

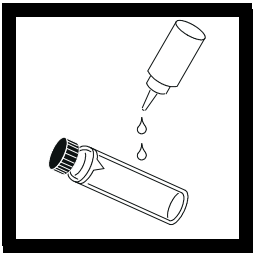
Clean the inside and outside of the cells by washing with laboratory detergent. Follow with multiple rinses of distilled or deionized water. Allow cells to air dry. Handle cells only by the top to minimize dirt, scratches and fingerprints in the light path.

2.3.2 Oiling the Sample Cell

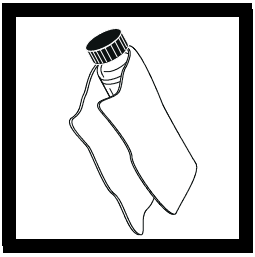
Applying a thin coat of silicone oil will mask minor imperfections and scratches which may contribute to turbidity or stray light. Use silicone oil equivalent to Hach Cat. No. 1269-36. This silicone oil has the same refractive index as glass. When applied in a thin, uniform coat, the oil fills in and masks minor scratches and other imperfections in the glass. Apply the oil uniformly by wiping with a soft, lint-free cloth.
Avoid application of excess oil. Applying excess oil may retain dirt and contaminate the instrument's cell compartment.



1. Thoroughly clean the sample cell.



2. Apply a small bead of silicone oil from the top to the bottom of the cell-- just enough to coat the cell with a thin layer of oil.



3. Using a soft, lint-free cloth, spread the oil uniformly, then wipe off the excess so that only a thin coat of oil is left. The cell should appear nearly dry with little or no visible oil.

Note: *Soft, lint-free cloth (velvet) works well for oiling. Store the oiling cloth with the sample cells and keep it free of dirt. After a few applications of oil, the cloth will contain enough residual oil that simply wiping the cell with the oiled cloth will provide a sufficient oil coat on the sample cell. Periodically, add a small amount of oil to the sample cell surface to replenish the oil in the cloth.*

Note: *Only a thin coat of oil on the sample cells is necessary.* ***Avoid using excessive amounts of oil.***

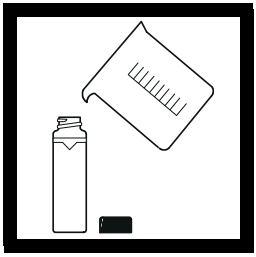
2.3.3 Orienting Sample Cells

Note: *When orienting and matching cells, it may be more efficient to use the continuous reading mode. The instrument performs continuous readings if the* **READ** *key is pressed and held. As long as the key is held, the lamp remains on and the display is updated every 1.2 seconds. The instrument cannot be used in continuous read mode if the Signal Averaging mode is on.*

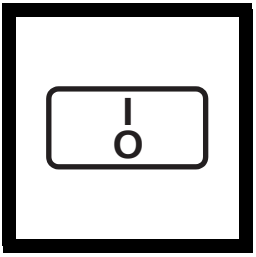
Precise measurements for very low turbidity samples require using a single cell for all measurements or optically matching the cells. Using one cell provides the best precision and repeatability. When one cell is used, an orientation mark (other than the factory-placed diamond) can be placed on the cell so it's inserted into the instrument with the same orientation each time.

2.3.3.1 Orienting a single cell

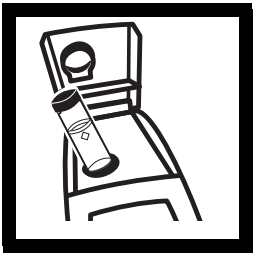
When using a single cell, make an index or orientation mark on the cell as follows:



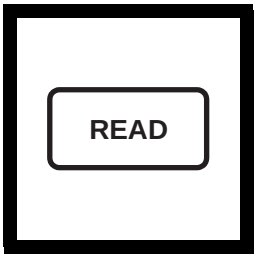
1. Fill the clean sample cell to the line with high quality water (< 0.5 NTU). Cap and wipe with lint-free cloth. Apply silicone oil. See *Section 3.6.2.2* on page *40* for more information about high quality water.



2. Press: **I/O** to turn the instrument on.



3. Insert the sample cell into the sample compartment. Close the cover.



4. Press: **READ**

Record the cell's position in the cell compartment and the displayed reading.

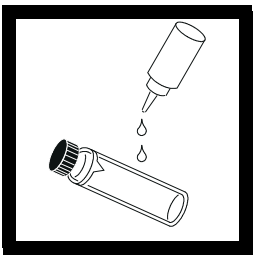
Note: *This procedure may be easier if the user holds the* **READ** *key through the whole process. This allows the lamp to remain on and make continuous readings.*

5. Remove the cell, rotate it slightly and reinsert it into the cell compartment. Close the cover, then press **READ**. Record the cell's position and the displayed reading.

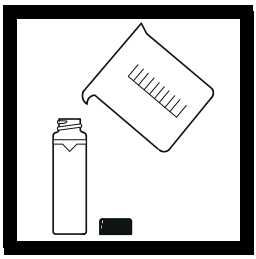
6. Repeat *step 5* until the lowest reading is displayed. Place an orientation mark on the cell's marking band near the top of the cell so the cell can be consistently inserted in the position that yields the lowest reading. When using the cell, always place it in the instrument so the orientation mark aligns with the raised mark on the instrument.

2.3.4 Matching multiple sample cells

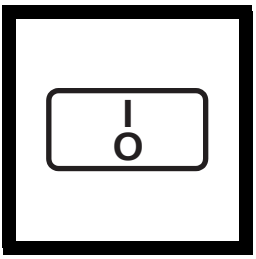
Precise measurements of very low turbidity samples require the cells be optically matched or a single cell be used for all measurements. If more than one cell is used, follow this procedure to match (index) the cells:



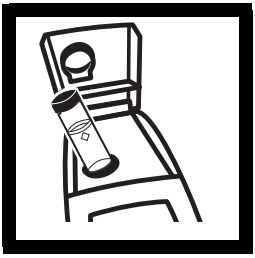
1. Clean and oil the sample cells as instructed in *Section 2.3.1* on page *23* and *Section 2.3.2* on page *23*.



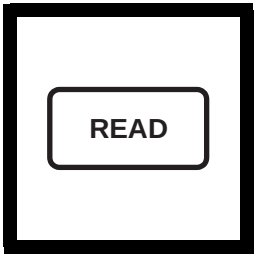
2. Fill the clean sample cells to the line with the same sample.



3. Press: **I/O** to turn the instrument on.



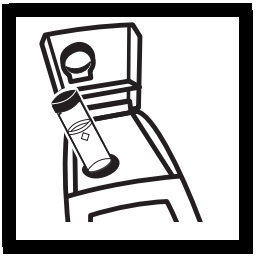
4. Insert the **first** sample cell into the sample compartment and close the cover.



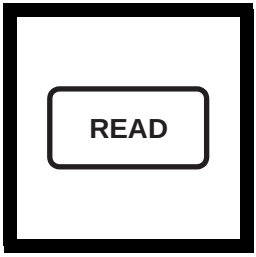
5. Press: **READ**

Record the cell's position in the cell compartment and the displayed reading. Place an orientation mark on the cell's marking band.

Note: *This procedure may be easier if the user holds the* **READ** *key through the whole process. This allows the lamp to remain on and make continuous readings.*



6. Insert the **second** sample cell into the cell compartment and close the cover.



7. Press: **READ**

Record the cell's position in the cell compartment and the displayed reading.

8. Remove the cell, rotate it slightly and reinsert into the cell compartment. Close the cover, then press **READ** again. Record the cell's position and the displayed reading.

9. Repeat *step 8* until the value displayed for the second cell is within 0.01 NTU (or 1%) of the value obtained for the first cell. Place an orientation mark on the second cell's marking band so it is consistently inserted in this position.

Note: *Due to variability in glass, it may not be possible to match all cells.*

10. Repeat *step 6* through *step 9* if matching other sample cells.

2.3.5 Removing Bubbles (Degassing)

Before measurement, removing air and other trapped gasses from the sample is strongly recommended, even if bubbles are not visible. Four degassing methods are commonly used:

1. applying a partial vacuum
2. adding a surfactant
3. using an ultrasonic bath
4. heating the sample

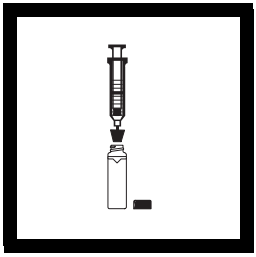
In some cases, more than one method may be necessary for effective bubble removal. For example, use of both a surfactant and ultrasonic bath may be necessary for some severe conditions. Use care with these techniques. If misused, sample turbidity can be altered.

Removing air bubbles by letting the sample stand for a period of time is not recommended. Particulates that cause turbidity may settle and the sample temperature may change. Both conditions may alter sample turbidity, resulting in measurements not representative of the original turbidity.

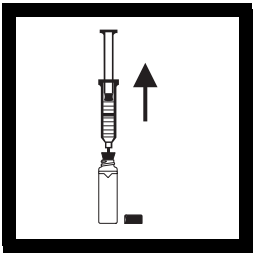
2.3.5.1 Application of vacuum

Apply a vacuum with any convenient, clean, oil-free vacuum source. The vacuum lowers the atmospheric pressure, allowing trapped bubbles to escape into the air above the sample. Vacuum works well with non-viscous samples (such as water) that don’t contain volatile components. Applying vacuum to viscous, volatile-containing samples (paint resins) may cause the volatile components to come out of solution and aggravate the bubble problem.

To apply a vacuum, use a sample degassing kit equivalent to Cat No. 43975-00 (Degassing Kit) or 43975-10 (Degassing and Filtration Kit). These kits contain a syringe and rubber stopper for vacuum degassing. An electric or hand-operated pump equivalent to Cat No. 14283-00 or 14697-00, respectively, may also be used.



1. Fill a sample cell to the mark with sample. Insert a #2 single-hole rubber stopper and syringe into the cell. If using a pump, insert a piece of glass tubing into the stopper.



2. **Slowly** apply the vacuum by carefully pulling the plunger upward, then holding it. If using a hand or electric pump, connect the tubing to the vacuum pump with vacuum hose. Apply vacuum until visible gas bubbles disappear. **Slowly** release the vacuum. Remove the vacuum apparatus and cap the cell.

2.3.5.2 Adding a surfactant

Surfactants should be limited to severe problems when other degassing methods are ineffective. Surfactants change the surface tension of the water, which releases trapped gases. Hach recommends a surfactant such as Triton X-100 or the equivalent, Hach Cat No. 14096-37. Put one drop of Triton X-100 in the sample cell before adding sample.

Note: *Any turbidity contributed by surfactant addition is negligible.*

This technique is very effective when the water is super-saturated with air. However, changing the surface tension may accelerate settling of turbidity-causing particles. Mix the sample gently, but thoroughly, and analyze as soon as possible after adding the surfactant. Avoid vigorous mixing as the surfactant may foam. Rinse the sample cells thoroughly between samples to prevent surfactant accumulation.

2.3.5.3 Using an ultrasonic bath

Note: *The time necessary to expel bubbles may vary from a few seconds to a minute or more. To avoid excessive application of ultrasound, a simple procedure can be followed. First, apply ultrasound until all visible bubbles are absent. Then measure the sample turbidity. Apply ultrasound for a short time period and again measure turbidity. Continue for several repetitions, noting the treatment time and turbidity readings. If turbidity begins to increase instead of decrease, the ultrasound waves have probably started to alter the suspended particles. Note the time it takes for this to occur and record it as the maximum time limit for ultrasonic treatment.*

Ultrasonic baths effectively remove gas bubbles from most samples, especially viscous liquids. However, the ultrasonic waves which cause degassing may also alter the characteristics of the particles causing the turbidity. Turbidity depends on the size, shape, composition and refractive index of the suspended particles. Excessive ultrasound application may alter particle size and shape, thus changing sample turbidity. In some cases, ultrasound may aggravate air bubble removal by fracturing the bubbles, making degassing more difficult.

1. Fill a clean sample cell to the line with sample. Leave uncapped.

2. Immerse the cell (1/2 to 2/3 immersed) in an ultrasonic bath and allow it to stand until visible bubbles are expelled.

3. Remove the cell, cap, then thoroughly dry the cell. Apply silicone oil as directed.

2.3.5.4 Application of heat

Whenever possible, avoid using heat to degas samples because heat may change the characteristics of the suspended particles and cause volatile components to come out of solution. Gentle heating may be helpful for degassing some very viscous samples when combined with application of vacuum or ultrasound. If heat is necessary, heat the sample only until degassing occurs. The simplest technique is to prepare a warm water bath and partially immerse the filled sample cell. Use the shortest time necessary for expelling visible bubbles. Cool sample to original sample temperature before taking measurements.

2.3.6 Measuring Overrange Samples

Nephelometric turbidity measurement depends on detection of light scattered from particles suspended in the liquid. If the turbidity is very high, a significant amount of light is blocked or absorbed by the particles and only a small amount of light reaches the detector. This results in a negative interference – the measured turbidity is lower than the actual turbidity. This condition is called “going blind”. A multidetector ratioing instrument, such as the Hach 2100P Turbidimeter, minimizes this effect and extends the instrument range. Highly turbid samples may also be diluted, but this should be avoided when possible since it may alter the characteristics of the suspended particles and produce erroneous results.

Light absorbing particles such as activated carbon and highly colored samples may also cause an instrument to “go blind”. Dilution may not correct for these interferences. A ratioing instrument will correct for the presence of light absorbing particles and color.

2.3.7 Condensation (fogging)

Condensation may occur on the outside of the sample cell when measuring a cold sample in a warm, humid environment. Condensation interferes with turbidity measurement, so all moisture must be thoroughly wiped off the sample cell before measurement. If fogging recurs, let the sample warm slightly by standing at room temperature or immersing it in a warm bath for a short period. After warming, mix the sample thoroughly before measurement. Allowing samples to warm can alter sample turbidity, so it is best to avoid warming samples before measurement when possible.

2.3.8 Calibration

Turbidimeters must be properly calibrated with a primary standard. Hach recommends formazin or StablCal Stabilized Formazin for calibration. For U.S. Environmental Protection Agency (USEPA) reporting, calibrate at least as often as required by the appropriate regulatory agencies. The frequency of calibration depends on environmental conditions (humidity, temperature) and use. If necessary, calibrate more frequently.

Use secondary standards for periodic calibration checks. Please note that Gelex® standards must be assigned values after StablCal Stabilized Formazin calibration or formazin calibration and before use as secondary standards. Gelex standards must be recalibrated (values assigned) each time the instrument is calibrated with StablCal Stabilized Formazin or formazin. See *Section 3.6* on page *37* for detailed information on the use of StablCal Stabilized Formazin, formazin, and Gelex standards.

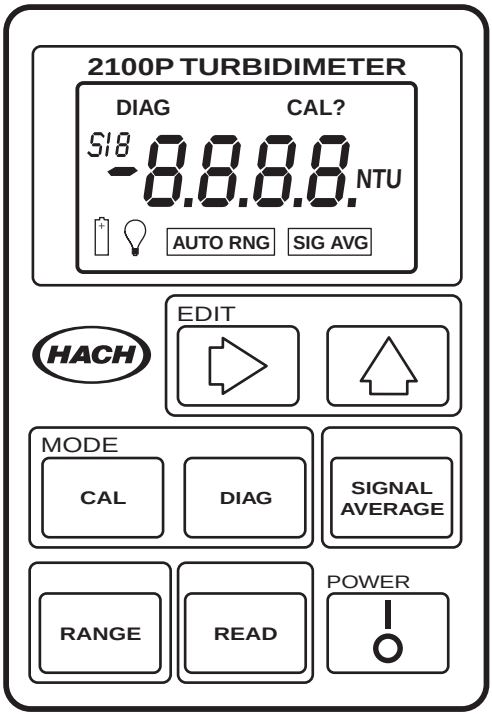
2.3.9 Representative Sampling

A representative sample accurately reflects the true condition of the water source from which the sample was taken. To ensure a representative sample, gently, but thoroughly, mix every sample before aliquots are taken. Do not allow the sample to settle.

When sampling from a tap in a distribution system or treatment plant, allow the water to run for at least five minutes before sampling. When sampling from a stream, reservoir, clarifier, or storage tank, collect at least one liter (l quart) and thoroughly mix before measurement. If the water source is not uniform, it may be necessary to sample several locations at varying depths and combine the samples into a single, well-mixed composite sample before measurement.

SECTION 3 OPERATION

3.1 Operational Controls and Indicators



Key	Description
I O	Power key to turn instrument on and off. If no keys are pressed for 5.5 minutes, the instrument turns off automatically.
READ	Depressed to perform a measurement. To conserve battery power, the lamp turns on only when **READ** is depressed. A reading is displayed about 12 seconds after the key is depressed. During the delay, a flashing **NTU** is displayed. After the reading is displayed, the lamp turns off and the reading continues to be displayed. Continuous readings may be done by holding this key if **not** in the Signal Averaging mode. After the initial delay, the reading is updated every 1.2 seconds.
CAL	Used to perform a calibration or review calibration data. Also terminates a calibration or calibration review and returns to the 2100P measurement mode.

Key	Description
△	Edits a flashing digit in the calibration mode or sequences through the calibration standards (S0,S1, S2, S3) or diagnostic menu.
▷	Used to move the editing cursor to the digits being edited in the calibration mode or initiate editing of a standard value.
SIGNAL AVERAGE	Turns the signal averaging function on or off.
DIAG	Selects the diagnostic mode.
RANGE	Selects Auto Range or Manual Range (one of three manual modes).

Display Icon	Description
DIAG	Turns on after the **DIAG** is pressed to access the diagnostic mode. A number displayed under the **DIAG** icon (1-9) indicates which diagnostic function is active. See *Section 5.1* on page *67* for more information on diagnostic codes.
CAL	Turns on after the **CAL** key is pressed to access the calibration mode and remains on during the calibration.
CAL?	Appears after calibration if a value entered during calibration is outside an acceptable range. May indicate an operator error or possible instrument malfunction. Flashing **CAL?** indicates the default calibration coefficients are being used (even after a user-calibration has been done) or that no calibration data is currently stored.
S__	Displayed during calibration. The S is followed by a number to indicate which standard value is currently being edited or displayed. Flashing number is prompting user for measurement of **S0**, **S1**, **S2** or **S3** to establish a calibration. Steady number identifies which standard's value is being displayed.
+	Flashes when the battery voltage drops to 4.4 volts as an indication to change batteries. At <4.0 volts, the instrument automatically shuts off.
	The lamp symbol is constantly on when the lamp is on and flashes after a reading if a marginal light level reaches the transmitted light detector. A flashing icon indicates the sample may be too turbid (not within measurement range) and needs dilution or the lamp needs replacing.
SIGNAL AVERAGE	Indicates the signal averaging mode is on. The icon turns off if signal averaging is not selected.

Display Icon	Description
AUTO RNG	Indicates instrument is in automatic range mode. The icon turns off when manual range mode is selected.
8888	The 4-digit display is active when the instrument is on (measurements are displayed to three digits). After the **READ** key is pressed **- - - -** is displayed during wait periods.
NTU	Identifies the measurement units- Nephelometric Turbidity Units. This icon is active during measurements and in the calibration mode.

3.2 Using the Read Key

To preserve battery power and prolong lamp life, the lamp turns on only after the **READ** key is pressed. Pressing the key turns the instrument lamp on; after about 12 seconds, the lamp turns off, but the measurement value continues to be displayed. After the first measurement, a four-second recovery time occurs before another measurement can be started. If **READ** is pressed during the recovery time, the display will begin flashing, but the lamp will not turn on until the full four seconds have passed. If no other key strokes occur within 5.5 minutes, the instrument turns off.

3.2.1 Continuous Reading

The instrument cannot be used in continuous read mode if the Signal Averaging mode is on.

The instrument will perform continuous readings if the **READ** key is pressed and held. As long as the key is held, the lamp remains on and the display is updated every 1.2 seconds.

3.3 Using the Signal Averaging Key

The signal averaging mode compensates for reading fluctuations caused by drifting of sample particles through the light path. Signal averaging is turned on or off by pressing the **SIGNAL AVERAGE** key. The **SIG AVG** icon is displayed when signal averaging is on.

Signal averaging measures and averages ten measurements while displaying intermediate results. The initial value is displayed after about 11 seconds and the display is updated every 1.2 seconds until all ten measurements are taken (about 22 seconds). After 22 seconds, the lamp

turns off, but the final measured turbidity value continues to be displayed until another key is pressed.

When signal averaging is off, the instrument takes three measurements, the microprocessor averages them, then displays the average. If the **READ** key is held during measurement, the initial value is displayed in 12 seconds and is updated every 1.2 seconds as long as the **READ** key is held.

When the instrument is turned on, the instrument defaults to the signal averaging mode which was used during the last measurement.

3.4 Using the Range Selection Key

As shipped, the instrument defaults to automatic range mode. The first time the **RANGE** key is pressed, the instrument goes into manual range mode. The second, third, and fourth key strokes put the instrument in the 0.00-9.99, 10 to 99.9 or 100-1000 NTU range, respectively. Another key stroke brings the selection back to automatic range mode. When the automatic range mode is selected, the **AUTO RNG** icon is displayed. Range selection can be done any time except when a measurement or calibration is in progress.

When the instrument is turned on, the instrument defaults to the range mode and measurement range which was used during the last measurement.

3.5 Restoring the Default Calibration

To restore and use the default calibration, turn the instrument off. Press and hold **DIAG**, then press and release **I/O**. Release **DIAG** when the software version number disappears from the display. (For models with serial number less than 920300000800, **2100** disappears). This clears any user-entered calibration from memory; the 2100P will use the default calibration for measurement. **CAL?** will appear and continue to flash until a user-entered calibration is successfully completed.

For best results, a user-entered calibration should be done every three months.

3.6 Calibration

Calibration of the 2100P Turbidimeter is based on formazin, the primary standard for turbidity. The instrument's electronic and optical design provide long-term stability and minimize the need for frequent calibration. The two-detector ratioing system compensates for most fluctuations in lamp output. **A formazin recalibration should be performed at least once every three months,** more often if experience indicates the need. When calibration is necessary, use a primary standard such as StablCal™ Stabilized Standards or formazin standards.

Hach Company only recommends the use of StablCal® Stabilized Formazin or formazin standards for the calibration of Hach turbidimeters. Hach Company cannot guarantee the performance of the turbidimeter if calibrated with co-polymer styrene divinylbenzene beads or other suspensions.

Important Note: DO NOT calibrate with Gelex® Secondary Standards. Gelex standards are designed for instrument verification, not calibration.

3.6.1 StablCal Stabilized Formazin Standards*

Most consistent results will be achieved with the use of StablCal Stabilized Formazin Standards for calibration. Refer to *Section 3.6.1.2* and *Section 3.6.1.3* for information on preparing the standards for use.

Note: *Hach StablCal Stabilized Formazin in 20-, 100-, and 800-NTU values is packaged in convenient sets for calibration of the 2100P Turbidimeter. The set may be ordered in 500-mL size bottles by specifying Cat. No. 26594-00, in 100-mL size bottles by specifying Cat. No. 26594-10 or in sealed vials by ordering Cat. No. 26594-05. (See OPTIONAL ACCESSORIES AND REAGENTS on page 74.)*

3.6.1.1 Storing and Handling StablCal Stabilized Standards

For optimum results when using StablCal Stabilized Standards, adhere to the following recommendations:

* StablCal Stabilized Formazin is cited as a primary standard in Hach Method 8195, an acceptable version of USEPA Method 180.1.

- Do not transfer the standard to another container for storage.
- Do not return standard from the sample cell back into the its original container. Standard contamination will result.
- Store standards between 0 and 25 °C.
- For long-term storage, refrigeration at 5 °c is recommended. Do not store above 25 °C.
- Allow the standard to acclimate to ambient instrument conditions before use (not to exceed 40 °C).
- Store away from direct sunlight. Store vials in their respective kit or shipping box with the cover in place.

3.6.1.2 Preparing Bulk StablCal Stabilized Standards

Bulk standards that have been sitting undisturbed for longer than a month must be shaken to break the condensed suspension into its original particle size. Start at *step 1* for these standards. If the standards are used on at least a weekly interval, start at *step 3*.

Important Note: These instructions do not apply to <0.1-NTU* StablCal Standards; <0.1NTU StablCal Standards should not be shaken or inverted.

1. Shake the standard vigorously for 2-3 minutes to resuspend any particles.
2. Allow the standard to stand undisturbed for 5 minutes.
3. Gently invert the bottle of StablCal 5 to 7 times.
4. Prepare the sample cell for measurement using traditional preparation techniques. This usually consists of oiling the sample cell (see*Section 2.3.2* on page *23*) and marking the cell to maintain the same orientation in the sample cell compartment (see *Section 2.3.3* on page *24*). This step will eliminate any optical variations in the sample cell.

* Used in place of the dilution water standard when performing a calibration.

5. Rinse the sample cell at least one time with the standard and discard the rinse.

6. Immediately fill the sample cell with the standard. Cap the sample cell and let it stand for one minute. The standard is now ready for use in the calibration procedure, *Section 3.6.3*.

3.6.1.3 Preparing StablCal Stabilized Standards in Sealed Vials

Sealed vials that have been sitting undisturbed for longer than a month must be shaken to break the condensed suspension into its original particle size. Start at *step 1* for these standards. If the standards are used on at least a weekly interval, start at *step 3*

Important Note: These instructions do not apply to <0.1-NTU* StablCal Standards; <0.1NTU StablCal Standards should not be shaken or inverted.

1. Shake the standard vigorously for 2-3 minutes to resuspend any particles.

2. Allow the standard to stand undisturbed for 5 minutes.

3. Gently invert the vial of StablCal 5 to 7 times.

4. Prepare the vial for measurement using traditional preparation techniques. This usually consists of oiling the vial (see *Section 2.3.2* on page *23*) and marking the vial to maintain the same orientation in the sample cell compartment (see *Section 2.3.3* on page *24*). This step will eliminate any optical variations in the sample vial.

5. Let the vial stand for one minute. The standard is now ready for use in the calibration procedure, *Section 3.6.3*.

3.6.2 Formazin Primary Standards

Perform the procedure in *Section 3.6.2.1* to prepare a 4000-NTU standard. Alternately, order a 4000-NTU stock solution from Hach by specifying Cat. 2461-49. Prepare the dilutions from the 4000-NTU stock solution by following the instructions in *Section 3.6.2.4*.

3.6.2.1 Preparing Formazin Stock Solution

Dilute formazin standard solutions from a 4000 NTU stock solution equivalent to Hach Cat. No. 2461-49. The prepared stock solution is stable for up to one year when properly prepared. An alternative to purchasing the 4000 NTU stock solution is preparing a stock solution as follows:

1. Dissolve 5.000 grams of reagent grade hydrazine sulfate ($N_2H_4 \cdot H_2SO_4$) in 400 mL of distilled water.

2. Dissolve 50.000 grams of pure hexamethylenetetramine in 400 mL of distilled water.

3. Pour the two solutions into a 1000-mL volumetric flask and dilute to the mark with distilled water.

4. Let the solution stand for 48 hours at 25 °C (77 °F) to develop the 4000-NTU stock suspension. The standing temperature is critical for correct formation of formazin polymers.

5. Mix the 4000 NTU suspension for at least ten minutes before use. Then it can be diluted with distilled or demineralized water to achieve a solution of the desired NTU value.

Instead of diluting a formazin stock solution, StablCal Stabilized Formazin Standards may be used. Order the StablCal Calibration Set for the 2100P Turbidimeter, Cat.No. 26594-00 (500-mL bottles), Cat. No. 26594-10 (100 mL bottles), or Cat. No. 26594-05 (sealed vials). (See *OPTIONAL ACCESSORIES AND REAGENTS* on page *74*.)

3.6.2.2 Correcting for Turbidity of Dilution Water

The 2100P Turbidimeter automatically compensates for turbidity contributed by dilution water when calculating the true value of the lowest formazin standard. Use high quality distilled or deionized water less than 0.5 NTU. The instrument will display E 1 after calibration if

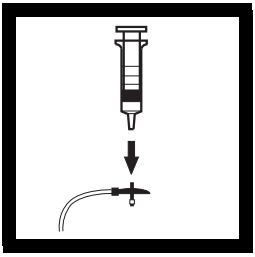
the dilution water turbidity is greater than 0.5 NTU. In this case, prepare the water as directed below.

The value of the dilution water can be arbitrarily forced to zero (see calibration procedure). This is not recommended for most applications and, if done, should be done only if the dilution water turbidity is less than 0.2 NTU.

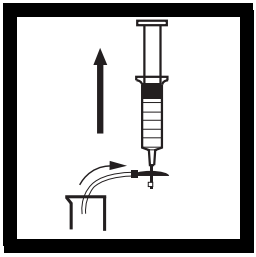
3.6.2.3 Preparing Dilution Water

Note: *Use the same dilution water for all dilutions and the sample blank.*

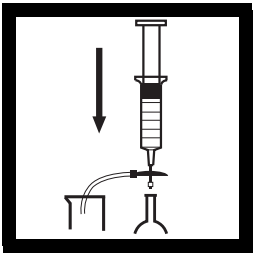
Collect at least 1000 mL of high quality dilution water (distilled or deionized water). The 2100P Turbidimeter, as received from the factory, is precalibrated and may be used to check the dilution water turbidity. If the turbidity is greater than 0.5 NTU, filter the water with the Sample Filtration and Degassing Kit (Cat. No. 43975-10) or equivalent. When measuring low range turbidity, clean all glassware with 1:1 hydrochloric acid and rinse several times with dilution water. If the glassware is not used immediately, use stoppers to prevent contamination from small particles.



1. Attach the syringe to the 3-way valve by gently twisting the square end into the syringe tip. Attach the connector, tubing and a 0.2 micron filter (clear part faces syringe) as shown. Be sure the connections are tight.



2. Fill a beaker or container with the water to be filtered. Insert the tubing into the container. Slowly draw the water into the syringe by pulling up on the syringe plunger.



3. Draw about 50 mL of sample into the syringe. Slowly push on the plunger to force the water through the filter and into a graduated cylinder or volumetric flask. Repeat Steps 2 and 3 until the desired amount of water is obtained.

Note: *As the filter clogs, it gets more difficult to push water through it. At this point, discard the filter and attach a new filter. Replacement filters are available in packages of 10 (Cat. No. 23238-10).*

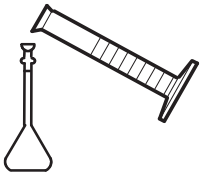
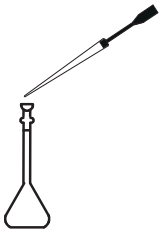
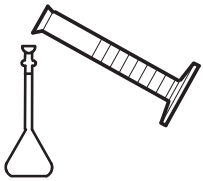
3.6.2.4 Preparing Formazin Dilutions (Factory recommended)

Hach Company recommends using 20, 100, and 800 NTU formazin standards for calibrating the 2100P Turbidimeter. Dilutions with other NTU values can be prepared and used (see *Section 3.6.3.1* on page *48*). If problems occur when using alternate solutions, use the dilutions specified here.

Prepare all formazin dilutions immediately before use and discard after calibration. The 4000 NTU solution is stable for up to a year, but dilutions deteriorate more rapidly. Use the same high quality water (turbidity <0.5 NTU) for the dilutions and the blank.

Preparing the 20, 100 and 800 NTU standards

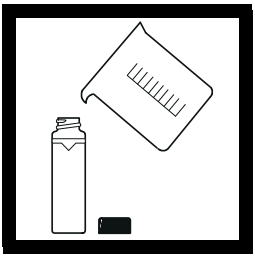
Table 1 Formazin Standard Preparation

	Step 1	Step 2	Step 3
Standards			
20 NTU	Add 100 mL of dilution water to a clean **200-mL** class A volumetric flask.	With a TenSette* pipet, add 1.00 mL of well-mixed 4000 NTU Formazin stock solution to the 200-mL flask.	Dilute to the mark with dilution water. Stopper and mix.
100 NTU	Add 100 mL of dilution water to a clean **200-mL** class A volumetric flask.	With a TenSette pipet, add 5.00 mL of well-mixed 4000 NTU Formazin stock solution to the 200-mL flask.	Dilute to the mark with dilution water. Stopper and mix.
800 NTU	Add 50 mL of dilution water to a clean **100-mL** class A volumetric flask.	With a TenSette pipet, add 20.00 mL of well-mixed 4000 NTU Formazin stock solution to the 100-mL flask.	Dilute to the mark with dilution water. Stopper and mix.

* A class A volumetric pipet may be used in place of a TenSette Pipet.

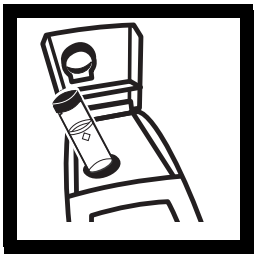
3.6.3 Calibrating the Turbidimeter

Note: *For best accuracy use the same sample cell or four matched sample cells for all measurements during calibration. Always insert the cell so the orientation mark placed on the cell during the matching procedure is correctly aligned. (See Section 2.3.4 on page 26 for matching sample cells).*



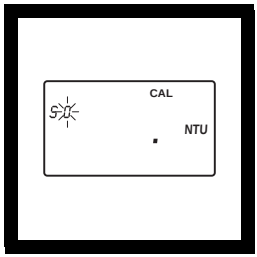
1. Rinse a clean sample cell with dilution water several times. Then fill the cell to the line (about 15 mL) with dilution water or use StablCal <0.1 NTU standard.

Note: *The same dilution water used for preparing the standards must be used in this step.*



2. Insert the sample cell in the cell compartment by aligning the orientation mark on the cell with the mark on the front of the cell compartment. Close the lid. Press **I/O**.

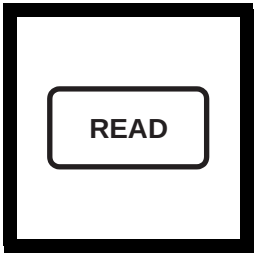
Note: *Choose signal average mode option (on or off) before pressing* **CAL** *– the* **SIGNAL AVERAGE** *key is not functional in calibration mode.*



3. Press: **CAL**

The **CAL** and **S0** icons will be displayed (the **0** will flash). The 4-digit display will show the value of the S0 standard for the previous calibration. If the blank value was forced to 0.0, the display will be blank (as shown). Press → to get a numerical display.

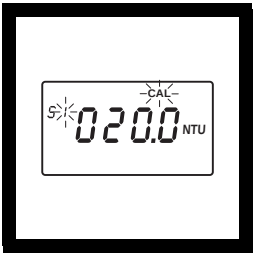
Hach Company only recommends the use of StablCal® Stabilized Formazin or formazin standards for the calibration of Hach turbidimeters. Hach Company cannot guarantee the performance of the turbidimeter if calibrated with co-polymer styrene divinylbenzene beads or other suspensions. DO NOT calibrate with Gelex® Secondary Standards.



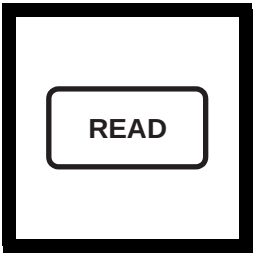
4. Press: **READ**

The instrument will count from 60 to 0, (67 to 0 if signal average is on), read the blank and use it to calculate a correction factor for the 20 NTU standard measurement. If the dilution water is ≥ 0.5 NTU, E 1 will appear when the calibration is calculated (See *Section 3.6.2.3* on page *41* for more dilution water information). The display will automatically increment to the next standard. Remove the sample cell from the cell compartment.

Note: *The turbidity of the dilution water can be "forced" to zero by pressing → rather than reading the dilution water. The display will show* **S0 NTU** *and the ↑ key must be pressed to continue with the next standard.*



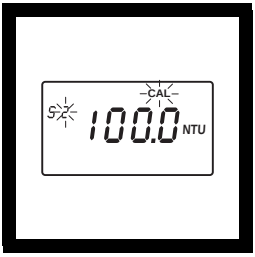
5. The display will show the **S1** (with the 1 flashing) and **20 NTU** or the value of the S1 standard for the previous calibration. If the value is incorrect, edit the value by pressing the → key until the number that needs editing flashes. Use the ↑ key to scroll to the correct number. After editing, fill a clean sample cell to the line with **well mixed** 20 NTU StablCal Standard or 20 NTU formazin standard. Insert the sample cell into the cell compartment by aligning the orientation mark on the cell with the mark on the front of the cell compartment. Close the lid.



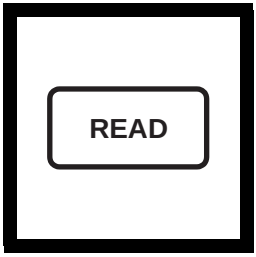
6. Press: **READ**

The instrument will count from 60 to 0 (67 to 0 if signal average is on), measure the turbidity and store the value. The display will automatically increment to the next standard. Remove the sample cell from the cell compartment.

Note: *For potable water applications with low turbidity values the instrument calibration may be stopped after the 20 NTU StablCal Standard has been read. Press* **CAL** *after reading the 20 NTU standard. The instrument calibration is now complete for the range of 0-20 NTU only. The instrument will continue to read turbidity values above 20 NTU. These values were not updated during the 0-20 NTU calibration.*

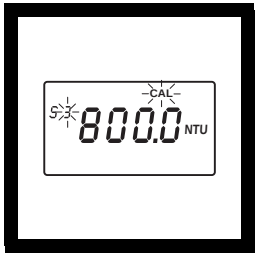


7. The display will show the **S2** (with the 2 flashing) and **100 NTU** or the value of the S2 standard for the previous calibration. If the value is incorrect, edit the value by pressing the → key until the number that needs editing flashes. Use the ↑ key to scroll to the correct number. After editing, fill a clean sample cell to the line with **well mixed** 100 NTU StablCal Standard or 100 NTU formazin standard. Insert the sample cell into the cell compartment by aligning the orientation mark on the cell with the mark on the front of the cell compartment. Close the lid.

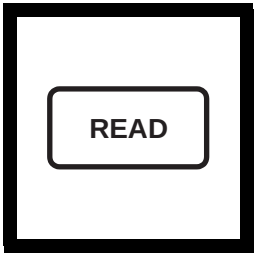


8. Press: **READ**

The instrument will count from 60 to 0 (67 to 0 if signal average is on), measure the turbidity and store the value. Then, the display will automatically increment to the next standard. Remove the sample cell from the cell compartment.



9. The display will show the **S3** (with the 3 flashing) and **800 NTU** or the value of the S3 standard for the previous calibration. If the value is incorrect, edit the value by pressing the → key until the number that needs editing flashes. Use the ↑ key to scroll to the correct number. After editing, fill a clean sample cell to the line with **well mixed** 800 NTU StablCal Standard or 800 NTU formazin standard. Insert the sample cell into the cell compartment by aligning the orientation mark on the cell with the mark on the front of the cell compartment. Close the lid.



10. Press: **READ**

The instrument will count from 60 to 0 (67 to 0 if signal average is on), measure the turbidity and store the value. Then the display will increment back to the S0 display. Remove the sample cell from the cell compartment.

11. Press: **CAL** to accept the calibration. The instrument will return to measurement mode automatically.

Note: *Pressing* **CAL** *completes the calculation of the calibration coefficients. If calibration errors occurred during calibration, error messages will appear after* **CAL** *is pressed. If* **E 1** *or* **E 2** *appear, check the standard preparation and review the calibration; repeat the calibration if necessary. If* **CAL?** *appears, an error may have occurred during calibration. If* **CAL?** *is flashing, the instrument is using the default calibration.*

NOTES

- If the **I/O** key is pressed during calibration, the new calibration data is lost and the old calibration will be used for measurements. Once in calibration mode, only the **READ, I/O,** ↑, and → keys function. Signal averaging and range mode must be selected before entering the calibration mode.

- If **E 1** or **E 2** are displayed, an error occurred during calibration. Check the standard preparation and review the calibration; repeat the calibration if necessary. Press **DIAG** to cancel the error message (**E 1** or **E 2**). To continue without repeating the calibration, press **I/O** twice to restore the previous calibration. If **CAL?** is displayed, an error may have occurred during calibration. The previous calibration may not be restored. Either recalibrate or use the calibration as is.

- To review a calibration, press **CAL** and then ↑ to view the calibration standard values. As long as **READ** is never pressed and **CAL** is not flashing, the calibration will not be updated. Press **CAL** again to return to the measurement mode.

3.6.3.1 Preparing User-selected Formazin Dilutions

The formazin solutions should span the entire range of the instrument. Hach recommends preparing three standards:

1. 10 to 30 NTU

2. 90 to 110 NTU

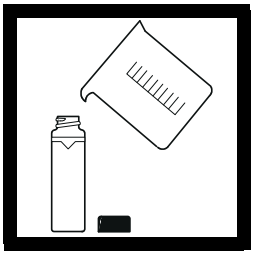
3. 700 to 900 NTU

The standards must have a difference of at least 60 NTU.
In addition, a blank made from the dilution water should be prepared.

Prepare the formazin standard solutions from the well mixed 4000 NTU stock solution as specified in *Section 3.6.2.4* on page *42* and dilution water as specified in *Section 3.6.2.2* and *Section 3.6.2.3* on page *41*. Make the standards **immediately** before use and discard them after calibration is done.

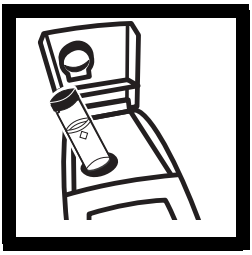
3.6.3.2 Calibrating with User-selected Standards

Note: *For best accuracy use the same sample cell or four matched sample cells for all measurements during calibration. Always insert the sample cell with the same orientation.*



1. Fill a clean sample cell to the line (about 15 mL) with dilution water.

Note: *The same dilution water used for preparing the standards must be used in this step.*

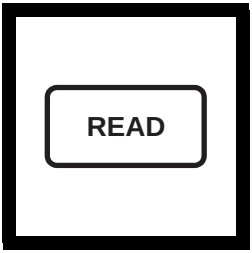


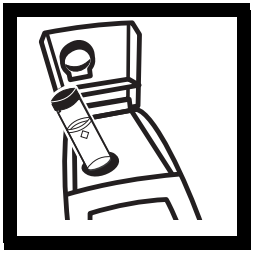
2. Insert the sample cell into the cell compartment and close the lid. Press **I/O**.

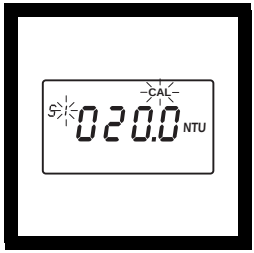
Note: *Choose signal average mode option (on or off) before pressing* **CAL** *– the* **SIGNAL AVERAGE** *key is not functional in calibration mode.*

3. Press: **CAL**.

The **CAL** and **S0** icons will appear (the **0** will flash). The 4-digit display will show the value of the S0 standard for the previous calibration.





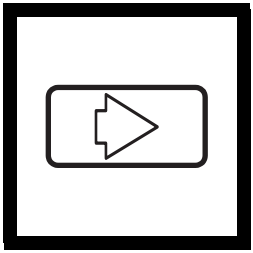


4. Press: **READ**.

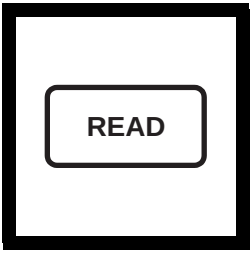
The instrument will count from 60 to 0 (67 to 0 if signal average is on), measure the blank and use it to calculate a correction factor for the lowest standard. If the dilution water is ≥0.5 NTU, **E 1** will appear (see *Section 3.6.2.3* on page *41* for more dilution water information). The display will automatically increment to the next standard. Remove the sample cell from the cell compartment.

5. Thoroughly mix the 10 to 30 NTU range standard, then fill a clean sample cell to the line with the standard. Insert the sample cell into the cell compartment

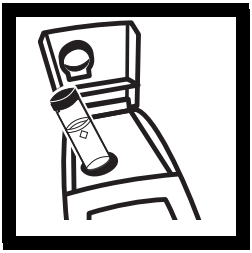
6. The display will show the **S1** icon (with the **1** flashing) and **20 NTU** or the value of the S1 standard for the previous calibration.



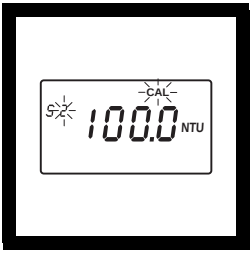
7. Edit the standard concentration by pressing →. The **1** will stop flashing and the left digit in the display will flash. Press ↑ to scroll the digit up to the appropriate number. Press → again to move the cursor to the next digit and edit it in the same manner.



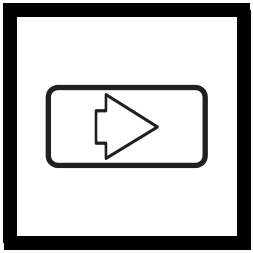
8. When all the digits show the appropriate value, press **READ**. The instrument will count from 60 to 0 (67 to 0 if signal average is on), measure the turbidity and store the value. The display will automatically increment to the next standard. Remove the sample cell from the cell compartment.



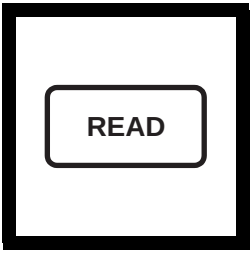
9. Thoroughly mix the 90 to 110 NTU standard, then fill a clean sample cell to the line with the standard. Insert the cell into the cell compartment.



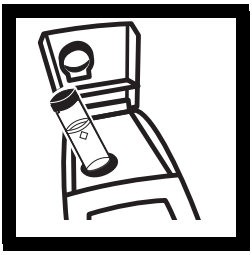
10. The display will show the **S2** icon (with the **2** flashing) and **100 NTU** or the value of the S2 standard for the previous calibration.



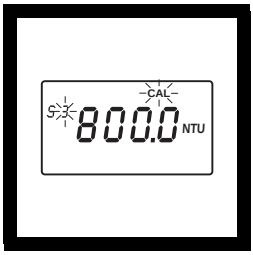
11. Edit the standard concentration by pressing →. The **2** will stop flashing and the left digit in the display will flash. Press ↑ to scroll the digit up to the appropriate number. Press → again to move the cursor to the next digit and edit it in the same manner.



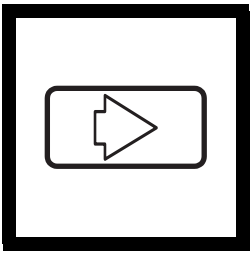
12. When all the digits show the appropriate value, press **READ**. The instrument will count from 60 to 0 (67 to 0 if signal average is on), measure the turbidity and store the value. Remove the sample cell from the cell compartment.



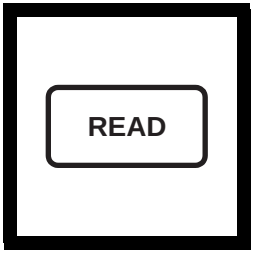
13. Thoroughly mix the 700 to 900 NTU standard, then fill a clean sample cell to the line with the standard. Insert the cell into the cell compartment.



14. The display will show the **S3** icon (with the **3** flashing) and **800 NTU** or the value of the S3 standard for the previous



15. Edit the standard concentration by pressing →. The **3** will stop flashing and the left digit in the display will flash. Press ↑ to scroll the digit up to the appropriate number. Press → again to move the cursor to the next digit and edit it in the same manner.



16. When all the digits show the appropriate value, press **READ**. The instrument will count from 60 to 0 (67 to 0 if signal average is on), measure the turbidity and store the value. The instrument will increment back to **S0**. Remove the sample cell from the cell compartment.

17. Press: **CAL**.

The instrument will store the new calibration data and return the instrument to the measurement mode. It will use the new calibration to calculate turbidity for subsequent measurements.

Note: *Pressing* **CAL** *completes the calculation of the calibration coefficients. If calibration errors occurred during calibration, error messages will appear after* **CAL** *is pressed. If* **E 1** *or* **E 2** *appear, check the standard preparation and review the calibration; repeat the calibration if necessary. If* **CAL?** *appears, an error may have occurred during calibration. If* **CAL?** *is flashing, the instrument is using the default calibration.*

NOTES

- If the **I/O** key is pressed during calibration, the new calibration data is lost and the old calibration will be used for measurements. Once in calibration mode, only the **READ, I/O,** ↑, and → keys function. Signal averaging and range mode must be selected before entering the calibration mode.

- If **E 1** or **E 2** are displayed, an error occurred during calibration. Check the standard preparation and review the calibration; repeat the calibration if necessary. If the error messages recur, calibrate using the factory specified standards, *Section 3.6.2.4* on page *42* and *Section 3.6.3* on page *44*. Press **DIAG** to cancel the error message (**E 1** or **E 2**). To continue without repeating the calibration, press **I/O** twice to restore the previous calibration. If **CAL?** is displayed, an error may have occurred during calibration. The previous calibration may not be restored. Either recalibrate or use the calibration as is.

- To review a calibration, press **CAL** and then only ↑ to view the calibration standard values. As long as **READ** is never pressed and **CAL** isn't flashing, the calibration will not be updated. Press **CAL** again to return to the measurement mode.

3.6.4 Using Gelex® Secondary Turbidity Standards

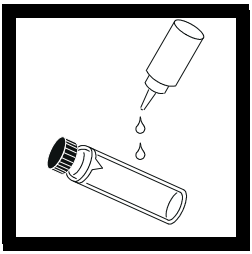
Note: *Store Gelex standards at room temperature. Do not allow to freeze or exceed 50 °C.*

Gelex Secondary Standards (included with 4650000 only) are particulate suspensions similar to formazin primary standards in light scattering characteristics. NTU values on the Gelex standards indicate the range for which they should be used. Due to minor variations in glass and individual instrument optical systems, the true value of the Gelex standards must be determined against formazin in the same instrument they will be used with for later calibration checks.

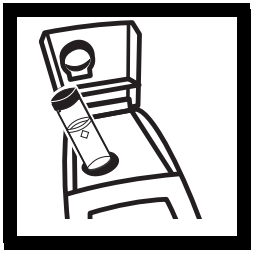
3.6.4.1 Assigning Values to Gelex Standards

1. Calibrate the instrument with formazin.

2. Select automatic range mode using the **RANGE** key.

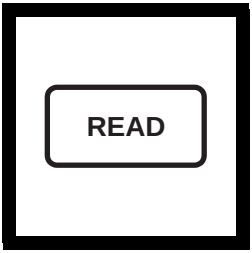


3. Thoroughly clean the outside of the Gelex vials and apply a thin coating of silicone oil.



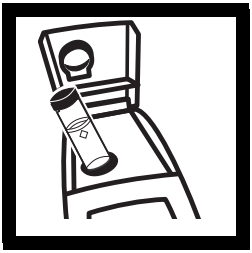
4. Place the 0-10 NTU Gelex standard in the cell compartment so the diamond on the vial aligns with the orientation mark on the instrument. Close the sample lid.

Note: *Correct cell orientation is essential to obtain accurate Gelex values. Always orient the cell so the diamond mark aligns with the orientation mark on the instrument.*



5. Press: **READ**.

Record the displayed value, remove the vial from the instrument and mark the value on the band near the top of the vial.



6. Repeat *step 3* through *step 5* for the other Gelex standards, being careful to orient the cells properly.

Re-assign with every formazin calibration

7. Re-assign values to the Gelex standards each time the instrument is calibrated with formazin.

3.6.4.2 Routine Calibration Check With Gelex Standards

The 2100P Turbidimeter does not require standardization before every measurement as some turbidimeters do. Periodically, as experience dictates, check the instrument calibration using the appropriate Gelex Secondary Standard. Be sure the Gelex standards are aligned correctly when inserting them (diamond aligns with orientation mark). If the reading is not within 5% of the previously established value, recalibrate the instrument with StablCal Stabilized Formazin Primary Standard or formazin primary standard (*Section 3.6.3* on page *44*).

Important Note: DO NOT calibrate with Gelex® Secondary Standards. Gelex standards are designed for instrument verification, not calibration.

MAINTENANCE

Some of the following manual sections contain information in the form of warnings, cautions and notes that require special attention. Read and follow these instructions carefully to avoid personal injury and damage to the instrument. Only personnel qualified to do so, should conduct the maintenance tasks described in this portion of the manual.

Certains des chapitres suivants de ce mode d'emploi contiennent des informations sous la forme d'avertissements, messages de prudence et notes qui demandent une attention particulière. Lire et suivre ces instructions attentivement pour éviter les risques de blessures des personnes et de détoriation de l'appareil. Les tâches d'entretien décrites dans cette partie du mode d'emploi doivent être seulement effectuées par le personnel qualifié pour le faire.

Algunos de los capítulos del manual que presentamos contienen muy importante información en forma de alertas, notas y precauciones a tomar. Lea y siga cuidadosamente estas instrucciones a fin de evitar accidentes personales y daños al instrumento. Las tareas de mantenimiento descritas en la presente sección deberán ser efectuadas únicamente por personas debidamente cualificadas.

Einige der folgenden Abschnitte dieses Handbuchs enthalten Informationen in Form von Warnungen, Vorsichtsmaßnahmen oder Anmerkungen, die besonders beachtet werden müssen. Lesen und befolgen Sie diese Instruktionen aufmerksam, um Verletzungen von Personen oder Schäden am Gerät zu vyyyermeiden. In diesem Abschnitt beschriebene Wartungsaufgaben dürfen nur von qualifiziertem Personal durchgeführt werden.

Algumas das seguintes secções do manual contêm informações em forma de advertências, precauções e notas que requerem especial atenção. Leia e siga atentamente as presentes instruções para evitar ferimentos pessoais e não danificar o instrumento. As tarefas de manutenção descritas nesta parte do manual só poderão ser executadas por pessoal qualificado.

SECTION 4 MAINTENANCE

4.1 Cleaning

Keep the turbidimeter and accessories as clean as possible and store the instrument in the carrying case when not in use. Avoid prolonged exposure to sunlight and ultraviolet light. Wipe spills up promptly. Wash sample cells with non-abrasive laboratory detergent, rinse with distilled or demineralized water, and air dry. Avoid scratching the cells and wipe all moisture and fingerprints off the cells before inserting them into the instrument. Failure to do so can give inaccurate readings. See *Section 2.3.1* on page *23* for more information about sample cell care.

4.2 Battery Replacement

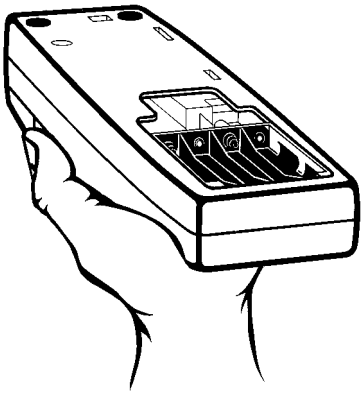
AA alkaline cells typically last for about 300 tests with the signal averaging mode off, about 180 tests if signal averaging is used. The “battery” icon flashes when battery replacement is needed. Refer to *Section 1.4.2* on page *16* for battery installation instructions. If the batteries are changed within 30 seconds, the instrument retains the latest range and signal average selections. If it takes more than 30 seconds, the instrument uses the default settings.

If, after changing batteries, the instrument will not turn off or on and the batteries are good, remove the batteries and reinstall them. If the instrument still won't function, contact Hach Service or the nearest authorized dealer.

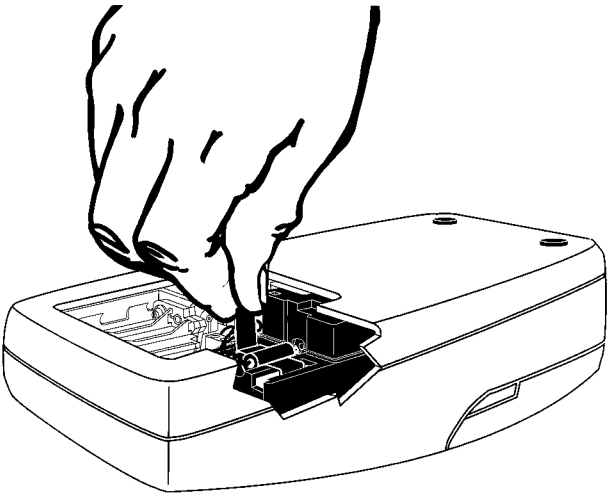
4.3 Lamp Replacement

The procedure below explains lamp installation and electrical connections. Use a small screwdriver to remove and install the lamp leads in the terminal block. The instrument requires calibration after lamp replacement.

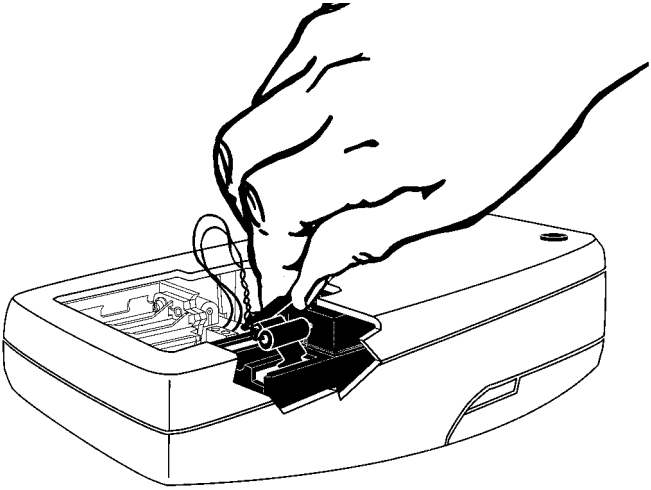
1. Orient the instrument so it is upside down and the top faces away from you. Remove the battery cover and at least one battery.



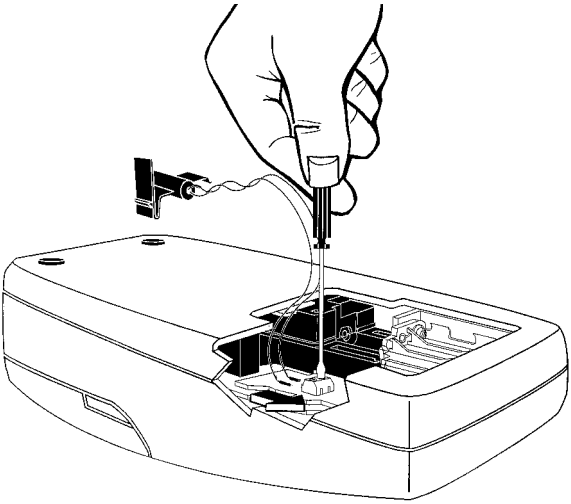
2. Remove the lamp assembly by grasping the tab on the left side of the assembly. Firmly, but gently, slide the assembly towards the rear of the instrument.



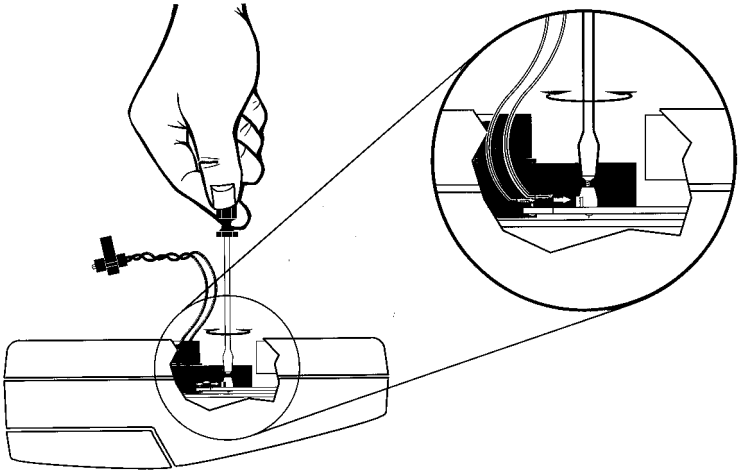
3. Rotate the tab towards the nearest outside edge. The assembly should release and slip out easily.



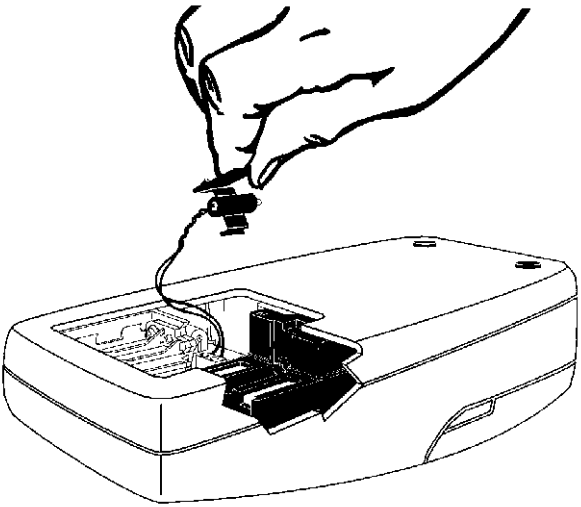
4. Back the terminal block screws **partially** out (1 to 2 turns) and remove the old lamp leads.



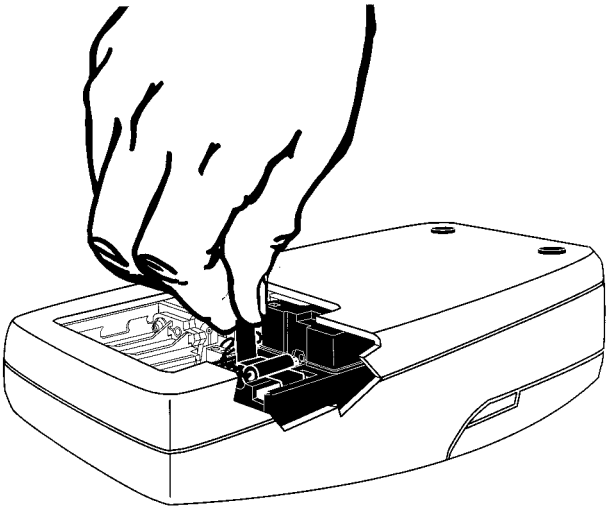
5. Gently bend the wires of the new lamp assembly into an “L” shape so they fit easily into the housing. Insert the leads into the terminal screws and tighten with clockwise turns. Gently tug on the wires to make sure they are connected to the terminal block.



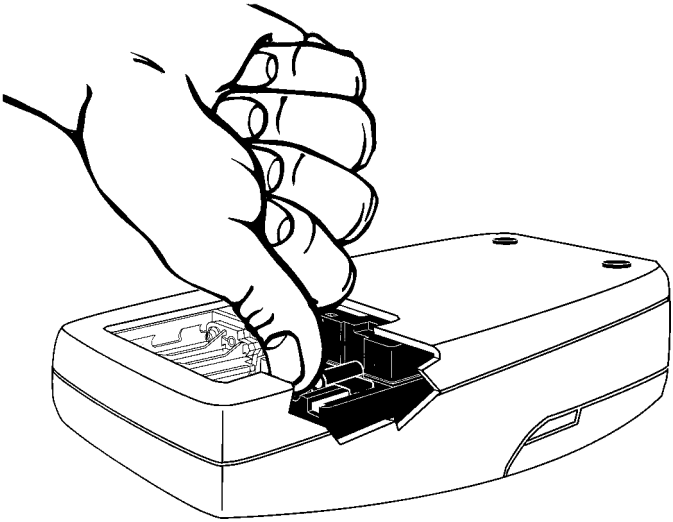
6. Hold the new lamp assembly by the tab with the lamp facing the tope (keyboard) of the instrument. Slide the small catch on the other side of the assembly into the black plastic slot (towards the nearest edge of the instrument).



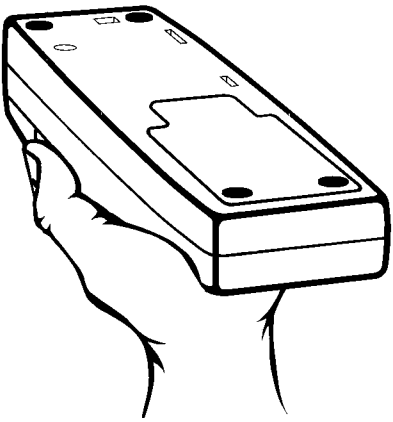
7. Snap the U-shaped bottom of the tab into the slot on the left side of the black plastic that holds the lamp assembly.



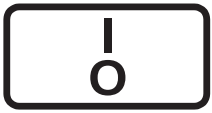
8. With your thumb, firmly slide the assembly forward until it stops. Again, push firmly against the tab to make sure the lamp is seated correctly.



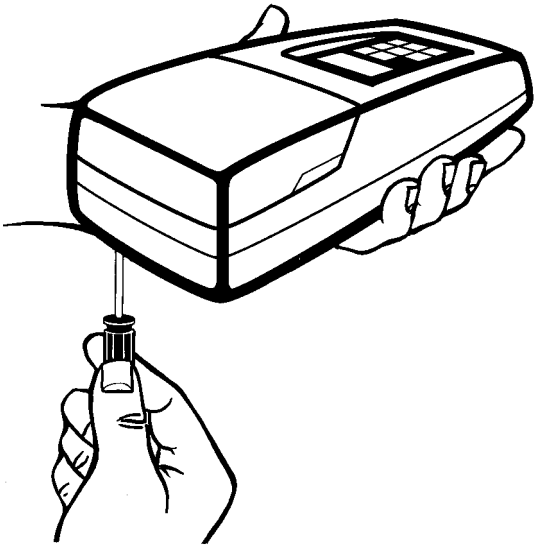
9. Replace the battery(s) and battery cover.



10. Insert the 800 NTU formazin standard into the sample cell. Press and hold **READ**. Then press **I/O**. Release the **READ** key after the software version number disappears from the display (for models with serial numbers less than 920300000800, **2100** disappears).



11. Adjust the scattered light amplifier output by inserting a small flat-bladed screwdriver into the trimpot hole (located on bottom). Adjust the display to read 2.5 ± 0.3 volts (2.0 volts for models that display **2100** when turned on).



12. Press **I/O** to exit gain adjust mode.

13. Perform a formazin calibration according to *Section 3.6.3* on page *44* or *Section 3.6.3.1* on page *48*.

SECTION 5 TROUBLESHOOTING

5.1 Using the Diagnostic Functions Key

Enter the diagnostic mode by pressing the **DIAG** key. Exit this mode at any time by pressing the key again. The diagnostic mode allows access to information about instrument function which may be useful for servicing and troubleshooting.

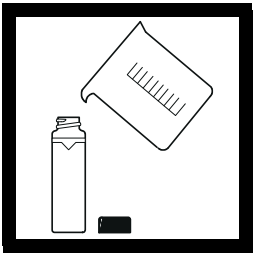
5.1.1 Basic Diagnostic Codes

The diagnostic codes are:

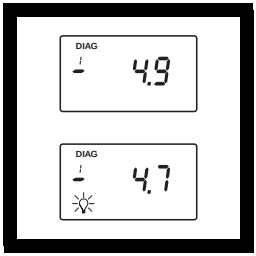
Code	Description
1	Checks the battery voltage with the lamp on, then with the lamp off. This is a dual diagnostic code.
2	Displays calibration coefficient a_o
3	Displays calibration coefficient a_1
4	Displays calibration coefficient b_o
5	Displays calibration coefficient b_1
6	Displays the lamp voltage (about 3 volts)
7	Displays the dark voltage of the transmitted light detector amplifier with the lamp off and the detector amplifier voltage with the lamp on.
8	Displays the high gain dark voltage of the 90° detector amplifier with the lamp off and the detector amplifier voltage with the lamp on.*
9	Displays the low gain dark voltage of the 90° detector amplifier with the lamp off and the detector amplifier voltage with the lamp on.

* Samples with turbidity >10 NTU may display **- - -** for the lamp-on amplifier voltage.

5.2 The Diagnostic Procedure

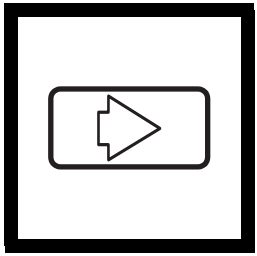


1. Fill a clean sample cell to the line with clear water, cap the cell and place it in the cell compartment. Press the **READ** key and wait until the reading is finished.

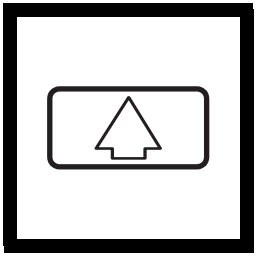


2. Press: **DIAG**

The **DIAG** icon will turn on and **1** will be displayed below the icon. The instrument will measure the battery voltage with the lamp off and display the result in volts (V). Then the lamp icon will turn on and the instrument will measure the voltage with the lamp on. The value is briefly displayed before defaulting to the lamp-off reading. To repeat the measurement, press **READ**.



3. To continuously display the lamp-on voltage, press →. The lamp icon will flash. Press → to turn the lamp icon off (the lamp is not on during this display).



4. Press the ↑ key to scroll through the other diagnostics. Each press of the key increments the digit in the small numerical display below the **DIAG** icon and the result of the diagnostic measurement is then displayed. Each press of the **READ** key updates the value. For measurements made with the lamp off and again with the lamp on, the measurement with the lamp off is displayed when the diagnostic is entered. To see the second measurement with the lamp on, press the → key (only works with diagnostic codes 1, 7, 8, & 9). The lamp icon will flash and the lamp-on measurement will be displayed in volts. Press → to turn the lamp icon off.

Note: *DIAG 8 will display* ---- *for the lamp-on voltage if a of >10 NTU is placed in the cell compartment.*

5.3 Other Instrument Diagnostics

5.3.1 Display Test

Pressing and holding the **I/O** key turns on all the display icons and elements so you can determine if all the elements and icons are functioning. The display test sequence will cycle as long as the key is held down.

5.4 Error Messages

Error messages indicate sample interferences and/or instrument malfunction.

5.4.1 Flashing Numeric Display

If the highest value in the range selected is flashing in the display, the sample is too turbid (or overrange) for the selected range. In automatic or manual range, **1000** flashes if the sample is over the instrument's range. In manual range mode, select the next higher range mode if **9.99** or **99.9** flashes. See *Section 2.3.6* on page *31* for measuring overrange samples. The display will stop flashing if a sample within range is inserted and read.

5.4.2 E Messages

An error message indicates either an instrument failure or an operation cannot be performed. **An error message can be cleared by pressing DIAG** (display will return to previous measurement or calibration value). The meter continues to operate as best it can. If the message occurs during a calibration, calibration can continue. If the error message occurs when a calibration is being calculated, the instrument will discard the new calibration and retain the old calibration. Error messages and corrective actions are listed below.

5.4.3 CAL?

A flashing **CAL?** appears when the instrument is using the default calibration programmed at the factory. It will appear if the analyst has erased the user-entered calibration using the procedure to restore the default calibration or after and E 4 error is cleared by pressing DIAG. Recalibrate as soon as possible when **CAL?** appears. **CAL?** (not flashing) appears when a calibration has questionable validity.

Message*	Probable Cause	Corrective Action
E1	Dilution water is ≥ 0.5 NTU.	Start calibration over with better quality dilution water or use a membrane filter to filter the water before use.
E2	Two standards have the same value or their difference is less than 60 NTU. Not all standards were read during the calibration. Standard 1 is too low (<10 NTU).	Recheck preparation of standards and repeat calibration.
E3	Low light error.	Re-read measurement. Check lamp** Check for obstructed light path. Dilution may be necessary.
E4	EEPROM malfunction.	Check sum failed. Press **I/O**. If E 4 reappears, call Hach service. If **CAL?** appears, recalibrate.
E5	A/D overrange.	Check for obstructed light path. Call Hach Service.
E6	A/D underrange.	Check for open lid during reading and re-read. Check for obstructed light path. If persists, call Hach Service.
E7	Light Leak.	Close lid before pressing **READ** key.
E8	Bad lamp circuit.	Reinsert lamp leads at terminal block-make sure the lead ends are not touching each other.If this fails, call Hach Service.

* Error messages 4, 5, and 6 may indicate a failure in the internal electronics.
** Check lamp by inserting a pencil or piece of paper into the cell compartment and pressing READ. Light should be visible on the inserted object.

GENERAL INFORMATION

At Hach Company, customer service is an important part of every product we make.

With that in mind, we have compiled the following information for your convenience.

Replacement Parts & Accessories

REPLACEMENT PARTS

Description	Cat. No.
StablCal Calibration Set for 2100P, Sealed Vials: <0.1 NTU, 20 NTU, 100 NTU, and 800 NTU	26594-05
AA Batteries, 4/pkg	19380-04
Battery Door	46005-00
Carrying Case	46506-00
Gelex® Standards, set (includes standards and 3 sample cells)	24641-05
Instrument Manual	46500-88
Lamp Assembly, with leads	46539-00
Oiling Cloth	47076-00
Sample Cells, 1 inch, with cap, 6/pkg	24347-06
Silicone Oil, 15 mL	1269-36

OPTIONAL ACCESSORIES AND REAGENTS

Deionized Water, 3.78 L	272-17
Bath, Ultrasonic, 2.8 L (0.75-gal), w/heater	24895-00
Battery Eliminator, 120 V	46079-00
Battery Eliminator, 230 V	46080-00
Filter, 0.2 micron, 10/pkg	23238-10
Formazin, 4000 NTU, 500 mL	2461-49
Formazin, 4000 NTU,100 mL	2461-42
Hexamethylenetetramine, 500 g	1878-34
Hydrazine Sulfate, 100 g	742-26
NiCad Rechargeable Battery (4 required)	16077-00

OPTIONAL ACCESSORIES AND REAGENTS, continued

Description	Cat. No.
Pipet, TenSette®*, 1-10 mL	19700-10
Pipet Tips, for 1-10 mL TenSette Pipet, 50/pkg	21997-96
Pipet Tips, for 1-10 mL TenSette Pipet, 1000/pkg	21997-28
Pipet, Volumetric, Class A, 1.00 mL	14515-35
Pipet, Volumetric, Class A, 5.00 mL	14515-37
Pump, Vacuum, Hand-Operated	14283-00
Sample Degassing Kit	43975-00
Sample Filtration and Degassing Kit	43975-10
StablCal® Calibration Set for 2100P Turbidimeter	
<0.1, 20, 100, 800 NTU, 500 mL each	26594-00
<0.1, 20, 100, 800 NTU, 100 mL each	26594-10
<0.1 NTU** StablCal®*** Stabilized Formazin Standard,100 mL	26597-42
20 NTU StablCal® Stabilized Formazin Standard, 100 mL	26601-42
100 NTU StablCal® Stabilized Formazin Standard, 100 mL	26602-42
800 NTU StablCal® Stabilized Formazin Standard, 100 mL	26605-42
Triton-X Solution, 118 mL (4 oz)	14096-32
Volumetric Flask, 100 mL	14574-42
Volumetric Flask, 200 mL	14574-45

* TenSette™ is a Hach Company trademark.

** <0.1 NTU StablCal® Standard is used in place of dilution water standard when performing a calibration.

*** StablCal® is a registered trademark of Hach Company.

HOW TO ORDER

By Telephone:

6:30 a.m. to 5:00 p.m. MST
Monday through Friday
(800) 227-HACH
(800-227-4224)
By FAX: (970) 669-2932

By Mail:

Hach Company
P.O. Box 389
Loveland, CO 80539-0389
U.S.A.

Ordering information by E-mail: orders@hach.com

Information Required

- Hach account number (if available)
- Your name and phone number
- Purchase order number
- Brief description or model number
- Billing address
- Shipping address
- Catalog number
- Quantity

Technical and Customer Service (U.S.A. only)

Hach Technical and Customer Service Department personnel are eager to answer questions about our products and their use. Specialists in analytical methods, they are happy to put their talents to work for you. Call **1-800-227-4224** or E-mail **techhelp@hach.com**.

International Customers

Hach maintains a worldwide network of dealers and distributors. To locate the representative nearest you, send E-mail to **intl@hach. com** or contact:

In Canada, Latin America, Africa, Asia, Pacific Rim:
Telephone: (970) 669-3050; FAX: (970) 669-2932

In Europe, the Middle East, or Mediterranean Africa:
HACH Company, c/o
Dr. Bruno Lange GmbH
Willstätterstr. 11
D-40549 Düsseldorf
Germany
Telephone: +49/[0]211.52.88.0
Fax: +49/[0]211.52.88.231

REPAIR SERVICE

Authorization must be obtained from Hach Company before sending any items for repair. Please contact the HACH Service Center serving your location.

In the United States:
Hach Company
100 Dayton Avenue
Ames, Iowa 50010
(800) 227-4224 (U.S.A. only)
Telephone: (515) 232-2533
FAX: (515) 232-1276

In Canada:
Hach Sales & Service Canada Ltd.
1313 Border Street, Unit 34
Winnipeg, Manitoba
R3H 0X4
(800) 665-7635 (Canada only)
Telephone: (204) 632-5598
FAX: (204) 694-5134
E-mail: canada@hach.com

In Latin America, the Caribbean, the Far East, the Indian Subcontinent, Africa, Europe, or the Middle East:
Hach Company World Headquarters
P.O. Box 389
Loveland, Colorado, 80539-0389
U.S.A.
Telephone: (970) 669-3050
FAX: (970) 669-2932
E-mail: intl@hach.com

WARRANTY

Hach warrants most products against defective materials or workmanship for at least one year from the date of shipment; longer warranties may apply to some items.

HACH WARRANTS TO THE ORIGINAL BUYER THAT HACH PRODUCTS WILL CONFORM TO ANY EXPRESS WRITTEN WARRANTY GIVEN BY HACH TO THE BUYER. EXCEPT AS EXPRESSLY SET FORTH IN THE PRECEDING SENTENCE, HACH MAKES NO WARRANTY OF ANY KIND WHATSOEVER WITH RESPECT TO ANY PRODUCTS. HACH EXPRESSLY DISCLAIMS ANY WARRANTIES IMPLIED BY LAW, INCLUDING BUT NOT BINDING TO ANY WARRANTY OF MERCHANTABILITY OR FITNESS FOR A PARTICULAR PURPOSE.

LIMITATION OF REMEDIES: Hach shall, at its option, replace or repair nonconforming products or refund all amounts paid by the buyer. **THIS IS THE EXCLUSIVE REMEDY FOR ANY BREACH OF WARRANTY.**

LIMITATION OF DAMAGES: IN NO EVENT SHALL HACH BE LIABLE FOR ANY INCIDENTAL OR CONSEQUENTIAL DAMAGES OF ANY KIND FOR BREACH OF ANY WARRANTY, NEGLIGENCE, ON THE BASIS OF STRICT LIABILITY, OR OTHERWISE.

This warranty applies only to Hach products purchased and delivered in the United States.

Catalog descriptions, pictures and specification, although accurate to the best of our knowledge, are not a guarantee or warranty.

For a complete description of Hach Company’s warranty policy, request a copy of our Terms and Conditions of Sale for U.S. Sales from our Customer Service Department.

APPENDIX D

Bristol Standard Operating Procedures

BRISTOL ENVIRONMENTAL REMEDIATION SERVICES, LLC

SOIL SAMPLING

STANDARD OPERATING PROCEDURE BERS-01

Record of Changes

Revision No.	Date	Prepared by	Approved by
0	01/15/08	B. Allen	S. Ruth
1	10/14/09	L. Maserjian	P. Curl
2	02/17/10	L. Maserjian	B. Allen

SOIL SAMPLING

STANDARD OPERATING PROCEDURE

Summary: Soil samples may be collected using a variety of methods and equipment. The methods and equipment used are dependent on the depth of the desired sample, the type of the sample required, and the soil type. Manual techniques and equipment, such as hand augers, are usually used for surface or shallow, subsurface soil sampling. Power-operated equipment is usually associated with collecting deep samples, but this equipment can also be used for collecting shallow samples.

Soil samples collected for volatile organic compound (VOC) analysis are handled in a manner that minimizes the loss of contaminants due to volatilization and biodegradation. Where required to meet project objectives, field extraction and preservation with methanol are performed to ensure sample integrity and representativeness during sample handling and transport.

Health and Safety: Sampling activity should only be conducted in accordance with an approved Site Health and Safety Plan.

Personnel Qualifications: Sampling personnel will be trained and certified as hazardous site workers per Title 29 Code of Federal Regulations, Part 1910.120(e) [29 CFR 1910.120(e)]. If applicable, additional qualification requirements will be specified in the site Quality Assurance Project Plan (QAPP) and will be met.

Equipment and Materials: Prior to deployment in the field, the requisite sampling equipment and materials will be identified, secured, and inspected for signs of damage or potential contamination. Sampling equipment will be selected to preserve the chemical and physical integrity of the samples. Equipment selection will be based on the depth of the samples to be collected and, to a certain extent, the characteristics of the material being sampled. Equipment used for sampling trace contaminants should be constructed of inert materials, such as Teflon® or stainless steel. Ancillary equipment, such as auger flights, post hole diggers, etc., may be constructed of other materials if this equipment does not come in contact with the samples. However, plastic, chromium, galvanized, painted, or rusted equipment should not be used for routine soil sampling operations.

- Sample containers for collecting samples using the methanol extraction and preservation method must be prepared and weighed in advance by the laboratory performing the analysis. Required equipment may include disposable plastic syringes and a clean, sharp utility knife.
- Surface soil is generally classified as soil between the ground surface and 6 inches below ground surface (bgs). Surface soil sampling equipment typically consists of spoons, shovels, hand-augers, push tubes, and post-hole diggers.

- Subsurface soil is generally soil that is at least 6 inches bgs, and can be collected using manual or powered sampling devices. Manual sampling devices consist of hand augers, push tubes, and post-hole diggers. Powered devices typically consist of power augers; split-spoon samplers, which are driven with a drill rig drive-weight assembly or hydraulically pushed using drill rig hydraulics; continuous split-spoon samplers; specialized hydraulic cone penetrometer rigs; and/or backhoes.
- In addition to soil sampling equipment, sampling support equipment may include Global Positioning System (GPS) or survey equipment for locating sample points, organic vapor analyzer with a photoionization detector (PID), tape measures, survey stakes or flags, stainless steel buckets/bowls or disposable aluminum pie pans, canvas/plastic sheet, pre-cleaned sample containers, decontamination supplies and equipment, safety equipment, logbooks, camera, chain-of-custody forms, and supplies for sample labeling, packaging, and shipping.
- Sample containers will be of the type and size specified in the governing QAPP.

Field Screening

Field-screening samples are typically collected either before or concurrently with laboratory analytical samples. Field screening before sample collection can help guide the selection of the most appropriate location to collect a laboratory analytical sample. Collecting field-screening samples concurrently with laboratory samples can help establish a correlation between screening and analytical results at a particular site. Field screening is commonly performed using an organic vapor analyzer, such as a PID.

Headspace PID Screening

Headspace PID screening samples are collected by filling a resealable Ziploc® bag approximately one-third to one-half full of freshly exposed or uncovered soil, and immediately sealing the bag. The soil should be agitated and then allowed to warm for approximately five minutes to an hour, to allow the headspace vapors to develop. After the headspace vapors have developed, insert the tip of a calibrated PID into the void headspace of the bag, and record the highest reading.

In-Situ PID Screening

In some instances, where a limited volume of soil is available for field screening, such as soil cores from a direct-push rig or split spoon, using the headspace method may use up material that potentially could be used for a laboratory analytical sample. In these cases, field screening of the soil may be performed by making small divots approximately every 6 inches along the length of the core, and inserting the calibrated PID tip just above the freshly exposed divot, taking care not to touch the material. Record the highest reading at each location in the field logbook or field form.

Surface Soil Sampling

1. If a thick, matted root zone is encountered at or near the surface, remove it before collecting the sample.
2. Carefully remove the top layer of soil or debris to the desired sample depth with a pre-cleaned spade.
3. Using a pre-cleaned, stainless steel scoop, spoon, or trowel, remove and discard a thin layer of soil from the area that came in contact with the spade.
4. Collect samples following procedures described in the General Soil Sampling Procedures Section.

Subsurface Soil Sampling: Subsurface samples can be collected using hand or power augers, Geoprobes®, split-spoon samplers, or from backhoes.

- Augering is the most common method used to collect shallow subsurface samples. The auger is used to bore a hole to the desired sampling depth. VOC samples are generally collected directly from the sampling device. Non-VOC samples are collected after thorough mixing. If a core sample is required, the auger tip is replaced with a thin wall tube sampler, and the system is lowered into the borehole and driven to the required sample depth. The system is withdrawn and the core is collected from the thin wall tube sampler.
- Geoprobe sampling uses a direct-push system that employs percussion power to essentially "hammer" sampling equipment (Macro-Core® samplers) into the subsurface to extract soil for laboratory analysis. The advantage of using a Geoprobe is that there is a much smaller hole diameter and minimal soil cuttings. The Macro-Core sampler is a solid barrel that is pushed into the subsurface for collecting continuous core samples of unconsolidated materials at depth. The Macro-Core soil samples are collected in a 4 to 5 foot long Teflon, polyvinyl chloride (PVC), or polyethylene terephalate glycol (PETG) liner; the samples can be obtained by splitting the liner or capped to it to preserve the samples for future analysis.
- On underground storage tank or contaminated soil excavation sites, a backhoe bucket is commonly used for collecting soil for samples. When a trench or excavation is deeper than four feet, the bucket of the backhoe or excavator will be used to collect soil, so that personnel do not climb into an unprotected hole.
- Power hand augers are commonly used to aid in the collection of subsurface soil samples at depths where hand augering is impractical. This equipment is a sampling aid, and not a sampling device; the typical lower depth range available with these devices is 20 to 25 feet. The power auger is used to advance a hole to the required sampling depth, at which point a hand auger is usually used to collect the sample.
- Split-spoon sampling provides for the collection and extraction of undisturbed soil cores of 18 or 24 inches in length. A series of consecutive cores may be extracted to

give a complete soil column profile, or an auger may be used to drill down to the desired depth for sampling. The split spoon is then driven to the sampling depth through the bottom of the augered hole, and the core is extracted. When used in conjunction with drilling, split-spoon samplers are usually driven either inside a hollow-stem auger or inside an open borehole after the rotary or cable tool drilling equipment has been temporarily removed.

- When split spoon or Macro-Core sampling is performed to gain geologic information, sampling will be performed in accordance with SOP BERS-06 Borehole Logging.
- Continuous split-spoon samplers may be used to obtain five-foot-long, continuous samples, approximately 3 to 5 inches in diameter. These devices are placed inside a five-foot section of hollow-stem auger and advanced with the auger during drilling. As the auger advances, the central core of soil moves into the sampler.
- Cone Penetrometer Rigs use a standard split spoon that is modified with a releasable tip to keep the spoon closed during the sampling push. Upon arrival at the desired depth, the tip can be remotely released and the push continued. During the subsequent push, the released tip floats freely up the inside of the sample barrel as the soil core displaces it. Split-spoon soil samples, therefore can be collected without drilling by simply pushing the device to the desired depth. This technique is particularly beneficial at highly contaminated sites, because cuttings are not produced as with drill rigs. This results in limited investigation-derived waste (IDW) and minimal exposure to sampling personnel.

Sampling using a Hand Drill with an Auger Attachment

1. Attach the auger bit to a drill-rod extension, and attach the “T” handle to the drill rod.
2. Clear the area to be sampled of any surface debris (e.g., twigs, rocks, and litter). It is generally advisable to remove the first three to six inches of surface soil.
3. Begin augering, periodically removing the auger from the hole and depositing accumulated soils onto a plastic sheet spread near the hole.
4. After reaching the desired depth, slowly and carefully remove the auger from the boring.
5. Remove auger tip from drill rods and replace with a pre-cleaned, thin-wall tube sampler. Install the proper cutting tip.
6. Carefully lower the tube sampler down the borehole. Gradually force the tube sampler into the soil. Take care to avoid scraping the borehole sides. Avoid hammering the drill rods to facilitate coring as the vibrations may cause the boring walls to collapse.
7. Remove the tube sampler and unscrew the drill rods.
8. Remove the cutting tip and the core from the device.
9. Discard approximately 1 inch off the top of the core. Place the remaining core into a labeled sample container without mixing.

10. Collect samples following procedures described in the General Soil Sampling Procedures Section.
11. If another sample is to be collected in the same hole, but at a greater depth, reattach the auger bit to the drill and assembly and follow steps 3 through 10, making sure to decontaminate the auger and tube sampler between samples.
12. Abandon the hole according to applicable state regulations and corporate procedures.

Sampling using a Geoprobe with a Macro-Core Attachment

1. Clear the area to be sampled of any surface debris (e.g., twigs, rocks, and litter). It is generally advisable to remove the first three to six inches of surface soil.
2. Begin hammering the Geoprobe.
3. After hammering the depth of the Macro-Core (either 4 or 5 feet in length), slowly and carefully remove the Macro-Core from the hammer.
4. Open the Macro-Core tubing using a two-razor tool designed for the Macro-Core.
5. Use a pre-cleaned stainless steel spoon or knife to obtain soil from the core at selected locations, based on PID field screening.
6. Collect samples following procedures described in the General Soil Sampling Procedures Section. The Macro-Core is not reusable. Remove any soil on the core material, place the used core in a plastic trash bag (with as many used cores as will fit), tie the ends of the plastic bag, and dispose of it in a trash receptacle.
7. Abandon the hole according to applicable state regulations and corporate procedures.

Sampling with a Hand Auger

1. Insert the hand auger into the material to be sampled at a 0° to 45° angle from the horizon.
2. Rotate the auger once or twice to cut a core of material.
3. Slowly withdraw the auger, with the slot facing upward.
4. Collect samples following the procedures described in the General Soil Sampling Procedures Section.

Sampling with a Split Spoon

1. Assemble the sampler by aligning both sides of barrel, then screw the drive shoe on the bottom and the headpiece on top.
2. Place the sampler perpendicular to the material sampled.
3. Using a well ring, drive the tube. Do not drive the tube past the bottom of the head piece, or the sample may be compressed.

4. Record the length of the tube used to penetrate the material being sampled, and the number of blows required to obtain this depth in the site logbook or on field data sheets.
5. Withdraw the sampler and open by unscrewing the bit and head, and splitting the barrel. Record the amount of recovery and soil type on the boring log. If a split sample is required, use a clean stainless steel knife to divide the tube contents in half, lengthwise.
6. Without disturbing the core, collect samples following the procedures described in the General Soil Sampling Procedures Section.

Sampling from a Backhoe

1. If backhoe buckets are not cleaned in between sample locations, collect samples from material in the center of the bucket.
2. Prior to collecting samples from soil in the backhoe bucket, dress the surface with a stainless steel shovel, spatula, knife, or spoon, to remove at least six inches of surface layer of soil, which may have been smeared across the trench wall as the bucket passed.
3. Be aware of "sluff" material in the bucket that is not representative of the specified sample depth.
4. Collect samples following procedures described in the General Soil Sampling Procedures Section.

General Soil Sampling Procedures:

1. If the QAPP requires field screening for VOCs using a PID, follow the procedures described in the Field Screening Section.
2. Collect samples in appropriate containers in order of volatility, with the most volatile samples collected first. Containers should be either pre-labeled or labeled immediately after sample collection. Follow the procedures for collecting volatile samples described in the following section.
3. For non-VOC samples, place the material into the appropriate container.
4. If a composite non-VOC sample is required, place the material from the designated sampling intervals or locations into a mixing bowl, mix thoroughly, and collect the sample from the mixture into the appropriate container.
5. If non-VOC duplicate, split, duplicate, or other quality assurance/quality control (QA/QC) samples are required, collect twice the routine amount of sample material, mix thoroughly, and fill two identical sets of sample containers.
6. Fill sample containers to the top with measures taken to prevent soil from remaining in the lid threads prior to being sealed.
7. After sample containers are filled, immediately seal them, chill them, and process them for shipment to the laboratory.

Volatile Sample Collection – Sample Collection for Methanol or other Preservative Extraction

General procedures for all volatile sample collection:

- Soil samples for volatile analysis can be collected using any of the sampling methods described above.
- When collecting soil for volatile sample analysis, always submit a separate non-preserved sample for moisture analysis/dry weight calculation, unless already submitting non-volatile samples from the same location.
- Never composite VOC samples.
- If VOC duplicate, split, or other QA/QC samples are required, collect and containerize samples that are co-located, not composited.
- If VOC samples are required, transfer the sample into a labeled sample container with a stainless steel laboratory spoon, or equivalent, and secure the cap tightly.
- Avoid placing pebbles or other large particles into the sample. To the extent practical, the sample should consist of sand, silt, or clay, with care to avoid rocks or pebbles.
- Ensure that the threads on the sample container and cap are free of soil particles. Wipe with a clean brush or paper towel if needed. The sample container should be open for the shortest time possible to prevent evaporation of the methanol and surrogate solution.
- After soil is placed in methanol or other preservative, it should be gently agitated or swirled so that the soil is immersed in the preservative. Do not shake the sample, as it may cause undue volatilization.

The different methods of collecting volatile samples with field extraction, using methanol or another preservative, are described in general below. Refer to the project QAPP for site-specific information on specific soil and methanol volumes required for the appropriate analytical method:

<u>Measuring 10 grams of soil into a VOA vial containing methanol</u>:

1. "Zero" one 40-milliliter volatile organic analyte (VOA) vial containing 10 milliliters of methanol on a small scale.
2. Use a disposable scoop to collect soil.
3. Very gently, transfer the soil into the vial until 10 grams of soil is weighed. Try not to let any soil drop outside the sample container onto the scale. Immediately cap the vial.
4. Ensure that the methanol does not splash. If methanol splashes or spills from the sample container, discard the container and re-sample.
5. Record the tare weight onto the sample sheet or label.

6. Repeat the process for the second VOA vial containing methanol.
7. Place the samples in a protective sleeve and store on ice until delivery to the laboratory.

Using a sampling coring device to collect soil for VOC analysis:

1. Coring devices (for example, En Core® or Terracore®) are disposable, and are not to be reused after each sample.
2. Push the core sampler into freshly exposed soil until the sample chamber is filled. Most of these devices deliver approximately 5 grams of soil.
3. Once the core is filled with soil, retrieve the coring device from the soil.
4. Wipe all soil from outside of the sampler. The soil plug should be flush with the mouth of the sampler.
5. If the QAPP requires using a preservative (for example methanol or sodium bisulfate), use the following preservation procedure:
    a. Place the mouth of the sampler into a pre-tared VOA vial containing the appropriate preservative, and extrude the sample by pushing the plunger down. Immediately cap the VOA vial.
    b. Place the labeled sample in protective padding and on ice.
6. If the QAPP requires freezing the samples unpreserved, use the following procedure:
    a. Place the mouth of the sampler into a pre-tared VOA vial containing exactly 5 milliliters of deionized water, and extrude the sample by pushing the plunger down.
    b. Cap the VOA vial and be sure the soil is below the water level. Gently swirl the vial.
    c. Repeat the process to collect a second soil vial.
    d. Immediately place the labeled sample in protective padding and on ice.
    e. As soon as practical, freeze sample in a freezer or by placing in a cooler containing dry ice.
    f. When freezing the soil vials, it is recommended that the vials be placed at a 45° angle to reduce the likelihood of vial breakage due to freezing.
7. If the QAPP requires submitting unpreserved, unfrozen samples that were collected using an EnCore device, use the following procedure:
    a. Immediately place the cap on the open end of the core. Place the capped core inside the foil sample bag. Make sure that the sample bag is labeled.
    b. Place the sample bag on ice for shipment to the laboratory for analysis within 48 hours.

Quality Control:

The following procedures apply:

- Samples will be packaged, handled, and shipped in accordance with SOP BERS-03 *Sample Management Procedures.*
- Equipment will be operated and used in accordance with the manufacturer's instructions, unless otherwise specified in the site QAPP.
- Equipment examination activities should occur prior to field deployment, and they should be documented.
- An equipment rinsate blank is generally required per matrix, and for each sampling event, to evaluate the potential of cross contamination from sampling equipment. Equipment rinsate blanks will be collected by pouring analyte-free water over the decontaminated sampling equipment.
- Depending on the needs of the project, a field blank may be required per matrix and for each sampling event to evaluate whether contaminants have been introduced into the samples during the sampling process. Field blank samples will be obtained by pouring analyte-free water into a sampling container at the sampling point.

Interferences and Potential Problems:

There are two primary problem areas associated with soil sampling: cross-contamination and improper sample collection.

Cross-contamination can be eliminated or minimized through the use of dedicated sampling equipment. If this is not possible or practical, field personnel will decontaminate sampling equipment as described in the site Sampling and Analysis Plan (SAP). Improper techniques may include using contaminated equipment, disturbing the matrix, compacting the sample, and inadequate homogenization of the samples, any of which can produce non-representative samples.

To safeguard against collecting non-representative soil samples, the following guidelines and techniques should be adhered to during sampling:

- Samples for VOC analysis should be collected before other samples are collected, and should be transferred immediately from the sampling device into the sample container to reduce volatilization. Step-by-step instructions for field extraction and preservation with methanol are described above.
- Anytime a vertical or near vertical surface, such as that which is achieved when shovels or backhoes are used for subsurface sampling, the surface should be dressed to remove the outer smear zone. This is necessary to minimize the effects of cross contamination due to smearing of materials from other levels.

- It is extremely important that soil samples intended for non-VOC analyses be mixed as thoroughly as possible to ensure that each sample is representative of the material sampled. The most common method of mixing is referred to as quartering. Where required by the QAPP, quartering will be performed as follows:
  a. Divide the material in the sample pan into quarters and mix each quarter individually.
  b. Mix two quarters to form halves.
  c. Mix the two halves to form a homogenous matrix.
  d. Repeat this procedure until the sample is adequately mixed.
  e. If round bowls are used for sample mixing, stir the material in a circular fashion, reversing direction, and occasionally turning the material over.
- One trip blank per cooler is generally required when submitting samples for VOC analysis. Trip blanks are prepared and sealed by the laboratory. They are transported to the field and returned, unopened, to the laboratory in the same cooler as the samples collected for VOC analysis.
- Methanol blanks may also be required when soil samples designated for VOC analysis are preserved with methanol.
- Blanks will be collected at the frequency and locations specified in the site QAPP. Blanks will be analyzed for the same target analytes as the associated field samples. Each blank will be assigned a unique sample number, and submitted blind to the laboratory.

BRISTOL ENVIRONMENTAL REMEDIATION SERVICES, LLC

GROUNDWATER SAMPLING

STANDARD OPERATING PROCEDURE BERS-02

Record of Changes

Revision No.	Date	Prepared by	Approved by
1	10/14/09	B. Allen	L. Maserjian
2	02/16/2010	J. Clark	B. Allen/ J. Clark

GROUNDWATER SAMPLING

STANDARD OPERATING PROCEDURE

Summary: Groundwater samples are usually obtained from either temporarily or permanently installed groundwater monitoring wells. In order to obtain a representative groundwater sample, the stagnant water in the well casing and the water immediately adjacent to the well are purged before sample collection. Depending on the needs of the project, purging can be performed either by traditional methods (purging several full well volumes), or by the low stress/low flow method. Once purging is complete, samples are collected using a sampling device that does not affect the integrity or representativeness of the sample.

Health and Safety: Sampling activity should only be conducted in accordance with an approved Site Health and Safety Plan. Electric generators must be grounded to prevent possible electrical shock.

Interferences and Potential Problems: The primary problems associated with groundwater sampling are the collection of non-representative samples, and sample contamination from equipment or the environment. These can be eliminated or minimized through implementation of strict well purging and sample collection and handling procedures, and by the use of qualified personnel.

To safeguard against collecting non-representative stagnant water, the following guidelines and techniques should be adhered to during sampling:

- Monitoring wells should be pumped or bailed prior to sampling. This should be done in a manner that minimizes alterations to the water chemistry.
- The well should be sampled as soon as possible after purging and stabilization of indicator field parameters.
- Analytical parameters typically dictate whether the sample should be collected through the purging device or through separate sampling equipment.
- Portions of water that have been tested with a field meter probe will not be collected for chemical analysis.
- Excessive pre-pumping of the well should be avoided.

Personnel Qualifications: Sampling personnel will be trained and certified as hazardous site workers per Title 29 Code of Federal Regulations, Part 1910.120e [29 CFR 1910.120(e)]. If applicable, additional qualification requirements will be specified by the Bristol Quality Control Manager prior to any on-site sampling activity.

Equipment and Materials: Prior to deployment in the field, the requisite sampling equipment and materials will be identified, secured, and inspected for signs of damage or potential contamination.

- Ideally, purging and sample withdrawal equipment should be completely inert, economical, easily cleaned, reusable, able to operate at remote sites in the absence of power resources, and capable of delivering variable rates for sample collection. Adjustable rate, submersible and peristaltic pumps are preferred. Peristaltic pumps are only effective if groundwater depths are approximately 25 feet below the ground surface or shallower. When sampling for volatile contaminants, a pump that minimizes or eliminates volatilization should be selected. The use of inertial pumps is discouraged because of their tendency to cause greater disturbance during purging and sampling.
- Sampling and purging equipment (e.g., bailers, bladders, pumps, and tubing) should be made from stainless steel, Teflon®, polypropylene, or glass.
- The use of 1/4 or 3/8-inch inner diameter tubing is preferred. Clean, pharmaceutical grade tubing should be used in drawing and sampling groundwater. Water level measuring devices should be capable of measuring to 0.01-foot accuracy.
- In addition to groundwater sampling equipment, sampling support equipment may include water level indicators, depth sounder, water quality meter (such as YSI), keys for well caps, organic vapor screening device (such as photoionization detector [PID]), plastic sheeting, tubing, pre-cleaned sample containers, sample preservatives, decontamination supplies and equipment, safety equipment, logbooks, field forms, camera, chain- of-custody forms and seals, coolers and ice packs, and labeling, packaging, and shipping supplies. Sample containers will be of the type and size specified in the governing Quality Assurance Project Plans (QAPPs).

Field Preparation: Perform the following steps before any purging or sampling activities:

1. Pre-label and ready all the required sample containers.
2. To the extent known, plan to sample wells in order of increasing contamination.
3. Check the well for security damage or evidence of tampering, and record observations.
4. Record location, time of day, and date in field notebook.
5. Remove locking well cap and well casing cap.
6. Screen well headspace with a PID or equivalent, to determine the presence or absence of volatile organic compounds. Record instrument readings in the field logbook or field form.
7. Lower a water-level measuring device into the well until water surface is encountered and the instrument alarms.

8. Measure distance from water surface to reference measuring point on well casing or protective barrier post, and record in the field logbook or on the field form. If there is no reference point, measure from the top of the steel casing, top of PVC riser pipe, from ground surface, or some other position on the wellhead, and note in the field logbook or field form.
9. Measure the total depth of the well and record in the field logbook or field form. Measure well depth either the day before sampling or after all sampling in that well has been completed. Take care to minimize disturbance of the water column.
10. Calculate the volume of water in the well using the following calculations and data reduction:

    Well volume: $V = 0.041d^2h$

    V = volume of one well casing of water in *gallons*

    d = inner diameter of the well casing in *inches*

    h = total height of the water column in *feet*

    Based on this equation, one well volume can be calculated simply by multiplying the height of the water column in feet by the appropriate conversion factor, which is based on the casing diameter as follows:

Diameter	2-inch	3-inch	4-inch	5-inch	6-inch
Volume (gal/ft.):	0.1632	0.3672	0.6528	1.02	1.4688

11. Select the appropriate purging and sampling equipment based on requirements in the site-specific QAPP.

Purging: To ensure that a representative groundwater sample is collected, a well is typically purged prior to sample collection. Well purging is accomplished either by using low-flow procedures or removing a prescribed volume of water from the well (usually a minimum of three to five well volumes). During both purging methods, water quality parameters should be monitored for stabilization.

Purging may be performed by using bailers or pumping mechanisms. In general, a pump is preferred over a bailer for purging and sampling because it will not stress the well like dropping a bailer into the well. If using a pump, select a low removal rate in order to not stress the well. Tubing should remain filled with water, so as to minimize possible changes in water chemistry upon contact with the atmosphere.

If possible, avoid purging wells to dryness by slowing the purge rate. If the well has a poor recharge rate and is purged dry, sample the well once the water level has recovered sufficiently to collect the appropriate volumes for all required analyses. Record in the field logbook or on the field form that samples were collected, even though water quality parameters did not stabilize or the required volume of water was not removed.

If water quality parameters have not stabilized after 1 hour of purging, options include continued purging until stabilization is achieved, or collecting samples although stabilization has not been achieved. Record all actions taken in the field logbook or field form.

Once the purging requirements have been met, the groundwater sample can be collected. Collect and dispose of purge water and solid investigation-derived waste (IDW) as prescribed in the site-specific QAPP.

These procedures are used for sampling events that require purging prior to sampling. For some projects, sampling may be performed without purging the well first. Refer to the non-purge sampling procedures.

Low-flow purging

For low-flow purging and sampling, the Region 1 U.S. EPA Low Flow Guidance Document [*Low Stress (low flow) Purging and Sampling Procedure for the Collection of Ground Water Samples from Monitoring Wells*, July 30, 1996, Revision 2] will be followed, and is summarized below.

1. After the water level and total well depth have been measured, lower the submersible pump or tubing (Teflon, polyethylene, or other approved material) for peristaltic pump slowly (to minimize disturbance) into the well to the middle of the submerged, screened interval of the well, or appropriate depth based on site-specific conditions. Placing the pump or tubing in this manner will reduce the risk of drawing down the water table to below the pump intake, thus preventing the introduction of air into the sample tubing.
2. Before starting the pump, measure the water level and record it on the Groundwater Low Flow Purging Form.
3. Start the pump at its lowest speed setting and slowly increase the speed until discharge occurs. Check water level. Adjust pump speed until there is little or no water level drawdown (less than 0.3 feet). If the minimal drawdown that can be achieved exceeds 0.3 feet, but remains stable, continue purging until indicator field parameters stabilize (described in Number 5, below).
4. Monitor and record water level and pumping rate every 3 to 5 minutes during purging. If a flow rate meter is present, record the pumping rate every 3 to 5 minutes as well. Record any pumping rate adjustments (both time and flow rate). Pumping rates should, as needed, be reduced to the minimum capabilities of the pump to ensure stabilization of indicator parameters. Adjustments are best made in the first 15 minutes of pumping. The final purge volume must be greater than the stabilized drawdown, plus the extraction tubing volume.
5. Monitor indicator field parameters every 3 to 5 minutes during purging, with a calibrated combination type meter (i.e., YSI, etc.). The following field parameters will be monitored: turbidity, temperature, specific conductance, pH, oxidation-

reduction potential (ORP), and dissolved oxygen (DO). All measurements, except turbidity, must be obtained using a flow-through cell. Transparent flow-through cells are preferred. This allows the field personnel to watch particulate buildup within the cell. If the cell needs to be cleaned during purging, continue pumping and disconnect the cell for cleaning. Then reconnect and continue monitoring.

6. Groundwater samples can be collected after the field parameters stabilize within the following limits:
   - Turbidity: +/- 10% for values greater than 1 nephelometric turbidity units (NTUs)
   - DO: +/- 10 %. Note: DO may not stabilize unless using a flow-thru cell. If not using a flow-thru cell, disregard this parameter for the purpose of establishing stability
   - Specific conductance: +/- 3%
   - Temperature: +/- 3%
   - pH: +/- 0.1 pH units
   - ORP: +/- 10 millivolts

Purging is considered complete and sampling may begin when all of the above indicator field parameters have stabilized. Do not change the flow rate of the pump prior to sampling. Remove the flow through cell prior to collecting the groundwater samples, and collect directly from the pump discharge.

General well purging – removing specified volume of water

During general well purging, a specified minimum volume of water (usually three to five well casing volumes) should be purged prior to sampling. Water temperature, pH, turbidity, DO, ORP, and specific conductance should be periodically measured during purging using a calibrated combination type meter (i.e., YSI, etc.). These parameters should be measured and recorded approximately every three to five minutes, or after each well volume is removed. The sample can be collected after the required volume of water has been purged and the parameters have stabilized within the limits described above in Number 6 of the low-flow purging section.

Purging Methods

Pumping mechanisms – peristaltic pumps, submersible pumps, non-contact gas bladder pumps, and suction pumps, etc.

1. Assemble the pumping unit. For more information on pump assembly and operation, refer to the specific user's manual for the type of pump used.
2. Lower the tubing (peristaltic pump) or pump/tubing assembly (submersible pumps)

into the well to the midpoint of the zone to be sampled. If possible, keep the tubing or pump intake at least 2 feet above the bottom of the well, to minimize mobilization of particulates present in the bottom of the well.

3. Attach a water quality meter to the outlet tubing to monitor water quality parameters.
4. If required, attach a flow meter to the outlet tubing to measure the volume and rate of water purged.
5. Attach the power supply (typically a battery, generator, etc.). Use a ground fault circuit interrupter (GFCI), or ground the generator to avoid electric shock.
6. Start the pump at its lowest speed setting and slowly increase the speed until discharge occurs. Adjust the pump speed until there is little or no water level drawdown (less than 0.3 feet). If the minimal drawdown that can be achieved exceeds 0.3 feet, but remains stable, continue purging until indicator field parameters stabilize.
7. During purging, monitor water quality parameters and water level drawdown.
8. After water parameters have stabilized, disconnect the water quality meter and flow meter, then collect sample.

Bailer purging

1. Attach the line to the bailer and slowly lower until completely submerged, be careful not to drop the bailer to the water, which would cause turbulence and the possible loss of volatile contaminants.
2. Pull bailer out, while ensuring that the line either falls onto a clean area of the plastic sheeting or that it never touches the ground.
3. Empty the bailer into a pail of known volume (for example, a five-gallon bucket, preferably graduated). Use the volume of the pail to estimate the amount of water removed.
4. During purging, monitor water quality parameters.
5. Remove the required amount of water.
6. If water quality parameters have stabilized, the sample can be collected. If parameters have not stabilized, continue purging until stabilization has been achieved, or collect sample if directed to do so by the project manager.

Sampling: Sampling may be accomplished using pumping mechanisms or bailers. Care must be exercised during the use of bailers because of their tendency to disturb sediment, leading to increased turbidity.

General procedures

1. If using a pumping mechanism, do not change the flow rate maintained during purging.
2. Remove the water quality and flow rate meters, if used.
3. If using a pumping mechanism, collect non-filtered samples directly from the outlet tubing into the sample bottle. For filtered samples, connect the pump outlet tubing directly to the filter unit. The pump pressure should remain decreased so that the pressure buildup on the filter does not blow out the pump bladder, or displace the filter.
4. For certain projects, sampling may be performed without purging the well first, typically using a bailer. It is preferable to record the water quality parameters (turbidity, DO, specific conductance, temperature, pH, and ORP) before the sample is collected. Non-purge sampling will be performed in accordance with the steps below.
5. If using a bailer, lower the bailer slowly and gently into the well, taking care not shake the casing sides or to splash the bailer into the water. Stop lowering at a point adjacent to the screen. Allow the bailer to fill and then slowly and gently retrieve the bailer from the well, avoiding contact with the casing, so as not to knock flakes of rust or other foreign materials into the bailer. If the bailer comes with a Bottom Emptying Devise (BED), place the BED into the bottom of the bailer. Fill the sample containers from the BED. A specific BED for volatile samples is recommended because it reduces the outflow to a very low laminar rate. This device is typically purchased separately from the bailers.
6. Collect samples in appropriate containers in order of volatility, with the most volatile samples collected first. Containers should be either pre-labeled or labeled immediately after sample collection. For collecting volatile samples using the zero-headspace procedure, follow procedures specified at the end of this section.
7. Fill containers slowly (avoid turbulence).
8. Filter and preserve samples as specified in the site-specific QAPP.
9. If duplicate samples, split samples, or other quality assurance/quality control (QA/QC) samples are required, collect them at the same time as the primary sample.
10. Cap sample containers tightly and place into a sample cooler. Samples must be chilled and maintained at a temperature of 4 degrees Celsius. Do not allow samples to freeze.
11. Replace the well cap.
12. Log all samples in the field notebook or on field forms.
13. Package samples and complete requisite paperwork.
14. Dispose of all liquid and solid IDW in accordance with project planning documents.

Volatile sampling using zero-headspace procedure

1. Open the sample vial, set cap in clean place, and fill the vial just to overflowing. Do not rinse the vial or allow excessive overflowing. There should be a meniscus on the top of the filled vial.
2. Check that the cap has not been contaminated and carefully cap the vial. Slide the cap directly over the top and screw down firmly. Do not over tighten because the cap may break.
3. Invert the vial and tap gently. It is imperative that no air is entrapped in the sample vial. If an air bubble appears that is smaller than approximately 1.0 millimeter, the sample is still viable. If the bubble(s) are larger, discard the sample and begin again.
4. Place the vial in a protective foam sleeve, and then place into the cooler.

Quality Control: The following procedures apply:

- Samples will be packaged, handled, and shipped as prescribed in BERS-03 *Sample Management Standard Operating Procedure.*
- Equipment will be operated and used in accordance with the manufacturer's instructions, unless otherwise specified in the site-specific QAPP.
- Equipment examination activities should occur prior to field deployment, and they should be documented. It is especially important to check that the correct number and type of sample bottles are being sent/taken to the field prior to starting the field activities.
- Depending on the needs of the project, if using non-disposable equipment, collect an equipment rinsate blank to evaluate the potential for cross contamination from the purging or sampling equipment. Collect equipment rinsate blanks by pouring analyte-free water over the decontaminated sampling equipment.
- Depending on the needs of the project, a field blank may be required per matrix and for each sampling event to evaluate whether contaminants have been introduced into the samples during the sampling process. Field blank samples will be obtained by pouring laboratory-grade, certified organic-free water (for organics) or deionized water (for metals) into a sampling container at the sampling point.
- One trip blank per cooler is required when submitting samples for volatile organic analysis. Trip blanks for water and soil samples are prepared and sealed by the laboratory. They are transported to the field and returned, unopened, to the laboratory in the same cooler as the samples collected for volatile organic compound (VOC) analysis.
- Blanks will be collected at the frequency and locations specified in the site-specific QAPP. Blanks are analyzed for the same target analytes as the associated field samples. Each blank receives a unique sample number and is submitted blind to the laboratory.

BRISTOL ENVIRONMENTAL REMEDIATION SERVICES, LLC

SAMPLE MANAGEMENT

STANDARD OPERATING PROCEDURE BERS-03

Record of Changes

Revision No.	Date	Prepared by	Approved by
0	01/15/08	B. Allen	S. Ruth
1	2/23/2010	M. Faust	B. Allen

SAMPLE MANAGEMENT

STANDARD OPERATING PROCEDURE

Method Summary: To ensure the quality and integrity of analytical data, samples will be managed in accordance with rigorous sample handling, shipping, and custody protocols at all times. Pertinent protocols will be determined prior to initiation of field sampling activity and will apply to sampling, transport, and analysis activities.

Health and Safety: Sampling activity should only be conducted in accordance with an approved Site Health and Safety Plan.

Interferences and Potential Problems: Improper sample management may result in a number of problems, including, but not limited to:

- Inability to collect samples during the field event due to lack of appropriate sample containers and/or preservatives.
- Contamination and/or loss of samples or sample constituents through improper storage and handling, tampering, or breakage.
- Inability to validate resulting data.
- Development of erroneous conclusions regarding site contamination based on inaccurate data and/or problems correlating data and sample locations at the site.
- Mishandling of residual sample material following analysis.

Personnel Qualifications: Sample management personnel will be trained and certified as hazardous site workers per Title 29 Code of Federal Regulations, Part 1910.120(e) [29 CFR 19 10.120(e)] and trained in applicable DOT sample shipping regulations of 49 CFR Part 172, Subpart H. If applicable, additional qualification requirements will be specified in the site-specific Quality Assurance Project Plan (QAPP) and met by designated personnel.

Equipment and Materials: Equipment selection will be based on the objectives of the sampling program and the analytes of concern. Prior to deployment in the field, the requisite sampling equipment and materials will be identified, secured, and inspected for signs of damage or potential contamination.

Sample Identification and Labeling: Sample identification and labeling protocols will follow the procedures specified in the governing program QAPP.

Each collected sample will be assigned a unique sample identification number. The designated sample number will be included on the sample label and referenced on associated sample tags, field logbooks, chain-of-custody forms, analysis request forms, and all data reports related to the samples.

To prevent misidentification of samples, the field team will affix legible labels to each sample container. The labels will be sufficiently durable, and an indelible pen will be used to record data on the labels, so that sample identification information remains legible even when wet. Markers should never be used for sample labeling, as they can be a source of volatile compounds and potential contamination of the sample. Additional labeling requirements will be presented in the site-specific QAPP.

Information that is generally included on the container label and/or sample tag includes:

- Sample identification number;
- Sample collector's name or initials;
- Date and time of sample collection;
- Chemical/physical preservatives used;
- Type of sample (composite, grab, filtered); and
- Analytical parameters requested

Sample Containers and Coolers: Sample containers will be selected, prepared, cleaned, and controlled in accordance with EPA Office of Solid Waste and Emergency Response (OSWER) Directive #9240.0-05A *Specifications and Guidance for Contaminant-Free Sample Containers* (EPA *540/R-93/05* 1, December 1992), and as specified in the governing program QAPP. In advance of each sampling event, the subcontract laboratory should prepare a complete set of precleaned sample containers.

Prior to field activity, field personnel will implement the following steps:

1. Check all sample containers against the specifications of the site-specific QAPP. Ensure that the sample containers and caps are in good condition and free of obvious contamination, constructed of the appropriate material (i.e., plastic or glass), contain appropriate preservative solutions, and will hold sufficient volume for planned analyses, if specified.
2. Verify that sample identification labels are properly affixed to each container.
3. Verify that an adequate quantity of each type and volume of sample container is available for the anticipated environmental and quality control samples. Verify that extra containers are readily available to field staff as contingency for damaged or potentially contaminated containers, and for collecting samples of opportunity.
4. Ensure that containers and coolers are stored in clean areas to prevent exposure to fuels, solvents, and other potential contaminants.

Sample Collection: Field personnel will collect samples as prescribed in the governing QAPP. Samples should be transferred in the field from the sampling equipment directly into

a container that has been specifically prepared for that sample (based on the analytes of concern, preservation requirements, and the type of analysis to be performed).

To minimize the potential for cross-contamination and loss of sample constituents, sample fractions should be collected and containerized in the order of volatilization sensitivity of the analytes of interest. The following sample collection order is recommended:

- Volatile organic compounds (VOCs)
- Purgeable organic carbon
- Purgeable organic halogens
- Total organic halogens
- Total organic carbon
- Extractable organic compounds
- Metals
- Phenols
- Cyanide
- Sulfate and chloride
- Turbidity
- Nitrate and ammonia
- Radionuclides
- Ignitability
- Corrosivity
- Reactivity

As the samples are being collected, or immediately thereafter, the field sampling team will document the date and time of sample collection, pertinent field information (e.g., sampling depth), and the identity of sampling personnel, on each container label. Additional detail on the sampling event may be documented in the site logbook as appropriate.

Sample Custody: BERS will ensure the integrity and security of all samples under their control, using a stringent chain-of-custody protocol. This will be supplemented as needed to meet all work assignment requirements.

During the sampling event, field personnel will prepare a chain-of-custody form documenting each sample collected as follows:

- Sample numbers, date and time of collection, sampling location, name of the person who collected the samples, preservatives used, and the analyses requested.
- Document each sample transfer on the custody sheet. Ensure that this form remains with the samples until they arrive at, and are processed by, the laboratory.
- When samples are relinquished to a commercial carrier for transport to the laboratory, sign the chain-of-custody form under "Relinquished By," enter the name of the carrier organization under "Received By," and document the date and time of transfer. Upon receipt of the samples, the laboratory sample custodian will similarly sign and date the chain-of-custody form.

Under no circumstance is there to be a break in custody.

Sample Packaging: Unless otherwise specified in the site-specific QAPP, field personnel will implement the following steps when packaging environmental samples for shipment:

- Tighten all sample lids. Verify that all containers are labeled and intact. Verify that all container labels are secure, legible, and complete.
- Bag samples individually in appropriate-sized plastic bags (e.g., Ziploc®) and seal. Up to 3 VOC vials may be packed together in container bags.
- Secure and tape the drain plug on the cooler with fiber or duct tape.
- Spread inert packing material (rubber foam, air pillows, or "bubble" wrap) in the bottom of the bag inside the cooler and place sample bags on top of the packing material.
- Include a temperature blank (a small container filled with water) to be used by the laboratory to determine the internal temperature of the cooler upon receipt at the laboratory.
- Place ice packs (e.g., blue ice) into cooler. If ice packs are unavailable, place ice into doubled heavy-duty polyethylene bags and seal with tape. Put double-bagged ice on top of, and in between, samples. Fill in remaining space with packing material.
- Place the chain-of-custody record into a plastic sealable bag (e.g., Ziploc), seal the bag, and tape it to the inside of the cooler lid.
- Close the cooler and tape the top of the cooler shut. Affix custody seals to the top and sides of the cooler, such that the cooler cannot be opened without breaking at least one seal.
- Mark the cooler with "This End Up" and arrows to indicate the proper upward position.
- Tape a label containing the name and address of the destination to the outside of the cooler.

Sample Scheduling, Delivery, and Holding Times: In work assignments where analytical services are procured from a subcontractor laboratory, the laboratory will be required to designate a point of contact (POC) for both normal business hours, and for emergency situations during off-hours. In addition, the laboratory will be required to designate a sample custodian, who will be notified by the BERS field sampling supervisor each time samples are shipped.

Unless otherwise approved, samples will be delivered to, and received by, the laboratory within 24 hours of collection.

Sample holding time tracking begins with the collection of samples, and continues until the analysis is complete. The site-specific QAPP will specify holding time requirements for each analyte of interest to the project.

Quality Control: No additional QC procedures apply.

Data Management and Records Management: Sampling records will be generated and maintained as prescribed in this procedure and the governing QA plans. Sampling data will be documented on field data sheets or in the logbooks.

(Intentionally blank)

BRISTOL ENVIRONMENTAL REMEDIATION SERVICES, LLC

FIELD MEASUREMENT AND TEST EQUIPMENT

STANDARD OPERATING PROCEDURE BERS-04

Record of Changes

Revision No.	Date	Prepared by	Approved by
0	01/15/09	B. Allen	L. Maserjian
1	2/5/10	M. Hannah	B. Allen

FIELD MEASUREMENT AND TEST EQUIPMENT

STANDARD OPERATING PROCEDURE

Summary: Various types of instruments are used to measure the physical and chemical characteristics of a sample in the field. In general, field measurement and test equipment (M&TE) are maintained and operated according to the manufacturer's instructions specific to each instrument. Field M&TE are inspected for function and damage on a regular basis and prior to each use. All findings are recorded in the appropriate logbook. Field M&TE are calibrated in accordance with the manufacturer's specifications. Calibrations are checked on a regular basis and prior to and after use in the field. When daily calibrations are required, calibrations and/or checks are performed at the beginning and end of the day, and the results are recorded in the field logbook. When daily calibrations are not required during field use, checks against appropriate standards are performed.

Health and Safety: Field activities will only be conducted in accordance with an approved Site Health and Safety Plan.

Interferences and Potential Problems: When multiple measurements are taken from the same sample material, the order in which the measurements are made becomes very important. Conductivity may be affected by temperature of the measured solution; therefore, temperature of the sample should be read first, so that appropriate adjustments can be made in accordance with the manufacturer's instructions.

Personnel Qualifications: Field personnel will be trained and certified as hazardous site workers per Title 29 Code of Federal Regulations, Part 1910.120(e) [29 CFR 1910.120(e)]. If applicable, additional qualification requirements will be specified in the site Quality Assurance Project Plan (QAPP).

Equipment and Materials: Prior to deployment in the field, the appropriate equipment and materials will be identified, secured, and inspected for signs of damage or potential contamination. Manufacturer's instructions and specifications for each instrument used will be maintained in the project files. Materials used for calibration of instrumentation, such as standard solutions, must be traceable to relevant, recognized performance standards.

Planning Considerations: Procedures used for the maintenance and use of field equipment, including those performed by subcontractors and suppliers, will be outlined preceding field activities and in accordance with the procedures outlined in this SOP. Equipment must be inspected prior to use in the field for damage and function. Calibration and maintenance of field equipment will be performed according the manufacturer's instructions for that particular instrument. The required frequency of calibration varies between instruments. While some instrumentation must be calibrated only annually or semi-annually, other instrumentation must be calibrated daily during use in the field. Instrumentation that does not require field calibration usually requires a check against a standard. Attention should be paid

to specific requirements for each instrument used in the field, and it is important to remember that the requirements for each instrument may differ.

Instructions for the Maintenance and Use of Field Equipment: Refer to the following sections for instructions on the proper calibration, maintenance, and use of field instrumentation used to measure physical/chemical properties of sample material:

Multi-parameter Water Quality Meter: Many water quality meters are capable of measuring several parameters, such as temperature, conductivity, pH, dissolved oxygen (DO), and oxygen reduction potential (ORP). The following sections provide general instructions for calibrating each parameter. The field personnel will adhere to the calibration instructions for the each instrument used.

Temperature

Temperature, defined as a measure of hotness or coldness on a defined scale, is measured using a thermometer. Three types of thermometers are commercially available: digital (thermocouple) thermistor; glass bulb, mercury-filled thermometer; and bi-metal strip dial indicator thermometer.

Calibration

Thermometers will be calibrated in accordance with the manufacturer's instructions or calibrated semi-annually against a National Institute of Standards and Technology (NIST)-certified thermometer. Thermistors should be checked against a mercury bulb thermometer in water prior to use, and should agree within ± 0.5° degree Celsius (°C).

Maintenance

All thermometers should be inspected regularly and prior to use for leaks, cracks, and function.

Use

Measurements should be made in situ, when possible. To measure the temperature of sample material, perform the following steps:

1. Clean the probe with deionized water, and immerse into the sample.
2. Swirl the thermometer in the sample.
3. Allow the thermometer to equilibrate with the sample.
4. Suspend the thermometer away from the sides and bottom to observe the reading.
5. In a logbook, record the reading to the nearest 0.5° C.
6. Report results to the nearest 0.5° C.

Conductivity: Conductivity, the quality or power of conducting or transmitting, is typically measured using the Wheatstone bridge meter. Conductivity is measured in millisiemens per centimeter (mS/cm) at 25° C. While the sample temperature may be lower, nearly all conductivity meters will convert specific conductance (which is not corrected for temperature) to conductivity.

Calibration

Conductivity will be calibrated in accordance with the manufacturer's instructions. During use in the field, checks against a one-point standard will be performed to ensure the accuracy of the meter, and results will be recorded in a field logbook. The following steps will be implemented both before and after use of the meter to measure the conductivity of sample material in the field:

1. Check and record the temperature of the standard solutions.
2. Rinse the probe with analyte-free water before immersing it in the standards solution.
3. Turn the probe on, immerse it in the standard solution, and record the results.
4. If the meter is not accurate to within ± 10% of the standards, correct the problem before proceeding.

Maintenance

All conductivity meters should be inspected regularly and prior to use for damage and function. Conductivity sensors may become fouled with minerals or other materials, and may require cleaning in the field. Cleaning is accomplished by passing a nylon brush along the sensor surface in a light scrubbing motion, until a metallic shine appears on the sensor. Follow up the cleaning with a fresh or deionized water rinse. DO NOT use a metal brush to clean the sensor surface.

Use

1. Collect the sample and record its temperature.
2. Correct the instrument's temperature adjustment to the temperature of the sample (if necessary).
3. Immerse the probe in the sample. Keep the probe away from the sides and bottom of the container, and ensure that the sensor is in full contact with the sample.
4. Record the results in a logbook.
5. Rinse the probe.
6. Report results to the nearest ten units for readings below 1,000 mS/cm at 25° C and the nearest one hundred units for readings above 1000 mS/cm at 25° C.

Hydrogen Ion Concentration (pH): The pH of a solution is defined as the negative logarithm of the effective hydrogen ion concentration in gram equivalents per liter. The pH is used to measure acidity and alkalinity on a scale ranging from 0 to 14, with 7 representing neutrality. Orion and YSI Water Quality Monitoring System meters are examples of commercially available meters used to measure the pH of liquid-state material.

Calibration

Any pH meter will be calibrated in accordance with the manufacturer's instructions. During use in the field, a two-point or three-point standard will be used to ensure the accuracy of the meter. Results will be recorded in a field logbook. The expected pH of the sample to be collected, estimated from either historical data or by using four-color pH paper, should fall between the two buffering points. Both prior to and after use in the field, the following procedures should be followed as a minimum:

1. Remove the meter from storage and allow it to equilibrate to ambient temperature.
2. Select either pH 4 and pH 7, or pH 7 and pH 10, as the appropriate standard solutions as described above.
3. Use a thermometer to determine the temperature of the buffering solutions, and record the temperature.
4. Rinse the probe with analyte-free water, and immerse it into the pH 7 buffer and set the meter to 7. If the solution temperature is not at 25°C, a table with corrected pH values can be found on the calibration solution bottle or in the operations manual.
5. Rinse the probe with analyte-free water and immerse it into the second buffer, and record the reading.
6. Rinse and store the probe in a container filled with analyte-free water.

Maintenance

All pH meters should be inspected for damage and function regularly and prior to use. During use, periodically check the calibration of the meter by rinsing it with analyte-free water and immersing it into the pH 7 buffer solution.

Use

Follow these steps when measuring the pH of a sample:

1. If measuring temperature, record temperature prior to measuring pH.
2. Immerse the probe in the sample, keeping it away from the sides and bottom of the container. Allow the probe to equilibrate with the sample material.
3. With the probe suspended away from the container surface, record the pH.
4. Rinse the probe with analyte-free water and store in a container filled with analyte-free

water until the next sample is ready.

5. Record results to the nearest 0.1 Standard Unit (SU).

Storage

After use, rinse the unit with fresh water or Alconox®, followed by fresh water, at contaminated sites. Leave a small amount (20mL) of pH 4 solution in the storage cup before sealing the unit in order to keep the pH sensor moist during storage.

Dissolved Oxygen (DO): The membrane/electrode (ME) is the most commonly used instrument for measuring the dissolved oxygen present in a sample.

Calibration

Calibrate the DO probe according to the manufacturer's instructions, either in air-saturated water, or in a water-saturated air environment.

Maintenance

The DO probe should be inspected regularly and prior to use for damage and function. The membrane of the DO meter should be inspected for air bubbles, holes, and dryness. If the membrane is dry, replace and soak it in analyte-free water prior to calibration of the meter. If the metallic sensor is discolored, or does not appear shiny, use the fine-grit sandpaper (supplied with the DO sensor replacement kit) and buff the metal surface in a circular pattern until the surface shines. Rinse the sensor with deionized water before installing a new membrane.

Use

When measuring DO in situ with a field probe, follow these steps:

1. Allow the DO reading to stabilize.
2. Read the dial to the nearest 0.1 mg/L, and record the measurement.

Oxygen Reduction Potential (ORP): ORP, also known as redox potential, is the tendency of a chemical species to acquire electrons and thereby be reduced. Each species has its own intrinsic reduction potential; the more positive the potential, the greater the species' affinity for electrons and tendency to be reduced.

Calibration

Calibrate the ORP probe according to the manufacturer's instructions in a standardized calibration solution. The ORP is affected by temperature. Refer to the calibration solution or operations manual to correct for temperature during calibration.

Maintenance

The ORP probe should be inspected regularly and prior to use for damage and function.

Use

When measuring ORP in situ with a field probe, follow these steps:

1. Immerse the probe in the sample, keeping it away from the sides and bottom of the container. Allow the probe to equilibrate with the sample material.
2. With the probe suspended away from the container surface, record the ORP to the nearest 1.0 millivolt.
3. Rinse the probe with analyte-free water and store in a container filled with analyte-free water until the next sample is ready. Do not store the unit in deionized water.

Turbidity Meter: A nephelometer/turbidmeter is used to measure the turbidity of a liquid sample by determining how much light can pass through it. The Hach® Turbidimeter is the most commonly used commercially available meter for measuring the turbidity of a sample. Turbidity is measured in nephelometric turbidity units (NTUs).

Calibration

Calibration of turbidity meters will be performed in accordance with manufacturer's instructions. Any turbidity meter must be calibrated at both the beginning and end of the day during use in the field, and results will be recorded in a field logbook. The following procedures will be used to calibrate a turbidity meter in the field:

1. Turn the meter "ON" and allow 2 minutes for the lamp to stabilize.
2. Rinse the sample cell with organic-free or deionized water.
3. To "zero" the calibration, fill the cell to the fill line with organic-free or deionized water and then cap the cell.
4. Use lens paper to wipe off excess water and streaks from the outside of the cell.
5. Open the cover and insert the cell (arrow to the front) into the unit and close the cover.
6. Press "Blank" and wait for the "light bulb" icon to go off. Record the reading.
7. Hach turbidity meters require calibration with known standards. Refer to the operations manual for information on calibrating the meter.
8. Using the Gelex Turbidity Standards, repeat steps 4, 5, and 6. Record all findings.

Maintenance

Turbidity meters should be inspected regularly and prior to use for damage and function. During use, periodic checks should be performed using the standards to ensure continued proper calibration of the instrument. If error codes appear on the unit display, refer to the owner's manual to resolve the error.

Use

Follow these steps to measure the turbidity of a sample:

1. Pour sample material into the cell to the fill line and replace the cap on the cell.
2. Wipe excess water and any streaks from the outside of the cell with lens paper.
3. Place the cell inside the measurement chamber with the arrow towards the front and close the cover.
4. Press "READ" and wait for the "light bulb" icon to turn off
5. Record the reading.
6. Empty the cell and rinse with organic or analyte-free water.

Quality Control: The following procedures apply:

- Equipment will be operated and used in accordance with the manufacturer's instructions, unless otherwise specified in the site-specific work plan or its equivalent.
- Equipment examination activities will occur prior to field deployment, and they should be documented.

Calculations and Data Reduction: Does not apply.

Data Management and Records Management: Equipment calibration and maintenance records will be generated and maintained as prescribed in the governing QAPPs.

(Intentionally blank)

BRISTOL ENVIRONMENTAL REMEDIATION SERVICES, LLC

EQUIPMENT DECONTAMINATION

STANDARD OPERATING PROCEDURE BERS-05

Record of Changes

Revision No.	Date	Prepared by	Approved by
1	10/14/09	B. Allen	L. Maserjian
2	2/23/10	L. Maserjian	B. Allen

EQUIPMENT DECONTAMINATION

STANDARD OPERATING PROCEDURE

Summary: Disposable tools and equipment should be used when possible. However, where non-disposable items are used, appropriate decontamination will be accomplished according to the type of equipment being used and the type of samples being collected. In general, field equipment will be decontaminated by means of the following steps:

1. Perform non-phosphate detergent and tap water wash, using a brush if necessary.
2. Perform tap-water rinse.

When sampling for trace organic compounds, the following step will be added:

3. Perform deionized/distilled water rinse.

Health and Safety: Field activities should only be conducted in accordance with an approved Site Health and Safety Plan. Decontamination hazards and precautions include the following:

- Hazardous substances may be incompatible with decontamination materials. For example, the decontamination solution may react with contaminants to produce heat, explosion, or toxic products. Also, vapors from decontamination solutions may pose a direct health hazard to workers by inhalation, contact, fire, or explosion. The Site Health and Safety Plan will provide procedures and identify responsibilities to ensure that incompatible materials are identified and segregated from each other.
- The Site Health and Safety Plan will specify the use of personal protective equipment (PPE) that is appropriate for both the contaminants of concern and the decontamination chemicals used. The PPE selection will take into account that decontamination materials may degrade protective clothing or equipment, and that some solvents can permeate protective clothing.
- Solvent rinsing operations will be performed in well-ventilated areas.
- Investigation-derived waste (IDW) generated from decontamination activities will be managed as prescribed in SOP BERS-09: *IDW Management.*
- Material Safety Data Sheets (MSDS) will be kept with all decontamination solvents or solutions as required by the Hazard Communication Standard.
- Phosphate-containing detergents will not be used in jurisdictions where they are banned.

Interferences and Potential Problems: Potential problems related to equipment decontamination can be eliminated by the use of appropriate materials, reagents, and techniques.

- The use of distilled and/or deionized water commonly available from commercial vendors may be acceptable for decontamination of sampling equipment.

- The use of an untreated potable water supply is not an acceptable substitute for tap water. Tap water may be used from any municipal or industrial water treatment system.
- If acids or solvents are utilized in decontamination, they raise health and safety and waste disposal concerns.
- Washing complex and sophisticated sampling equipment with acids or solvents can damage the equipment.
- If not used immediately, cleaned equipment will be stored to prevent recontamination.
- PVC and plastic items will not be rinsed with solvents.

Personnel Qualifications: Field personnel will be trained and certified as hazardous site workers per Title 29 Code of Federal Regulations, Part 1910.120(e) [29 CFR 1910.120(e)]. If applicable, additional qualification requirements will be specified in the site Quality Assurance Project Plan (QAPP).

Equipment Requirements: Prior to deployment in the field, the requisite sampling equipment and materials will be identified, secured, and inspected for signs of damage or potential contamination. Decontamination equipment, materials, and supplies are generally selected based on availability. Other considerations include the ease of decontaminating or disposing of the equipment.

The following standard materials and equipment are recommended for decontamination activities:

- Non-phosphate detergent.
- Tap water.
- Distilled/deionized water
- Pesticide grade solvent
- Long- and short-handled brushes
- Bottle brushes
- Drop cloth/plastic sheeting
- Paper towels
- Plastic or galvanized tubs or buckets
- Pressurized sprayers (H_20)
- Solvent sprayer with Teflon nozzle
- Aluminum foil
- Plastic sheeting

- PPE
- Trash bags
- Trash containers
- 55-gallon drums
- Metal/plastic buckets/containers for storage and disposal of decontamination solutions.

The appropriate materials and equipment will be selected as needed on a site-specific basis.

Planning Considerations: Equipment decontamination activities, including those performed by subcontractors and suppliers, will be planned in advance of field activities and in consultation with program health and safety personnel.

Decontamination: Depending on the nature of the work, field equipment requiring decontamination may include heavy equipment, downhole equipment, sampling equipment, and groundwater pumping equipment.

Heavy Equipment Decontamination: Heavy equipment includes the drilling rig and backhoe. Field personnel will implement the following steps to decontaminate heavy equipment:

1. Set up a decontamination pad that is large enough to fully contain the equipment to be cleaned. Use one or more layers of heavy plastic sheeting to cover the ground surface.
2. Spray areas of the equipment that may have been exposed to contaminated soils using steam or high-pressure sprayer and detergent. Be sure to spray down all surfaces, including the rear area of the undercarriage.
3. Rinse the equipment with potable water.
4. Remove equipment from the decontamination pad and allow to air dry.

Downhole Equipment Decontamination: Downhole equipment includes hollow-stem augers and drill pipes. Well casings and screens will be decontaminated as decribed under "Sampling Equipment". Field personnel will implement the following steps to decontaminate downhole equipment:

1. Set up a centralized decontamination area, if possible. This area should be set up to contain contaminated rinse waters, and to minimize the spread of airborne spray.
2. Set up a "clean" area upwind of the decontamination area to receive cleaned equipment for air drying. At minimum, clean plastic sheeting must be used to cover the ground, tables, or other surfaces where decontaminated equipment is to be placed.
3. Wearing the required PPE, use a high-pressure sprayer or steam unit and detergent to clean the contaminated equipment. Aim downward to avoid spraying outside the decontamination area. Be sure to spray inside corners and gaps. If necessary, use a brush to dislodge dirt or debris.

4. Rinse the equipment using potable water.
5. Remove the equipment from the decontamination area and place in the clean area to air dry.
6. Cover the equipment to prevent contamination if the equipment is not used immediately.
7. Collect all contaminated waters, plastic sheeting, and disposable gloves, boots, and clothing in the designated containers. Receptacles containing contaminated items must be properly labeled for disposal. Containerize liquids and solids separately.

Sampling Equipment Decontamination: Sampling equipment includes split spoon samplers, spatulas, compositing bowls, and other utensils that come into direct contact with samples.

Field personnel will collect disposable sampling equipment in the designated containers and dispose of them as prescribed in the Site Health and Safety Plan and SOP BERS-09: *IDW Management.* Field personnel will implement the following steps to decontaminate non-disposable equipment:

1. Set up a decontamination line on plastic sheeting. The decontamination line should progress from dirty to clean, and end with an area for drying decontaminated equipment. At minimum, use clean, plastic sheeting to cover the ground, tables, or other surfaces on which decontaminated equipment will be placed. Set up a containment system for collecting wash/rinse waste.
2. Wash the item thoroughly in a bucket of soapy water. Use a stiff-bristle brush to dislodge dirt or debris. Before washing, disassemble items that might trap contaminants internally. Do not re-assemble until decontamination is complete.
3. Rinse the item in potable water. Rinse water should be replaced as needed, generally when cloudy.
4. Allow to air dry.
5. Collect all contaminated waters, plastic sheeting, and disposable gloves, boots, and clothing in the designated containers. Receptacles containing contaminated items must be properly labeled for disposal. Liquids and solids must be drummed separately.

Groundwater Sampling Pumping Equipment Decontamination: Field personnel will implement the following steps to decontaminate sampling pumps:

1. Set up a decontamination area and a separate clean storage area using plastic sheeting to cover the ground, tables, and other porous surfaces where decontaminated equipment will be placed. Set up three clean containers of the appropriate size and shape for immersing the pump assembly. Fill the first container with dilute, non-foaming soapy water, and the second with potable water. Use the third container for waste discharge.

2. If decontaminating an electric submersible pump (e.g., Grundfos® Redi-Flo), remove the bottom screw plug to flush the cooling water. Replace this water with deionized water after the decontamination process is complete.
3. Set up the pump assembly in the same configuration as used for sampling. Submerge pump intake and all downhole wetted parts (tubing, piping, and foot valve) in the soapy water container. Place the discharge outlet in the waste container above the level of wastewater. Pump soapy water through the pump assembly until it discharges to the waste container.
4. Move the pump assembly to the rinse water container while leaving discharge outlet in the waste container. Ensure that all downhole wetted parts are immersed in the potable water rinse. Pump potable water through the pump assembly until it runs clear.
5. Pump a sufficient amount of analyte-free water through the hose to flush out the tap water, then purge with the pump in reverse mode. Rinse the outside of the pump using analyte-free water. Decontaminate the discharge outlet by hand following the steps for decontamination of sampling equipment.
6. Remove the decontaminated pump assembly to the clean area and allow to air-dry.
7. Cover intake and outtake orifices with aluminum foil to prevent the entry of airborne contaminants or particles.
8. Place pump in clean plastic bag.

Quality Control: The following procedures apply:

- Equipment will be operated and used in accordance with the manufacturer's instructions, unless otherwise specified in the site-specific work plan or its equivalent.
- Equipment examination activities should occur prior to field deployment, and should be documented.
- After decontamination activities, the field personnel should make a record of the equipment type, date, time, and method of decontamination in the field logbook.
- If sampling equipment requires the use of plastic tubing, dispose of it as contaminated. Replace with clean tubing before conducting additional sampling.

Calculations and Data Reduction: Does not apply.

Data Management and Records Management: Generate and maintain decontamination records as prescribed in the governing QAPPs.

(Intentionally blank)

BRISTOL ENVIRONMENTAL REMEDIATION SERVICES, LLC

BOREHOLE LOGGING

STANDARD OPERATING PROCEDURE BERS-06

Record of Changes

Revision No.	Date	Prepared by	Approved by
0	01/15/09	B. Allen	L. Maserjian
1	2/23/10	L. Maserjian	B. Allen
2	4/01/10	L. Kleppin	L. Maserjian

BOREHOLE LOGGING

STANDARD OPERATING PROCEDURE

Summary: Boreholes are typically advanced for the purposes of well or piezometer installation, subsurface soil sampling, or rock coring. Drill rigs (typically hollow-stem auger, air rotary, or cable tool), or direct-push technologies (i.e., Geoprobe®) are the most common methods for advancing borings. Because borehole logging typically involves the collection of subsurface soil samples to describe the geologic material, this procedure supplements what is presented in the downhole sampling portion of the SOP BERS-01: *Soil Sampling Procedures.*

To facilitate uniformity in description, boreholes are generally logged on the basis of their identifiable physical characteristics, rather than assigning interpreted depositional soil types or rock names (i.e., glacial till) that may represent the bias or experiences of the field geologist/engineer. This procedure uses the term soil to identify any unconsolidated geologic material, typically of sedimentary or volcanic origin. The term rock is used to describe any consolidated or indurated geologic material.

Health and Safety: Subsurface sampling should only be conducted in accordance with an approved Site Health and Safety Plan. Prior to the commencement of drilling activities, all boring locations must be verified free and clear of underground and overhead utilities.

Interferences and Potential Problems: Several factors can potentially result in problems in the logging and physical description of a soil boring, such as incomplete subsurface sampling, sample collection from unknown depth intervals, and inconsistent description of soil or rock types by two different geologists/engineers.

Field personnel will take the following considerations into account during soil boring activities:

- When collecting a subsurface soil sample with an apparatus such as a split-spoon, recovery of the entire sample interval is often incomplete. Even if the entire length of the split-spoon is driven, factors of soil-pore pressure, density, or texture often result in partial sample recovery. In these circumstances, it is important to note that the recovered portion of the sample is not necessarily representative of the entire sample interval.
- Depending on the project objectives, continuous subsurface sampling may not be specified in the work plan. Consequently, gaps are present in the borehole log regardless of sample recovery. It must be recognized that interpolating the geology between sample intervals contains an element of uncertainty, the magnitude of which is dependent of the heterogeneity of the geologic material.
- After advancing the drill bit to the sample depth interval, it is necessary to thoroughly remove all cuttings and loose material from the bottom of the hole prior to sampling. Failure to perform this task will result in the sampling of "sluff," i.e., soil material from unknown depth intervals. Sluff can often be identified by low-density, homogeneous material at the top of the sample interval, and is not representative of the undisturbed soil.

- When logging a borehole through the description of drill cuttings (as opposed to undisturbed sampling), it is important to recognize the lag time between when the drill bit cuts the soil or rock material, and when these cuttings appear at the surface. Accordingly, cuttings at the surface are not representative of the material being drilled at the bottom of the hole. The duration of the lag time is a function of the depth of the boring, and the method of drilling.
- Problems resulting from the inconsistent description of identical soil or rock types by two different geologists/engineers can be minimized through the use of standard soil/rock classification and grain size charts that reduce the subjective nature of the description. Effective communication between members of the field team will also minimize these problems.

Personnel Qualifications: Personnel will be trained and certified as hazardous site workers per Title 29 Code of Federal Regulations, Part 1910.120(e) [29 CFR 1910.120(e)]. If applicable, additional qualification requirements will be specified in the site Quality Assurance Project Plan (QAPP) and met.

Equipment and Materials: Prior to deployment in the field, the requisite drilling and downhole sampling equipment and materials shall be identified, secured, and inspected for signs of damage or potential contamination.

The types and quantities of equipment and supplies necessary to complete a boring depend on the drilling and sampling method as specified in the work plan. In general, the drilling subcontractor will provide the equipment and supplies necessary for the completion of the boring in compliance with applicable state and federal regulations.

This equipment includes a fully tooled drill rig and all supplies necessary to either complete a well in the boring, or abandon the hole. Such items may include: the drill rig and support vehicles, auger flights, steel casing, hydraulic jacks, welding supplies, well screen and casing, sand, bentonite chip and/or grout, water tanks, steam cleaner, cement, 55-gallon drums, pumps, well development supplies, surface monuments, shovels, and all tools necessary to complete the job. Although the drilling subcontractor typically provides these items, it is the responsibility of the project manager and project geologist/engineer to ensure that all required equipment and supplies are specified and present on site.

Bristol typically provides all items pertaining to health and safety, field screening, sampling, as well as descriptive logging of the borehole. The descriptive logging of a borehole requires certain reference materials to provide an accurate physical description of the geologic material, including: soil and rock classification charts, Munsell color chart, grain size chart, hand lens, scales, an assortment of pens and markers, field logbook, and the Boring Log Form.

Other materials related to health and safety, field screening, or sampling may include: personnel protective equipment, photoionization detector (PID), folding table, spoons, utility knife, shovel, water level indicator, tape measure (graduated in tenths of feet), survey stakes or flags,

laboratory-supplied sample containers, coolers, ice, decontamination supplies, camera, logbooks, chain-of-custody forms, and labeling, packaging, and shipping supplies.

Borehole Logging Procedures: Prior to the initiation of any drilling, sampling, or logging activities, it is necessary to organize and stage all equipment and materials at the borehole location. This may include the following:

1. Decontaminate the drill rig and all sampling equipment.
2. Ensure that utilities have been checked and cleared.
3. Ensure that the water supply is from an approved source.
4. Stage drums or containers for investigation-derived waste (IDW) near the borehole.
5. Establish a convenient area, preferably a solid platform or table, for use while examining geologic material or preparing samples.
6. Have all reference material (e.g., classification and color charts), logbooks, and boring log forms ready and accessible.
7. At the beginning of drilling activities, monitor for organic vapors with a PID in accordance with the SOP BERS-10: *General Air Monitoring and Sampling Procedures*. Air monitoring of the breathing zone around the borehole should continue intermittently (or as specified in the work plan) until total depth (TD) is attained.
8. During drilling activities, record any observations or unusual motions of the rig that may be a function of the hardness or density of the geologic materials. This includes rate and ease of penetration, bit chatter, and bouncing. Record these observations in the field logbook and/or the Boring Log Form.
9. Containerize soil and rock cuttings in open-top 55-gallon drums, or as specified for project IDW. Label each drum with the site name, date, location, depth intervals of contained material, and the nature of the material.
10. Collect subsurface samples at the depths and frequency specified in the site QAPP. For subsurface soil sampling, this will typically be accomplished by driving a split-spoon or pushing a sample sleeve with the drill rig. If a split-spoon sampler is used, drive the spoon using a 140-pound hammer, dropped 30 inches. Record the number of hammer blows (blow count) required to attain full penetration of the sampler (in a series of three, 6-inch intervals). Refer to American Society for Testing Materials (ASTM) Method Dl586 if needed.
11. Upon retrieval of the sample spoon/sleeve, collect all required samples prior to performing any descriptive logging activities. This is especially critical for sample aliquots designated for volatile organic analyses. These procedures are described in detail in the SOP BERS-01: *Soil Sampling Procedures.*
12. Describe the soil on the basis of the physical characteristics (i.e., texture) of the soil sample. Unless otherwise specified in the site QAPP, perform soil identification and description in accordance with the Unified Soil Classification System (USCS, ASTM

Method D2487). Fill material should be described in the same depth of detail as soil samples. Because visual determinations of grain size and grain size distribution (i.e., grading or sorting) are critical to the assignment of a USCS Group Symbol, a grain size reference card will be available to facilitate this estimation, and to provide consistency between different field geologists/engineers. Sample description are listed, in the following order:

1. **Soil Type** (USCS description)
2. **USCS Classification Symbol**
3. **Consistency or Relative Density** (based on blow count)
4. **Moisture Content** (dry, moist, wet)
5. **Color** (based on Munsell chart)
6. **Structures present**, if any (e.g., laminated, bedded)
7. **Additional modifying information** (e.g., grain sizes, particle shape, cementation) and observations or characteristics which may affect the engineering or chemical fate properties of the material (e.g., presence of fill, organics, or rooting)
8. **Odors, sheens, or visual evidence of contamination** (slight petroleum odor, strong solvent odor, etc.)

The following are examples of typical entries to be placed on the Boring Log Form and/or the field logbook:

- SANDY GRAVEL (GP) – medium dense, moist, gray (GLEY 6/N) subrounded to 2-inch maximum diameter, slight petroleum odor
- SILTY SAND (SM) – medium dense, wet, reddish brown (5YR 5/3) fine to medium grained
- SILTY CLAY (CL) – medium stiff, wet, dark greenish gray (GLEY 4/1) occasional fine-grained sand seams 3 inches thick
- GRAVELLY SANDY SILT (ML) – very stiff, moist, dark brown (7.5YR 3/2) abundant cobbles

  (Note: "very stiff" consistency describes silt that is plastic, i.e., like clay)
- SANDY SILT (ML) – medium dense, moist, yellow (10YR 7/6) fine-grained sand

  (Note: "medium dense" relative density describes silt that is non-plastic, i.e., like sand)

USCS Classification Symbol

Two-letter USCS codes should follow the USCS description. Do not use dual symbols such as CL-ML on field logs; use dual symbols when laboratory test results indicate that soil should be

classified as such, for example: If the results of a laboratory grain-size distribution test indicate there is 49 percent gravel, 40 percent sand, and 11 percent silt, the soil should be classified as: SANDY GRAVEL with SILT (GP-GM). If the results of an Atterberg Limit Test indicate that the soil has a plasticity index of 5 and a liquid limit of 20, the soil should be classified as: CLAYEY SILT (CL-ML).

Consistency and Relative Density

Consistency. An estimate of the consistency (if available, use standard penetration resistance as a guide) should accompany descriptions of all fine-grained soils (silts and clays where more than 50 percent of the material would pass the No. 200 sieve). The following consistency terminology should be used:

SPT N-value*	Consistency	Identification Procedure	Approximate Shear Strength (psf)
0 – 2	Very soft	Very easily penetrated several inches with fist	Less than 250
2 – 4	Soft	Easily penetrated several inches by thumb	250 – 500
4 – 8	Medium stiff	Penetrated several inches by thumb with moderate effort	500 – 1,000
8 – 15	Stiff	Readily indented by thumb, but penetrated only with great effort	1,000 – 2,000
15 – 30	Very stiff	Readily indented by thumb nail	2,000 – 4,000
greater than 30	Hard	Indented with difficulty by thumb nail	Greater than 4,000

Note: *SPT (standard penetration test) N-value is defined as the total number of blows required to drive the second and third 6 inches of penetration (the first 6 inches' blow count is also recorded), while driving a 2-inch-OD sampler with a 140-pound hammer falling from a height of 30 inches.

OD = outer diameter

psf = pounds per square inch

If laboratory strength tests are performed, the logs should be corrected if necessary.

Relative Density. Descriptions of all coarse-grained soil (sand and gravel where less than 50 percent of the material would pass the No. 200 sieve and 100 percent would pass the 3-inch size) should be accompanied by an estimate of the relative density. The following guideline terminology should be used:

SPT N-value*	Relative Density
Less than 4	Very loose
4-10	loose
10-30	Medium dense
30-50	Dense
Over 50	Very dense

Note: *SPT (standard penetration test) N-value is defined as the total number of blows required to drive the second and third 6 inches of penetration (the first 6 inches' blow count is also recorded), while driving a 2-inch outer diameter sampler with a 140-pound hammer falling from a height of 30 inches.

Moisture Content

Moisture content should be estimated using only the terminology described below. An estimate of the moisture content should be included immediately following the estimated consistency or relative density. The following terms should be used:

Description	Criteria
Dry	Absence of moisture, dry to the touch
Moist	Damp, but no visible water
Wet	Visible free water, usually below water table

Color

Color is correctly described by comparing the soil sample with a Munsell color chart and applying the correct designations and descriptions. If the Munsell color chart is not available, colors should be described using only red, green, yellow, purple, blue, black, brown, gray, white, or orange. Descriptions such as tan, buff, etc., should be avoided, though modifiers such as light or dark are acceptable. Dual color descriptions (red-brown) can also be used.

Structure

The structural characteristics of undisturbed samples often provide important information. Some of the more common terms are defined below and should be used when applicable.

- Stratified – Alternating layers of different types or colors of soil. If the layers are less than about ¼ inch thick, describe as laminated
- Fissured – Tendency to shear along definite fracture planes with little resistance
- Slickensided – Soils containing polished, glossy surfaces on soil particles or fractures
- Blocky – Easily broken into small angular lumps that resist further breakdown

- Lensed – Inclusion of small pockets of different texture, such as small pockets of sand scattered through a clay mass
- Angularity – Angularity can be described of the coarse grains as follows:

Description	Criteria
Angular	Particles have sharp edges and relatively plane sides with unpolished surfaces
Subangular	Particles are similar to angular description, but have rounded edges
Subrounded	Particles have nearly plane sides, but have well-rounded corners and edges
Rounded	Particles have smoothly curved sides and no edges

Additional Modifying Information

Other descriptive information that should be included, if applicable, are approximate percentages of clay, silt, sand, and gravel; average grain size and maximum size of particles; shape/angularity of coarse grains; general composition or mineralogical description of grains (e.g., granitic, micaceous, etc.) coatings on coarse grains; plasticity; organic content; cementation; and local or geologic name.

- Editorial comments and notes on drilling procedure or difficulty should follow the stratum identification.
- The term "grading" is often misused:

  Misuse – grading lighter
  Better – becomes light brown at 5 feet

  Misuse – grading sandier at 10 feet
  Better – grades to sand at (or below) 10 feet

Odors, Sheens, or Visual Evidence of Contamination

An odor description should always indicate the type of odor (e.g., petroleum, solvent, etc.) and may include modifiers such as strong or slight.

Bedrock Logging Procedure

Consolidated rock material will typically be described and logged through either drill cuttings or diamond coring. At the present time, there is not a unified system for classifying rock. Because there are numerous types of rock classification schemes in common use, the specific format to be used will be specified in the site QAPP, depending on the local geology and terminology. Formal stratigraphic nomenclature can also be used in addition to the rock name. Rock is classified as rock and not as soil, as it sometimes appears in the cuttings. Conversely, rock fragments or coarse gravel should not be referred to as "rock", unless larger than on cubic yard (see also definition of cobbles and gravel).

A systematic procedure is presented below for qualitatively evaluating the physical condition of a rock mass that was originally developed for logging rock encountered in boreholes; however, it is equally applicable to rocks exposed by natural processes or by excavation. A typical description is presented below, followed by discussions of the description.

Typical Descriptions:

- SANDSTONE, gray. little fractured, hard, moderately strong, moderately weathered.
- TALC SCHIST, yellow, thin-bedded, crushed, low hardness, weak, deep weathering, strike NW, dip 50 degrees NE.

Color. Use Munsell numerical designations to describe color names.

Bedding and Splitting. Rock in place varies in character from massive to thinly laminated or foliated. These planar features (layers) were either formed at the time of deposition (as in the case of sedimentary and some volcanic rocks), or may have resulted from forces acting on the rock at depth (as in the case of foliated metamorphic rocks, such as schist).

The following quantitative classification has been developed to describe layered rock.

Splitting Property	Stratification	Thickness
A. Massive	Very thick-bedded	Greater than 4.0 feet
B. Blocky	Thick-bedded	2.0 to 4.0 feet
C. Slabby	Thin-bedded	0.2 to 2.0 feet
D. Flaggy	Very thin-bedded	0.05 to 0.2 foot
E. Shaley or Platey	Laminated	0.01 to 0.05 foot
F. Papery	Thinly laminated	Less than 0.01 foot

Strike and Dip. Strike and dip of rock bedding planes are obtained from oriented rock cores or surface outcrops. Strike is the direction of a horizontal line on a bedding plane related to the points of the compass (e.g., N 30 degrees E). Dip is the vertical angle below a horizontal plane and the direction down dip related to the four quadrants of the compass (e.g., 45 degrees SE). Always note strike and dip on the field log if they are known or can be estimated.

Fracturing. Fractures include joints, faults, shears, and other more or less continuous ruptures in the rock. Joints are fractures (no relative movement of the rock on either side) and can be regular or irregular and discontinuous. Faults or shears are fractures on which movement has taken place. These features tend to reduce the overall mass hardness and strength of the rock. You should describe whether or not fractures contain open voids, or, if they are filled, describe the type of filling (e.g., clay, quartz, etc.)

Categories of fracturing are based on measurements of the lengths of recovered diamond drill core lengths and inspections of trench exposures.

Intensity	Size of Pieces (feet)
Crushed	Less than 0.05
Intensely fractured	0.05-01.
Closely fractured	0.1 – 0.5
Moderately fractured	0.5 – 1.0
Little fractured	1.0 – 4.0
Massive	Greater than 4.0

Hardness. Hardness and strength are interrelated in many ways, and the meaning of the term depends on how it is being used. Hardness is evaluated with a pocket knife on an unfractured specimen. When equating mass hardness, the degree of fracturing should always be considered, as follows:

- Soft – Reserved for plastic material alone
- Low Hardness – Can be gouged deeply or carved with a pocket knife
- Moderately Hard – Can be readily scratched by a knife blade, scratch leaves heavy trace of dust and is readily visible after the powder has been blown away
- Hard – Can be scratched with difficulty, scratch produces little powder and is often faintly visible
- Very Hard – Cannot be scratched with pocket knife, leaves a metallic streak

Strength. Evaluating the strength of rock in the field is usually a somewhat subjective activity in that it depends on both the observer's interpretation and on the way unfractured specimens respond to hammer blows. Consistency between observers can be evaluated through laboratory test results. Some categories of rock strength include:

- Plastic
- Friable – Crumbles by rubbing with fingers
- Weak – An unfractured specimen of such material will crumble under light hammer blows
- Moderately Strong – Specimen will withstand a few heavy ringing hammer blows before breaking

- Strong – Specimen will withstand a few heavy ringing hammer blows and usually yields large fragments
- Very Strong – Rock will resist heavy ringing hammer blows and with difficulty will yield only dust and small flying fragments

Weathering. Both hardness and strength are influenced by weathering, which usually decomposes or disintegrates the mineral constituents of a rock mass. Permeability is also an important control on the depth of weathering. Pre-existing fractures act as access for water and, therefore, as avenues along which the rate of weathering is accelerated.

Quality Control: Appropriate quality control measures for borehole logging may include the following:

- If more than one geologist/engineer is present on site, collaborate on the description of selected soil samples to ensure uniformity of description.
- Collect an assortment of soil samples representative of the variety of geologic materials, and send these samples to a geotechnical laboratory for analysis. In most cases, a grain size (sieve) analysis, followed by a hydrometer test to divide the fine fraction will be suitable. This test (ASTM Method D422) will provide a grain size distribution curve, and identify a USCS Group Symbol on the overall sample. This serves as a control check for the field geologist/engineer.

BRISTOL ENVIRONMENTAL REMEDIATION SERVICES, LLC

MONITORING WELL INSTALLATION

STANDARD OPERATING PROCEDURE BERS-07

Record of Changes

Revision No.	Date	Prepared by	Approved by
0	01/15/09	B. Allen	L. Maserjian
1	01/04/10	L. Maserjian	B. Allen

MONITORING WELL INSTALLATION

STANDARD OPERATING PROCEDURE

Summary: A borehole is drilled into the ground at the pre-designated location and to the pre-defined depth. Depending on the hydrogeologic conditions of the site and the project objectives, a variety of drilling methods can be used. The most common of these include hollow-stem augering, direct-push drilling, mud rotary drilling, and air rotary drilling.

Well drilling and construction activities will follow all applicable state and local regulations and standards. In the event that a monitoring well is abandoned, all applicable state regulations will be followed. In general, the borehole is sealed, the well casing and screen are removed, and the borehole is cleaned and backfilled. Monitoring wells will be constructed by a driller holding a valid well-driller license, issued by the state where the work is performed.

Health and Safety: Field activities should only be conducted in accordance with an approved Site Health and Safety Plan.

Drilling rigs and equipment present a variety of safety hazards. Field personnel working around drilling rigs should know the position of the emergency "kill" switch. Wires, lines, and ropes should be inspected, and frayed or damaged sections discarded. Swivels and blocks should turn freely. Gages should be operational and controls clearly marked. Underground utilities should be clearly marked, and drillers should be aware of any overhead hazards, such as power lines. Drilling should be avoided in these areas. Ear protection should be worn when working around drilling equipment for extended periods of time, particularly air rotary equipment.

Personnel Qualifications: Field personnel will be trained and certified as hazardous site workers per Title 29 Code of Federal Regulations, Part 1910.120(e) [29 CFR 1910.120(e)]. If applicable, additional qualification requirements will be specified in the site Quality Assurance Project Plan (QAPP), and will be met.

Equipment and Materials: Prior to deployment in the field, the requisite sampling equipment and materials will be identified, secured, and inspected for signs of damage or potential contamination.

The following equipment is necessary for the project's on-site geologist/hydrogeologist:

- Depth sounder or measuring tape
- Water level indicator
- All required health and safety gear
- Description aids (e.g., Unified Soil Classification System [USCS] classification chart, Munsell color chart, grain size charts, etc.)

- Field logbook
- Photoionization detector (PID).

Equipment and tools to install the well are normally provided by the drilling contractor and include the following:

- Drilling equipment appropriate to the site conditions, drilling depth, and other project requirements.
- Threaded flush-jointed riser pipe of an approved material (e.g., Schedule 40 polyvinyl chloride [PVC], 2-inch diameter).
- Threaded flush-jointed slotted screen, made of an approved material (e.g., Schedule 40 PVC, 2-inch diameter) to meet design criteria.
- Well centralizers.
- Properly sized and washed filter pack material (silica sand).
- Bentonite pellets or chips.
- Powdered bentonite.
- Portland cement (American Society for Testing and Materials [ASTM] Types I or II).
- Steel-protective casing with locking cap.
- Tremie pump/box and pipe.

Planning: Drilling and well installation programs must be planned and supervised by an environmental professional. The following steps will be used to plan well installation activities:

- Review existing data on site geology and hydrogeology, including publications, air photographs, water quality data, and existing maps.
- Conduct an on-site assessment to determine potential access problems for drill rig. Locate water supply sources, establish equipment storage area, and observe outcrops.
- Perform utilities check. Note location of underground utilities and overhead electrical wires.
- Select drilling, sampling, and well development methods.
- Determine well construction specifications (e.g., casing and screen materials, casing and screen diameter, screen length and screen interval, filter pack, and screen slot size).
- Determine disposition requirements for drill cuttings and fluids.
- Prepare the work plan, site QAPP, and Site Health and Safety Plan.
- Prepare and execute the drilling subcontract.

Well Construction Materials

All well construction casing and downhole equipment will be thoroughly cleaned prior to downhole use.

Casing Material – The well casing material should not interact with the groundwater. Well casings for environmental projects are usually constructed of PVC or stainless steel. Carbon steel or other appropriate material can also be used, if approved by the regulatory agency. Casing sections can be joined using welded or threaded joints. If using PVC casing, Schedule 40 should be used for shallow wells, and Schedule 80 may be required for deeper wells (greater than 60 feet).

Borehole Diameter – When installing a standard well using hollow-stem auger, the borehole diameter will be a minimum of 4 inches larger than the casing diameter, to allow for emplacement of sand and sealant. When installing a prepacked well, or when using the direct-push drilling method, the borehole diameter may not be 4 inches larger than the casing diameter.

Casing Diameter – 2-inch diameter casing is recommended for most monitoring wells. Larger diameter casing may be required for some applications, and when using the direct-push drilling method, it may be necessary to use smaller diameter casing.

Well Screen – The most common well screen length is 10 feet, though other lengths can be used as required by site-specific conditions, and as allowed by regulatory agencies. The well screen will consist of continuous-slot, machine slotted, or other manufactured PVC or stainless steel well screen. The screen must be installed across the water table. The screen will be installed at a depth that will prevent the water table from ever being above the top of the screen or below the bottom of the screen, taking into account seasonal water table fluctuation. A slot size of 0.010 inches is generally adequate for most installations.

Bottom Cap – A threaded bottom cap will be used.

Filter Pack – The filter pack material will consist of clean silica sand, and be properly sized to prevent fine particles in the formation from entering the well. Clean, medium-to-coarse silica sand is generally adequate as filter pack material for a 0.010-inch slotted well screen. The filter pack will extend at least 2 feet above the top of the screen, unless the water table is shallow enough to prevent optimal well completion.

Bentonite Seal – A bentonite seal will be placed above the filter pack. The bentonite seal will be composed of bentonite chips or pellets (3/8-inch in size or smaller). The bentonite seal will be at least 2 feet in thickness, unless the water table is shallow enough to prevent optimal well completion.

Annular Space Above Bentonite Seal – The annular space above the bentonite seal will be composed of any of the following:

- Bentonite-cement grout (5 pounds of powdered bentonite to 94 pounds of Portland cement, with 6.6 to 8.5 gallons of clean water);
- Neat cement grout (94 pounds of Portland cement, with 5 to 6 gallons of clean water); or,
- Bentonite grout (20 percent solids, created by mixing 50 pounds of bentonite grout with 24 gallons of clan water).

Monument Boxes – For abovegrade monument boxes, a concrete pad (2-foot minimum radius, 4-inch minimum thickness) will be poured around the shroud and wellhead. The concrete and surrounding soil will be sloped to direct rainfall and runoff away from the wellhead. Bollards (steel posts) are recommended around the monument box to protect the wellhead from vehicle damage. For below-grade monitoring wells, a concrete pad (2-foot minimum radius, 4-inch minimum thickness) will be poured around the monument box. The concrete and surrounding soil will be sloped to direct rainfall and runoff away from the well vault.

Well Construction Methods

Overburden (Unconsolidated) Wells: Any of the drilling methods discussed in this SOP can be used to drill or set a well in the unconsolidated material (overburden). The hollow stem or direct-push method are the preferred choices for shallow (<100 feet) overburden wells.

Field personnel will take the following precautions when constructing overburden wells:

1. The project engineer/geologist will determine the screen slot size based on sand pack size. The length of screen used will be site dependent.
2. Under no circumstances should the sand pack extend into more than one aquifer. In most cases, the well design can be modified to allow for a sufficient sand pack without threat of crossflow between producing zones through the sand pack.

Field personnel will implement the following steps when constructing overburden wells:

1. Advance the borehole to the required depth using the selected drilling method. The hole should be drilled approximately one foot deeper than required for the combined length of casing and screen. The final completion depth should be sounded with a decontaminated, weighted tape before continuance of well placement.
2. Withdraw the drill rods if using the rotary or direct-push method. Check the hole depth with a weighted surveyor's tape.
3. Prepare the casing and screen for installation. Attach a threaded cap to base of well screen. Decontaminate the casing and screen using a steam pressure water sprayer before assembly, if not previously done. Tighten joints to manufacturer's specifications.
4. Place at least one foot of filter pack on the bottom of the boring to provide a firm footing for the well.

5. When the casing string is set to the desired depth, hang the centered casing in place. For deeper wells (greater than 60 feet), centralizers may be used near the top and bottom of the well screen.
6. There should be 2 to 3 feet of stickup above the ground surface once the well has been lowered to its final position, unless the wellhead is flush mounted because of its location. Place the well screen across the water table. When constructing a well in a confined aquifer, the top of the well screen is generally positioned just above the interface of the saturated zone and the overlying confining layer.
7. Place a filter pack of clean, silica sand between the well screen and the side of the boring by slowly pouring the sand between the well casing and the boring annulus (gravity feed). Check the elevation of the sand pack periodically by sounding with a weighted tape measure. The filter pack should extend at least 2 feet above the top of the screen to prevent bentonite from penetrating into the screened section and altering groundwater geochemistry. The filter pack may extend less than 2 feet above the top of the screen, if groundwater is shallow enough to prevent optimal well completion.
8. The auger flights/drill casing should be withdrawn slowly, so that the filter pack is placed evenly around the screen without bridging.
9. Allow the filter pack to settle. Check the depth to the top of the filter pack with a weighted tape.
10. In materials that will not maintain an open hole using hollow-stem augers, the temporary or outer casing will be withdrawn gradually during placement of sand pack/grout. For example, after filling 2 feet with sand pack, the outer casing should be withdrawn 2 feet. This step of placing more sand and withdrawing the outer casing should be repeated until the level of the sand pack is approximately 3 feet above the top of the well screen. This ensures that there is no locking of the permanent (inner) casing in the outer casing.
11. Place a bentonite seal of a minimum of 3-foot vertical thickness in the annular space above the sand pack to separate the sand pack from the cement surface seal. Using a tremie pipe, or using gravity feed for shallow wells (<35 feet), place bentonite pellets or chips (not powder) onto the top of the filter pack. Bentonite pellets can be used if the seal is to be seated below the water table. Slurried bentonite may be used if an unusually high water level exists in the well and precludes the use of bentonite pellets. If a tremie pipe is used, slowly withdraw the pipe as the bentonite is added, to ensure even placement of the bentonite seal around the annulus. Check the depth with a weighted tape.
12. If the bentonite seal is installed above the water table, hydrate according to the manufacturer's specifications. Allow adequate hydration time before dismantling.
13. If a slurry of bentonite is used as an annular seal, it is prepared by mixing powdered or granular bentonite with potable water. The slurry must be of sufficiently high specific gravity and viscosity to prevent its displacement by the grout to be emplaced above it. As a precaution (regardless of depth), and depending on fluid viscosity, a few handfuls of bentonite pellets may be added to solidify the bentonite slurry surface.

14. To prevent pellets from entering the well casing, a cap will be placed over the top of the well casing before pouring the bentonite pellets.
15. Place cement and/or bentonite grout from the top of the bentonite seal to the ground surface.
16. Fill the annulus with grout from the top of the annular seal until undiluted grout flow from the annulus at the ground surface. Grout may be placed by pouring or by pumping the grout through a tremie pipe (normally a 1.25-inch PVC or steel pipe).
17. If the well is completed above ground, a protective casing will be installed. The protective casing will be constructed of metal, and will be at least 5 feet in length. The protective casing will extend approximately 1.5 to 3 feet above the ground surface, and will be set in concrete or cement grout. The protective casing diameter should be 4 inches greater in diameter than the well casing. A 0.5-inch drain hole may be installed near ground level. A flush-mount protective casing may be used in areas of high traffic, or where access to other areas would be limited by a well stick-up.
18. Install a protective steel cap, and secure it to the protective casing by a padlock.
19. Steel bollards should be installed around the protective casing in areas where vehicle traffic may be a problem. Posts should have a minimum diameter of 3 inches, with a minimum height of 4 feet.
20. Label and date monitoring wells with paint, engraving, or steel tags.

Bedrock Wells: Bedrock wells will be drilled using the air or mud rotary method and completed as an open-hole, providing that borehole cave-in is not a possibility. Crystalline rock wells are usually drilled most efficiently with the air rotary method, while consolidated sedimentary formations are drilled using either the air rotary or mud rotary method.

Well Development: Well development is the process by which the aquifer's hydraulic conductivity is restored by removing drilling fluids and fine-grained formation material from newly installed wells. The monitoring well should be developed so that formation water flows freely through the screen and is not turbid, and all sediment and drilling disturbances are removed from the well.

The most common method of well development is surging and bailing. A well is considered developed when the pH and conductivity of the groundwater stabilizes, and the measured turbidity is less than 50 nephelometric turbidity units (NTUs).

Field personnel will implement the following steps to develop the well:

- Measure the total depth (TD) of the well and depth to water (DTW).
- Using an appropriately-sized surge block, surge 5-foot sections of well screen, using 10-20 up/down cycles per section. Periodically remove the surge block and bail accumulated sediment from the well, as required. Ensure that the surge block and bailer are properly decontaminated before use.

- Water quality parameters will be measured periodically during well development. When turbidity is less than 50 NTU and pH and conductivity have stabilized, development is complete.
- For some wells, it may not be possible to achieve turbidity of less than 50 NTU, even when pH and conductivity have stabilized. In these instances, field personnel should note the final turbidity on the Well Development Form.

If used for collection of water quality parameters, the pump selected must be rated to achieve the desired yield at a given depth. The pump system should include the following:

- A check valve to prevent water from running back into the well when the pump is shut off.
- Flexible discharge hose.
- Safety cable or rope to remove the pump from the well.
- Flow meter monitoring system (measuring bucket or inline flow meter).

The amount of water removed during development will be recorded on the Well Development Form. The pump rate and water quality parameters (pH, conductivity, and turbidity) will also be recorded every 5 to 10 minutes.

Well Abandonment: When a decision is made to abandon a monitoring well, the borehole should be sealed in such a manner that the well cannot act as a conduit for migration of contaminants from the ground surface to the water table or between aquifers.

To properly abandon a well, the preferred method is to completely remove the well casing and screen from the borehole, clean out the borehole, and backfill with a cement or bentonite grout, neat cement, or concrete. The grout should be pumped from the bottom of the borehole to the ground surface using a tremmie pipe. If the casing cannot be removed, bentonite-cement grout, neat cement grout, or bentonite grout must be emplaced in the well using a tremie pipe from the bottom of the well to the ground surface.

In order to comply with state well abandonment requirements, the appropriate state agency should be notified (if applicable) of monitoring well abandonment. However, some state requirements are not explicit, so a technically sound well abandonment method should be designed based on the site geology, well casing materials, and general condition of the well(s).

Data Management and Records Management: Well construction, development, and abandonment records will be generated and maintained as prescribed in the governing QAPPs. Well installation data will be documented on field data sheets or in the logbooks.

Interferences and Potential Problems: The main source of problems in well installation is a flawed design, usually resulting in the use of drilling or installation procedures that are not appropriate or optimal for the site conditions. The design and installation of permanent

monitoring wells involve drilling into various types of geologic formations that exhibit varying subsurface conditions. Designing and installing permanent monitoring wells in these geologic environments may require several different drilling methods and installation procedures.

The selection of drilling methods and installation procedures should be based on field data collected during a hydrogeologic site investigation and/or a search of existing data. Each permanent monitoring well should be designed and installed to function properly throughout the duration of the monitoring program. When designing monitoring wells, the following should be considered:

- short-and long-term objectives
- purpose(s) of the well(s)
- probable duration of the monitoring program
- contaminants likely to be monitored
- types of well construction materials to be used
- surface and subsurface geological/hydrogeological conditions (e.g., presence of heaving sand or artesian conditions)
- properties of the aquifer(s) to be monitored
- well screen placement
- general site conditions
- potential site health and safety hazards.

The advantages and disadvantages of each drilling method are evaluated based on these considerations. Advantages and disadvantages of the various drilling methods are summarized below.

Direct-Push Drilling

The advantages of direct-push drilling are:

- Zero cuttings (except from Macro-Core® sampling)
- Fast in shallow situations
- Sampling cores (Macro-Cores) 4 to 5 feet long
- Can be used to install temporary or permanent wells
- Small rig can access tight areas; easy to transport to remote locations.

The disadvantages of direct-push drilling are:

- Small rigs have limited depth
- Can only install wells up to 2 inches in diameter.

Auger Drilling

The advantages of auger drilling are:

- Relatively fast and inexpensive
- Because augers act as temporary casings, drilling fluids are not used, resulting in reduced well development.

The disadvantages of auger drilling are:

- Very slow or impossible to use in coarse materials, such as cobble or boulders
- Cannot be used in some consolidated formations, and is generally limited to depths of approximately 100 feet.

Mud Rotary Drilling

The advantages of mud rotary drilling are:

- Fast, more than 100 feet of borehole advancement per day is common
- Provides an open borehole, necessary for some types of geophysical logging and other tests
- Can drill through consolidated bedrock.

The disadvantages of mud rotary drilling are:

- Potential for cross-contamination of water-bearing zones
- Drill cuttings may be mixed and not accurately represent lithologies at a given drilling depth
- Drilling mud may alter the groundwater chemistry
- Water levels can only be determined by constructing wells
- Drilling mud may change local permeability of the formation, and may not be entirely removed during well development
- Disposal of large volumes of drilling fluid and cuttings may be necessary if they are contaminated.

Air Rotary Drilling

The advantages of air rotary drilling are:

- Fast, more than 100 feet of borehole advancement a day is possible
- Preliminary estimates of well yields and water levels are often possible
- No drilling fluid to plug the borehole
- Can drill through consolidated bedrock

The disadvantages of air rotary drilling are:

- Generally cannot be used in unconsolidated formations.
- In contaminated zones, the use of high-pressure air may pose a significant hazard to the drill crew because of transport of contaminated material up the hole.
- Introduction of air to the groundwater could reduce concentration of volatile organic compounds (VOCs).

Sonic Drilling

The advantages of sonic drilling are:

- One of the fastest drilling methods available
- Minimizes generation of waste (soil cuttings)
- Can drill through some types of bedrock
- Allows for continuous core sampling

The disadvantages of sonic drilling are:

- More expensive than most drilling methods
- Occasionally requires use of water or other drilling fluid
- In some consolidated formations, may generate heat which could volatize VOCs prior to sample collection.

BRISTOL ENVIRONMENTAL REMEDIATION SERVICES, LLC

WATER LEVEL MEASUREMENT

STANDARD OPERATING PROCEDURE BERS-08

Record of Changes

Revision No.	Date	Prepared by	Approved by
0	01/15/08	B. Allen	S. Ruth
1	2/23/10	M. Faust	B. Allen

WATER LEVEL MEASUREMENT

STANDARD OPERATING PROCEDURE

Purpose and Scope: The purpose of this document is to provide sufficient and appropriate instructions for the determination of the depth-to-water and floating chemical product (i.e., gasoline, kerosene) in an open borehole, cased borehole, monitoring well, or piezometer.

Summary: Prior to measurement, water levels in piezometers and monitoring wells are allowed to stabilize for a minimum of 24 hours after well construction and development. A survey mark is placed on the casing for use as a reference point for measurement. The distance from water surface to reference point on well casing is measured at least twice and recorded.

Health and Safety: Field activities should only be conducted in accordance with an approved Site Health and Safety Plan.

Interferences and Potential Problems: Generally, water level measurements taken in boreholes, piezometers, or monitoring wells are used to construct water table or potentiometric surface maps, and to determine flow direction, as well as many other aquifer characteristics. Situations that may impact the accuracy of water level measurements include:

- The magnitude of the observed changes between wells appears too large;
- Atmospheric pressure changes;
- Aquifers that are tidally influenced;
- Aquifers affected by river stage, impoundments, and/or unlined ditches;
- Aquifers stressed by intermittent pumping of production wells;
- Aquifers being actively recharged due to precipitation event;
- Occurrence of pumping; and
- During storm events over a shallow aquifer where recharge is rapid.

Additional sources of error may include the following:

- The chalk used on steel tape may contaminate the well.
- Cascading water may obscure the water mark, or cause it to be inaccurate.
- Many types of electric sounders use metal indicators at 5-foot intervals around a conducting wire. To ensure accuracy, these intervals should be checked with a survey tape (preferably with units divided in hundredths of a foot).

- If there is product or oil present on the water, it can insulate the contacts of the probe on an electric sounder, or give false readings due to thickness of the oil. If this situation is suspected, it is recommended that interface probes be used to determine the thickness and density of the oil layer in order to determine the correct water level.
- Turbulence in the well and/or cascading water can make water level determination difficult with either an electric sounder or steel tape.

Personnel Qualifications: Field personnel will be trained and certified as hazardous site workers per Title 29 Code of Federal Regulations, Part 1910.120(e) [29 CFR 1910.120(e)]. If applicable, additional qualification requirements will be specified in the site-specific Quality Assurance Project Plan (QAPP) and will be met.

Equipment and Materials: Prior to deployment in the field, the requisite sampling equipment and materials will be identified, secured, and inspected for signs of damage or potential contamination.

There are a number of devices that can be used to measure water levels. The device must be capable of attaining an accuracy of 0.02 feet, and calibrated on a regular basis.

Field equipment for performing water level measurements include:

- Air monitoring equipment (e.g., photoionization detector [PID] or flame ionization detector [FID])
- Well depth measurement device
- Electronic water level indicator
- Metal tape measure
- Chalk
- Ruler
- Watch
- Logbook
- Paper towels
- Groundwater water level data forms
- pH meter (optional)
- Specific conductivity meter (optional)
- Thermometer (optional).

Site Preparation: The following steps will be followed before measurement activities are performed:

- Determine the extent of the sampling effort, the sampling methods to be employed, and the types and amounts of equipment and supplies needed.
- Obtain necessary sampling and monitoring equipment.
- Decontaminate or pre-clean equipment, and ensure that it is in working order.
- Perform a general site survey prior to site entry in accordance with the Site-Specific Health and Safety Plan.
- Identify and mark all sampling locations.

Water Level Measurement: A survey mark should be placed on the north side of the casing for use as a reference point for measurement. Generally, the reference point is marked on the top of the well casing, and is established at the time the well is surveyed. The measuring point should be documented in the site logbook and on a groundwater level data form. Every attempt should be made to notify future field personnel of such reference points in order to ensure comparable data and measurements.

Prior to measurement, water levels in piezometers and monitoring wells should be allowed to stabilize for a minimum of 24 hours after well construction/development. In low-yield situations, recovery may take longer. Measurements should be made to the closest 0.01 feet.

The following procedures will be followed to determine groundwater elevation:

1. Make sure that water level measuring equipment is in good operating condition.
2. To the extent known, measure wells in order of increasing contamination.
3. Clean all equipment entering the well.
4. Remove locking well cap, note well ID, time of day, elevation (top of casing) and date in site logbook or an appropriate groundwater level data form.
5. Remove well casing cap.
6. If required by site-specific conditions, monitor headspace of well with a PID or FID to determine presence of volatile organic compounds, and record in site logbook.
7. Lower electric water level measuring device or equivalent into the well until water surface is encountered.
8. Measure the distance from the water surface to the reference measuring point on the well casing or protective barrier post, and record in the site logbook. In addition, note that the water level measurement was from the top of the steel casing, the top of the PVC riser pipe, the ground surface, or some other position on the wellhead.
9. Groundwater level data should be documented as follows:
    - Logger Name – Person taking field notes;
    - Site Name;

- Date the water levels are measured;
- Location – Monitor well number and physical location;
- Time (24-hour clock) at which the water level measurement was recorded;
- Depth to Water – Water level measurement in feet, tenths, or hundredths of feet, depending on the equipment used. Two measurements are required to ensure accuracy;
- Comments – Any information the field personnel deems applicable may be included here;
- Measuring Point – Marked measuring point on PVC riser pipe, protective steel casing, or concrete pad surrounding well casing, from which all water level measurements for individual wells should be measured. This provides consistency in future water level measurements.

10. Measure total depth of well (at least twice to confirm measurement) and record in field logbook or on groundwater level data form.
11. Remove all downhole equipment; replace well casing cap and locking steel caps.
12. Rinse all downhole equipment and store for transport to next well.
13. Decontaminate all equipment.
14. Note any physical changes, such as erosion or cracks in protective concrete pad or variation in total depth of well, in field logbook and on groundwater level data form.

Quality Control: The following procedures apply:

- Equipment will be operated and used in accordance with the manufacturer's instructions, unless otherwise specified in the site-specific work plan or its equivalent.
- Equipment examination activities should occur prior to field deployment, and they should be documented.
- Each well should be tested at least twice in order to compare results.

Calculations and Data Reduction: Calculations and data reduction will be performed using the following equations and rules:

Groundwater elevation above mean sea level: $E_w = E - D$

where:

E_w = Elevation of water above mean sea level or local datum (feet or meters)

E = Elevation above sea level or local datum at point of measurement (feet or meters)

D = Depth to water (feet or meters)

BRISTOL ENVIRONMENTAL REMEDIATION SERVICES, LLC

INVESTIGATION-DERIVED WASTE (IDW) MANAGEMENT

STANDARD OPERATING PROCEDURE BERS-09

Record of Changes

Revision No.	Date	Prepared by	Approved by
0	01/15/09	B. Allen	L. Maserjian
1	02/23/10	L. Maserjian	B. Allen

IDW MANAGEMENT

STANDARD OPERATING PROCEDURE

Summary: Investigation-derived waste (IDW) includes any material discarded after use during a field investigation at a hazardous waste site, and it includes personal protective equipment (PPE), disposable equipment, such as sampling equipment, drilling mud, soil cuttings, purge, or well-development water. IDW is classified as either hazardous or nonhazardous, depending on the properties of the waste. Whenever feasible, all IDW will be disposed of on site at active facilities.

If IDW is suspected to be hazardous, the material will be tested for proper classification. If the test determines the material to indeed be hazardous, it will be stored on site no longer than 90 days and then disposed of at a permitted treatment or disposal facility. Alternatively, it will be placed in the facility's waste treatment system, if appropriate. Whenever possible, nonhazardous IDW will be disposed of in the facility's Dumpster, waste treatment system, or on the ground in or near the source area, as appropriate. If on-site disposal is not feasible, nonhazardous IDW will be disposed of in a Dumpster or landfill.

Health and Safety: Field activities should only be conducted in accordance with an approved Site Health and Safety Plan.

Interferences and Potential Problems: Care should be taken to ensure segregation of hazardous 1DW from nonhazardous materials. The volume of spent solvent generated from field equipment decontamination procedures should be kept to a minimum, by applying only the minimum amount of solvent necessary and capturing it separately from the wash water. All hazardous waste will be containerized. Project planning will address procedures and responsibilities for the proper handling and disposal of project IDW.

Personnel Qualifications: Field personnel will be trained and certified as hazardous site workers per Title 29 Code of Federal Regulations, Part 1910.120(e) [29 CFR 19 10.120(e)]. If applicable, additional qualification requirements will be specified in the site Quality Assurance Project Plan (QAPP) and will be met.

Equipment and Materials: Prior to deployment in the field, the materials necessary for the management of IDW wastes in the field, such as 55-gallon drums and 5-gallon buckets, will be identified and secured.

Types of IDW: Materials which may become IDW include, but are not limited to, the following:

- PPE, including disposable coveralls, gloves, booties, respirator canisters, splash suits, etc.

- Disposable equipment, including plastic ground and equipment covers, aluminum foil, conduit pipe, composite liquid waste samplers, tubing, and broken or unused sample containers, sample container boxes, or tape, etc.
- Soil cuttings from drilling or hand augering activities.
- Drilling mud or water used for water rotary drilling.
- Groundwater obtained through well development or well purging.
- Cleaning fluids, such as spent solvents and wash water.

Management of Hazardous IDW: The site QAPP will specify disposal practices for hazardous or suspected hazardous IDW. If appropriate, these wastes will be disposed of on site by placement into the facility's waste treatment system, or they will be disposed of in the source area from which they originated, if doing so does not endanger human health or the environment. If on-site disposal is not possible, appropriate tests will be performed to characterize the waste for proper disposal. If the wastes are determined to be hazardous, they will be properly contained and labeled, and then stored on site for a maximum of ninety days before they are manifested and shipped to a permitted treatment or disposal facility.

The generation of hazardous IDW will be kept to a minimum. Nonhazardous materials will be segregated from hazardous materials to prevent cross-contamination. The most commonly produced type of IDW will probably be spent solvent from decontamination procedures and purged groundwater. Segregating the solvent from the wash water during equipment decontamination procedures will minimize the volume of spent solvent IDW generated during field activities.

Field personnel will implement the following procedures when managing hazardous IDW from specific practices:

- Disposable PPE – Containerize in 5-gallon bucket with tight-fitting lid. Identify and leave on site with permission of site operator. Otherwise, arrange for proper off-site disposal.
- Reusable PPE – Decontaminate following procedures described in the SOP BERS-05: *Equipment Decontamination*. Otherwise, follow procedures for disposable PPE.
- Spent Solvents – Containerize in original containers with contents clearly identified. Leave on site with permission of site operator.
- Soil Cuttings – Containerize in 55-gallon drum with a tight-fitting lid. Identify and leave onsite with permission of site operator.
- Groundwater – Containerize in 55-gallon drum with a tight-fitting lid. Identify and leave on site with permission of site operator. Otherwise, arrange for testing and proper off-site disposal.

- Decontamination Water – Containerize in 55-gallon drum with a tight-fitting lid. Identify and leave on site with permission of site operator. Otherwise, arrange for testing and proper off-site disposal.
- Disposable Equipment – Containerize in 55-gallon drum or 5-gallon bucket with a tight-fitting lid. Identify and leave on site with permission of site operator. Otherwise, arrange for testing and proper off-site disposal.

Management of Nonhazardous IDW: The site QAPP will specify disposal practices for nonhazardous IDW. If the waste site is active, permission will be sought from the site operator for on-site disposal of nonhazardous PPE, disposable equipment, and/or paper/cardboard wastes in the facility's Dumpsters. If on-site disposal is not feasible, the materials will be taken to a nearby permitted landfill.

If the facility is active, permission will be sought to place nonhazardous IDW, including drill cuttings, purge or well-development water, decontamination wash water, and drilling mud, etc., in the facility's waste treatment system. When appropriate, nonhazardous drill cuttings will be spread around the borehole, or, if they were removed for a temporary well, they will be placed back into the borehole. Otherwise, cuttings, purge water, and development water will be placed in a pit in or near the source area. Nonhazardous monitoring well purge or development water may also be poured onto the ground downgradient of the monitoring well. Purge water from functioning private potable wells will be discharged directly onto the ground surface. If on-site disposal is not feasible, these items will be placed into a unit with an environmental permit, such as a landfill or sanitary sewer. These types of materials will not be placed in Dumpsters.

Field personnel will implement the following procedures when managing nonhazardous IDW from specific practices:

- Disposable PPE – Place waste in double bag, and place in site Dumpster, with permission of site operator. Otherwise arrange for testing and disposal.
- Reusable PPE - Decontaminate following procedures described in the SOP BERS-05: *Equipment Decontamination.*
- Soil Cuttings – Containerize in 55-gallon drum with a tight-fitting lid. Identify and leave on site with permission of site operator. Otherwise, arrange for testing and disposal.
- Groundwater – Containerize in 55-gallon drum with a tight-fitting lid. Identify and leave on site with permission of site operator. Otherwise, arrange for testing and disposal.
- Decontamination Water – Containerize in 55-gallon drum with a tight-fitting lid. Identify and leave on site with permission of site operator. Otherwise, arrange for testing and disposal.
- Disposable Equipment – Containerize in 55-gallon drum or 5-gallon bucket with tight-fitting lid. Identify and leave on site with permission of site operator. Otherwise, arrange for testing and disposal.

- Trash – Place waste in double bag, and place in site Dumpster with permission of site operator. Otherwise, arrange for proper disposal.

Quality Control: The following procedures apply:

- Proper handling and disposal activities will be planned prior to commencement of field activities. All planning decisions will be documented in the site QAPP.
- IDW will be handled, stored, and disposed of in accordance with the site QAPP and relevant facility plans.

Calculations and Data Reduction: N/A

Data Management and Records Management: Records concerning the management of IDW will be generated and maintained as prescribed in the governing QA plans.

BRISTOL ENVIRONMENTAL REMEDIATION SERVICES, LLC

FIELD DOCUMENTATION

STANDARD OPERATING PROCEDURE BERS-11

Record of Changes

Revision No.	Date	Prepared by	Approved by
0	01/05/10	L. Maserjian	B. Allen

FIELD DOCUMENTATION

STANDARD OPERATING PROCEDURE

Method Summary: To ensure the quality and integrity of field and analytical data, field activities will be documented in the project field notebook. In the event that more than one person is working on the site and performing different activities, more than one field notebook will be designated for the site. When the field notebook is filled, a new notebook will be started. Pertinent protocols for documenting field activities are provided below.

Notebook Cover: The cover of each field notebook will contain the following information:

- Job title
- Job number
- Name of company
- Name of personnel in charge of notebook
- Date of field activities covered in the notebook.

First Page of Each Day: The following information must be provided in the beginning of each day of work:

- Job title
- Names of all personnel on site
- Weather conditions
- Location, if multiple sites
- Health and Safety meeting notes.

Each Page of Notebook: The following information must be provided on each page of the field notebook:

- Date
- Initials or signature of person taking notes (bottom of page)
- Location, if you have changed during the day
- Page number, if not on the notebook.

Required General Information for Field Notebooks:

- Do not erase mistakes/errors – draw a line through the deletion and initial it.
- Do not leave pages blank. If a page is skipped, draw a diagonal line across the page and initial the line.
- Record persons arriving and leaving site (guests to site, clients, regulatory agency personnel).
- Record health and safety issues that arise (close calls or accidents should also be documented on required forms).
- Note photographs taken and direction in which photograph was taken.
- Take an overview photograph of site before digging/drilling, etc.
- Include a photograph of the site after it is restored (if applicable).

Required Documentation for Sample Collection Activities:

- Instrument name;
- Calibration record (when, by whom, results, gas type);
- Sampling location map with North arrow (field-screening and analytical samples);
- Sample ID, with description of soil material;
- Duplicate information;
- Sample time, each sample;
- Sample depth;
- List what analyses sample will be analyzed for;
- Field-screening measurements;
- Type of machinery used if not already recorded on field forms (Macro-Core sampler, split spoon, pumps, sampling meters);
- If Global Positioning System (GPS) is used, make note of where it was used;
- Delivery or pick-up information (airway bill #, Fed Ex tracking #, Fed Ex pick up information).

Required Documentation for Underground Storage Tank (UST)/Aboveground Storage Tank (AST) Removal Activities:

- UST or AST dimensions;
- Dimensions of tank excavations, depth to groundwater, and depth of excavation;

- Footage of fuel piping (how many feet from dispenser to tanks);
- Where vent lines, fill ports, dispensers and pipe runs are located;
- Location of piping joints;
- Amount of sludge/water removed from tanks prior to decommissioning;
- Amount of contaminated soil/media (cubic yards of stockpiles);
- Amount of contaminated soil or debris hauled from site (number of truckloads);
- Amount of clean fill brought to the site;
- Type of machinery used.

Required Documentation for Monitoring Well/Soil Boring Activities (This list does not include the documentation that will be provided on a boring log and groundwater sample collection form.):

- Always collect swing-tie measurements to monitoring wells (even if you have a GPS);
- If drillers add water during well installation, note how much was added;
- Well screen slot size;
- Well filter sand pack size;
- Depth of top and bottom of well screen;
- Total depth of well;
- Amount of well construction materials used for each well (e.g., bags of silica sand, concrete, amount of screened casing, and amount of blank casing);
- Location of sand filter pack, bentonite seal, and grout used;
- Amount of water removed during development (unless you are using a well development form);
- Drill rig type;
- Changes in level of the water table/ aquifer.

Interferences and Potential Problems: Improper documentation of field activities may result in a number of problems, including, but not limited to:

- Inability to find sample collection locations that is needed for maps or finding areas for further assessment/excavation;
- Inability to create an as-built map;
- Inability to legally support data due to poor documentation;

- Development of erroneous conclusions regarding site contamination based on inaccurate data and/or problems correlating data and sample locations at the site;
- Difficulty in writing thorough reports due to poor documentation.

BRISTOL ENVIRONMENTAL REMEDIATION SERVICES, LLC

EXCAVATION AND TRENCHING

STANDARD OPERATING PROCEDURE BERS-12

Record of Changes

Revision No.	Date	Prepared by	Approved by
0	03/9/10	C. Croley	G. Jarrell
1	4/20/10	M. Faust	P. Curl

EXCAVATION AND TRENCHING

STANDARD OPERATING PROCEDURE

Summary: The standards covering excavation and trenching safety are included in Title 29 Code of Federal Regulations, Part 1926.650-652 (Subpart P) [29 CFR 1926.650-652, subpart P], and U.S. Army Corps of Engineer's Safety and Health Requirements Manual 385-1-1 (15 September 2008). The Federal Standards require protective systems to be in place when anyone enters an excavation or trench that is more than 5 feet (1.5 meters) in depth. Bristol Environmental Remediation Services, LLC (Bristol), as a member of the Bristol Alliance of Companies, through their Corporate Health and Safety Manual, follows a more conservative guideline of requiring protective systems to be in place for excavations or trenches of 4 feet (1.2 meters) in depth.

Definitions:

1. A "Protective System" means a method of protecting employees from cave-ins, from material that could fall or roll from an excavation face or into an excavation, or from collapse of adjacent structures. Protective systems include support systems, sloping and benching systems, shield systems, and other systems that provide the necessary protection.
2. An "Excavation" means any man-made cut, cavity, trench, or depression in an earth surface, formed by earth removal. Bristol's use of excavations is normally in conjunction with the installation or removal of underground storage tank facilities.
3. A "Trench" (trench excavation) means a narrow excavation (in relation to its length) made below the surface of the ground. In general, the depth is greater than the width, but the width of a trench (measured at the bottom) is not greater than 15 feet (4.6 meters). Trenching is normally used in conjunction with contaminated site assessments where samples are collected for field screening or analysis, or when piping is being installed or removed.
4. A "Confined or Enclosed Space" means any space having a limited means of egress, which is subject to the accumulation of toxic or flammable contaminants or has an oxygen deficient atmosphere. Confined or enclosed spaces include, but are not limited to, storage tanks, process vessels, bins, boilers, ventilation or exhaust ducts, sewers, underground utility vaults, tunnels, pipelines, and open-top spaces more than 4 feet in depth, such as trenches, pits, tubs, vaults, and vessels.

Personnel Qualifications: All personnel associated with trenching and excavation will be trained in the safe practices applicable to excavating and trenching. Personnel will be trained in the applicable elements of 29 CFR1926.651, 1926.652 and subpart P. Additional training may be required for trenches that are considered to be a confined space, or present other work-related hazards.

Intent: It is not intended that Bristol personnel routinely conduct work activities in any excavation or trench.

Excavation/Trenching Plans: Written plans, although not always required, are suggested as an effective checklist prior to beginning excavation/trenching activities. Any excavation/trenching activities that fall under the USACE 385-1-1 safety guidelines require a written and approved plan prior to the start of work.

Work Permit: Excavations will require a Confined Space Work Permit when the depth of an excavation exceeds 4 feet, and personnel will access the excavation (Attachment 1).

Equipment and Materials: Prior to deployment in the field, the requisite trenching equipment and materials will be identified, secured, and inspected for signs of damage or potential contamination. General equipment requirements for trenching include.

- Excavator or backhoe.
- Materials required to demark the excavation/trench and equipment from the general public.
- Pre-engineered protective system (i.e., trench box) if personnel are to be entering an excavation/trench in excess of four feet in depth.
- Manufactured materials and equipment used for protective systems must be used and maintained in a manner that is consistent with the recommendations of the manufacturer, and in a manner that will prevent personnel exposure to hazards.
- Materials and equipment used for protective systems must be free from damage or defects that might impair their proper function.

Health and Safety Requirements: Excavation/Trenching should only be conducted in accordance with an approved site health and safety plan. General safety requirements are listed below:

- Prior to the commencement of trenching activities, all locations must be verified free and clear of underground and overhead utilities.
- Each person in an excavation must be protected from cave-ins by an adequate protective system, except when:
  - Excavations are made entirely in stable rock; or
  - Excavations are less than 4 feet in depth and examination of the ground provides no indication of a potential cave-in.
- Spoil piles should be kept a minimum of 2 feet from any edge of an excavation/trench, no matter what the sidewall angle of repose may be.

- Protective systems must have the capacity to resist without failure all loads that are intended or could reasonably be expected to be applied or transmitted to the system.
- Daily inspections of excavations, the adjacent areas, and protective systems, must be made for evidence of a situation that could result in possible cave-ins, indications of failure of protective systems, hazardous atmospheres, or other hazardous conditions. An inspection must be conducted prior to the start of work and as needed throughout the shift. Inspections must also be made after every rainstorm or other hazard-increasing occurrence.
- Adequate barrier physical protection must be provided at all trenches. During excavation, appropriate warning signs, flagging, or barricading shall be in place as fall protection. Upon completion of exploration and similar operations, trenches must be backfilled.
- Excavations located in close proximity to recognized roadways must be barricaded on the traffic side with illuminated or reflective materials barricades.
- Walkways or bridges with standard guardrails must be provided where personnel or equipment are to cross over trenches that are 4 feet in depth or greater.
- While the excavation is open, underground installations must be protected, supported, or removed as necessary to safeguard personnel.
- A stairway, ladder, ramp, or other safe means of egress must be located in shored trench excavations that are 4 feet or more in depth, so as to require no more than 25 feet of lateral travel for personnel.
- Personnel are not permitted underneath loads handled by lifting or digging equipment. Personnel are required to stand away from any vehicle being loaded or unloaded to avoid being struck by any spilled or falling materials. Operators may remain in the cabs of vehicles being loaded or unloaded when the vehicles are equipped to provide adequate protection for the operator during loading and unloading operations.
- Where oxygen-deficiency (atmospheres containing less than 19.5% oxygen) or a hazardous atmosphere exists, or could reasonably be expected to exist, such as in trenches in landfill areas or in areas where hazardous substances are stored nearby, the atmospheres in the excavation must be tested before personnel enter excavations regardless of depth.
- If the stability of adjoining buildings, walls, or other structures is endangered by excavation operations, support systems, such as shoring, bracing, or underpinning, must be provided (and inspected and approved by a Professional Engineer) to ensure the stability of such structures for the protection of personnel.
- Personnel may not work in trenches in which there is accumulated water, or where water is accumulating, unless adequate precautions have been taken to protect personnel against the hazards posed by water accumulation. The precautions necessary to protect personnel adequately vary with each situation, but could include special support or shield systems

- to protect from cave-ins, water removal to control the level of accumulating water, or use of a safety harness and lifeline.
- If evidence of a situation that could result in possible cave-ins, slides, failure of protective systems, hazardous atmospheres, or other hazardous conditions is identified, exposed workers shall be removed from the hazard and all work in the excavation/trench stopped until all necessary safety precautions have been implemented.
- Adequate protection must be provided to protect personnel from loose rock or soil that could pose a hazard by falling or rolling from an excavation face. Such protection must consist of scaling to remove loose material; installation of protective barricades at intervals as necessary on the face to stop and contain falling material; or other means that provide equivalent protection.
- The slopes and configurations of sloping and benching systems must be selected and constructed by the employer or his designee and must be in accordance with the requirements of the following:
  - Soil type must be determined utilizing the guidelines set forth in CFR 1926 Subpart P.
  - Benching and sloping requirements will be based on the determination of soil type and are listed in CFR 1926 subpart P.
  - If benching and sloping will not be utilized, then a pre-engineered shoring system shall be utilized to protect personnel from cave-in.
    - If trench boxes are used, the top of the trench box must extend a minimum of 18 inches above the point where the vertical soil wall meets the soil slope.

ATTACHMENT 1

Confined Space Entry Permit

Bristol **Confined Space Entry (CSE) Permit** **Display at Site**

Client:		Location:	
Date/Time Issued:		Date/Time Expires:	
Permit Initiator:		Entry Supervisor:	
Purpose for CSE:		Work to be Performed	

Standby Personnel:	1. 2. 3. 4.	Authorized Entrants:	1. 2. 3. 4.

Atmospheric Check (Prior to Entry) **Tester's Signature: _____**

Time:	% Oxygen:	% LEL:	Toxic ppm:

Energy Isolation Complete: ☐ yes ☐ no **Signature: _____**

Ventilation: ☐ Mechanical ☐ Natural

Atmospheric Check (During Entry) **Tester's Signature: _____**

Time:	% Oxygen:	% LEL:	Toxic ppm:
Time:	% Oxygen:	% LEL:	Toxic ppm:
Time:	% Oxygen:	% LEL:	Toxic ppm:
Time:	% Oxygen:	% LEL:	Toxic ppm:

Communication Procedures: _____

Rescue Procedures: _____

Rescue Equipment: _____

PPE Required: _____

Entry Supervisor Signature: _____

CSE Owner/Client signature: _____

Debrief required: ☐ yes ☐ no Return form to project manager when CSE complete.

BRISTOL ENVIRONMENTAL REMEDIATION SERVICES, LLC

GENERAL BACKFILL AND COMPACTION

STANDARD OPERATING PROCEDURE BERS-13

Record of Changes

Revision No.	Date	Prepared by	Approved by
0	03/9/10	C. Croley	G. Jarrell

GENERAL BACKFILL AND COMPACTION

STANDARD OPERATING PROCEDURE

Summary: After an excavation or trench has been completed, it will be backfilled and compacted. Proper backfill and compaction are important for maintaining the integrity of the ground surface. Compaction is defined as the method of mechanically increasing the density of soil. Backfill and compaction is commonly associated with underground storage tank (UST) removals and contaminated soil excavation. Backfill and compaction criteria will be based on the final use of the finished grade, i.e., foundation, traffic, or non-traffic areas. Backfill and compaction procedures for sites where density testing is not required are described in this Standard Operating Procedure.

There are five principle reasons to compact soil:

- Increases the load-bearing capacity;
- Prevents soil settlement and frost damage;
- Provides stability;
- Reduces water seepage, swelling, and contraction; and
- Reduces settling of soil.

Personnel Qualifications: Personnel will be trained and certified as hazardous site workers per 29 CFR 1910.120(e). Lead field personnel will have at least two years field experience with supervision of heavy equipment operators. If applicable, additional qualification requirements will be specified in the site QA Project Plan and met.

General Procedures for Backfill and Compaction:

Backfill material will be specified in the site work plan or QA plan. Generally, common fill from a local supplier will be used with the goal of using non-frost susceptible (NSF) materials, if possible, when working in cold climates.

The backfill material will be placed into the excavation or trench and spread into one foot lifts. If possible, each lift will be wetted. Each lift will be compacted using the heavy equipment (tracks or bucket). By following this procedure, compaction densities of 90% or greater are typically achieved. The surface backfill will be slightly mounded to provide positive drainage. Reseeding requirements will be specified in the site work plan or QA plan.

If clean sand and/or pea gravel is used as backfill for the excavation/trench compaction is not necessary.

If a clean, fine-grained soil (sand) is used for backfill in an excavation/trench, that is in native soil that has large, clean properties (coarse clean gravel), a filter fabric may have to be placed in the bottom of the excavation/trench, prior to backfilling operations to prevent soil migration, which would result in settlement.

Health and Safety Requirements: Backfilling should only be conducted in accordance with an approved site health and safety plan. General safety requirements are listed below.

- Personnel will stay clear of heavy equipment during operation
- The bucket of the excavator will be put on the ground while not in use
- All site personnel will wear specified personal protective equipment (PPE)/reflective vests
- Backup alarms will be required on all heavy equipment

Interferences and Potential Problems: Improper compaction may cause settlement of the soil, which may result in unnecessary maintenance costs where structures are present. Improper compaction can cause erosion problems.

BRISTOL ENVIRONMENTAL REMEDIATION SERVICES, LLC

Anchorage, Alaska

PHOTOIONIZATION DETECTOR

STANDARD OPERATING PROCEDURE BERS-16

Record of Changes

Revision No.	Date	Prepared by	Approved by
0	03/14/13	M. Faust	P. Yang

PHOTOIONIZATION DETECTOR

STANDARD OPERATING PROCEDURE

Summary: Photoionization detectors (PIDs) are devices used to screen for concentrations of volatile organic compound (VOC) vapors. The main components of the device include an UV lamp, a sensor detector, an ionization chamber, and a sampling pump. A PID works by drawing an air sample into the device with a sampling pump. The air sample is then bombarded with photons emitted by an UV lamp. The VOCs present in the air sample become ionized (positively charged) and produce an electric current. The PID then detects the current, and displays a numerical reading on a light-emitting diode (LED) screen. The numerical reading is proportionate to the magnitude of the current produced by the ionized VOCs. The greater the concentration of VOCs in the air sample, the greater the electrical current produced by photoionization, and the greater the reading displayed on the LED screen.

Photoionization detectors are used for a variety of applications in industries, including as safety devices to determine whether a work environment is safe. For environmental applications, PIDs are typically used to provide qualitative or semi-quantitative data about petroleum hydrocarbon contamination in soil. The following SOP is oriented towards environmental applications and provides information about field screening methods, calibration, and maintenance. This SOP is not necessarily model-specific, although much of the detail is oriented towards the MiniRAE Lite because this is Bristol's preferred PID model. If a different model is used, refer to your instrument's operating manual for model-specific procedures.

Health and Safety: Sampling activity should only be conducted in accordance with an approved Site Safety and Health Plan.

Personnel Qualifications: Sampling personnel will be trained and certified as hazardous site workers per Title 29 Code of Federal Regulations, Part 1910.120(e) [29 CFR 1910.120(e)]. If applicable, additional qualification requirements will be specified in the site Quality Assurance Project Plan and will be met.

Equipment and Materials: Prior to deployment in the field, the requisite PID, ancillary equipment, and materials will be identified, secured, and inspected for signs of damage or potential contamination.

- **PID –** This SOP is written using the MiniRAE Lite as the primary model, which is a product of RAE Systems, Inc.; however, most of the information is relevant to other PID units as well. **Note:** The PID should be fully charged, calibrated, and calibration checked prior to deployment in the field. Procedures for charging, calibration, and calibration checking are detailed below.
- **Calibration gas –** The standard calibration gas to be used is 100 parts per million (ppm) compressed isobutylene gas in a 34-liter compressed-gas steel cylinder. **Note:** Consider how the calibration gas will be transported to the job site. Shipping

- compressed gas requires special labeling and packaging procedures. Another option is to fill Tedlar bags with calibration gas and bring them into the field, which avoids the need to ship compressed gas.
- **Regulator –** Regulators are affixed to calibration gas cylinders to allow calibration gas to be released from the gas cylinder and drawn into the PID. Regulators come in two main varieties: fixed flow and on-demand. If the PID being calibrated utilizes a sampling pump, an on-demand regulator can be used and is preferred to economize the use of calibration gas. **Note:** Regulators can be male or female-threaded. Verify that the regulator matches the calibration gas cylinder prior to field deployment.
- **Tubing –** Tubing is used both during instrument calibration and during field screening activities. Tubing must be flexible, inert, and have the proper inner diameter to connect tightly to the PID inlet and moisture traps.
- **Moisture Traps –** Moisture traps are small plastic discs with inlet and outlet connectors and they contain 0.45-micrometer pore size filters. When attached with flexible tubing to the PID inlet, the traps can protect the PID sensor and lamp from moisture. Verify that the outlet connecter of the filter is attached to the PID inlet (using inert flexible tubing) prior to use. Spare moisture traps should be brought into the field, as they can wear out relatively quickly.
- **Battery and battery charger –** PIDs will typically be used with a rechargeable lithium ion battery. Spare lithium ion batteries and/or adapters for use with AA alkaline batteries may also be used. Lithium ion batteries may be charged using a charger stand or a travel charger. **Note:** Ensure that the AC/DC adaptor for the charger stand or travel charger is in the PID case prior to deployment to the field.
- **Maintenance kit –** A maintenance kit should be provided in the PID case, which includes: a bottle of methyl alcohol, latex finger cots, cotton swabs, and optical lens paper. The only maintenance that is typically performed in the field is cleaning the PID lamp. The cleaning procedures are presented below.
- **Manual –** A user's manual should always be included in the PID case brought into the field.
- **Calibration and Maintenance Log –** A fieldbook containing a calibration and maintenance log should be included in the PID case and should be updated each time the PID is calibrated and/or maintenance is performed.

Turning the Instrument On and Off: Procedures for powering on and off the specific PID model that is being used may be found in the user's manual for each model. Procedures for the MiniRAE Lite PID are as follows:

1. To turn on the PID, press and hold the middle button for approximately one second. The PID will go through the power-on procedures, including testing the sampling pump.

2. To turn off the PID, press and hold the middle button for approximately 7 seconds. The PID will beep repeatedly and the display will show a 5-second countdown as it powers off.

Charging the Battery: The PID must be charged prior to deployment in the field and at the end of each field day that it is used. Charging procedures for the specific PID model being used should be found in the user's manual for each model. For charging a MiniRAE Lite PID using a travel charger, procedures are as follows:

1. Leave the rechargeable lithium ion battery in the PID. Visually inspect the contacts on the bottom of the PID and on the travel charger. If they are dirty, wipe with a soft cloth (do not use solvents or other cleaners).

2. Plug the AC/DC adapter's round connector into the travel charger, and plug the adapter into an electrical outlet.

3. Attach the travel charger to the PID. This is accomplished by holding the travel charger firmly against the bottom of the PID in a position where the electrical contacts and attachment screws line up correctly, and then turn the attachment screws clockwise until they stop and the charger is firmly secured to the PID.

4. The PID will begin charging automatically. The green LED light on the travel charger will flash and the PID display screen will read "Charging…". When the PID is fully charged, the LED light will glow continuously and the PID display screen will read "Fully Charged".

Calibration Procedure: The required frequency with which calibration is performed may vary depending on the requirements of the job and the client. MiniRAE Lite PIDs can theoretically maintain a calibration for up to 30 days; however, most often they are recalibrated at the beginning of every field day. Refer to the Work Plan for the specific job to verify the calibration requirements.

Calibration is typically performed using the PID, a canister of 100-ppm compressed isobutylene gas, a regulator (either fixed flow or on-demand), and a short (approximately 6-inch) piece of inert flexible tubing that fits tightly on the regulator outlet and the PID inlet.

Standard procedure is for a 2-point calibration to be performed, including a fresh air/ zero-calibration and a span-gas calibration. Calibration procedures for the specific PID model that is being used may be found in the user's manual for each model. Calibration procedures for the MiniRAE Lite PID are as follows:

1. Turn the PID on. The display will show the home screen as "Ready… Start Sampling?". Press the middle button and the N/- button simultaneously for several seconds until the password screen is shown on the display. It is not standard

procedure to use a password for calibration, so you may bypass this prompt. Simply press the middle "Enter" button once to enter calibration mode.

2. First perform a zero calibration:

    a. Select "Zero Calibration" on the PID display. Press the Y/+ button ("Select").

    b. The next screen will display "Apply zero gas…". In a situation (for example, a site highly contaminated with petroleum) where the ambient air contains abundant fumes and it is impractical to move to an area of fresh air, it may be necessary to use a canister of compressed 0-ppm gas. For most situations, it is more practical to simply verify that the PID inlet is located in an area of relatively "fresh" air (for example, no obvious petroleum fumes from contaminated soil or vehicle traffic detectable to the human nose).

    c. Press the Y/+ ("Start") button and wait for the display screen to count down from 30 to 0. If performed correctly, the screen should display "Zero done! Reading = 0.0 ppm".

3. Next, perform a span-gas calibration:

    a. Select "Span Calibration" on the PID display. Press the Y/+ button ("Select"). The display screen should now indicate the type of calibration gas to be used (isobutylene) and the concentration of the gas (100 ppm).

    b. Attach the regulator (either an on-demand or 500 cubic centimeters (cc) per minute flow limiting regulator) to the 100-ppm compressed isobutylene gas canister. Connect the outlet of the regulator to the inlet of the PID using the short piece of inert flexible tubing. Press the Y/+ button ("Start") on the PID.

    c. The PID screen will then display a 30-second countdown. At the end of the 30 seconds, the screen will display the message "Span 1 is done!" and a concentration reading that should be very close (within 1.0 ppm) of the concentration of the span gas (100 ppm).

4. When the 2-point calibration is complete, press the middle button ("Back") and the screen will display "Updating settings". The display will return to the home screen within a few seconds.

5. Update the calibration log that is kept in the PID case with the date of calibration, zero calibration final reading, and span-gas final reading.

Calibration Check: A calibration check is sometimes referred to as a "bump test". On certain jobs, it may be acceptable to perform a calibration check at the beginning of each field day, rather than a calibration. Check the Work Plan to verify. Calibration checks may also be

conducted periodically throughout a field day to verify that the PID calibration has been maintained.

To perform a calibration check for a PID, apply calibration gas to a calibrated PID. Verify that the reading is within 10 percent (%) of the concentration of VOCs in the calibration gas. If the reading is outside of range, perform recalibration. If the inaccurate reading persists, the device may need to be taken in for maintenance prior to field use.

Lamp Selection: PIDs are outfitted with a UV lamp that breaks down VOCs into ions. Various lamps with different photon energies (measured in electron volts [eV]) are available for installation. To select the appropriate lamp for a particular job, it is important to know the ionization potential (IP) of the compound(s) targeted for detection. In order for the lamp to break down the compound so that it can be detected by the PID, the photon energy of the lamp must be greater than the IP of the target compound(s).

The most common PID lamps used have photon energies of 10.6 eV or 11.7 eV. A common application of a PID is for field screening soil samples for evidence of petroleum hydrocarbon contamination. In this application, a 10.6-eV lamp is appropriate, as most of the key contaminants of concern (COCs) associated with petroleum hydrocarbons (including benzene, toluene, ethylbenzene, and xylenes) have IPs that are below 10.6 eV. Lamps with higher photon energies (for example, 11.7 eV) should not be outfitted in a PID unless required to detect an important COC, as these lamps have shorter shelf lives and are less accurate for the relatively low IP COCs.

PID Field Screening: Field screening samples are typically collected either before or concurrently with laboratory analytical samples. Field screening before sample collection can help guide the selection of the most appropriate location from which to collect a laboratory analytical sample. Collecting field screening samples concurrently with laboratory samples can help establish a correlation between screening and analytical results at a particular site.

Two common methods of PID field screening are the headspace method and the in-situ method. Both are detailed below.

Headspace PID Screening: The headspace PID field screening method is the most accurate way to use a PID in the field and is typically required by the Work Plan for obtaining the highest quality field screening data.

1. For this method, field screening samples are collected by filling a resealable, ziptop, polyethylene bag approximately one-third to one-half full of freshly exposed or uncovered soil, and immediately sealing the bag. It is important that the soil be deposited into the bag as soon as practical after being exposed to ambient air, as the VOCs will immediately start volatizing, resulting in a low bias for the field screening result.

2. The bags should be agitated for approximately 15 seconds, and then allowed to warm for at least 10 minutes, but no more than one hour to allow the headspace vapors to develop. The soil must be warmed to at least 40º degrees Fahrenheit. The bags may be placed in a heated vehicle or simply in the sun if conditions are warm enough to achieve this temperature.

3. After the headspace vapors have developed, the bag should be agitated again for approximately 15 seconds. Insert the tip of a calibrated PID into the approximate midpoint of the void headspace of the bag, taking care to keep the opening as small as possible to minimize loss of VOCs. Measurement procedures for the specific PID model that is being used may be found in the user's manual. Procedures for the MiniRAE Lite PID are as follows:

    a. From the home screen (where the display reads: "Ready . . . Start sampling?"), press the Y/+ button ("Yes") to enter sampling mode. The sampling pump will engage and the display screen will show a concentration reading (initially should be close to 0.0 ppm).

    b. To prevent moisture or particles from being sucked into the PID and fouling the detector, a moisture trap is typically attached to the inlet of the PID using two short pieces of inert flexible tubing.

    c. Insert the tip of the flexible tubing into the headspace being measured and watch the PID display. If there are detectable compounds, the concentration reading will start climbing before eventually dropping again as these compounds are consumed by the ionizing lamp. Record the highest reading displayed (should occur within 5 to 10 seconds).

    d. If you are concerned that the highest reading was missed or forgotten, the PID records peak measurements. To view this value, press the N/- button ("→") twice to view the peak reading screen. Record this number, and then press the Y/+ button ("Clear"). Press the Y/+ button again ("Yes"), and then press the N/- ("→") three times to return to sampling mode. **Note:** It is important to clear the peak value because if the next peak value is lower, it will not be recorded.

In-Situ PID Screening: In some instances, where a limited volume of soil is available for field screening, such as soil cores from a direct-push rig or split spoon, using the headspace method may use up material that potentially could be used for a laboratory analytical sample. In these cases, field screening of the soil may be performed by making small divots approximately every 6 inches along the length of the core and inserting the calibrated PID tip just above the freshly exposed divot, taking care not to touch the material. Record the highest reading at each location in the field logbook or field form.

The in-situ method may be used by field personnel who want to quickly gather a large amount of qualitative field screening data in order to identify the zones with the highest level of contamination. Please note that VOC concentrations obtained using this method are typically lower and less accurate than concentrations obtained using the headspace field screening method. In-situ field screening results will not likely be accepted by regulatory agencies, and may need to be supplemented by targeted headspace field screening samples or laboratory analytical samples once the zones of contamination have been identified.

Interferences and Potential Problems: The most common PID problems that may occur in the field are detailed below, along with possible solutions:

Moisture Interference: Soil moisture may volatize during headspace development and present as moisture in the headspace. This moisture will commonly be ionized by the PID lamp and picked up by the detector, resulting in field screening results that are biased high. The best protection against moisture interference is to use a moisture trap. If you suspect a high bias as a result of moisture, record the concentration as displayed but make a note of the possible interference in the fieldbook. If a moisture trap is not used, it is possible for liquids to be sucked into the PID through the integrated porous metal filter and to damage the PID sensor and/or lamp. If this happens, the sensor and/or lamp may need to be cleaned or replaced.

Pump Failure: If the inlet port becomes blocked while the sampling pump is running, the PID will start beeping and the pump icon on the display will begin to flash. If this occurs, stop sampling (on a MiniRAE Lite, press the N/- button [“→”] and then the Y/+ button [“Yes”] when prompted to “Stop Sampling”) and ensure that the blockage has been cleared. The PID will have to be powered down, and then back on in order to reset the pump alarm.

Erratic Readings: If erratic readings occur, first recalibrate the instrument. If the problem persists, it may be that the sensor and/or lamp have become dirty or damaged. If in the field, try cleaning the lamp as described below under the maintenance section. Cleaning the sensor, or replacing the sensor or lamp, may be required but are not procedures that are typically undertaken in the field.

Inability to Zero between Measurements: It is commonly the case that the PID will not reach 0.0 ppm between field screening measurements. This may occur because of VOCs that have accumulated in the tubing or moisture trap. If the reading is relatively low (for example, below 1.0 ppm) it is usually fine to ignore it and consider it to be functionally zero. It may also be helpful to remove the tubing and moisture trap to allow them to air out for a few minutes. It is often necessary to replace the tubing and/or moisture trap if the elevated readings persist in fresh air. If the elevated readings persist after replacing the tubing and moisture trap, and you are relatively certain that the ambient air is clean, you may be able to solve the problem by repeating the zero point (fresh air) calibration procedure.

Maintenance: Only maintenance procedures that would typically be undertaken in the field are detailed here. Any procedures above and beyond those described here would typically be performed by in-house personnel in the controlled environment of the home office, or by outside instrument specialists. Anytime that a PID is dissembled, great care should be taken to avoid allowing dirt into the PID assembly. Make sure your hands and the work area are clean, and use the finger cots included in the maintenance kit on any finger that will come in contact with the inner workings of the PID. Do not disassemble outside if there is significant wind, to prevent grit from blowing into the inner workings of the PID.

Cleaning the Lamp Housing: For instructions specific to the PID that you are using, refer to the user's manual for each model. The following instructions are specific for the MiniRAE Lite PID: Gently unscrew the lamp housing cap, and remove the inlet probe/metal filter assembly. Pull the PID sensor straight out; some gentle rocking may be required. Clean the lamp window surface and lamp housing with methanol and cotton swabs. Hold the lamp up to the light at an angle to detect any remaining dirt. Repeat the wiping process until the lamp surface and housing are clean. Thoroughly dry the lamp housing and surface with optical lens paper. Reassemble the PID when it is completely dry.

Examining the PID Sensor: For instructions specific to the PID that you are using, refer to the user's manual for each model. The following instructions are specific for the MiniRAE Lite PID: Gently unscrew the lamp housing cap, and remove the inlet probe/metal filter assembly. Pull the PID sensor straight out; some gentle rocking may be required. Turn over the sensor unit to view the sensor electrodes. The electrodes should be flat and straight with no evidence of corrosion. If the sensor has become corroded or otherwise damaged, it will need to be replaced. If the sensor electrodes appear to be out of alignment, <u>gently</u> bend them until they are in alignment and do not touch the sensor housing. Reassemble the PID.

Clearing the Metal Filter: If it seems that the porous metal filter located within the sensor cover assembly has become clogged, possibly causing the sampling pump to stall and resulting in a pump failure, it may be possible to clear the filter. This will require a canister of compressed air (electronics duster) that is not typically included in the PID maintenance kit. Remove the inlet probe assembly and sensor cover assembly from the PID. Aim the compressed air at the <u>inner</u> side of the filter, and blow the compressed gas <u>backwards</u> through the filter. Replace the two assemblies and restart the sampling pump to see whether flow rates have improved.

Quality Assurance Review Checklist

Date Submitted: _____

Date Report Due: _____

Job#: _____ Name: _____ Project Manager: _____

Report Type: _____ Submitted by: _____

	Type of Review	**Date Due**	**Authorized Reviewers**	**Approved for Release to Client**
				Initial and Date
1	****Conformance to Federal and State Regulations and Statutes (as applicable)**		Bristol Senior Project Manager (Review must occur at the Planning Stage and Final Submittal)	
2	****Conformance to Statement of Work** (SOW must accompany)		Director of Operations, or Program Manager or Senior Manager	
3	Editorial Review		Technical Editor	
4	****Health and Safety Plan, OSHA Requirements, Special Site Conditions and/or Hazards**		Bristol Health and Safety Manager and CIH/CHSP for Special Site Conditions and/or Hazards	
5	Analytical Review and/or Data Verification		Environmental Chemist	
6	Quality Review		QA Manager or Independent Senior Manager	
7	****Approval for Release**		Director of Operations or Program Manager	
8	Final QC Prior to Release		Program Manager	

Comments:

**Required for all Submittals

APPENDIX E

All LUST Sites Covered by this QAPP

APPENDIX E.1

Western Gas LUST Site
Yakama Indian Reservation
Toppenish, Washington

TABLE OF CONTENTS

SECTION **PAGE**

1.0 SITE-SPECIFIC INFORMATION 1
1.1 Site-Specific Description 1
1.2 Site-Specific History 1
2.0 PROGRAM MANAGEMENT 7
2.1 A5 – Site-Specific Problem Definition/Background 7
2.2 A6 – Site-Specific Project/Task Description and Schedule 8
2.2.1 ACTIVITY – SITE ASSESSMENT 9
2.3 A7 – Site-Specific Quality Objectives and Criteria 14
2.3.1 Program Quality Objectives 14
2.3.2 Program Objectives 14
3.0 SITE-SPECIFIC DATA GENERATION AND ACQUISITION 17
3.1 B1 – Sampling Process Design 17
3.2 B2 – Sampling Methods 19
3.3 B3 – SAMPLE HANDLING AND CUSTODY 19
3.4 B4 – ANALYTICAL METHODS 20

TABLES

Table E.1-1 MTCA A Cleanup Levels for Groundwater 9
Table E.1-2 Groundwater Analytical Requirements 12
Table E.1-3 Action Levels for COCs and analytical results 15
Table E.1-4 Field Parameters for Groundwater 18
Table E.1-5 Sample Identification System 20

FIGURES

Figure E.1-1 Site Location Map
Figure E.1-2 Site Map
Figure E.1-3 Treatment Area Map

ATTACHMENT

Attachment E.1 Western Gas Corrective Action Plan (CAP)

ACRONYMS AND ABBREVIATIONS

AST	aboveground storage tank
bgs	below ground surface
BTEX	benzene, toluene, ethylbenzene, and xylene
CAP	Corrective Action Plan
EPA	U.S. Environmental Protection Agency
MTCA	Model Toxics Control Act
PCS	petroleum-contaminated soil
RegenOx	chemical oxidizer
TPH-diesel	Total petroleum hydrocarbons - diesel range
TPH-gas	Total petroleum hydrocarbons - gasoline range
UST	underground storage tank

1.0 SITE-SPECIFIC INFORMATION

1.1 SITE-SPECIFIC DESCRIPTION

The Western Gas LUST site, U.S. Environmental Protection Agency (EPA) Facility No. 4260105, is located within the Yakama Indian Reservation at 401 South Elm Street in Toppenish, Washington (Figure E.1-1). This site has previously been known as Kuki Mart, Toppenish Shell, and Brand X. The site encompasses 0.9 acre, and is bound by 1st Avenue to the north, a Subway restaurant and Advance America Cash Advance to the south, Elm Street to the east, and El Gallo Giro Grocery and Taco Shop to the west. The site supports a concrete pad, a gas station island with canopy and Western Gas (Figure E.1-2). The majority of the site is paved. The property is currently owned by Mr. Praveen Dewan of P.R. Distributors, LLC.

1.2 SITE-SPECIFIC HISTORY

The site history is based on information provided by the EPA and is contained in the *Western Gas Corrective Action Plan* (CAP) (Attachment E.1).

The property has been on EPA Region 10's active LUST list since 1991 when a release was first documented. White Shield, who performed a site assessment for another party, initially notified the EPA of contamination in May 1991. From the early 1950s through 1991, the property operated as a bulk plant for petroleum product distribution, and it hosted a minimart that retailed petroleum products. The minimart had an attached carwash.

The western side of the property operated as the bulk plant and had five aboveground storage tanks (ASTs) and two 1,500-gallon abandoned underground storage tanks (USTs). The eastern portion of the site was occupied by a small mini-mart with an attached carwash to the south. Two 6,000-gallon USTs north of the minimart contained regular and

unleaded gasoline, while two 4,000-gallon USTs east of the carwash contained regular and super unleaded gasoline.

Retail dispensers were north and east of the building, and a diesel dispenser associated with the ASTs was located in the central portion of the site.

Groundwater is located approximately 13 feet below ground surface (bgs), with seasonal variations. In general, groundwater flows to the southeast. There is a municipal water supply well approximately 1,000 feet from and to the east-southeast of the site. Site soils consist of clayey silt mixed with sand and gravel to a depth of 7–10 feet bgs and silty, sandy, gravel with cobbles below this depth. Three adjacent properties operate or had onsite fuel USTs.

During the 1991 site assessment, three test pits were dug downgradient of the USTs in areas most likely to contain petroleum contamination. Petroleum above Model Toxics Control Act (MTCA) cleanup levels was found in two test pits, and "high" levels of petroleum contaminants were reported in soil at 7 feet bgs in the third test pit, on the eastern portion of the site along Elm Street. Because of concerns about petroleum-contaminant migration, two additional test pits were dug at the Serv Um Self, immediately east of the site and across Elm Street. Analytical soil samples were not collected from the two offsite test pits, but groundwater samples were collected from all five of the test pits. One of the groundwater samples contained diesel and lead at concentrations that exceed MTCA cleanup levels; no detectable concentrations of contaminants were reported for the other groundwater samples. The 1991 *Site Assessment Repor*t recommended removing the unused USTs and cleaning the contaminated media.

A soil-gas and water survey report was prepared in April 1994 for the Indian Lands Resource Conservation and Recovery Act site at Toppenish by Lockheed Environmental Systems and Technology Company. Benzene, toluene, ethylbenzene, and xylenes (BTEX)

were the target analytes for soil-gas, soil, and water samples, while diesel was only analyzed for the soil and water samples. Samples collected from three locations along the eastern and southeastern side of the Western Gas site contained BTEX in groundwater but not in soil. Diesel range petroleum hydrocarbons were not detected.

The EPA performed a field inspection at the site in July 1994; they measured the depth to the bottom of the six USTs and the product within the USTs. There was 0.625 inches of product in one of the two 6,000-gallon USTs, the other UST was dry; the two 1,500-gallon USTs contained 3.75 and 3.25 inches of product, respectively, and the two 4,000-gallon USTs contained only oily water.

In 1995, the USTs were removed and Technico Environmental Services, Inc. initiated remedial efforts. They removed four USTs from three excavations. Two of the tank basins contained petroleum-contaminated soil (PCS), while the third tank basin did not. Diesel contamination above MTCA cleanup levels was also reported at the soil-water interface at the diesel dispenser associated with the ASTs, located in the central portion of the site. All PCS had been removed from one of the tank basins but there was still contaminated soil along the south and east of the tank basin adjacent to Elm Street (by existing MW-2). No further excavation to the south and east could take place at this tank basin because it would endanger Elm Street. The vertical extent of this excavation was to 20 feet bgs with the majority of the contamination located in the smear zone, 16–18 feet bgs. Approximately 300 cubic yards of PCS was removed from the site.

During the 1995 removal action, three monitoring wells were installed to evaluate groundwater contamination. Monitoring Well 1 (MW-1) was placed north and slightly east of the diesel dispenser and upgradient of the tank basin adjacent to Elm Street, which contained residual contamination; MW-2 was placed along the southern edge of the site, west of the contaminated tank basin; and MW-3 was placed against the southeastern

sidewall in the contaminated smear zone, near the tank basin adjacent to Elm Street. Gasoline and diesel were not detected in any of the three wells, while BTEX concentrations exceeded MTCA cleanup levels in MW3. The wells were not tested for lead.

In 1996, PLSA Engineering & Surveying performed a site assessment and cleanup so that the site could be developed for new construction (the Maid O'Clover Service Station and convenience store). Onsite structures were demolished prior to the removal of PCS. During removal activities, two additional USTs were encountered and removed. One was located in the northeast portion of the site; the location of the other UST, a waste oil tank, was not reported. Both had been previously decommissioned. Approximately 300 cubic yards of PCS was removed and disposed of off-site. PLSA's detailed their activities in the *Site Assessment RH Bowles LUST Site* report, which concluded that gasoline and BTEX contamination sources were removed to the extent practicable and groundwater quality was expected to improve in the near future. There was no evidence that the petroleum contamination had impacted neighboring properties, and since the site was to be covered by the new structures and pavement, the risk was minimal. An addendum to the report, issued in March 1997, states that there are three monitoring wells on site, including one in the fuel dispensing island (MW-4), indicating that MW-1 was no longer there. Also, the addendum stated that the PCS originating from the waste oil UST had been characterized for offsite disposal, but soil sampling had not been performed at the location of the former waste oil tank.

The monitoring well designations changed during the course of the above referenced investigations. After the construction of the Maid O'Clover Service Station and convenience store, the numbering of the wells changed (original MW-1 was destroyed). Present day MW-1 is the same as MW-2 referenced in pre-1996 reports, and present day

MW-2 is the same as MW-3 referenced in older reports. MW-3 (formerly called MW-4) was located across Elm Street to the east.

In October 2012, the EPA identified four, 4-inch PVC monitoring wells: MW-1, MW-2, and the two release detection wells associated with the active station. No other onsite wells were located by the EPA during the site visit; in November 2012 Bristol confirmed that the four wells identified by the EPA were the only the wells on site.

A site assessment to document the current status of the petroleum contamination was performed by Bristol in November 2012. Tasks accomplished include the installation of six GeoProbe borings using direct push technology, field screening the soil cores, collecting two soil samples from each boring, redeveloped the two existing groundwater monitoring wells on site, installation of three temporary monitoring wells in three of the six Geoprobe borings, and collecting groundwater samples from the two onsite monitoring wells, the southern- most release detection well associated with the active station, and three temporary monitoring wells placed in selected borings. Results indicated that petroleum contamination concentrations had naturally attenuated since the last site investigation; however, petroleum contaminants above MTCA A cleanup levels were still present in groundwater. The analytical laboratory case narrative from the groundwater sample collected from MW-2 in November 2012 stated that the results in the C10-C24 range(s) are due to what most closely resembles a complex mixture of weathered gasoline/kerosene product.

Bristol planned and executed the CAP which consisted of two in-situ groundwater remediation events (September 2013 and March 2014) in which a chemical oxidizer (RegenOx) was injected upgradient of the compliance well (MW-2) into the subsurface through six direct-push borings. The EPA collected groundwater samples after the first injection and despite the cleanup efforts conducted to date the concentration of gasoline

range hydrocarbons in the site compliance well MW-2 is still above MTCA Method A cleanup levels. Attainment of Method A cleanup levels at the point of compliance, represented by MW-2, is necessary to achieve clean closure at the site.

2.0 PROGRAM MANAGEMENT

2.1 A5 – SITE-SPECIFIC PROBLEM DEFINITION/BACKGROUND

The Western Gas site has petroleum contamination, specifically weathered gasoline range organics in groundwater above MTCA A cleanup levels. Groundwater monitoring is conducted at the onsite compliance well, which will be sampled and monitored until the analytical results show that the petroleum hydrocarbon concentrations are below MTCA A cleanup levels.

Monitoring well MW-2 was designated as the compliance well for the site, but it is in poor condition and needs to be replaced. Monitoring well MW-1 is no longer used. Both monitoring wells will be decommissioned

A replacement compliance well, designated MW-2B, will be installed approximately 10 feet away from MW-2. As specified by EPA, the top of casing for the new well will not be surveyed as the groundwater flow direction in Toppenish in known to flow to the southeast. Groundwater samples will be collected from the well and analyzed for TPH-gas and TPH-diesel. The EPA will determine the frequency of groundwater sample collection and analysis. The results will be compared to the MTCA A groundwater cleanup levels and will be used to evaluate current contaminant concentrations.

The site is undergoing the remedial treatment detailed in the 2013 CAP: chemical oxidation and enhanced bioremediation through oxidant injection into the subsurface at or around the groundwater-soil interface. The EPA and Bristol chose the approach of in-situ treatment through chemical oxidation and enhanced bioremediation. Regenesis provided technical assistance with design of the injection parameters based on site-specific analytical data, soil lithology types, and hydrogeology.

The Regenesis remediation product that was selected for use during the first stage of treatment is called RegenOx, which is a remediation product that directly destroys contaminants through oxidation reactions. ORC-Advanced, another remediation product that will be used at the site, accelerates naturally occurring aerobic bioremediation by supplying oxygen to the contaminated media.

In-situ remedial treatment was performed in September 2013 and in March 2014 with RegenOx. Regenesis recommended a combination of RegenOx and ORC-Advanced for this round of treatment. Using both products will give very fast results from chemical oxidation (RegenOx), along with a lingering oxygen source (ORC-Advanced) to help stimulate bioremediation for approximately 12 months. The treatment material is applied through injection into the subsurface hydrocarbon-impacted zone through direct-push borings, and is injected upgradient of the compliance well. Six injection points are used for each application of the chemical oxidant.

2.2 A6 – Site-Specific Project/Task Description and Schedule

Project tasks include site assessment and remediation. For the site assessment portion Bristol will decommission two onsite monitoring wells, install and develop one replacement well, sample and analyze groundwater, and remediate through the application of a chemical oxidant into the petroleum hydrocarbon impacted zone.

MTCA A groundwater cleanup levels are listed in Table E.1-1.

Table E.1-1 MTCA A Cleanup Levels for Groundwater

Analyte	NWTPH Method	MTCA A Cleanup Level (mg/L)
TPH-gas[1]	Gx	1.0
TPH-diesel	Dx extended	0.5

Notes:
Bristol will use TestAmerica in Tacoma, Washington as the analytical laboratory.
[1]This represents the cleanup level when benzene is not present in the groundwater.
EPA = U.S. Environmental Protection Agency
mg/L = milligram per liter
MTCA = Washington Department of Ecology Model Toxics Control Action
NWTPH = Northwest Total Petroleum Hydrocarbon
TPH-diesel = total petroleum hydrocarbons – diesel range
TPH-gas = total petroleum hydrocarbons – gasoline range

The fieldwork is anticipated to take approximately five calendar days. Bristol will mobilize to the site in August 2014:

Date	Activity
August 18 – 22, 2014	Mobilize and conduct field activities.
October 17, 2014 (or within 60 calendar days after completion of field work)	Submit draft Technical Memorandum for EPA review.
15 work days after receipt of SME's comments on the draft Summary Report	Submit final Technical Memorandum to EPA, Yakama Nation (YN) Environmental Management Program, YN Water Code Administration, and Praveen Dewan (property owner).

Notes:
EPA = U.S. Environmental Protection Agency
YN = Yakama Nation

2.2.1 ACTIVITY – SITE ASSESSMENT

Bristol will facilitate access to the property, apply for and obtain all required permits, and locate underground utilities prior to beginning site work. Required permits include three from the Yakama Nation Water Code Administration, specifically a well decommissioning permit, a well installation permit, and an interim hydraulic permit for the injection work. The first two permits are for one-time use, while the hydraulic permit is good for a year. After completing site activities and prior to demobilizing, Bristol will return the asphalt lot of the Western Gas station to its previous condition.

2.2.1.1 Monitoring Well Decommissioning

The two 4-inch diameter monitoring wells currently onsite, MW-1 and MW-2, are old and in poor condition, and the EPA believes these wells are no longer capable of providing representative groundwater samples. Bristol will abandon the wells in accordance with Washington State rules titled Minimum Standards for Construction and Maintenance of Wells (WAC 173-160) and SOP BERS-07 (included in Appendix D). Both wells are flush mounted and approximately 16 feet deep.

2.2.1.2 Monitoring Well Installation

One replacement well will be installed within 10 feet of the existing compliance well MW-2 (Figure E.1-3). The exact location will be determined in the field by the EPA SME. The new monitoring well will serve as a point of compliance for future site closure decisions and will be identified as MW-2B.

The replacement monitoring well MW-2B will be 2-inch diameter, schedule-40 PVC, flush mounted, and installed to a total depth of 19 feet, with a 10 foot 2-inch pre-packed screen screened interval from 8 to 18 feet bgs. The new well will be drilled, installed, and developed in accordance with Washington State standards described in WAC 173-160. Field screening will not be conducted during drilling, no analytical samples will be collected, and the boring will not be logged.

A bentonite pellet or chip seal will be placed above the sand to 2 feet above the filter sand and will be approximately 2 feet thick. Granular bentonite will be used to seal the well to near ground surface. The groundwater monitoring well will be completed flush with the ground surface and will have a lockable cap. The flush mount groundwater monitoring well will be completed with a 2 x 2 x 4-inch thick cement pad. Additional well installation methodology is described in SOP BERS-07 (included in Appendix D).

2.2.1.3 Well Development

Bristol will develop the new well within 24 hours of installation, in accordance with SOP BERS-07. While conventional practice is to wait 24 hours to develop and sample a well, in this case granular bentonite will be used to seal the well instead of grout. The granular will hydrate in an hour or so and will not be pulled down into filter pack like grout. Well development will consist of routinely surging and purging the well until relatively clear water is produced and water quality parameters, as measured with the YSI and turbidimeter, have stabilized. Total depth of the well and depth to water will be measured before and after well development. Well sampling may take place immediately after well development, or the following day.

2.2.1.4 Groundwater Sampling and Analysis

Groundwater samples will be collected from replacement well MW-2B using the low-flow technique of Puls and Barcelona (EPA, 1996), as described in SOP BERS-02. Field parameters will be measured using both a YSI meter and a turbidimeter. Field parameters will include pH, dissolved oxygen, conductivity, temperature, and oxidation-reduction potential. Groundwater samples will be collected when these parameters stabilize. Groundwater samples will be analyzed for TPH-gas, and TPH-diesel. Analytical methods, containers, sample preservation, and holding time requirements are described in Table E.1-2.

Table E.1-2 Groundwater Analytical Requirements

Contaminants of Concern	Analytical Method	Holding Time	Preservative	Sample Size and Container	QC Samples1
TPH-gas	NWTPH-Gx	14 days	HCl, cool 2-6°C	Three 40-mL glass VOA vials with teflon septum and screw top lids	4
TPH-diesel	NWTPH-Dx extended	14 days to extract, 40 days to analyze	HCl, cool 2-6°C	250 mL amber glass	3

Notes:

[1]QC will include one field duplicate at a rate of 1:10, an MS/MSD pair at a rate of 1:20 for environmental samples of the same matrix, and a trip blank to be included with coolers containing volatile samples.

°C = degrees Celsius
EPA = U.S. Environmental Protection Agency
HCl = hydrochloric acid
mL = milliliter
NWTPH = Northwest Total Petroleum Hydrocarbon
TPH = total petroleum hydrocarbon
VOA = volatile organic analysis

2.2.1.5 Remedial Treatment

The treatment area is defined in the CAP as a 30- by 15-foot rectangle, located between the former soil excavation and Elm Street. Regenesis recommended a combination of RegenOx and ORC-Advanced for this round of treatment. Using both products will give very fast results from chemical oxidation (RegenOx) along with a lingering oxygen source (ORC-Advanced) to help stimulate bioremediation for approximately 12 months.

As described in the 2013 CAP, six injection points will be used to introduce the chemical solution into the treatment zone, which the EPA specified is a 6 foot interval, 12 to 18 feet bgs for this round of treatment.

Based on the six application points and a 6-foot treatment zone, Regenesis recommended 640 pounds of RegenOx (400 pounds of RegenOx Part A, 240 pounds of RegenOx Part B) and 240 pounds of ORC-Advanced. To create the treatment solution, 911 gallons of water are added to the 400 pounds of RegenOx Part A to form a solution with 5% solids content.

The 240 pounds of Part B is added to the Part A solution. Once RegenOx Part A and Part B have been mixed together, the ORC-Advanced is mixed into the solution.

One third of the RegenOx-ORC Advanced solution designated for that injection point will be injected into each of three depths within the treatment interval. At each injection point, the pre-probe with an expendable tip will be initially driven to the bottom of the treatment interval (18 feet bgs) using standard 1-inch diameter drive rods, then the expendable tip will be disconnected.

One-half of the solution needed for an injection point will be mixed in a 55-gallon drum outfitted with a valve at the bottom. The drum will be staged on a platform so that the valve is located above the injection pump, allowing it to be gravity-fed into the pump's hopper. For each batch of solution, a premeasured volume of water will be added to the drum, followed by a premeasured amount of RegenOx Part A; the solution will be mixed until the oxidizer is dissolved. A premeasured amount of RegenOx Part B will then be added and the solution mixed for at least 5 minutes. Finally, the ORC-Advanced will be added and the solution well mixed. The mixed solution will be gravity fed into the injection pump hopper. The hopper has a maximum volume of 10 gallons, so each batch of the treatment reagents will fill hopper twice.

Once the hopper is prepared, an injection manifold (outfitted with a pressure gauge and a release valve) will be connected to the top of the drive rods, and the injection pump connected to the injection manifold with a hose. The RegenOx-ORC Advanced solution will then be injected into the treatment interval with the bottom of the pre-probe at three different depths, to be determined in the field. At each depth, one third of the chemical solution prepared for that injection point will be injected. When the injection is complete, the pre-probe and drive rods will be removed and the open borehole will be slowly filled with bentonite pellets to within 0.5 feet of the ground surface.

2.3 A7 – Site-Specific Quality Objectives and Criteria

2.3.1 Program Quality Objectives

The overall program quality objective is to develop and implement sampling, sample handling, and field measurement procedures that will provide data that are of the quantity and quality required for good decision making.

2.3.2 Program Objectives

The program objectives are to collect field information and samples from the replacement groundwater monitoring well MW-2B for laboratory analysis. Groundwater analytical and field data will be used to determine the current concentrations of petroleum contamination and attenuation parameters, such as dissolved oxygen, in the replacement compliance well MW-2B after performing the injection of RegenOx-ORC Advanced treatment solution.

Groundwater samples will be tested for TPH-gas and TPH-diesel. Per the analytical laboratory case narrative for sample collected in November 2012, the results in the C10-C24 range are due to what most closely resembles a complex mixture of weathered gasoline/kerosene product.

Action levels for the COCs and analytical results pre- and post-treatment are presented in Table E.1-3. As shown below, the analytical results for compliance well MW-2 from samples collected pre-treatment (November 2012) and post-remedial treatment. Samples were collected in February 2014 after the first injection of RegenOx.

Table E.1-3 Action Levels for COCs and analytical results

COC	MTCA A Cleanup Level	Unit	Pre-treatment (November 2012)	Post-treatment (February 2014)
TPH-gas	0.8 [a] (1.0)	mg/L	2.9	4.1
TPH-diesel	0.5	mg/L	1.2	1.4

Notes:

[a]The cleanup level is based on protection of groundwater for noncarcinogenic effects during drinking water use. Two cleanup levels are provided.

COC = contaminant of concern

mg/L = milligrams per liter

MTCA = Model Toxics Control Act

(Intentionally blank)

3.0 SITE-SPECIFIC DATA GENERATION AND ACQUISITION

Standard operating procedures for field environmental measurement and sample collection are presented in Appendix D. The procedures are described in detail to ensure that the data are representative of environmental conditions.

Bristol received permission to conduct work on the Western Gas property from the property owner via email on June 16, 2014. Bristol will coordinate with the property owner upon arrival onsite to establish the work zone. Since a portion of the work zone is in the driveway into the gas station, Bristol will coordinate with the property owner to ensure the customers have access to the active station while the site assessment and remediation work is being conducted.

3.1 B1 – SAMPLING PROCESS DESIGN

Bristol will install the replacement groundwater monitoring well, MW-2B, to determine the current concentrations of gasoline and diesel remaining in the groundwater at the point of compliance. Samples and field data will be collected from the replacement groundwater monitoring well prior to injecting the treatment solution.

The locations of the existing monitoring wells are shown on Figure E.1-2. The replacement well will be installed within 10 feet of existing MW-2; the exact location will be determined in the field by the EPA SME.

Groundwater sample analytical requirements are listed in Table E.1-2. Field parameters and instruments that will be used for groundwater monitoring and sampling are listed in Table E.1-4. Field forms and equipment manuals are included in Appendix C.

All information collected from the site is deemed critical information. As this site has been in an active cleanup status with the EPA for a number of years, no data will be collected for “informational purposes” only.

Sources of variability among groundwater sample results may include variability in the groundwater elevation (within or below the smear zone) since the water table in Toppenish fluctuates seasonally when irrigation of the agricultural fields begins in late spring, and the fact the new compliance well MW-2B will be located a few feet from the location of the original compliance well MW-2. All other variables should be within reasonable control as Bristol will use the same drilling/injection subcontractor, the same field technician, the same analytical laboratory, methods, and sampling techniques as were previously used by Bristol at the site. The SOPs and analytical methods will be the same that have been used for samples collected from this site in the past by Bristol.

Table E.1-4 Field Parameters for Groundwater

Field Parameters	Units	Instrument
Temperature	°C	YSI 556 Multi-Parameter Meter
Dissolved Oxygen	mg/L	
Specific Conductivity	µS/cm	
pH	--	
Oxidation-Reduction Potential	mV	
Water Level	Feet	Solinst Interface Probe Model 122

Notes:
°C = degrees Celsius
µS/cm = microsiemens per centimeter
mL = milligrams per liter
mV = millivolts

3.2 B2 – SAMPLING METHODS

Bristol will collect a groundwater sample from replacement groundwater monitoring well MW-2B using the low-flow purging and sampling procedures detailed in SOP BERS-02 (Appendix D).

- Before purging and sampling, the well will be checked for the presence of free-product, and then the depth to groundwater from a mark on the top of casing will be established with a water level sounder to an accuracy of 0.01 foot.
- During purging, parameters will be measured with a YSI water quality instrument and a tubidimeter. Parameters to be measured and recorded include pH, dissolved oxygen, conductivity, temperature, oxidation-reduction potential, and turbidity.
- Once parameters stabilize, samples for laboratory analysis will be collected.
- Personnel collecting the samples will wear new, disposable nitrile gloves for each sample.
- Water samples will be collected using precleaned containers provided by the laboratory.

Potential COCs, analytical methods, and sample container requirements are summarized in Table E.1-3. Water samples to be analyzed for VOCs will be containerized before all other water samples.

3.3 B3 – SAMPLE HANDLING AND CUSTODY

Laboratory sample requirements, which include the sample containers, preservatives, and technical hold times, as well as the frequency for sample QC requirements are listed in Table E.1-2.

Samples will be identified using the system described in Table E.1-5.

Sample handling and custody procedures are described in SOP BERS-03 (Appendix D), and in the TestAmerica QAM (Appendix A). Additional information is included in Section 3.3 of the QAPP.

Table E.1-5 Sample Identification System

Sample ID Component	Example	Note
Project Identifier	WG	Western Gas LUST Site
Sample location Identifier	MW-2B	Monitoring Well
Date	YYYYMMDD	-

Note:
A groundwater sample collected on August 15, 2014 from MW-2B at the Western Gas LUST Site would have the identifier of WG-MW-2B-20140815.

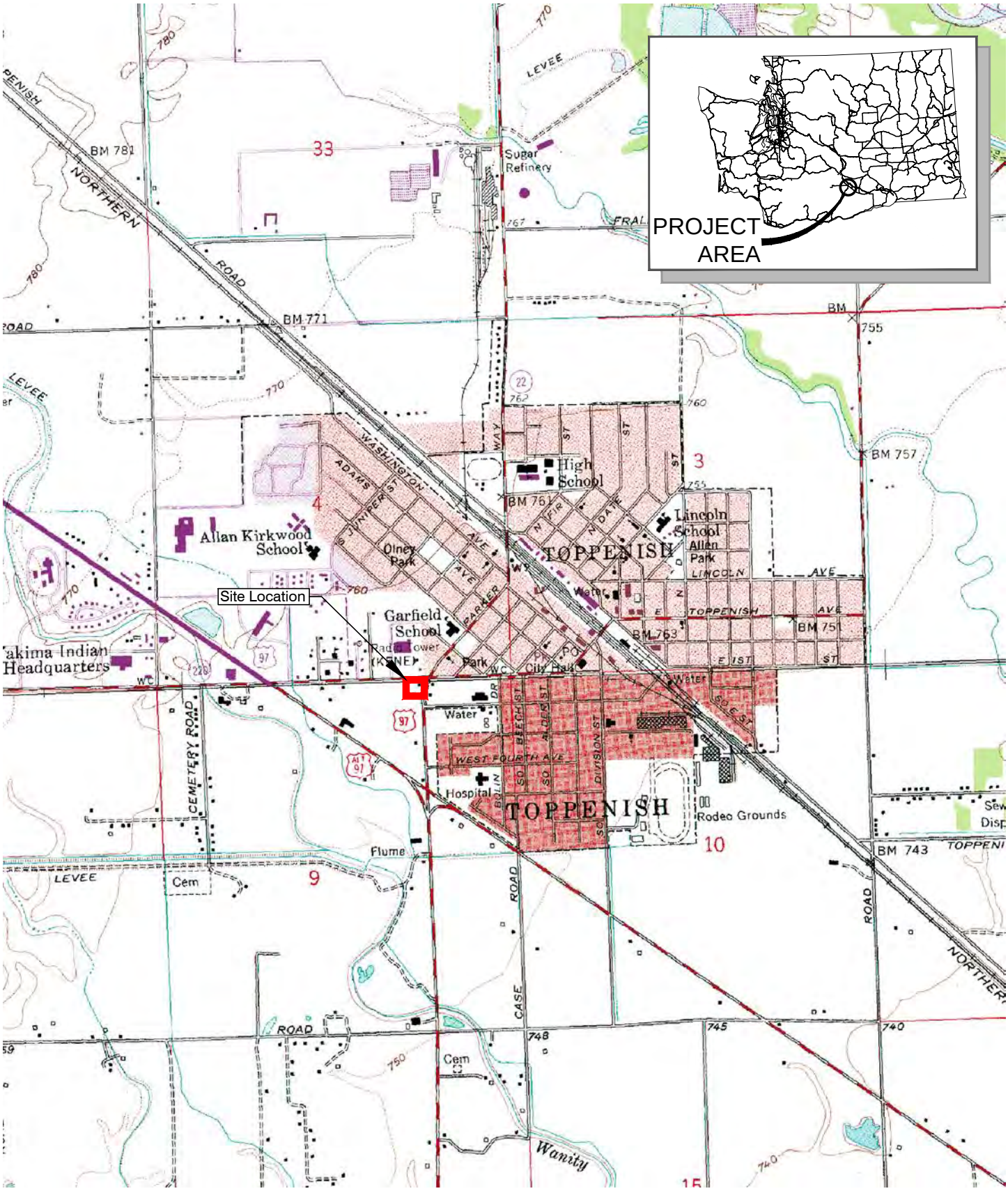
3.4 B4 – ANALYTICAL METHODS

Analytical methods, containers, sample preservation, and holding time requirements are described in Table E.1-2 (Groundwater Analytical Requirements).

Table E.1-4 shows the field parameters to be measured. Procedures for measuring the field parameters are included in SOP BERS-04 (Appendix C).

The operations and maintenance manuals for the YSI 556 Multi-Parameter Meter and the Solinst Interface Probe Model 122 are included in Appendix C. Copies of these manuals are maintained in the Bristol Anchorage, Alaska office and will also accompany the instruments into the field.

FIGURES



Topo Reference
USGS

N

0 1000 2000
1" = 2000 Feet

FIGURE E.1-1
EPA 1006 WESTERN GAS
401 S. ELM ST.
TOPPENISH, WA 98948
SITE LOCATION MAP

Bristol
ENVIRONMENTAL REMEDIATION SERVICES, LLC
Phone (907) 563-0013 Fax (907) 563-6713

DATUM:	NAD27
PROJECTION:	UTM WA Z10 M
PROJECT NO.	34150026
DATE	06/13/2014
DWN.	NAP
SCALE	1" = 2000'
APPRVD.	JSD

Drawing: O:\JOBS\34150026 EPA 1006 WESTGAS\ACAD-ENVIRO\DWGS\QAPP-FIGUREE.1-1.DWG - Layout: 8.5X11
User: NPEACOCK Jun 13, 2014 - 10:45am Xrefs: - Images: 34422193.TIF

Drawing: O:\JOBS\34150026 EPA 1006 WESTGAS\ACAD-ENVIRO\DWGS\QAPP-FIGUREE.1-2.DWG - Layout: 8.5X11
User: SLEE Jun 25, 2014 - 9:01am Xrefs: - Images: GOOGLE-IMAGE.TIF POTENTIAL SAMPLE PLAN.JPG

Legend

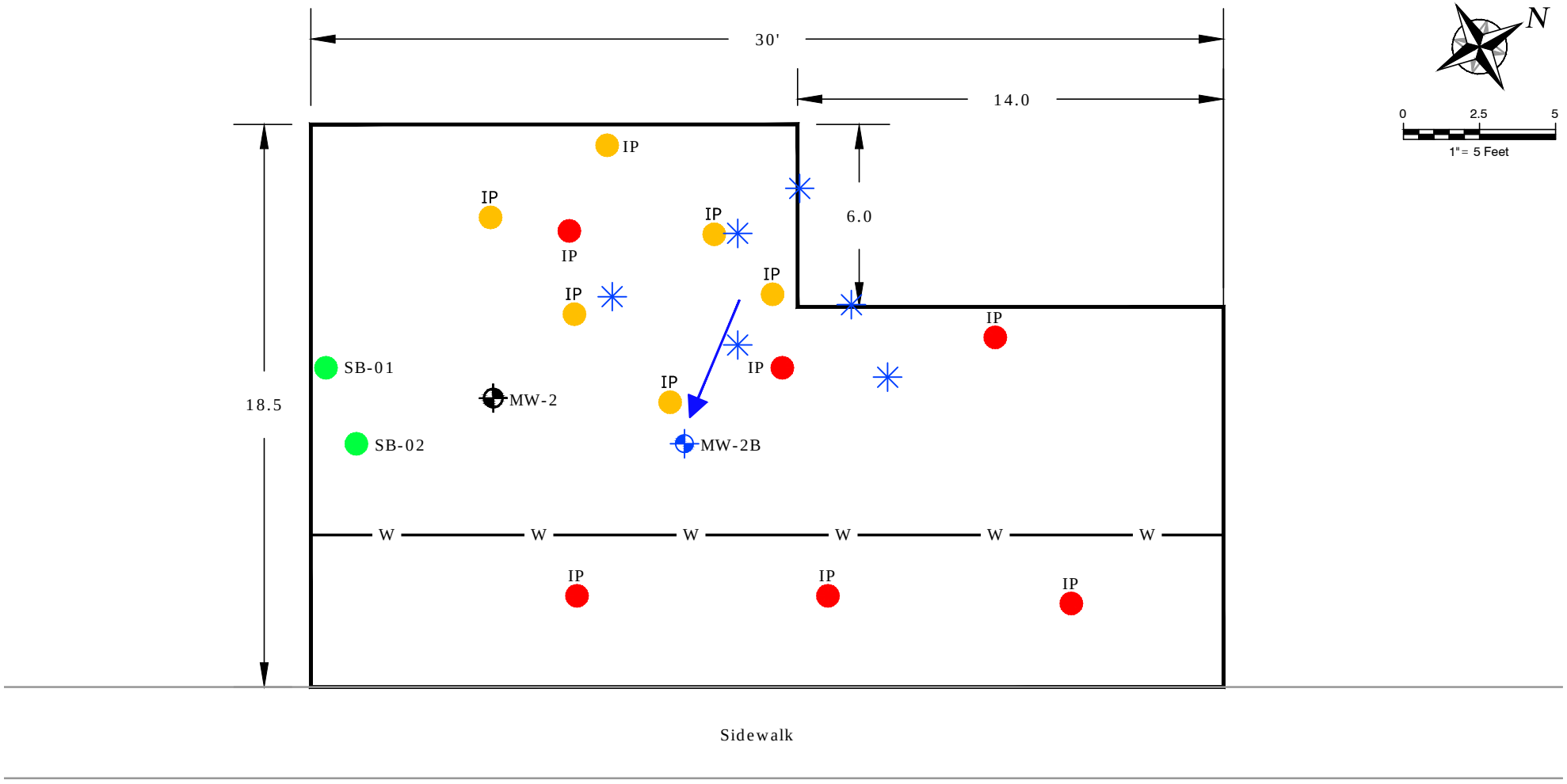
- EXISTING MONITORING WELL
- FORMER MONITORING WELL
- RELEASE DETECTION WELL
- WELL REFERENCE POINT
- GROUNDWATER FLOW DIRECTION
- BH BOREHOLE
- RW RELEASE DETECTION HOLE
- TW TEMPORARY HOLE

FIGURE E.1-2
EPA 1006 WESTERN GAS
401 S. ELM ST.
TOPPENISH, WA 98948
SITE MAP

Bristol
ENVIRONMENTAL REMEDIATION SERVICES, LLC
Phone (907) 563-0013 Fax (907) 563-6713

DATUM:	NAD 83
PROJECTION:	WA SP ZS FT
PROJECT NO.	34150026
DATE	6/25/2014
DWN.	STL
SCALE	SHOWN
APPRVD.	JSD

Drawing: \\BEESC-JOBS\BERS\JOBS\34150026 EPA 1006 WESTGAS\ACAD-ENVIRO\DWGS\QAPP-FIGUREE.1-3.DWG - Layout: 8.5X11
User: SLEE Jun 25, 2014 - 12:42pm Xrefs: - Images: INECTIONPOINTS.TIFF



Proposed August 2014 Injection Point

Proposed Well Location

Legend

Existing Monitoring Well

First Round RegenOx Injection Point (IP)

Second Round RegenOx Injection Point (IP)

Soil Boring

W — Water Line

Groundwater Flow Direction

FIGURE E.1-3
EPA 1006 WESTERN GAS
401 S. ELM ST.
TOPPENISH, WA 98948
TREATMENT AREA MAP

Bristol
ENVIRONMENTAL REMEDIATION SERVICES, LLC
Phone (907) 563-0013 Fax (907) 563-6713

DATUM:	NAD 83
PROJECTION:	WA SP ZS FT
PROJECT NO.	34150026
DATE	6/25/2014
DWN.	STL
SCALE	1" =5'
APPRVD.	JSD

ATTACHMENT E.1

Western Gas Corrective Action Plan (CAP)

U.S. ENVIRONMENTAL PROTECTION AGENCY

Western Gas

401 S. Elm Street, Toppenish, Washington

CORRECTIVE ACTION PLAN

EPA Facility No. 4260105
EPA Contract No. EP-W-12-009
Task Order B-7, Amendment 1

Revision 1

July 2013

Prepared for:

U.S. Environmental Protection Agency
Office of Underground Storage Tanks
Ariel Ross Building
1200 Pennsylvania Avenue, N.W.
Washington, DC 20460

Bristol
ENVIRONMENTAL REMEDIATION SERVICES, LLC
111 W. 16th Avenue, Third Floor, Anchorage, Alaska 99501

TABLE OF CONTENTS

SECTION **PAGE**

ACRONYMS AND ABBREVIATIONS iii
APPROVALS iv
DISTRIBUTION LIST iv
1.0 INTRODUCTION 1
1.1 Site Description and Location 1
1.2 Geology and Hydrology 2
1.3 Site History 3
1.4 Scope of Work 6
2.0 PROJECT ORGANIZATION 9
2.1 Project Organization 9
2.1.1 Bristol Program Manager 9
2.1.2 Bristol Project Manager 9
2.2 Contact Information 10
2.2.1 Subcontractors 11
2.3 Schedule 11
3.0 SITE CONDITIONS 13
3.1 Nature and Extent of Contamination 13
3.2 Conceptual Site Model 14
3.3 Contaminants of Concern 14
4.0 CORRECTIVE ACTION OBJECTIVE 15
5.0 PROPOSED CORRECTIVE ACTION 17
5.1 Selected Technology 17
5.2 Corrective Action Activities 18
5.2.1 Site Preparation 18
5.2.2 Sampling 19
5.2.3 Injection Test 20
5.2.4 RegenOx Injections 21

5.2.5 ORC Advanced Injection 23
6.0 FIELD SAMPLING PLAN 25
6.1 Sample Locations 25
6.2 Field Screening 25
6.3 Soil Sampling Collection Methods 26
6.3.1 Gasoline Range Organics 26
6.3.2 Diesel Range Organics 26
7.0 REPORTING 27
8.0 REFERENCES 29

TABLES

Table 1 Summary of Key Dates 12

FIGURES

Figure 1 Site Location Map
Figure 2 Site Map
Figure 3 Treatment Area Map

APPENDICES

Appendix A Quality Assurance Project Plan
Appendix B Regensis's Treatment Design
Appendix C Bristol's Standard Operating Procedures for Field Activities
Appendix D Field Forms

ACRONYMS AND ABBREVIATIONS

°C	degrees Celsius
AST	aboveground storage tank
bgs	below ground surface
Bristol	Bristol Environmental Remediation Services, LLC
BTEX	benzene, toluene, ethylbenzene, and xylenes
COC	contaminant of concern
DRO	diesel range organics
EPA	U.S. Environmental Protection Agency
FSP	Field Sampling Plan
GRO	gasoline range organics
LESAT	Lockheed Environmental Systems and Technology Company
LUST	leaking underground storage tank
mL	milliliter
MTCA	Model Toxics Controls Act
NWTPH-Dx	Northwest Total Petroleum Hydrocarbon Method (diesel)
NWTPH-Gx	Northwest Total Petroleum Hydrocarbons Method (gasoline)
PCS	petroleum-contaminated soil
PID	photoionization detector
PS&W	Pacific Soil and Water, Inc.
QA	quality assurance
QAPP	Quality Assurance Project Plan
QC	quality control
Regenesis	REGENESIS Remediation Technologies
SOW	scope of work
SSHP	Site Safety and Health Plan
TO	task order
UST	underground storage tank
VOC	volatile organic compound

APPROVALS

By their signatures, the undersigned approve this Corrective Action Plan.

Mary Millner — 07/16/2013
Mary Millner — Date
EPA Region 10 Subject Matter Expert

Gina Grepo-Grove — 07/17/13
Gina Grepo-Grove — Date
EPA Region 10 Quality Assurance Officer

Patricia Curl — 7-16-13
Patricia Curl — Date
Bristol Program Manager

Marty C. Hannah — 7-16-13
Marty Hannah — Date
Bristol Quality Assurance Manager

DISTRIBUTION LIST

Mary Millner, EPA Region 10 Subject Matter Expert

Lynn Depont, EPA Program Officer

Elizabeth Sanchey, Yakama Nation Environmental Program Manager

Mark Koffler, Yakama Nation Water Code Administration

1.0 INTRODUCTION

The U.S. Environmental Protection Agency (EPA) retained Bristol Environmental Remediation Services, LLC (Bristol), to prepare this Corrective Action Plan to detail the activities required to conduct corrective actions at the Western Gas site (formerly known as Kuki Mart, Brand X, and Toppenish Shell) (EPA Facility No. 4260105). Activities for the corrective action include 1) planning and initiating corrective action activities, to include injecting remedial products into the subsurface at the Western Gas site, 2) providing a report that describes the corrective action performed, and 3) making a recommendation for additional work or for closure. Bristol is conducting work on potential leaking underground storage tank (LUST) sites on Indian lands throughout the United States for the EPA. The EPA assigned this project to Bristol as Task Order (TO) No. B-7, Amendment 1, under Contract No. EP-W-12-009.

The regulatory guidance for this program includes EPA LUST guidance; Title 40 Code of Federal Regulations, Part 280; and the Washington State Department of Ecology Model Toxics Control Act (MTCA) site remediation guidance (Washington State Department of Ecology, 2007).

This Corrective Action Plan includes a Field Sampling Plan (FSP) (Section 6.0) for the limited sampling to be performed in support of this corrective action, and the site-specific Quality Assurance Project Plan (QAPP). The QAPP is presented as Appendix A.

1.1 SITE DESCRIPTION AND LOCATION

Western Gas is located on the Yakama Nation Indian Reservation at 401 S. Elm Street, Toppenish, Washington (Figure 1). It is 0.9 acre in size and is bounded by 1st Avenue to the north, a Subway restaurant and Advance America Cash Advance to the south, Elm Street to the east, and El Gallo Giro Grocery and Taco Shop to the west. The site supports a concrete pad, a gas station island with a canopy, and the Western Gas Service Station

(Figure 2). The majority of the site is paved. EPA Region 10 lists the site as an active LUST site (EPA, 2012).

1.2 Geology and Hydrology

Toppenish lies within the Columbia Plateau. The Columbia Plateau is defined by the extent of the Columbia River Basalt and covers a large area, including eastern Washington, western Idaho, and northern Oregon. Locally, Toppenish is located within the southeast portion of the Toppenish Creek Basin. The basin is bound to the north by the Rattlesnake Ridge, to the south by the Toppenish Ridge, and to the west by the Cascade Range. The Yakima River flows approximately 2 miles east of town in a southeasterly direction toward the Columbia River. Locally, the topography dips gently to the southeast. The Western Gas site is underlain by alluvial deposits consisting of the Yakima River flood silts. The flood silts are a clayey, sandy silt that comprise the upper 4 to 5 feet of substrate. Below the flood deposits lie the mainstream Yakima River gravels, which extend to a depth of 14 feet. The gravels consist of basaltic and andesitic cobbles and gravels that range in size from 2 to 6 inches in diameter (White Shield, Inc., 1991).

The Toppenish Creek Basin is divided by the Yakima River into two drainage areas. Surface water drains to the southeast, following the local topography. The closest body of water to the work site is the Wanity Slough, approximately one-quarter mile away to the southwest. Groundwater in the area is present in a shallow alluvial aquifer and a deeper basaltic aquifer. Groundwater at the site is within the alluvial aquifer, and was measured at a depth of about 13 feet below the ground surface (bgs) in November 2012 (Bristol, 2013). Groundwater levels exhibit seasonal variation as a function of irrigation and recharge and can fluctuate from 6 to 8 feet, with highest levels expected in September, and lowest levels expected in early April. The general trend of groundwater flow in the vicinity of the site is to the southeast; however, there are not enough data available to

determine the exact direction of flow. The nearest public water supply well is approximately 0.2 mile east-southeast from the site (White Shield, 1991).

1.3 Site History

Initial notification of contamination present at the site was provided to EPA in May 1991, through a site assessment performed for another party by White Shield (White Shield, 1991). From the early 1950s through 1991, the property operated as a bulk plant for distribution of petroleum products and supported a mini-mart that retailed petroleum products; the mini-mart had an attached car wash (White Shield, 1991). The western side of the property operated as the bulk plant and had five aboveground storage tanks (ASTs) and two abandoned underground storage tanks (USTs) of unknown size, but which were assumed to have held 1,500 gallons each. The eastern portion of the site was occupied by a small mini-mart, with an attached car wash to the south. Two 6,000-gallon USTs, containing regular and unleaded gasoline, were located to the north of the mini-mart, while two 4,000-gallon USTs containing regular and super unleaded gasoline were located to the east of the car wash. The two 1,500-gallon, two 6,000-gallon, and two 4,000-gallon USTs were unused at the time of the 1991 site assessment (EPA, 1997). Retail dispensers were located north and east of the building, and a diesel dispenser associated with the ASTs was located in the central portion of the site. Groundwater is located approximately 13 feet bgs, with seasonal variations. In general, groundwater flows to the southeast. There is a municipal water supply well located approximately 1,000 feet to the east-southeast of the site. Site soils consist of clayey silt mixed with sand and gravel to a depth of 7 to 10 feet bgs and silty, sandy gravel with cobbles below this depth. Three adjacent properties either currently have or previously had on-site fuel USTs.

During the 1991 site assessment, three test pits were dug downgradient of the USTs in areas most likely to contain petroleum contamination. Petroleum above MTCA cleanup levels was found in test pits 1 and 3; high levels of petroleum contaminants were reported

in soil at 7 feet bgs in Test Pit 3. Test Pit 3 is located on the eastern portion of the site along Elm Street. Because of concerns about migration of petroleum contaminants, two additional test pits were dug at the Serv Um Self located immediately east of the site across Elm Street. Analytical soil samples were not collected from the two off-site test pits; however, groundwater samples were collected from all five of the test pits. One of the groundwater samples collected from Test Pit 4 contained diesel and lead at concentrations that exceed MTCA cleanup levels; no detectable concentrations of contaminants were reported for the other groundwater samples. The report recommended removing the unused USTs and conducting a cleanup of the contaminated media.

A soil-gas and water survey report was prepared in April 1994 for the Indian Lands Resource Conservation and Recovery Act site at Toppenish (Lockheed Environmental Systems and Technology Company [LESAT], 1994). Benzene, toluene, ethylbenzene, and xylenes (BTEX) were the target analytes for soil-gas, soil, and water samples, while diesel was only analyzed for the soil and water samples. The results for samples collected from three locations along the eastern and southeastern side of the site show BTEX in groundwater samples, but not in the soil samples. Diesel range petroleum hydrocarbons were not detected (LESAT, 1994).

The EPA performed a field inspection at the site in July 1994 (EPA, 1994). The depth to the bottom of the six USTs was measured, as well as the product within the USTs. Product was measured in one of the two 6,000-gallon USTs (0.625 inches) and both of the 1,500-gallon tanks (3.75 and 3.25 inches), while the 4,000-gallon USTs contained only oily water.

In 1995, the USTs were removed and remedial efforts were initiated by Technico Environmental Services, Inc. Three areas were excavated; there were two USTs in each of the excavations. Two of the excavations, tank basins for USTs I and II and for III and IV, contained petroleum-contaminated soil (PCS), while the third tank basin for USTs V and

VI did not. Diesel contamination above MTCA cleanup levels was also reported at the soil-water interface at the diesel dispenser associated with the ASTs, which was located in the central portion of the site. All PCS was removed from the tank basin formerly containing USTs I and II, while PCS was left along the south and east of the tank basin formerly containing USTs III and IV. No further excavation to the south and east could take place at this tank basin since further excavation would endanger Elm Street. The vertical extent of this excavation was to 20 feet bgs, with the majority of the contamination located in the smear zone, which was 16 to 18 feet bgs. Approximately 300 cubic yards of PCS were removed from the site (Technico, 1995).

During the 1995 removal action, three monitoring wells were installed to evaluate groundwater contamination. Monitoring Well 1 was placed north and slightly east of the diesel dispenser and west of the tank basin for USTs I and II; MW2 was placed along the southern edge of the site, west of the tank basin formerly containing USTs III and IV; and MW3 was placed against the southeastern sidewall in the contaminated smear zone, near the tank basin formerly containing USTs III and IV. Gasoline and diesel were not detected in any of the three wells, but BTEX concentrations exceeded MTCA cleanup levels in MW3. Testing for lead was not performed.

In 1996, PLSA Engineering & Surveying performed a site assessment and cleanup at the site (PLSA, 1996), so that the site could be developed for new construction (the Maid O'Clover Service Station and convenience store). On-site structures were demolished prior to the removal of PCS. During removal activities, two additional USTs were encountered and removed. One was located in the northeast portion of the site; the location of the other UST, a waste oil tank, was not reported. Both had been previously removed from service. Approximately 300 cubic yards of PCS were removed and disposed of off site. The report concluded that the sources of gasoline and BTEX contamination were removed to the extent practicable; therefore, groundwater quality was expected to

improve in the near future. There was no evidence that the petroleum contamination had impacted neighboring properties, and since the site was to be covered by the new structure(s) and pavement, the risk was minimal. A March 1997 addendum to this report, states that there are three monitoring wells on site, including a monitoring well installed in the fuel dispensing island (MW4), indicating that MW1 was no longer there. Also, the addendum stated that the PCS originating from inside the waste oil UST had been characterized for off-site disposal, but soil sampling had not been performed at the location of the former waste oil tank.

EPA provided a Sampling and Analysis Plan prepared by Blue Mountain Environmental Consulting, LLC (Blue Mountain, 2001). No further reports or documentation of site activities were provided by the EPA for this site.

In November 2012, Bristol performed additional site assessment activities, including driving direct-push soil borings, collecting subsurface soil samples, redeveloping existing groundwater monitoring wells, installing monitoring wells, and sampling existing and temporary monitoring wells (Bristol, 2013). Results of the site assessment indicate that groundwater contamination is only present in MW2 (Figure 2) and that natural attenuation appears to be taking place. The site assessment also concluded that the source of the groundwater contamination at MW2 is soil contamination that remains present in the southeast corner of the site. This remaining PCS is adjacent to Elm Street and cannot practically be removed.

1.4 Scope of Work

The currently authorized scope of work (SOW) for the corrective action for the Western Gas LUST site includes the following activities:

- Conducting limited subsurface soil sampling to reestablish baseline soil contamination levels
- Performing a pre-injection aquifer test

- Performing one round of injections of RegenOx at the site
- Preparing a Corrective Action Report, and provide recommendations for further work

This Corrective Action Plan includes details for an additional SOW that has not yet been funded, but includes:

- Performing additional rounds of RegenOx injections at the site
- Performing one ORC Advanced injection at the site

(Intentionally blank)

2.0 PROJECT ORGANIZATION

Descriptions of Bristol's key staff and their responsibilities and authority are provided below.

2.1 PROJECT ORGANIZATION

2.1.1 Bristol Program Manager

The Program Manager, Patricia Curl, is responsible for overall program performance and quality. Ms. Curl will be the primary program-level point of contact between the EPA and Bristol. She is responsible for the EPA program, both technically and administratively. Ms. Curl is empowered to access and direct environmental resources across the company. She is responsible for assigning Project Managers (PMs) and technical support staff based on the nature and complexity of the tasking. Ms. Curl will implement program management and quality assurance (QA) plans that:

- Guide managers and staff on the processes and procedures for completing the contractual and technical requirements for start-up of this support activity
- Provide technical oversight to the PMs
- Define responsibilities and levels of authority
- Establish procedures for project controls and forecasting
- Ensure that systems are in place for monitoring costs, schedule, and performance
- Assign responsibilities for QA and establish a system for both peer and quality reviews of deliverables
- Integrate the use of subcontractors and foster Bristol's commitment to small business

2.1.2 Bristol Project Manager

The Bristol PM is Julie Sharp-Dahl. Ms. Sharp-Dahl is responsible for the technical performance of the project and the day-to-day coordination with the EPA Subject Matter Expert (SME). As the technical leader, she is responsible for the technical content of the

work and the overall project quality. The PM's quality control-(QC-) related responsibilities are to:

- Ensure that all planning documents for the project are prepared and reviewed
- Assign project QC responsibilities to appropriately qualified personnel at the outset of the project
- Select additional technical reviewers for the project
- Communicate project scope requirements to project team members
- Communicate with the Client
- Ensure that all project deliverables and activities are in accordance with planning documents
- Respond to corrective action requests and ensure that deficiencies are corrected in a timely manner

The PM oversees the preparation of the Site Assessment Plan, FSP, Site Safety and Health Plan (SSHP), and QAPP. The Site Assessment Plan, FSP, SSHP, and QAPP are prepared to describe procedures to be used to conduct the site assessment. These documents describe the work to be performed and set the guidelines for verifying the quality and integrity of field activities performed, including environmental samples to be collected, analytical tests to be run, and QA/QC measures to be implemented. They also set standards for identifying safety hazards and ensure measures are implemented to protect workers.

2.2 Contact Information

Names, phone numbers, and email addresses for all key team members anticipated to work under this TO are as follows:

- Mary Millner, EPA Subject Matter Expert, 206-553-6061, Millner.Mary@epa.gov
- Patricia Curl, Bristol Program Manager, 907-563-0013, pcurl@bristol-companies.com
- Julie Sharp-Dahl, Bristol PM, 907-563-0013, jsharp-dahl@bristol-companies.com

- Mathew Faust or Patrick Braley, Bristol Field Scientist, 907-441-0261 or 907-242-6843 (cell), mfaust@bristol-companies.com or pbraley@bristol-companies.com
- Wil Badonie, Yakama Nation Environmental Program representative, 509-865-5121, x 6079, wbadonie@yakama.com

2.2.1 Subcontractors

Wherever possible, Bristol's practice is to subcontract services from local suppliers, with an emphasis on small and tribally owned business support. Bristol will implement subcontracts that provide best value to the government and the project. Subcontract types will be time and materials. Subcontractors may include laboratories and drilling companies.

The following are the major subcontractors that will provide services to Bristol for this TO:

- TestAmerica Laboratories, Inc., will provide analytical services. TestAmerica is located in Tacoma, Washington.
- REGENESIS Remediation Technologies (Regenesis) provided technical assistance in designing the corrective action approach, and will serve as a vendor supplying the remedial products.
- Pacific Soil and Water, Inc. (PS&W), will provide drilling and injection services for the corrective action. PS&W is located in Tigard, Oregon.

2.3 SCHEDULE

Bristol anticipates that fieldwork for the first round of RegenOx injections will begin in mid-September 2013 and will require approximately 4 days to complete. A summary of key dates is presented in Table 1.

Table 1 Summary of Key Dates

Date	Activity
May 14, 2013	Submit Budget Narrative and Cost Estimate to EPA
May 28, 2013	Submit Revised Budget Narrative and Cost Estimate to EPA
July 2013	Submit Draft Corrective Action to EPA for review/comment
14 days after Bristol's receipt of EPA's comments on the Draft Corrective Action Plan	Submit Final Corrective Action Plan to EPA
August 2013	Public Notification of Corrective Action
September 2013	Initial RegenOx Injection Event
45 days after completion of initial injection, unless the contract is modified to perform additional injection events	Submit Draft Corrective Action Report
14 days after Bristol's receipt of EPA's comments on the Draft Site Assessment Report	Submit Final Corrective Action Report
To be determined	Additional RegenOx Injection Event(s) (field effort to be performed by EPA personnel)
To be determined	ORC Advanced Injection Event (field effort to be performed by EPA personnel)
To be determined	Groundwater monitoring event(s) (to be performed by EPA personnel)

Notes:

Bristol = Bristol Environmental Remediation Services, LLC

EPA = U.S. Environmental Protection Agency

3.0 SITE CONDITIONS

The following section summarizes site conditions, including the nature and extent of contamination, the conceptual site model, and the contaminants of concern (COCs).

3.1 NATURE AND EXTENT OF CONTAMINATION

Sources of contamination at the site included leaks from USTs and/or fuel dispensers. The majority of PCS was removed during previous cleanup efforts (including approximately 300 cubic yards in 1995 [Technico, 1995] and an additional 300 cubic yards in 1996 [PLSA, 1996]). However, an unknown volume of PCS was left in place in the subsurface in the southeast corner of the site, where close proximity to Elm Street prevented further excavation. Based on the results of site assessment activities performed by Bristol in 2012, it is believed that the majority of subsurface soil contamination remaining on the site is located in the unexcavated zone along Elm Street in the southeast corner of the property (Bristol, 2013).

Results of the 1995 cleanup activities indicate that the majority of subsurface soil contamination is located in a “smear zone” at depths of 16 to 18 feet. As the most recent round of groundwater sampling measured depths to groundwater of approximately 13 feet in the southeast corner of the site, it is possible that the smear zone has become submerged, or that the 1995 estimate of depth was inaccurate.

Contamination present in subsurface soil has migrated to groundwater at the site, as evidenced by observations of smear-zone contamination during the 1995 soil excavation. The most recent round of groundwater sampling (performed in 2012) confirmed that gasoline range organics (GRO) and diesel range organics (DRO) concentrations in groundwater that exceed MCTA cleanup levels remain at MW2 (Bristol, 2013). MW2 is located in the southeast corner of the site in close proximity to the remaining subsurface soil contamination (Figure 2).

3.2 CONCEPTUAL SITE MODEL

Potentially complete subsurface soil exposure pathways include ingestion and dermal contact pathways in the event of future construction or excavation in the contaminated zone, as well as vapor intrusion pathways in the event that a structure is constructed over the contaminated zone. Potentially complete groundwater exposure pathways include ingestion, inhalation, and dermal contact pathways in the event that water supply wells are installed in the contaminated zone.

3.3 CONTAMINANTS OF CONCERN

The COCs at the Western Gas LUST site have historically included total petroleum hydrocarbons (TPH) (including GRO and DRO), volatile organic compounds (VOCs), polynuclear aromatic hydrocarbons, and lead.

For the limited soil sampling that will be performed in support of this corrective action, analytes and analytical methods include:

- GRO by Northwest Total Petroleum Hydrocarbon Method TPH-Gx (NWTPH-Gx)
- DRO by Northwest Total Petroleum Method Hydrocarbon TPH-Dx (NWTPH-Dx)

4.0 CORRECTIVE ACTION OBJECTIVE

The objective of the corrective action is to reduce concentrations of COCs in subsurface soil in the southeast corner of the property and to subsequently restore the groundwater quality at the site (as represented by well MW2) to concentrations below MTCA Method A groundwater cleanup levels and to minimize the potential for exposure to humans and the environment.

(Intentionally blank)

5.0 PROPOSED CORRECTIVE ACTION

The following section describes the proposed corrective action for the Western Gas site, including the selected remedial technologies, anticipated corrective action activities, and approximate project schedule.

5.1 SELECTED TECHNOLOGY

The selected technology for the corrective action is chemical oxidation and enhanced bioremediation achieved through injection of Regenesis's remediation products into the contaminated zone. The approach of in-situ treatment through chemical oxidation and enhanced bioremediation was selected by EPA and Bristol. Regenesis provided technical assistance with design of the injection parameters based on site-specific analytical data, soil lithology types, and hydrogeology. Regenesis's proposal (including design details) is included in Appendix B. The product will be applied to the contaminated zone using direct-push technology injection techniques.

The products proposed for use on the site include RegenOx and ORC Advanced. RegenOx is a remediation product that directly destroys contaminants through oxidation reactions. RegenOx is a two-part product consisting of an oxidizer and an activator, which are combined and then injected into the contaminated media (in either the saturated or vadose zone). RegenOx includes a sodium percarbonate complex with a multi-part catalytic formula, and can quickly destroy petroleum hydrocarbon and associated VOC contaminants through a chain of oxidation reactions. The chemical reaction that is produced is relatively safe, as the chemicals are non-corrosive and generate minimal amounts of heat and pressure.

ORC Advanced is a remediation product that accelerates naturally occurring aerobic bioremediation by supplying oxygen to the contaminated media. ORC Advanced consists of a calcium oxy-hydroxide compound, and (once injected into the subsurface) works by

sorbing onto soil particles and releasing oxygen in a controlled manner. ORC Advanced can release oxygen for up to 12 months after being hydrated.

Regenesis recommended using three to five injections of RegenOx to treat the soil contamination at the site, followed by one injection of ORC Advanced to treat the remaining low-level groundwater contamination (Appendix B). This estimate was based on soil concentrations from laboratory analyses performed 18 years ago; the number of injections required may be refined based on concentrations of COCs measured in soil samples collected during the first injection event. At this time, Bristol is only funded to perform one injection event of RegenOx, though procedures for additional events are described below. If additional injection events are required to bring groundwater concentrations below cleanup levels, they may be scheduled and funded at a future date.

5.2 Corrective Action Activities

Corrective action activities described below include soil sampling, an injection test, the initial funded RegenOx injection, and future additional injections of RegenOx and ORC Advanced.

5.2.1 Site Preparation

For the first injection event, Bristol will arrive on site at least a half-day before the drilling subcontractors arrives. Bristol will perform the following activities in preparation for site activities:

- Verify that utility locates have been completed.
- Locate the proposed 30-foot by 15-foot treatment area using the site map (Figure 2) and relative locations of site features (for example, the sidewalk edge and MW2).
- Mark the corners of the treatment area by driving survey nails into the asphalt.
- Identify the locations of the first and second round of RegenOx injection points (depicted on Figure 3) using swing ties and cloth measuring tapes (adjust locations if necessary to avoid subsurface utilities); mark the injection point locations with marking paint.

- Using swing ties and cloth measuring tapes, hand draw an accurate sketch of the treatment area that will allow accurate identification of injection point locations using distances from known points during future injection events.

5.2.2 Sampling

Limited soil sampling will be performed during the initial injection event in order to better characterize the remaining soil contamination in the treatment area. Samples have not been collected from this portion of the site since 1995, and concentrations of COCs may have decreased through natural attenuation since this time. Analytical results will be used by Regenesis and Bristol technical personnel to refine the number of injections needed to remediate the site.

Injection points will be driven at the site by a drilling subcontractor using Geoprobe direct-push equipment. Of the six injection points that will be driven during the first injection event, two will be cored to total depth (19 feet bgs) with 4-foot Macro-Core acetate sleeves to facilitate collection of subsurface soil samples. The borings that will be cored and sampled will be those closest to the source area in order to identify the highest levels of contamination that is still present. The borings that will be cored will also be the first injection points that are driven, so that the treatment interval can be refined if necessary. It is anticipated that the borings that will be sampled will be at injection points 01 and 04 (Figure 3).

The core recovered from each boring will be set aside during advancement of the boring, and the Macro-Core acetate sleeves will not be opened until the total depth of the boring has been reached and all of the core has been recovered. Depth intervals and top and bottom ends of each sleeve will be marked on the acetate sleeve with permanent marker as they are set aside to prevent confusion. Once the entire core has been recovered, the acetate sleeves will be opened and the core will be screened for organic vapors using a photoionization detector (PID) along the entire length of the core. Field screening will

be conducted approximately every 6 inches along the core using the rapid acquisition technique (described below in Section 6.2). When the entire length of core has been field screened, the PID readings will be evaluated, and a sample will be collected from the depth interval with the highest field screening result.

Additional details for the soil sampling procedures are outlined below in the Field Sampling Plan (Section 6.0).

Regenesis recommends that quarterly groundwater sampling be initiated at the site following the third injection of RegenOx and that the groundwater be analyzed for COCs, as well as additional parameters for evaluating the progress of aerobic bioremediation (including pH, dissolved oxygen, oxidation-reduction potential, specific conductance, chemical oxygen demand, biological oxygen demand, total and dissolved iron, and methane). Bristol is not contracted to perform groundwater monitoring activities at the site at this time; however, we have been informed that EPA intends to perform groundwater sampling utilizing EPA personnel.

5.2.3 Injection Test

Regenesis recommends that a pre-aquifer injection test be conducted during the initial injection event in order to ensure that the subsurface will accept the proposed volume of RegenOx. The injection test will be performed in the injection point located closest to MW2 (injection point 01), and the static water level in MW2 will be measured immediately prior to and following the injection test in order to provide qualitative information on the potential radius of influence of the injected products.

The drilling subcontractor will perform the injection test after the following general procedures are performed:

- Drive a 1.5-inch pre-probe with an expendable tip to 19 feet bgs (the bottom of the treatment interval) using standard 1-inch drive rods.

- Disconnect the expendable tip and attach the slurry pump to the drive rod using the quick-connect fitting, and put 20 gallons of potable water in the pump hopper.
- Withdraw the drive stem 3 feet so that the bottom of the pre-probe is located at approximately 16 feet bgs (the top of the treatment interval).
- Pump the 20 gallons of potable water into the treatment interval, observing pump pressure levels to estimate how well the formation will accept the RegenOx.
- Once the water has been completely pumped into the formation, lower the drive stem 3 feet so that the pre-probe is back at 19 feet bgs (bottom of the treatment interval) and prepare for the RegenOx injection.

Based on Bristol's knowledge of the subsurface soil lithology at the Western Gas site, it is anticipated that the injection test will be successful and will indicate that the subsurface will readily accept the remediation products. However, if the injection test indicates that the subsurface will not accept the product at the desired pumping rate, the corrective action may be modified by reducing the volume of injected material or modifying the injection interval.

5.2.4 RegenOx Injections

The approximate injection point locations for the first round of RegenOx injections are depicted on Figure 3. As recommended by Regenesis, boring locations are to be spaced on approximately 8 feet apart along two rows, and the rows are to be spaced 10 feet from each other (Regenesis's specific design recommendations are included in Appendix B). Figure 3 also shows the approximate injection point locations for a second round of RegenOx injections (currently not funded); Regenesis recommends that injection points be offset by approximately 4 feet for each round of injections to maximize distribution of RegenOx.

The treatment interval for RegenOx is anticipated to be from 16 to 19 feet bgs, based on the descriptions of the impacted soil interval from the 1995 soil removal. However, if the field-screening results from the cored and sampled boring indicate that the highest levels of soil contamination are located at a different depth, the treatment interval will be

adjusted to include the most impacted interval. Regardless of the adjustment, the length of the treatment interval will remain 3 feet.

Regenesis has recommended a 5% oxidizer solution for this project. For each injection point, Regenesis has specified the following volumes and quantities to be used to achieve this concentration in the solution: 57 gallons of water, 25 pounds of RegenOx Part A, and 11 pounds of RegenOx Part B.

The drilling subcontractor will perform the RegenOx injections following the general procedures listed below (more specific instructions for RegenOx injections written by Regenesis are included in Appendix B):

- Drive a 1.5-inch pre-probe with an expendable tip to 19 feet bgs (or the bottom of the treatment interval) using standard 1-inch drive rods.
- Withdraw the rod assembly several inches, and disconnect the expendable tip.
- Pour a premeasured volume of water into the mixing vessel, and then add the premeasured quantity of the RegenOx Part A oxidizer (**WARNING:** For safety reasons, always add oxidizer to water rather than the reverse). Mix the solution until the oxidizer is dissolved, and then add the premeasured quantity of the RegenOx Part B activator. Mix for at least 5 minutes until the solution becomes homogenous.
- The injection should begin as soon as possible after mixing is complete. Attach the slurry pump to the drive rod using the quick-connect fitting, and transfer the mixed solution to the slurry pump hopper.
- Pump the RegenOx solution into the subsurface while withdrawing the drive rods (either continuously or in 1-foot increments) until the bottom of the pre-probe is located at approximately 16 feet bgs (or the top of the treatment interval) and the RegenOx solution has been completely discharged.
- Remove the drive rods and complete the injection point borehole with clean sand directly above the treatment zone, and a bentonite seal to within 0.5 feet of the surface. Finish the injection point borehole at the surface with quick set concrete to create a surface seal.

5.2.5 ORC Advanced Injection

The injection point locations for the ORC Advanced injections are not depicted on Figure 3. Although the injection point spacing will be the same as for the RegenOx locations, the appropriate offset is unknown until the number of RegenOx injections to be performed is determined. As a general directive, the ORC Advanced injection points will be located in the center of each cluster of RegenOx injection points.

The treatment interval for ORC Advanced is anticipated to be from 14 to 19 feet bgs, based on descriptions of the impacted soil interval from the 1995 soil removal. The treatment interval may be adjusted based on field screening from the first RegenOx injection event. Regardless of the adjustment, the length of the treatment interval will be 5 feet.

Regenesis has recommended a 30% solution for this project. For each injection point, Regenesis has specified the following volumes and quantities be used to achieve this concentration in the solution: 11 gallons of water and 40 pounds of ORC Advanced.

The drilling subcontractor will perform the ORC Advanced injections following these general procedures (more specific instructions for ORC Advanced injections written by Regenesis are included in Appendix B):

- Drive a 1.5-inch pre-probe with an expendable tip to 19 feet bgs (or the bottom of the treatment interval) using standard 1-inch drive rods.
- Withdraw the rod assembly several inches, and disconnect the expendable tip.
- Create the injection slurry by mixing the premeasured volume of water with the premeasured quantity of ORC Advanced.
- The injection should begin as soon as possible after mixing is complete. Attach the slurry pump to the drive rod using the quick-connect fitting, and transfer the mixed solution to the slurry pump hopper.
- Withdraw the rod assembly 5 feet, and then pump the slurry into the treatment interval.

- Remove the drive rods and complete the injection point borehole with clean sand directly above the treatment zone, and then a bentonite seal to within 0.5 feet of the surface. Finish the injection point borehole at the surface with quick set concrete to create a surface seal.

6.0 FIELD SAMPLING PLAN

The following sections include details for the limited subsurface soil sampling that will be conducted in tandem with the first round of RegenOx injections, including sample locations, field screening methods, and soil sample collection methods.

The site-specific QAPP for TO B-7, along with a one-page attachment summarizing changes for Amendment 1, is included as Appendix A. Bristol's Standard Operating Procedures (SOPs) for sampling activities are included in Appendix C and field forms that may be used are included in Appendix D.

No field QC samples, such as field duplicates or blanks, will be collected during the corrective action, because the sample results are only to be used for refining the injection approach.

6.1 SAMPLE LOCATIONS

As described above in Section 5.2.2, one laboratory analytical sample will be collected from each of the two borings. The samples will be collected from the depth intervals corresponding with the highest field-screening results.

6.2 FIELD SCREENING

Field personnel will use a PID for field screening soil samples. Field screening for soil borings will be performed using a rapid acquisition technique, which does not require development of headspace vapors, in order to collect a large number of data points while leaving enough soil material for laboratory analysis. For this technique, small divots will be made approximately every 6 inches along the length of the core. The inlet of the PID will be immediately inserted into each freshly exposed divot, taking care not to touch the material. Field personnel will record the highest reading at each divot on the boring log and/or fieldbook.

6.3 SOIL SAMPLING COLLECTION METHODS

The borings will be constructed with 4-foot Macro-Core sleeves that field personnel will visually inspect for contamination upon opening. Contaminated soil within a core will be collected as a grab sample.

The methods that will be used for collecting analytical soil samples are presented below. Sample jars will be filled according to analyte volatility: first GRO, and then DRO.

6.3.1 Gasoline Range Organics

Soil samples to be analyzed for GRO will be containerized before DRO samples. For soil samples that are to be submitted to the laboratory for GRO analysis (NWTPH-Gx), a Terra Core sampling device will be used to transfer 10 grams of soil from the boring to two 40-milliliter (mL) containers. The 40-mL container will contain 10 mL of methanol. The methanol and Terra Core sampler will be provided by the laboratory. The 40-mL containers will be placed on ice and kept at a temperature between 0 and 6 degrees Celsius (°C) until delivery to the laboratory.

6.3.2 Diesel Range Organics

For soil samples that are to be submitted to the laboratory for DRO (NWTPH-Dx), the sample will be collected using clean gloved hands or a disposable sample scoop and placed into one 4-ounce jar. The soil will not be packed but will fill the container. The sample will be kept on ice at a temperature between 0 and 6°C until it is delivered to the laboratory.

7.0 REPORTING

The results from the corrective action will be presented in a Corrective Action Report. The report will contain an introduction, background, a description of corrective action activities, recommendations for future site activities, references, figures, tables, and appropriate appendices such as field notes, photographic log, investigation-derived waste disposal records, laboratory data packages, and data verification summaries. Presently, it is assumed that the report will be written following the initial RegenOx injection event and that it will be limited to summarizing the results of that event. However, if the EPA modifies the TO to include additional RegenOx injection events, or an ORC Advanced injection event, the preparation of the report may be delayed so that it can include a summary of the additional site activities.

(Intentionally blank)

8.0 REFERENCES

Blue Mountain Environmental Consulting, LLC. 2001. *Sampling and Analysis Plan for Maid O'Clover (Brand-X Tank'n Tummy) Gasoline Station.*

Bristol. 2013. *Western Gas LUST Site, Site Assessment Report, EPA Contract No. EP-W-12-009, Task Order B-7, Revision 1*. March.

Lockheed Environmental Systems & Technologies Company (1994). *Soil-Gas, Soil and Water Survey Report for the Indian Lands RCRA Site at Toppenish, Washington.*

PLSA Engineering & Surveying. 1996. *Site Assessment RH Bowles Leaking Underground Tank Site, No. 4-260105.*

Technico Environmental Services, Inc. 1995. *Site Characterization and Independent Cleanup Action Report for the Brand X Tank 'n' Tummy, 401 S. Elm Street, Toppenish, WA.*

U.S. Environmental Protection Agency (EPA). 1994. *Memo to Denise Baker from Jeff Keeler regarding the July 13 – 14, 1994 Trip Report*.

U.S. Environmental Protection Agency (EPA). 1997. *Basis of Decision, Brand X Tank 'n Tummy, Toppenish Washington*. EPA UST Facility No. 4-26105. May 27.

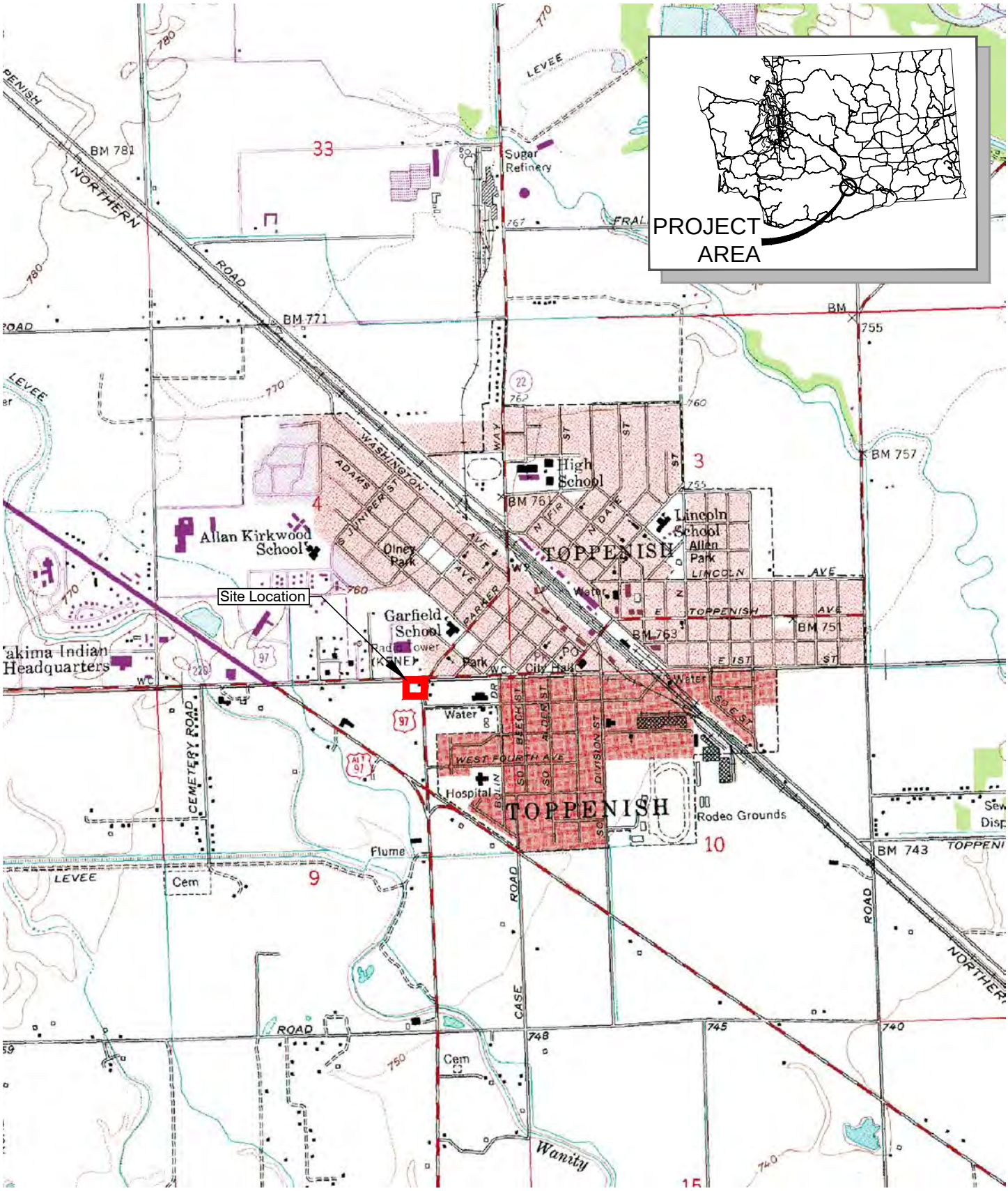
EPA. 2012. EPA Region 10 Active LUST Sites on Indian Lands. Available at: http://yosemite.epa.gov/r10/water.nsf/UST/UST+Info+Resources/$FILE/Active-IL-LUSTs.pdf . Accessed October 2, 2012.

Washington State Department of Ecology. 2007. *Model Toxics Control Act Statute and Regulation: Model Toxics Control Act, Chapter 70.105D RCW. Uniform Environmental Covenants Act, Chapter 64.70 RCW. MTCA Cleanup Regulation, Chapter 173-340 WAC*. Publication No. 94-06. Compiled by Washington State Department of Ecology Toxics Cleanup Program. Revised November 2007.

White Shield, Inc. 1991. *Site Assessment Report for Underground Storage Tank Closure at Brand X Tank & Tummy Toppenish, WA*.

(Intentionally blank)

FIGURES



Topo Reference
USGS

N

0 1000 2000
1" = 2000 Feet

FIGURE 1
EPA B-7 WESTERN GAS
401 S. ELM ST.
TOPPENISH, WA 98948
SITE LOCATION MAP

Bristol
ENVIRONMENTAL REMEDIATION SERVICES, LLC
Phone (907) 563-0013 Fax (907) 563-6713

DATUM:	NA
PROJECTION:	NA
PROJECT NO.	34130043
DATE	06/18/13
DWN.	MTG
SCALE	SHOWN
APPRVD.	JSD

Drawing: \\BEESC-JOBS\BERS\JOBS\34130043 EPA B-7 TOPPENISH SHELL\ACAD-ENVIRO\FIGURES-JUN2013\FIGURE-1.DWG - Layout: FIGURE-1
User: MGARCIA Jun 18, 2013 - 12:34pm Xrefs: - Images: 34422193.TIF

Drawing: \\BEESC-JOBS\BERS\JOBS\34130043 EPA B-7 TOPPENISH SHELL\ACAD-ENVIRO\FIGURES-JUN2013\FIGURE-2.DWG - Layout: FIGURE-2
User: MGARCIA Jun 18, 2013 - 1:08pm Xrefs: - Images: GOOGLE-IMAGE.TIF POTENTIAL SAMPLE PLAN.JPG

Legend

- existing monitoring well
- former monitoring well
- release detection well
- well reference point
- groundwater flow direction
- BH borehole
- RW release detection well
- TW temporary well

Notes:

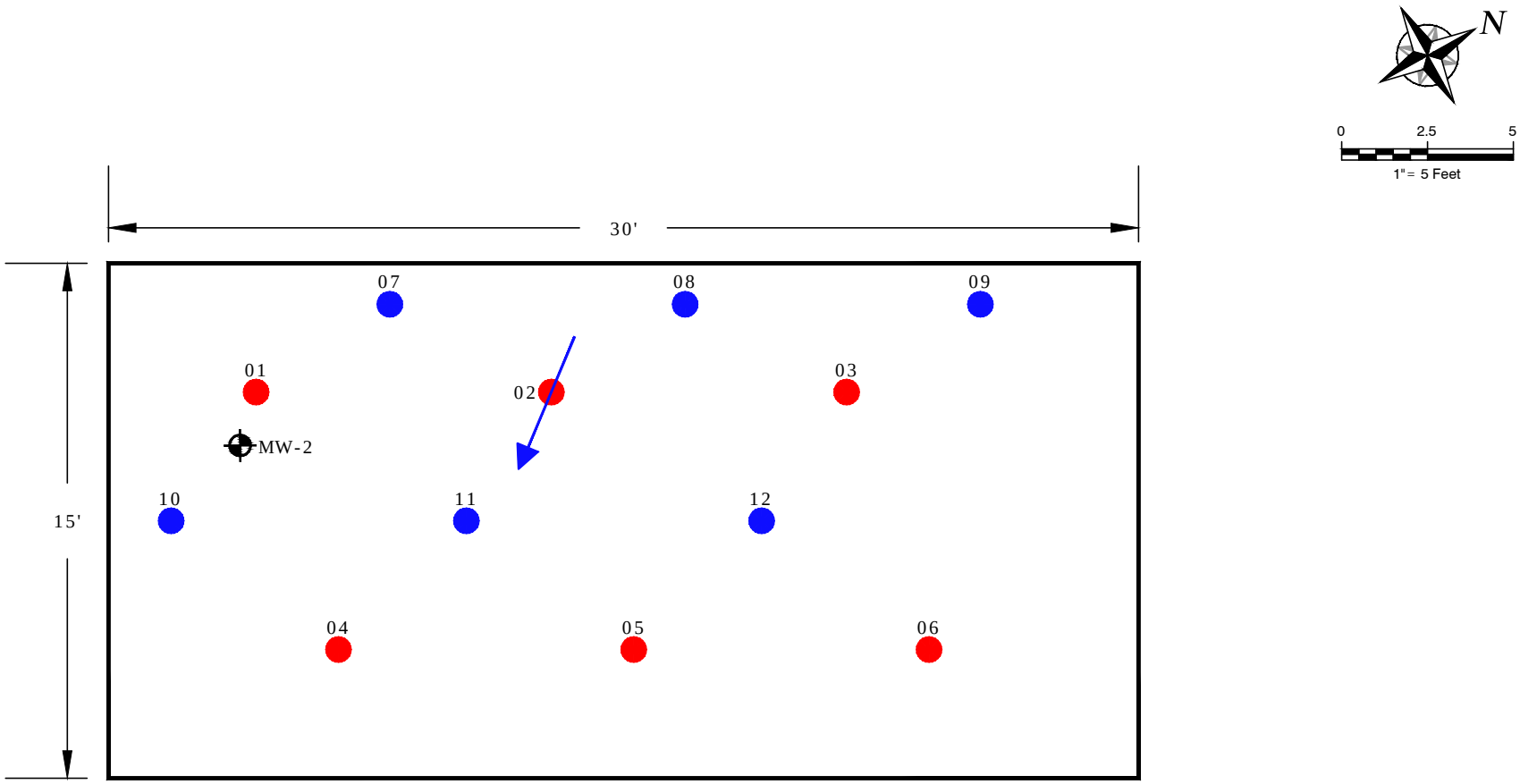
- Clayey silt with sand to depth of 7-10 feet below ground surface.
- Silty sandy cobbly gravel below 7-10 feet below ground surface.
- Groundwater ≈ 13 feet below ground surface.
- RW104 is a release detection well.
- MW3 is no longer present.

FIGURE 2
EPA B-7 WESTERN GAS
401 S. ELM ST.
TOPPENISH, WA 98948
SITE MAP

Bristol
ENVIRONMENTAL REMEDIATION SERVICES, LLC
Phone (907) 563-0013 Fax (907) 563-6713

DATUM:	NAD 83
PROJECTION:	WA SP ZS FT
PROJECT NO.	34130043
DATE	06/18/13
DWN.	MTG
SCALE	SHOWN
APPRVD.	JSD

Drawing: \\BEESC-JOBS\BERS\JOBS\34130043 EPA B-7 TOPPENISH SHELL\ACAD-ENVIRO\FIGURES-JUN2013\FIGURE-3.DWG - Layout: FIGURE-2
User: MGARCIA Jul 01, 2013 - 9:01am Xrefs: - Images:



Sidewalk

Legend

- Existing Monitoring Well
- First Round RegenOx Injection Point
- Second Round RegenOx Injection Point
- Groundwater Flow Direction

FIGURE 3
EPA B-7 WESTERN GAS
401 S. ELM ST.
TOPPENISH, WA 98948
TREATMENT AREA MAP

Bristol
ENVIRONMENTAL REMEDIATION SERVICES, LLC
Phone (907) 563-0013 Fax (907) 563-6713

DATUM:	NAD 83
PROJECTION:	WA SP ZS FT
PROJECT NO.	34130043
DATE	06/18/13
DWN.	MTG
SCALE	SHOWN
APPRVD.	JSD

APPENDIX A

Quality Assurance Project Plan

QAPP AMENDMENT FORM

Sample Alteration Form

Project Name and Number: Remediation at Western Gas, Toppenish, WA, EPA Facility # 4260105 (Bristol Project No. 34130043)

Material to be Sampled: Subsurface Soil

Measurement Parameter: Gasoline range organics (GRO) and Diesel range organics (DRO)

Standard Procedure for Field Collection & Laboratory Analysis (cite reference):

Soil Sampling SOP: BERS-01
GRO by Northwest Method TPH-Gx
DRO by Northwest Method TPH-Dx

Reason for Change in Field Procedure or Analysis Variation:

No change to procedure or analysis, just additional sampling beyond original scope of task order

Variation from Field or Analytical Procedure:

Two additional soil samples

Special Equipment, Materials or Personnel Required:

Geoprobe drill rig, Terra Core samplers, VOA vials, and 4-oz jars

Initiators Name: Julie Sharp Dahl (BERS) Date: 7/9/13

Project Manager: Mary Mulkins Date: 7/9/13

QA Officer: [illegible] Date: 7/11/13

TABLE OF CONTENTS

SECTION **PAGE**

ACRONYMS AND ABBREVIATIONS v
APPROVALS vi
1.0 INTRODUCTION 1
2.0 PROJECT DESCRIPTION 3
2.1 Applicable Documents 3
2.2 Program Task Descriptions 3
3.0 DATA QUALITY OBJECTIVES AND CRITERIA 5
3.1 Sampling Process and Design 5
3.2 Data Quality Objectives 5
3.3 Analytical Quality Control 6
3.3.1 Screening Data 6
3.3.2 Definitive Laboratory Data 6
4.0 SPECIAL TRAINING AND CERTIFICATIONS 13
5.0 DOCUMENTS AND RECORDS 15
5.1 Quality Documents and Records 15
5.2 Draft and Final Reports 15
6.0 DATA GENERATION AND ACQUISITION 17
6.1 Sampling Procedures 17
6.2 QC Sample Collection 17
6.2.1 Equipment Blanks 17
6.2.2 Duplicate Samples 18
6.2.3 Matrix Spike and Matrix Spike Duplicate Samples 18
6.2.4 Trip Blanks 18
6.2.5 Temperature Blanks 19
7.0 SAMPLE CUSTODY, HANDLING, AND SHIPPING PROCEDURES 21
7.1 Sample Custody 21
7.2 Chain-of-Custody 21

7.3 Field Procedures....21
7.4 Sample Labels....22
7.5 Chain-of-Custody Record....22
7.6 Sample Handling and Shipping....23
8.0 ANALYTICAL PROCEDURES AND DETECTION LIMITS....25
9.0 PREVENTATIVE MAINTENANCE....27
9.1 Instrument Inspection....27
10.0 PERFORMANCE CHECK/CALIBRATION PROCEDURES....29
10.1 Photoionization Detector....29
10.2 Supplies and Consumables....29
11.0 DATA MANAGEMENT AND ASSESSMENT PROCEDURES....31
11.1 Field Data....31
11.1.1 Precision....31
11.1.2 Accuracy....32
11.1.3 Representativeness....32
11.1.4 Completeness....32
11.1.5 Comparability....33
11.1.6 Sensitivity....33
11.2 Laboratory Data....33
11.2.1 Level II Data Deliverables....33
12.0 CHARACTERIZATION AND OVERSIGHT....35
12.1 Performance System Audits....35
12.2 Corrective Actions....36
12.3 Quality Assurance Reports....37
13.0 DATA VERIFICATION AND USABILITY....39
13.1 Field Measurements....41
13.2 Laboratory Measurements....41
13.2.1 Laboratory Data Reporting....41
14.0 QUALITY CONTROL REVIEW....43

15.0 REFERENCES.....45

TABLES

Table 1 PQLs and Cleanup Goals for Soil 8
Table 2 PQLs and Cleanup Goals for Groundwater 10

ATTACHMENTS

Attachment 1 TestAmerica Laboratories, Inc. Quality Assurance Manual
Attachment 2 Analytical Control Limits

(Intentionally blank)

ACRONYMS AND ABBREVIATIONS

ANSI	American National Standards Institute
Bristol	Bristol Environmental Remediation Services, LLC
CoC	chain-of-custody
DQI	data quality indicators
DQO	data quality objective
EPA	U.S. Environmental Protection Agency
FSP	Field Sampling Plan
GRO	gasoline range organics
ID	identification
LCS	laboratory control sample
LCSD	laboratory control sample duplicate
LUST	leaking underground storage tank
MS	matrix spike
MSD	matrix spike duplicate
NELAC	National Environmental Laboratory Accreditation Conference
NWTPH-Dx	Northwest Total Petroleum Hydrocarbons-diesel
NWTPH-Gx	Northwest Total Petroleum Hydrocarbons-gasoline
OUST	Office of Underground Storage Tanks
PID	photoionization detector
PM	Project Manager
PQL	practical quantitation limit
QA	quality assurance
QAPP	Quality Assurance Project Plan
QC	quality control
RPD	relative percent difference
SW	EPA Solid Waste Test Method
TestAmerica	TestAmerica Laboratories, Inc.
TO	task order
TPH	total petroleum hydrocarbons
VOC	volatile organic compound
WA	Work Assignment

APPROVALS

By their signatures, the undersigned approve this Quality Assurance Project Plan.

Mary Millner — 11/5/2012
Mary Millner — Date
EPA Region 10 Subject Matter Expert

Gina Grepo-Grove — 11/5/2012
Gina Grepo-Grove — Date
EPA Region 10 Quality Assurance Manager

Patricia Curl — 11-2-12
Patricia Curl — Date
Bristol Program Manager

Julie Sharp-Dahl — 11/2/12
Julie Sharp-Dahl — Date
Bristol Project Manager

Terri Torres — 11/5/12
Terri Torres — Date
TestAmerica Analytical Services Project Manager

DISTRIBUTION LIST

Lynn Depont, EPA Administrative Task Order Manager

Mary Millner, EPA Region 10 Subject Matter Expert

Elizabeth Sanchey, Yakama Environmental Program Manager

Mark Koffler, Yakama Water Code Administration

Terri Torres, TestAmerica Analytical Services Project Manager

1.0 INTRODUCTION

Bristol Environmental Remediation Services, LLC (Bristol), is committed to performing environmental and engineering services that meet or exceed quality standards. Bristol is supporting the U.S. Environmental Protection Agency's (EPA's) Office of Underground Storage Tanks (OUST) mission of ensuring that leaking underground storage tanks (LUSTs) in the United States no longer pose a threat to human health and the environment. Bristol's Quality Assurance (QA) Program is designed to achieve these standards through professional training and the implementation of program/project QA plans and site-specific quality control (QC) activities.

(Intentionally blank)

2.0 PROJECT DESCRIPTION

Under this Task Order (TO) (No. B-7), Bristol will perform a Site Assessment at the Western Gas site, located on the Yakama Nation Indian Reservation, at 401 S. Elm Street, Toppenish, Washington.

2.1 APPLICABLE DOCUMENTS

To support the EPA OUST for this project, and to perform the work in the best way possible, Bristol will review available guidance documents. Some of the available documents include:

- Requirements for Quality Management Plans (QA/R-2) (EPA, 2001a)
- Requirements for Quality Assurance Project Plans (QA/R-5) (EPA, 2001b)
- American National Standards Institute/American Society for Quality Control (ANSI/ASQ) E4 -2004, Quality Systems for Environmental Data and Technology Programs: Requirements with Guidance for Use (ANSI, 2004)
- Guidance for Quality Assurance Project Plans (QA/G-5) (EPA, 2002a)
- Data Quality Assessment: A Reviewers Guide (QA/G-9R) (EPA, 2006a)
- Data Quality Assessment: Statistical Methods for Practitioners (QA/G-9S) (EPA, 2006b)
- Guidance on Systematic Planning Using the Data Quality Objectives Process (QA/G-4) (EPA, 2006c)
- Guidance on Environmental Data Verification and Data Validation (QA/G-8) (EPA, 2002b)
- Test Methods for Evaluating Solid Waste, Physical/Chemical Methods. SW-846 (EPA, 2007)

2.2 PROGRAM TASK DESCRIPTIONS

The EPA TO No. B-7 site assessment will include the following activities at the Western Gas LUST site:

- Install six geoprobe borings.
- Collect two grab soil samples from each boring.

- Redevelop up to three existing groundwater monitoring wells and collect groundwater samples.
- Install three temporary monitoring wells in three of the six geoprobe borings and collect groundwater samples.
- Transport and dispose of investigation-derived waste at an approved facility.

3.0 DATA QUALITY OBJECTIVES AND CRITERIA

The overall QA objective for this program is to develop and implement sampling, sample handling, and analytical procedures that will provide data which can be used to fulfill the data quality objectives (DQOs), as developed for the site in accordance with EPA QA/G-4: *Guidance on Systematic Planning Using the Data Quality Objectives Process* (EPA, 2006c). The DQOs are qualitative and quantitative statements developed by data users to specify the quality of data from field and laboratory data collection activities needed to support specific decisions or regulatory actions. The DQOs for the sites describe which data are needed, why the data are needed, and how the data will be used to meet the needs of the project. The DQOs also identify numeric limits for the data to allow the data user (or reviewers) to determine whether the data collected are of sufficient quality for their intended use.

The following sections discuss the site-specific details of the project, including types of information needed, the appropriate sampling and analysis methods, the area of the project, and the contaminants of concern and action levels.

3.1 SAMPLING PROCESS AND DESIGN

The number of samples to be collected is presented in the Field Sampling Plan (FSP) portion of the Site Assessment Plan.

3.2 DATA QUALITY OBJECTIVES

The DQO development, as described in EPA guidance, is based on:

- Describing the problem
- Identifying project objectives
- Specifying the data necessary to meet project objectives
- Describing the methods that will yield data of acceptable quality and quantity to support the required decisions

The objective of this project is to perform a site assessment at the Western Gas LUST site to determine the nature and extent of petroleum-related contamination at the site.

3.3 ANALYTICAL QUALITY CONTROL

The two general categories of data used by Bristol to support the TO are: 1) screening data and 2) definitive laboratory data.

3.3.1 Screening Data

Screening data are generated by rapid methods of analysis with less rigorous sample preparation, calibration, and/or QC requirements than are necessary to produce definitive laboratory data. Screening data are obtained by use of approved field equipment, such as combustible gas indicators, field test kits, total organic vapor analyzers (photoionization detectors [PIDs] and flame-ionization detectors), and geophysical survey instruments. Examples of how these data are to be used include area evaluation of contamination, gross evaluation of contaminant concentrations in samples, and health and safety screening.

Screening data may also provide analyte identification and quantitation, although the quantitation may be relatively imprecise. Examples of these kinds of data include Hanby system colorimetric data, topographical survey data, pH data, turbidity or dissolved oxygen data, and data collected using field gas chromatographs. When adequately supported and confirmed, screening data can be used for site characterization, evaluation of alternatives, engineering design, and monitoring during sampling.

3.3.2 Definitive Laboratory Data

Definitive laboratory data are quantitative, have known precision and accuracy such as EPA reference methods, and are produced under controlled conditions using laboratory-grade instrumentation. The data are analyte-specific, and both identification and quantitation are confirmed. Definitive laboratory data are developed under strict and

standardized QC and documentation requirements. Data uses include risk assessment, site characterization, evaluation of alternatives, and engineering design.

Screening data collected from the Western Gas site will include soil PID data. Definitive data includes the following analytical laboratory data:

- Total petroleum hydrocarbon (TPH): gasoline range organics (GRO) by Northwest Total Petroleum Hydrocarbon-gasoline (NWTPH-Gx)
- TPH: diesel range organics (DRO) and oil range organics (ORO) by Northwest Total Petroleum Hydrocarbon-diesel (NWTPH-Dx) (extended)
- Volatile organic compounds (VOCs) by EPA Solid Waste Test Method (SW) 8260B
- Polynuclear aromatic hydrocarbons (PAHs) by SW8270 SIM (selective ion monitoring)
- Lead by SW6020

The PID data will be used to guide soil sample locations within borings.

The QC that will be performed while obtaining field-screening data, such as PID measurements, is detailed in Sections 9 and 10, and includes the manufacturer's instructions for calibrating, maintaining, and utilizing the field equipment, including understanding the limitations of the equipment in a variety of field conditions (such as cold temperature, high humidity, or rain).

The laboratory's QA manual is included in Attachment 1. Analytical QC for the definitive data, which includes the soil samples that will be analyzed, is presented in Attachment 2.

All data are limited by the PQLs. The PQLs are based on the extent to which the equipment, laboratory or field, or analytical process can provide accurate measurements of reliable quality for specific constituents in field samples. The PQL for a given analysis will vary, depending on instrument sensitivity and matrix effects. The PQLs for the definitive

soil data are presented in Table 1. The PQLs for the definitive groundwater data are presented in Table 2.

Table 1 PQLs and Cleanup Goals for Soil

Analyte	EPA Method	TestAmerica PQLs (mg/kg)	MTCA Cleanup Levels (mg/kg)
Benzene	8260B	0.001	0.03
Toluene	8260B	0.002	7
Ethylbenzene	8260B	0.001	6
Naphthalene	8260B	0.005	5
Xylenes	8260B	0.002	9
1,2-Dibromoethane (EDB)	8260B	0.001	0.005
GRO	NWTPH-Gx	4.0	100[a] (30)
DRO	NWTPH-Dx extended	25	2,000
ORO	NWTPH-Dx extended	50	2,000
Naphthalene	8270C SIM	0.005	5[c]
1-Methylnaphthalene	8270C SIM	0.005	5[c]
2-Methylnaphthalene	8270C SIM	0.005	5[c]
Acenaphthene	8270C SIM	0.005	0.1[b]
Acenaphthylene	8270C SIM	0.005	0.1[b]
Phenanthrene	8270C SIM	0.005	0.1[b]
Anthracene	8270C SIM	0.005	0.1[b]
Fluorene	8270C SIM	0.005	0.1[b]
Benzo(a)anthracene	8270C SIM	0.005	0.1[b]
Chrysene	8270C SIM	0.005	0.1[b]
Fluoranthene	8270C SIM	0.005	0.1[b]
Pyrene	8270C SIM	0.005	0.1[b]

Table 1 PQLs and Cleanup Goals for Soil (continued)

Analyte	EPA Method	TestAmerica PQLs (mg/kg)	MTCA Cleanup Levels (mg/kg)
Benzo(b)fluoranthene	8270C SIM	0.005	0.1[b]
Benzo(k)fluoranthene	8270C SIM	0.005	0.1[b]
Benzo(a)pyrene	8270C SIM	0.005	0.1
Dibenzo(a,h)anthracene	8270C SIM	0.005	0.1[b]
Indeno(1,2,3-cd)pyrene	8270C SIM	0.005	0.1[b]
Benzo(g,h,i)perylene	8270C SIM	0.005	0.1[b]
Lead	6010	1.5	250[c]

Notes:

[a]Cleanup level for gasoline mixtures without benzene and the total of ethylbenzene, toluene, and xylenes are less than 1% of the gasoline mixture. The cleanup level for all other gasoline mixtures is 30 mg/kg.

[b]If the presence of other carcinogenic PAHs are suspected at the site, test for them using the toxicity equivalent methodology in the MTCA regulation of 0.1 mg/kg as the total concentration that all carcinogenic PAHs must meet.

[c]Cleanup level based on preventing unacceptable blood lead levels.

DRO = diesel range organics
EPA = U.S. Environmental Protection Agency
GRO = gasoline range organics
mg/kg = milligrams per kilogram
MTCA = Model Toxics Control Act
NWTPH-Gx = Northwest Total Petroleum Hydrocarbon-gasoline
NWTPH-Dx = Northwest Total Petroleum Hydrocarbon-diesel
ORO = oil range organics
PAHs = polynuclear aromatic hydrocarbons
PQLs = practical quantitation limits
SIM = selective ion monitoring
TestAmerica = TestAmerica Laboratories, Inc.

Table 2 PQLs and Cleanup Goals for Groundwater

Analyte	EPA Method	TestAmerica PQLs (mg/L)	MTCA Cleanup Levels (mg/L)
Benzene	8260B	0.001	0.005
Toluene	8260B	0.001	1.0
Ethylbenzene	8260B	0.001	0.7
Naphthalene	8260B	0.004	0.16
Xylenes	8260B	0.001	1.0[a]
1,2-Dibromoethane (EDB)	8260B	0.001	0.00001
GRO	NWTPH-Gx	0.05	0.8 [b] (1.0)
DRO	NWTPH-Dx extended	0.125	0.5
Lead	6020	0.0004	0.015

Notes:

[a]Cleanup level based on xylene not exceeding the maximum allowed cleanup level in this table for total petroleum hydrocarbons (TPH) and based on prevention of adverse aesthetic characteristics. This is a total value for all xylenes.

[b]The cleanup level is based on protection of groundwater for noncarcinogenic effects during drinking water use. Two cleanup levels are provided. The higher value is based on the assumption that no benzene is present in the groundwater sample. If any detectable amount of benzene is present in the groundwater sample, then the lower TPH cleanup level must be used.

DRO = diesel range organics
EPA = U.S. Environmental Protection Agency
GRO = gasoline range organics
mg/L = milligrams per liter
MTCA = Model Toxics Control Act
NWTPH-Dx = Northwest Total Petroleum Hydrocarbon-diesel
NWTPH-Gx = Northwest Total Petroleum Hydrocarbon-gasoline
PQLs = practical quantitation limits
TestAmerica = TestAmerica Laboratories, Inc.

All PQLs are less than the regulatory levels for the contaminants of concern (as shown in Tables 1 and 2). There is one exception. The PQL in water for 1,2-Dibromoethane is higher than the cleanup level. However, the soil PQL is below the cleanup level; therefore, if this compound is detected in soil, it may also be present in groundwater given the shallow depth to groundwater in this area.

Concentrations that fall between the method detection limit and the PQL will be J-flagged as estimated. The matrix spike/matrix spike duplicate (MS/MSD) and laboratory

control samples/laboratory control sample duplicates (LCS/LCSD) acceptance ranges, MS/MSD and LCS/LCSD relative percent difference (RPD) limits, and surrogate recovery acceptance ranges for soil are included in Attachment 2.

Field QC for the screening and definitive data that will be generated at the analytical laboratory will be established through the submission of trip blanks and MS/MSDs, as detailed in Section 6.0.

(Intentionally blank)

4.0 SPECIAL TRAINING AND CERTIFICATIONS

Bristol will conduct the confirmation soil sampling. Bristol personnel meet the minimum qualifications established for their particular job and functional discipline. Senior managers are responsible for assigning only qualified personnel to project activities. Depending on specific project task needs, locations, or conditions, Bristol will also provide staff (or subcontractors) with special training or certifications that may include:

- Hazardous Waste Operations and Emergency Response Training
- Resource Conservation and Recovery Act Hazardous Waste Regulations
- Site-specific Health and Safety Training, including Hazard Communication, Respiratory Protection, Personal Protective Equipment, and other topics

(Intentionally blank)

5.0 DOCUMENTS AND RECORDS

5.1 QUALITY DOCUMENTS AND RECORDS

The Bristol Program Manager and Project Manager (PM) are responsible for project document control. All completed project records will be maintained in a secure location in the Bristol Anchorage, Alaska office where they can be accessed as necessary and protected from deterioration. Hard-copy and electronic data will be archived in project files and on electronic media for the duration of the project or a minimum of five years, whichever is longer.

Bristol will maintain electronic and hard-copy records sufficient to recreate each analytical event conducted pursuant to the TO. At a minimum, the records will contain the following:

- TO Quality Assurance Reports
- Annual Quality Assurance Reports;
- Chain-of-custody (CoC) forms
- Sample logs
- Initial and continuing calibration records
- Instrument tuning records (as applicable)
- Method blank results
- Spike and spike duplicate records and results
- Laboratory records/QC reports
- Raw data, including instrument printouts, work sheets, and quantitation reports
- Other corrective action reports
- Other method- and project-required QC sample records and results

5.2 DRAFT AND FINAL REPORTS

All key project-related documents, such as the field notebook, site photographs, other relevant project information such as EPA-provided reports, and the project completion reports, will be kept in the project files.

(Intentionally blank)

6.0 DATA GENERATION AND ACQUISITION

6.1 SAMPLING PROCEDURES

All of the sampling locations and procedures to be used for environmental sample collection are presented in the FSP. The FSP describes in detail the procedures that will be followed during sampling to ensure that the data are representative of environmental conditions.

All sample containers to be used for screening data collection will be purchased in pre-cleaned condition and stored in a clean, moderate temperature environment before sample collection. All samples will be labeled, handled, and shipped following the procedures outlined in Section 7 of this Quality Assurance Project Plan (QAPP).

For samples collected to generate definitive laboratory data, the National Environmental Laboratory Accreditation Conference (NELAC) accredited laboratory will provide sample containers. The types of sample containers and preservation required for each matrix and analysis are outlined in the FSP, as specified by the EPA reference method to be used.

6.2 QC SAMPLE COLLECTION

As discussed above, the sampling procedures for all of the environmental samples are described in the FSP. The following sections outline the QC samples that will be collected for this TO.

6.2.1 Equipment Blanks

Equipment blanks will be collected at a rate of one per day when non-dedicated or non-disposable equipment is used for sampling. However, Bristol does not anticipate the use of non-dedicated equipment during the sampling of any media. If equipment blanks are collected, they will be collected for each analytical parameter for which the associated environmental sample was collected. Equipment blanks will be collected immediately

after decontaminating sampling equipment by pouring deionized water over the sampling equipment, then collecting it in the appropriate sample containers.

6.2.2 Duplicate Samples

During this sampling program, duplicate samples will be collected as a percentage of the total number of soil samples taken for each scheduled analytical method. One QC (duplicate) sample will be collected for every 10 soil samples collected.

Duplicate sample results are used to assess precision of the sample collection process. A field duplicate sample is a second sample collected at the same location as the original sample. When used, duplicate samples are collected simultaneously or in immediate succession using identical recovery techniques and are treated in an identical manner during storage, transportation, and analysis, as the primary samples.

Duplicate sampling is often used to determine the precision of soil sample analysis.

6.2.3 Matrix Spike and Matrix Spike Duplicate Samples

The MS/MSD samples are designed to evaluate whether there are site-specific matrix interferences with the analytes, and to determine the overall accuracy of analytical results.

One soil MS/MSD pair will be collected for every 20 soil samples collected at the site for laboratory analysis. The MS samples produced in the laboratory for percent recovery studies will be conducted in accordance with the project laboratory's standard QC protocols.

6.2.4 Trip Blanks

Before any sampling, trip blanks will be prepared by the laboratory. Trip blanks will accompany all soil samples scheduled for VOC or GRO analysis. The trip blanks will

consist of two VOA vials filled with preservative and Ottawa sand. The bottles will be filled so that there is no headspace and will be capped with a Teflon® septum lid.

6.2.5 Temperature Blanks

A temperature blank will be kept in each sample cooler. The temperature blank verifies the proper storage requirement during transport of the samples and must be measured upon arrival at the laboratory.

(Intentionally blank)

7.0 SAMPLE CUSTODY, HANDLING, AND SHIPPING PROCEDURES

7.1 SAMPLE CUSTODY

To ensure that samples are identified correctly, the documentation and sample custody procedures specified in this section will be followed during sample collection and analysis. Standard sample documentation and custody procedures, as outlined below, will be used to maintain and document sample integrity during collection, transportation, storage, and analysis. The PM will be responsible for ensuring that proper documentation and custody procedures are initiated at the time of sample collection and that individual samples can be tracked from the time of sample collection until the samples are relinquished to the laboratory.

Bristol will be in custody of the samples at all times until the samples are signed over (on a chain-of-custody form) to the laboratory. The laboratory will be responsible for maintaining sample custody and documentation from the time the samples are relinquished to the laboratory, until final sample disposition.

7.2 CHAIN-OF-CUSTODY

The CoC procedures provide an accurate, written record of the possession of each sample from the time of collection in the field through laboratory analysis. A sample is considered in custody if one of the following applies:

- It is in an authorized person's immediate possession
- It is in view of an authorized person after being in physical possession
- It is in a secure area after having been in physical possession of an authorized person
- It is in a designated secure area, restricted to authorized personnel only

7.3 FIELD PROCEDURES

The sample custody and documentation procedures will be initiated at the time of sample collection. Sample collection details will be documented in a field notebook. Samples

will be labeled and the appropriate information will be recorded on the CoC form using indelible ink. Any errors will be corrected by drawing a single line through the incorrect entry, entering the correct information, and then initialing and dating the change.

7.4 SAMPLE LABELS

Sample labels will be completed and attached to sample containers at the time of sample collection. The following information will be included on the sample label:

- Project name/location
- Sample location
- Field sample identification
- Date and time of sample collection
- Type of analyses to be performed
- Preservative (if applicable)
- Sampler's initials

7.5 CHAIN-OF-CUSTODY RECORD

Properly completed CoC forms will ensure that sample custody is documented, appropriate sample fractions have been collected, and scheduled analyses are properly assigned. Unused portions of the CoC form will be crossed out and initialed. A completed CoC record will be included with each sample cooler. The sampler will retain a copy of the CoC. When shipping the sample cooler to the laboratory by a commercial carrier, the CoC will be signed, placed in a plastic bag, and taped to the inside of the shipping container used for sample transport. Signed airbills will serve as evidence of custody transfer between the field sampler and courier, and courier and laboratory. The sampler will retain and file copies of the CoC record and the airbill after the samples are shipped. The samples are relinquished to the laboratory upon arrival, and the laboratory personnel will then complete the CoC.

7.6 SAMPLE HANDLING AND SHIPPING

After each soil sample is collected, the samples will be placed in a cooler containing ice for shipment to the designated project laboratory. The samples will be placed upright in the cooler and then secured with inert cushioning material to prevent breakage. A completed CoC form will accompany all samples. Complete packaging and shipping procedures are as follows:

- The samples will be placed upright in a waterproof ice chest or cooler.
- Ice will be placed around, among, and on top of the sample bottles. Enough ice will be used so that the samples will be maintained at 4 degrees Celsius.
- To prevent the sample containers from sliding around the cooler, the cooler will be filled with inert cushioning material, such as shipping peanuts, additional bubble pack, or cardboard dividers.
- The completed CoC form will be placed in a waterproof plastic bag and taped to the inside of the cooler lid.
- The lid will be secured with strapping tape or duct tape by wrapping it completely around the cooler.
- The completed shipping label will be attached to the top of the cooler and "This Side Up" and "Fragile" labels will be placed on the side of the cooler.

(Intentionally blank)

8.0 ANALYTICAL PROCEDURES AND DETECTION LIMITS

Laboratories accredited under the EPA National Environmental Laboratory Accreditation Program, or authorized by the EPA Work Assignment Manager, will analyze all samples collected for definitive laboratory data. The contaminants of concern, analytical approach, and sampling rationale for the Work Assignment (WA) are described in the FSP. At a minimum, all samples will be prepared and analyzed using *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, EPA Publication SW-846* (EPA, 2007), or an equivalent methodology. A NELAC-approved laboratory, TestAmerica Laboratories, Inc. (TestAmerica), located in Tacoma, Washington, will be used. A copy of TestAmerica's QA Manual is included as Attachment 1.

(Intentionally blank)

9.0 PREVENTATIVE MAINTENANCE

The field equipment that may be used during site investigations, as described in Section 10.1, will be maintained and used according to the manufacturers' directions. Each piece of equipment will be inspected on a regular basis to ensure that the equipment is operational. Any repair conducted in the field will be recorded in the field logbook.

9.1 INSTRUMENT INSPECTION

All instruments will be properly maintained so that they will provide reliable data when used. Instruments that will be used in the field and that will require periodic maintenance, testing, and inspection include the PID.

This instrument will be inspected and tested by Bristol at the beginning and end of the workday. The PID will be calibrated at the beginning of the workday, as described in Section 10. A set of new batteries will accompany the instrument and additional batteries, if needed, can be purchased near the work site.

If the PID requires repair by the field sampler, those procedures will be documented in the fieldbook.

For laboratory instrumentation testing, inspections, and maintenance, refer to TestAmerica's QA manual, located in Attachment 1.

(Intentionally blank)

10.0 PERFORMANCE CHECK/CALIBRATION PROCEDURES

This section discusses general requirements for performance checks and calibration of field equipment. Criteria are specific to each method and instrument manufacturer. Any performance checks or calibrations will be documented in the fieldbook. The following paragraphs outline the procedures for the field equipment anticipated to be used in this program.

10.1 PHOTOIONIZATION DETECTOR

Field personnel will use a PID for field screening soil samples. The PID will be calibrated each day in accordance with the manufacturer's specifications. The calibrations and checks will be documented in the field notebook, so that they are traceable to the equipment used. These data will be used to evaluate any drift of variability and may be used to assess impacts to the screening data collected.

10.2 SUPPLIES AND CONSUMABLES

The Bristol Field Task Manager is responsible for checking that all field supplies and consumables have been inspected before their use in the field. In addition to checking that all supplies and consumables have been packaged and stored appropriately, special attention will be directed toward ensuring that materials subject to expiration dates, or limited shelf life, will be used within their appropriate time frames. Commercial materials with a limited shelf life, or expiration date, will not be accepted for use if the shelf life or expiration date is within 30 days before intended usage.

(Intentionally blank)

11.0 DATA MANAGEMENT AND ASSESSMENT PROCEDURES

Field data sheets and sampling forms are reviewed and checked by the Field Task Manager before transfer to the Bristol PM. Field samples are checked by the Field Task Manager (designated by the PM) for label completion before leaving the site and being transferred to the laboratory. The Field Task Manager is identified on the CoC and is responsible for the samples until the laboratory receives them. The laboratory is then responsible for testing, disposing of, recording, and transferring the sample analysis data to the Bristol PM. It is the PM's responsibility to coordinate with the QA Manager for review of the data once received from the laboratory. Upon data acceptance by the QA Manager, the PM is responsible for including the data in the Site Characterization Report.

The DQO procedures utilize the data quality indicator's (DQI's) precision, bias (accuracy), representativeness, comparability, completeness, and sensitivity, as guides for evaluating the acceptability and utility of the laboratory analytical data.

11.1 FIELD DATA

The PM and QA Manager or designee will assess field measurement data. The data quality evaluation, in terms of the DQIs, will focus primarily on the laboratory data. However, the field data will be evaluated qualitatively in terms of the DQIs. The following sections discuss how the DQIs will be used to evaluate the field data and field sampling procedures.

11.1.1 Precision

Precision is a measure of the reproducibility of concentrations reported for duplicate soil samples collected from the same location. Precision is calculated by determining the RPD between duplicate samples. The precision objective for field duplicates is an RPD of 33 percent. The RPD is calculated by using the following formula:

$$RPD = \frac{x_1 - x_2}{(x_1 + x_2)/2} x100$$

11.1.2 Accuracy

Accuracy is the degree to which the measurement data approach the "true" value for each analyte. Accuracy is assessed by calculating the percent recovery for a sample spiked with the analyte of concern (laboratory control samples, matrix spike surrogates).

11.1.3 Representativeness

Representativeness refers to the comparability of the sample collection procedures to those delineated in the Work Plan and the degree to which the analytical data represent the subsurface contaminant concentrations. Representativeness will be accomplished by adhering to consistent field sampling and analytical procedures for soil samples. Representativeness of the data will be evaluated by ensuring the following:

- Sample screening and collection procedures consistently followed in Section 3 of the Site Assessment Plan;
- Analytical data was generated per the CoC and per the laboratory's control limits described in Attachment 2.

11.1.4 Completeness

Completeness is defined as the ratio of acceptable, validated laboratory measurements to the total number of planned measurements for each investigation. The completeness objective for all samples collected during these investigations will be at 90 percent for

$$Percent\ Completeness = \left(\frac{Number\ of\ Validated\ Measurements}{Total\ Number\ of\ Planned\ Measurements} \right) * 100$$

each task.

11.1.5 Comparability

Comparability is an evaluation of the relative consistency of the laboratory measurement data. Since comparability cannot be measured quantitatively, professional judgment is relied upon. Internal comparability will be achieved by adhering to consistent sample collection procedures and analysis methods throughout each investigation.

11.1.6 Sensitivity

Sensitivity is the capability of a method or instrument to discriminate between measurement responses representing different levels of the variable of interest.

Any measurements made during the project will employ only methods and techniques determined to produce measurement data of a known and verifiable quality sufficient to meet the overall objectives of the project.

The PQLs presented in Attachment 2 and in Tables 1 and 2 are applicable for the analytical laboratory testwork.

11.2 LABORATORY DATA

Data package documentation from the laboratories will be implemented as prescribed by site-specific needs. Summary QC documentation will be obtained from the laboratory. Summary QC documentation will include the QA/QC data listed below for each parameter analyzed, and will be included with both hard copies of the analytical report and with electronic deliverables.

11.2.1 Level II Data Deliverables

Case narrative – The case narrative will address any QC deficiencies associated with the data set contained in the associated report, and provide a cross-reference for field and laboratory sample identification codes:

- CoC documentation
- Analytical results for each parameter requested

- Laboratory-established precision and accuracy limits
- Dates of sample collection, preparation, and analysis
- Method blank – matrix specific
- LCS, LCSD results and RPDs
- MS/MSD analysis and calculated RPDs
- Surrogate recoveries

12.0 CHARACTERIZATION AND OVERSIGHT

Internal QC evaluates whether a method is performing within acceptable limits of precision and accuracy. On the sampling level, QC samples used to assess field-sampling techniques and environmental conditions during sample collection and transportation include blind duplicates, trip blanks, and equipment blanks.

Oversight of field sampling techniques will be performed by qualified field personnel who have had formal, informal, classroom, and on-the-job training. Personnel will adhere to set sampling procedures documented in the FSP. Sampling will be performed to ensure that the collection of data is representative of conditions at the site. Cross-contamination will be prevented through use of appropriate personal protective equipment, decontamination procedures, and proper sampling techniques. Sampling will be performed using proven, accurate, and reproducible techniques, unless nonconventional sampling and analytical methods are otherwise required by the EPA.

Oversight of field sampling procedures may include conducting audits, verifying documentation, and conducting pilot studies, where appropriate. Audits may be performed to ensure that the quality of the final product achieves the DQOs for a project. Data documentation will be reviewed for accuracy by qualified personnel. Pilot studies may be used to evaluate the logistics, equipment, sampling plans, and analytical protocols before a full-scale study is implemented.

12.1 PERFORMANCE SYSTEM AUDITS

Oversight of field procedures will be the direct responsibility of the Bristol PM, who will review all elements of the QAPP to ensure that the objectives of the TO are met. In addition to an initial review, the sampling procedures will be reviewed as the fieldwork progresses, so that any necessary modifications can be made. If any problems relating to

the sampling procedures are discovered, they will be communicated by the Bristol PM to the Bristol Program Manager and QA Manager for resolution.

Internal audits of field activities (sampling and measurements) will be conducted by the Bristol QA Manager or designee following each field event. The audits will include examining field measurement records, field equipment calibration records, field sampling records, field instrument operation records, sample collection procedures, sample handling and shipping procedures, and CoC procedures. Field activities will be audited following each field event to verify that all of the procedures outlined in the FSP and QAPP are being followed. Where warranted, follow-up audits will be conducted to correct deficiencies and to verify that QA procedures are maintained throughout the project.

12.2 Corrective Actions

The field staff will be responsible for documenting and reporting all suspected technical and QA nonconformances and suspected deficiencies during any field activity. The nonconformances and/or deficiencies will be documented in the field logbook and reported to the Project Manager. If the problem is associated with field measurements or sampling equipment, the field staff will take the appropriate steps to correct the problem. Typical field procedures to correct problems include the following:

- Repeating the measurement to check for error
- Assuring that the meters or instruments are adjusted properly for the ambient conditions, such as temperature
- Checking or replacing batteries
- Recharging batteries
- Recalibrating the instruments
- Replacing the meters or provisions used to measure field parameters
- Stopping work until the problem is corrected (if necessary)

If a nonconformance or problem requires a major adjustment to the field procedures outlined in the FSP (e.g., changing sampling methodology), the PM, in conjunction with the QA Manager, will be responsible for initiating corrective actions. The PM will be responsible for the following:

- Evaluating the reported nonconformance
- Controlling additional work on nonconforming items
- Determining the appropriate corrective actions
- Maintaining a log of all nonconformances and corrective actions
- Ensuring that explanations of nonconformances and corrective actions are included in an appendix of the Summary Report scheduled for this investigation

The PM will ensure that no additional work that is dependent on the nonconforming activity is performed until the appropriate corrective actions are completed.

12.3 QUALITY ASSURANCE REPORTS

All of the analytical data collected during the investigation will be presented to the EPA PM once the quality of the data has been checked. The data verification for all data to be included in final reports will be presented in a QA Report, which includes the following:

- Sampling procedures (planned and implemented, problems, and corrective actions)
- Analytical procedures and detection limits
- Analytical data (environmental and QC sample results)
- Results of the data quality evaluation
- Conclusions and recommendations

Bristol will check the quality of the analytical data by verifying that all of the data is in accordance with EPA/240/R-02/004 QA/G-8: *Guidance on Environmental Data Verification and Data Validation* (EPA, 2002b). The purpose of the verification will be to determine the usability of the data. At a minimum, the following items will be checked as part of data verification:

- Sample receipt conditions (sample preservation, cooler temperatures, CoC conditions, and custody seals)
- Sampling procedures (trip blanks, equipment blanks, and QA/QC samples)
- Adherence to method criteria and project DQOs
- Adherence to holding time requirements
- Meeting analytical QC criteria, checking the following parameters against their respective criteria:
  - Surrogate recovery
  - Blank spike LCS and LCSD recoveries
  - RPD of LCS and LCSD
  - MS and MSD recoveries
  - RPD of MS and MSD
  - RPD of blind duplicates (field replicates)
- Close scrutiny of anomalies and issues identified in the laboratory's case narrative to determine their overall impact on the quality of data
- The adherence to all other criteria set forth in this QAPP
- An objective overall assessment as to whether the data make sense for what is known of the site and conditions

Additional items that may be reviewed upon request of the EPA and normally provided in data validation include:

- System performance criteria (instrument blanks, continuing calibration verification standards, internal standards, and instrument tunes for mass spectrometry methods)
- Instrument calibrations
- Raw data for samples, extraction QC samples, and QA/QC samples

Accuracy will mainly be determined by reviewing surrogate recoveries. Precision will mainly be determined by reviewing the results of blind duplicates. A data verification report will be generated, which includes the items outlined above and a listing of all sample results. Data that require flagging will be assigned appropriate data qualifiers.

13.0 DATA VERIFICATION AND USABILITY

All data obtained from field and laboratory measurements will be reviewed and verified for integrity and continuity, reasonableness, and conformance to WA requirements, and then verified against the DQOs developed for the WA. Data not supported by appropriate QC data, or which does not meet the defined DQOs, will be reported to the EPA with the appropriate qualifiers.

The Bristol PM is responsible for ensuring that field data are properly reviewed, verified, and submitted for reporting purposes. The Bristol QA Manager is responsible for ensuring that the data are verified.

All data will be verified to ensure that they are representative of the samples analyzed and locations where measurements were made, and that the data and associated QC data conform to project specifications. Verification of data will be performed using self-assessments and peer review, as appropriate to the TO, followed by technical review by the Bristol PM. The data to be verified are evaluated against TO specifications and are checked for errors in transcription, calculations, and data input. Potential outliers are identified by examination for unreasonable data or, in rare instances, are identified using computer-based statistical software. If a question arises or an error or potential outlier is identified, the PM responsible for generating the data will be contacted to resolve the issue.

Issues that can be corrected will be corrected and documented electronically, or by initialing and dating the associated paperwork. If an issue cannot be corrected, the Bristol PM will consult with the Program Manager, QA Manager, or Chief Executive Officer, to establish the appropriate course of action, or the data associated with the issue will be rejected.

The Bristol QA Manager is responsible for determining whether the verified data meet the measurement performance criteria and are reportable to the EPA. The verification process involves the QA Manager (or designee) evaluating the data again for anomalies. The PM must satisfactorily address any suspected errors or anomalous data identified during this review before data verification can be completed.

A chemist will perform Data Verification of the data provided by the laboratory. The chemist has data validation and analytical laboratory experience and is familiar with the QC requirements for the analytical methods being reviewed. An AECOM chemist will be responsible for the data review, as an independent third party reviewer.

Data verification precedes data validation and is a systematic process for evaluating whether data have been generated with acceptable QC, as defined in the QAPP.

The verification is a review of only the items listed below, as well as a check of the completeness of supporting documentation. This is a cursory review of the laboratory's QC and may suggest that a more thorough validation is needed.

- Timeliness and a Check for Errors, including requested deliverables, preservation, holding times, and CoC
- Blank Results and Checks for Contamination, including trip, method, and field blanks, reviewed against the QAPP's criteria for representativeness
- Surrogate and Internal Standard Recovery, where appropriate
- LCS Analysis, including QC Check Samples, standard, and LCS duplicate results
- MS/MSD Analysis, reviewed against precision and accuracy criteria specified by the method or by project DQIs
- Sample/Duplicate Sample Analysis, reviewed against precision criteria specified by the method or by project DQIs
- Compound Quantitation and Reported Detection Limits, checking reporting limits against the contract required limits
- Field Duplicates and Other QC Samples, if samples are identified, reproducibility will be evaluated

- An Overall Summary Assessment of Data Use

13.1 Field Measurements

Raw data from field measurements and sample collection activities will be documented in the field logbook and on the appropriate forms, as described in the FSP. The field measurements and data collected during sampling will be presented in the project report to the EPA. All field data generated during this investigation will be evaluated under the direction of the QA Manager before they are incorporated in the final report.

13.2 Laboratory Measurements

The laboratory review of the data includes assessing compliance with criteria, as set forth in the QAPP. The control limits for the recoveries of surrogates, LCS/LCSDs, and MS/MSDs, are presented in Attachment 2. The RPDs for the LCS/LCSDs and MS/MSDs are also in Attachment 2. The assessment of accuracy will mainly be determined by surrogate recoveries. The assessment of precision will mainly be done by blind duplicates. The RPD criteria for blind field duplicate samples will be 50 percent. The MS/MSD data will be used to assess the effect of the matrix on the analysis and can also be used for precision.

13.2.1 Laboratory Data Reporting

The analytical data will be reported in a format organized to facilitate data evaluation. All of the data, including QC data, will be reported in the chronological order in which they were produced. The following information will be included in each data package:

- A cover sheet that identifies the samples contained in the report, including QC samples and samples that do not meet QA/QC criteria
- A report for each completed environmental and QC sample analysis (equipment blanks, source-water blanks, LCSs, surrogate spike samples, and method blanks) that includes the following information:
  - Field sample identification (ID) number (if applicable)
  - Laboratory ID number

- Date the sample was collected
- Date the sample was received by the laboratory
- Date and method of sample extraction (if applicable)
- Date and method of sample analysis
- Tabulated results for each sample
- Surrogate spike recoveries (if applicable)
- Internal standard recoveries (if applicable)
- Associated method blank results
- Detection limit for each analyte
- Initial concentration of the surrogate spikes, MSs, and LCS spikes, as well as the percent recovery and acceptance limits of each spiked analyte
- Identification of samples analyzed in association with each QC sample

14.0 QUALITY CONTROL REVIEW

The work process will be monitored using inspections, tests, QC reviews, peer reviews, and audits. Documents prepared by Bristol staff should be reviewed for technical content by a member of Bristol's senior management. Documents will also be reviewed by a technical editor prior to submittal to the EPA. Persons conducting QC reviews will be technically qualified to perform QC reviews.

(Intentionally blank)

15.0 REFERENCES

American National Standards Institute. 2004. *ANSI/ASQ E4 - 2004. Quality Systems for Environmental Data and Technology Programs: Requirements with Guidance for Use*.

Bristol. 2011. *Status Memo Summary of the Current Status of LUST Site No. 4260105-Toppenish Shell (Brand X), Yakama Nation Indian Reservation, Washington.* March 15.

Bristol. 2012. *Summary of Activities for Task Order B-7 Toppenish Shell, Toppenish, Washington Period of August 8, 2012 Through April 18, 2014.* August 27.

EPA. 2001a. *Requirements for Quality Management Plans. QA/R-2*. Office of Environmental Information. EPA/240/B-01/002. March 2001.

EPA. 2001b. *Requirements for Quality Assurance Project Plans. QA/R-5*. Office of Environmental Information. EPA/240/B-01/003. March 2001.

EPA. 2002a. *Guidance for Quality Assurance Project Plans. QA/G-5M*. Office of Environmental Information. EPA/240/R-02/007. December 2002.

EPA. 2002b. *Guidance on Environmental Data Verification and Data Validation. QA/G-8*. Office of Environmental Information. EPA/240/R-02/004. November 2002.

EPA. 2006a. *Data Quality Assessment: A Reviewer's Guide. QA/G-9R*. Office of Environmental Information. EPA/240/B-06/002. February 2006.

EPA. 2006b. *Data Quality Assessment: Statistical Methods for Practitioners. QA/G-9S*. Office of Environmental Information. EPA/240/B-06/003. February 2006.

EPA. 2006c. *Guidance on Systematic Planning Using the Data Quality Objectives Process. QA/G-4*. Office of Environmental Information. EPA/240/B-06/001. February 2006.

EPA. 2007. *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods.* EPA Publication SW-846.

(Intentionally blank)

ATTACHMENT 1

TestAmerica Laboratories, Inc.
Quality Assurance Manual
(Provided on CD)

ATTACHMENT 2

Analytical Control Limits

Analysis Group Description	Method Description	Method Code
Soil	Northwest - Semi-Volatile Petroleum Products (GC)	NWTPH_Dx

Analyte Description	CAS Number	RL - Limit	MDL - Limit	Units	LCSREC - Recovery Low	LCSREC - Recovery High
o-Terphenyl	84-15-1			mg/Kg		
#2 Diesel (C10-C24)	STL00163	25	5.7	mg/Kg	70	125
Motor Oil (>C24-C36)	STL00299	50	9.1	mg/Kg	64	127

Soil	Ultrasonic Extraction	3550B
Soil	Northwest - Volatile Petroleum Products (GC)	NWTPH_Gx

Analyte Description	CAS Number	RL - Limit	MDL - Limit	Units	LCSREC - Recovery Low	LCSREC - Recovery High
4-Bromofluorobenzene (Surr)	460-00-4			mg/Kg		
Gasoline	STL00228	4	0.5	mg/Kg	68	120
Trifluorotoluene (Surr)	98-08-8			mg/Kg		
1-Methylnaphthalene	90-12-0			mg/Kg		
Toluene	108-88-3			mg/Kg		
1,4-Difluorobenzene	540-36-3			mg/Kg		

Soil	Closed System Purge and Trap	5035A_FM
Soil	Volatile Organic Compounds (GC/MS)	8260B

Analyte Description	CAS Number	RL - Limit	MDL - Limit	Units	LCSREC - Recovery Low	LCSREC - Recovery High
Fluorobenzene (Surr)	462-06-6			ug/Kg		
Toluene-d8 (Surr)	2037-26-5			ug/Kg		
Ethylbenzene-d10	25837-05-2			ug/Kg		
Dichlorodifluoromethane	75-71-8	1	0.3	ug/Kg	35	135
Chloromethane	74-87-3	1	0.3	ug/Kg	50	130
Vinyl chloride	75-01-4	1	0.3	ug/Kg	60	125
Bromomethane	74-83-9	1	0.4	ug/Kg	30	160
Chloroethane	75-00-3	1	0.3	ug/Kg	40	155
Trichlorofluoromethane	75-69-4	1	0.3	ug/Kg	25	185
1,1-Dichloroethene	75-35-4	5	0.3	ug/Kg	65	135
Methylene Chloride	75-09-2	15	3.2	ug/Kg	55	140
trans-1,2-Dichloroethene	156-60-5	1	0.3	ug/Kg	65	135
1,1-Dichloroethane	75-34-3	1	0.4	ug/Kg	75	125
2,2-Dichloropropane	594-20-7	1	0.4	ug/Kg	65	135
cis-1,2-Dichloroethene	156-59-2	1	0.3	ug/Kg	65	125
Chlorobromomethane	74-97-5	1	0.3	ug/Kg	70	125
Chloroform	67-66-3	1	0.3	ug/Kg	70	125
1,1,1-Trichloroethane	71-55-6	1	0.4	ug/Kg	70	135
Carbon tetrachloride	56-23-5	1	0.4	ug/Kg	65	135

1,1-Dichloropropene	563-58-6	1	0.4	ug/Kg	70	135
Benzene	71-43-2	1	0.3	ug/Kg	75	125
1,2-Dichloroethane	107-06-2	1	0.3	ug/Kg	70	135
Trichloroethene	79-01-6	1	0.3	ug/Kg	75	125
1,2-Dichloropropane	78-87-5	1	0.3	ug/Kg	70	120
Dibromomethane	74-95-3	1	0.3	ug/Kg	75	130
Dichlorobromomethane	75-27-4	1	0.3	ug/Kg	70	130
cis-1,3-Dichloropropene	10061-01-5	1	0.3	ug/Kg	70	125
Toluene	108-88-3	2	0.3	ug/Kg	70	125
trans-1,3-Dichloropropene	10061-02-6	1	0.3	ug/Kg	65	125
1,1,2-Trichloroethane	79-00-5	1	0.3	ug/Kg	60	125
Tetrachloroethene	127-18-4	1	0.3	ug/Kg	65	140
1,3-Dichloropropane	142-28-9	1	0.3	ug/Kg	75	125
Chlorodibromomethane	124-48-1	1	0.3	ug/Kg	65	130
Ethylene Dibromide	106-93-4	1	0.3	ug/Kg	70	125
Chlorobenzene	108-90-7	1	0.3	ug/Kg	75	125
Ethylbenzene	100-41-4	1	0.3	ug/Kg	75	125
1,1,1,2-Tetrachloroethane	630-20-6	1	0.3	ug/Kg	75	125
1,1,2,2-Tetrachloroethane	79-34-5	2	0.3	ug/Kg	55	130
m-Xylene & p-Xylene	179601-23-1	2	0.6	ug/Kg	80	125
o-Xylene	95-47-6	1	0.3	ug/Kg	75	125
Styrene	100-42-5	2	0.4	ug/Kg	75	125
Bromoform	75-25-2	1	0.3	ug/Kg	55	135
Isopropylbenzene	98-82-8	2	0.3	ug/Kg	75	130
Bromobenzene	108-86-1	2	0.3	ug/Kg	65	120
N-Propylbenzene	103-65-1	1	0.3	ug/Kg	65	135
1,2,3-Trichloropropane	96-18-4	1	0.4	ug/Kg	65	130
2-Chlorotoluene	95-49-8	2	0.3	ug/Kg	70	130
1,3,5-Trimethylbenzene	108-67-8	5	0.4	ug/Kg	65	135
4-Chlorotoluene	106-43-4	2	0.4	ug/Kg	75	125
tert-Butylbenzene	98-06-6	2	0.4	ug/Kg	65	130
1,2,4-Trimethylbenzene	95-63-6	2	0.4	ug/Kg	65	135
sec-Butylbenzene	135-98-8	2	0.4	ug/Kg	65	130
1,3-Dichlorobenzene	541-73-1	1	0.4	ug/Kg	70	125
4-Isopropyltoluene	99-87-6	2	0.4	ug/Kg	75	135
1,4-Dichlorobenzene	106-46-7	1	0.4	ug/Kg	70	125
n-Butylbenzene	104-51-8	2	0.4	ug/Kg	65	140
1,2-Dichlorobenzene	95-50-1	1	0.4	ug/Kg	75	120
1,2-Dibromo-3-Chloropropane	96-12-8	2	0.3	ug/Kg	40	135
1,2,4-Trichlorobenzene	120-82-1	2	0.4	ug/Kg	65	130

1,2,3-Trichlorobenzene	87-61-6	2	0.3	ug/Kg	60	135
Hexachlorobutadiene	87-68-3	1	0.4	ug/Kg	55	140
Naphthalene	91-20-3	5	0.3	ug/Kg	40	125
4-Bromofluorobenzene (Surr)	460-00-4			ug/Kg		
Trifluorotoluene (Surr)	98-08-8			ug/Kg		
Methyl tert-butyl ether	1634-04-4	1	0.3	ug/Kg	65	125

Soil	Closed System Purge and Trap	5035A_FW
Soil	Semivolatile Organic Compounds (GC/MS SIM)	8270C_SIM

Analyte Description	CAS Number	RL - Limit	MDL - Limit	Units	LCSREC - Recovery Low	LCSREC - Recovery High
1,4-Dichlorobenzene-d4	3855-82-1			ug/Kg		
Naphthalene-d8	1146-65-2			ug/Kg		
Acenaphthene-d10	15067-26-2			ug/Kg		
Phenanthrene-d10	1517-22-2			ug/Kg		
Chrysene-d12	1719-03-5			ug/Kg		
Perylene-d12	1520-96-3			ug/Kg		
Nitrobenzene-d5	4165-60-0			ug/Kg		
2-Fluorobiphenyl	321-60-8			ug/Kg		
Terphenyl-d14	1718-51-0			ug/Kg		
Naphthalene	91-20-3	5	2	ug/Kg	64	129
2-Methylnaphthalene	91-57-6	5	2	ug/Kg	65	125
1-Methylnaphthalene	90-12-0	5	1.5	ug/Kg	48	148
Acenaphthylene	208-96-8	5	1.5	ug/Kg	69	129
Acenaphthene	83-32-9	5	1.5	ug/Kg	65	130
Fluorene	86-73-7	5	1.5	ug/Kg	68	128
Phenanthrene	85-01-8	5	1.5	ug/Kg	65	125
Anthracene	120-12-7	5	1.5	ug/Kg	73	123
Fluoranthene	206-44-0	5	1.5	ug/Kg	61	121
Pyrene	129-00-0	5	1.5	ug/Kg	54	134
Benzo[a]anthracene	56-55-3	5	1.5	ug/Kg	64	124
Chrysene	218-01-9	5	1.5	ug/Kg	71	126
Benzo[b]fluoranthene	205-99-2	5	1.5	ug/Kg	66	136
Benzo[k]fluoranthene	207-08-9	5	1.5	ug/Kg	63	143
Benzo[a]pyrene	50-32-8	5	1.5	ug/Kg	68	128
Indeno[1,2,3-cd]pyrene	193-39-5	5	1.5	ug/Kg	59	139
Dibenz(a,h)anthracene	53-70-3	5	1.5	ug/Kg	57	142
Benzo[g,h,i]perylene	191-24-2	5	1.5	ug/Kg	57	142

Soil	Ultrasonic Extraction	3550B
Soil	Polychlorinated Biphenyls (PCBs) by Gas Chromatography	8082

Analyte Description	CAS Number	RL - Limit	MDL - Limit	Units	LCSREC - Recovery Low	LCSREC - Recovery High
Tetrachloro-m-xylene	877-09-8	0.01	0.1	mg/Kg		
DCB Decachlorobiphenyl	2051-24-3	0.01	0.1	mg/Kg		
PCB-1016	12674-11-2	0.01	0.0032	mg/Kg	40	140
PCB-1016 Peak 1	STL00132	0.01	0.0032	mg/Kg		
PCB-1016 Peak 2	STL00290	0.01	0.0032	mg/Kg		
PCB-1016 Peak 3	STL00232	0.01	0.0032	mg/Kg		
PCB-1016 Peak 4	STL00307	0.01	0.0032	mg/Kg		
PCB-1016 Peak 5	STL00085	0.01	0.0032	mg/Kg		
PCB-1221	11104-28-2	0.011	0.008	mg/Kg		
PCB-1221 Peak 1	STL00206	0.011	0.008	mg/Kg		
PCB-1221 Peak 2	STL00174	0.011	0.008	mg/Kg		
PCB-1221 Peak 3	STL00313	0.011	0.008	mg/Kg		
PCB-1232	11141-16-5	0.011	0.007	mg/Kg	50	150
PCB-1232 Peak 1	STL00280	0.011	0.007	mg/Kg		
PCB-1232 Peak 2	STL00210	0.011	0.007	mg/Kg		
PCB-1232 Peak 3	STL00110	0.011	0.007	mg/Kg		
PCB-1232 Peak 4	STL00108	0.011	0.007	mg/Kg		
PCB-1232 Peak 5	STL00130	0.011	0.007	mg/Kg		
PCB-1242	53469-21-9	0.01	0.0021	mg/Kg	57	128
PCB-1242 Peak 1	STL00059	0.01	0.0021	mg/Kg		
PCB-1242 Peak 2	STL00276	0.01	0.0021	mg/Kg		
PCB-1242 Peak 3	STL00020	0.01	0.0021	mg/Kg		
PCB-1242 Peak 4	STL00075	0.01	0.0021	mg/Kg		
PCB-1242 Peak 5	STL00148	0.01	0.0021	mg/Kg		
PCB-1248	12672-29-6	0.01	0.003	mg/Kg		
PCB-1248 Peak 1	STL00039	0.01	0.003	mg/Kg		
PCB-1248 Peak 2	STL00268	0.01	0.003	mg/Kg		
PCB-1248 Peak 3	STL00294	0.01	0.003	mg/Kg		
PCB-1248 Peak 4	STL00264	0.01	0.003	mg/Kg		
PCB-1248 Peak 5	STL00218	0.01	0.003	mg/Kg		
PCB-1254	11097-69-1	0.01	0.0021	mg/Kg	65	132
PCB-1254 Peak 1	STL00292	0.01	0.0021	mg/Kg		
PCB-1254 Peak 2	STL00297	0.01	0.0021	mg/Kg		
PCB-1254 Peak 3	STL00022	0.01	0.0021	mg/Kg		
PCB-1254 Peak 4	STL00042	0.01	0.0021	mg/Kg		
PCB-1254 Peak 5	STL00067	0.01	0.0021	mg/Kg		
PCB-1260	11096-82-5	0.01	0.003	mg/Kg	60	130
PCB-1260 Peak 1	STL00060	0.01	0.003	mg/Kg		

PCB-1260 Peak 2	STL00274	0.01	0.003	mg/Kg		
PCB-1260 Peak 3	STL00248	0.01	0.003	mg/Kg		
PCB-1260 Peak 4	STL00277	0.01	0.003	mg/Kg		
PCB-1260 Peak 5	STL00197	0.01	0.003	mg/Kg		

Soil	Ultrasonic Extraction	3550B
Soil	Metals (ICP)	6010B

Analyte Description	CAS Number	RL - Limit	MDL - Limit	Units	LCSREC - Recovery Low	LCSREC - Recovery High
Arsenic	7440-38-2	3	0.32	mg/Kg	80	120
Barium	7440-39-3	0.5	0.1	mg/Kg	80	120
Cadmium	7440-43-9	0.5	0.2	mg/Kg	80	120
Chromium	7440-47-3	1.3	0.39	mg/Kg	80	120
Lead	7439-92-1	1.5	0.15	mg/Kg	80	120
Selenium	7782-49-2	5	0.2	mg/Kg	80	120
Silver	7440-22-4	1	0.56	mg/Kg	75	120

Soil	Preparation, Metals	3050B
Soil	Mercury (CVAA)	7471A

Analyte Description	CAS Number	RL - Limit	MDL - Limit	Units	LCSREC - Recovery Low	LCSREC - Recovery High
Mercury	7439-97-6	0.02	0.0063	mg/Kg	80	120

Soil	Preparation, Mercury	7471A_Prep
Soil	Metals (ICP)	6010B

Analyte Description	CAS Number	RL - Limit	MDL - Limit	Units	LCSREC - Recovery Low	LCSREC - Recovery High
Lead	7439-92-1	1.5	0.15	mg/Kg	80	120

Soil	Preparation, Metals	3050B
Water	Northwest - Semi-Volatile Petroleum Products (GC)	NWTPH_Dx

Analyte Description	CAS Number	RL - Limit	MDL - Limit	Units	LCSREC - Recovery Low	LCSREC - Recovery High
o-Terphenyl	84-15-1			mg/L		
#2 Diesel (C10-C24)	STL00163	0.125	0.073	mg/L	70	140
Motor Oil (>C24-C36)	STL00299	0.25	0.048	mg/L	66	125

Water	Liquid-Liquid Extraction (Continuous)	3520C_14d
Water	Northwest - Volatile Petroleum Products (GC)	NWTPH_Gx

Analyte Description	CAS Number	RL - Limit	MDL - Limit	Units	LCSREC - Recovery Low	LCSREC - Recovery High
4-Bromofluorobenzene (Surr)	460-00-4			mg/L		
Gasoline	STL00228	0.05	0.01	mg/L	79	110

Trifluorotoluene (Surr)	98-08-8			mg/L		
1-Methylnaphthalene	90-12-0			mg/L		
Toluene	108-88-3			mg/L		
1,4-Difluorobenzene	540-36-3			mg/L		

Water	Purge and Trap	5030B

Analyte Description	CAS Number	RL - Limit	MDL - Limit	Units	LCSREC - Recovery Low	LCSREC - Recovery High
Prep Analyte	STL00048					

Water	Volatile Organic Compounds (GC/MS)	8260B_LL

Analyte Description	CAS Number	RL - Limit	MDL - Limit	Units	LCSREC - Recovery Low	LCSREC - Recovery High
1,2-Dichlorobenzene	95-50-1	0.2	0.05	ug/L	80	130
2-Chlorotoluene	95-49-8	0.1	0.03	ug/L	75	130
4-Bromofluorobenzene (Surr)	460-00-4			ug/L		
1,2,3-Trichloropropane	96-18-4	0.2	0.05	ug/L	75	120
Carbon tetrachloride	56-23-5	0.1	0.025	ug/L	75	140
cis-1,3-Dichloropropene	10061-01-5	0.1	0.025	ug/L	70	120
Chlorobenzene	108-90-7	0.1	0.025	ug/L	80	120
Vinyl chloride	75-01-4	0.02	0.013	ug/L	65	140
sec-Butylbenzene	135-98-8	0.1	0.025	ug/L	80	125
Dibromomethane	74-95-3	0.1	0.025	ug/L	80	130
Ethylbenzene-d10	25837-05-2			ug/L		
m-Xylene & p-Xylene	179601-23-1	0.2	0.05	ug/L	80	130
o-Xylene	95-47-6	0.1	0.025	ug/L	80	120
1,2,4-Trichlorobenzene	120-82-1	0.2	0.075	ug/L	60	125
Styrene	100-42-5	0.1	0.025	ug/L	75	130
Chlorobromomethane	74-97-5	0.1	0.025	ug/L	80	125
Dichlorobromomethane	75-27-4	0.1	0.025	ug/L	80	125
1,3-Dichlorobenzene	541-73-1	0.2	0.05	ug/L	80	120
Benzene	71-43-2	0.1	0.025	ug/L	80	120
Fluorobenzene (Surr)	462-06-6			ug/L		
Chloroethane	75-00-3	0.25	0.075	ug/L	75	140
trans-1,3-Dichloropropene	10061-02-6	0.1	0.025	ug/L	60	140
1,2,3-Trichlorobenzene	87-61-6	0.4	0.1	ug/L	60	125
N-Propylbenzene	103-65-1	0.1	0.025	ug/L	80	120
Trifluorotoluene (Surr)	98-08-8			ug/L		
4-Isopropyltoluene	99-87-6	0.2	0.05	ug/L	80	120
n-Butylbenzene	104-51-8	0.1	0.03	ug/L	75	125
1,1-Dichloropropene	563-58-6	0.1	0.025	ug/L	80	130

cis-1,2-Dichloroethene	156-59-2	0.1	0.025	ug/L	80	130
1,1,2,2-Tetrachloroethane	79-34-5	0.1	0.025	ug/L	75	125
1,2,4-Trimethylbenzene	95-63-6	0.1	0.03	ug/L	80	125
Toluene	108-88-3	0.1	0.025	ug/L	80	120
Naphthalene	91-20-3	0.4	0.1	ug/L	45	130
1,3,5-Trimethylbenzene	108-67-8	0.1	0.025	ug/L	80	125
1,3-Dichloropropane	142-28-9	0.1	0.025	ug/L	80	130
Chloroform	67-66-3	0.1	0.03	ug/L	80	130
4-Chlorotoluene	106-43-4	0.2	0.05	ug/L	75	130
Chlorodibromomethane	124-48-1	0.1	0.025	ug/L	70	120
Dichlorodifluoromethane	75-71-8	0.4	0.1	ug/L	30	180
1,1,2-Trichloroethane	79-00-5	0.1	0.025	ug/L	80	130
Toluene-d8 (Surr)	2037-26-5			ug/L		
tert-Butylbenzene	98-06-6	0.1	0.025	ug/L	80	130
Chloromethane	74-87-3	0.1	0.035	ug/L	50	140
Methylene Chloride	75-09-2	0.5	0.025	ug/L	60	145
1,1-Dichloroethene	75-35-4	0.1	0.03	ug/L	70	150
Isopropylbenzene	98-82-8	0.1	0.025	ug/L	75	120
1,2-Dichloroethane	107-06-2	0.1	0.025	ug/L	80	140
Tetrachloroethene	127-18-4	0.1	0.025	ug/L	40	180
1,1,1-Trichloroethane	71-55-6	0.1	0.025	ug/L	80	140
2,2-Dichloropropane	594-20-7	0.1	0.025	ug/L	60	150
1,2-Dibromoethane	106-93-4	0.1	0.025	ug/L	70	130
Bromoform	75-25-2	0.1	0.025	ug/L	65	130
1,2-Dibromo-3-Chloropropane	96-12-8	0.4	0.1	ug/L	55	120
Trichlorofluoromethane	75-69-4	0.1	0.025	ug/L	30	180
Trichloroethene	79-01-6	0.1	0.025	ug/L	80	130
Bromobenzene	108-86-1	0.1	0.035	ug/L	80	130
1,2-Dichloropropane	78-87-5	0.1	0.025	ug/L	80	120
1,1,1,2-Tetrachloroethane	630-20-6	0.1	0.025	ug/L	75	125
Ethylbenzene	100-41-4	0.1	0.03	ug/L	80	125
trans-1,2-Dichloroethene	156-60-5	0.1	0.025	ug/L	80	140
Hexachlorobutadiene	87-68-3	0.2	0.075	ug/L	75	135
1,1-Dichloroethane	75-34-3	0.1	0.025	ug/L	75	135
Bromomethane	74-83-9	0.1	0.025	ug/L	70	135
1,4-Dichlorobenzene	106-46-7	0.2	0.05	ug/L	80	120
BFB	460-00-4	0.01	0.01	ug/L		
Methyl tert-butyl ether	1634-04-4	0.1	0.025	ug/L	75	120

Water	Purge and Trap	5030B

Analyte Description	CAS Number	RL - Limit	MDL - Limit	Units	LCSREC - Recovery Low	LCSREC - Recovery High
Prep Analyte	STL00048					

Water	Semivolatile Organic Compounds (GC/MS SIM)	8270C_SIM

Analyte Description	CAS Number	RL - Limit	MDL - Limit	Units	LCSREC - Recovery Low	LCSREC - Recovery High
1,4-Dichlorobenzene-d4	3855-82-1			ug/L		
Naphthalene-d8	1146-65-2			ug/L		
Acenaphthene-d10	15067-26-2			ug/L		
Phenanthrene-d10	1517-22-2			ug/L		
Chrysene-d12	1719-03-5			ug/L		
Perylene-d12	1520-96-3			ug/L		
Terphenyl-d14	1718-51-0			ug/L		
Naphthalene	91-20-3	0.1	0.036	ug/L	65	125
2-Methylnaphthalene	91-57-6	0.13	0.03	ug/L	65	125
1-Methylnaphthalene	90-12-0	0.1	0.03	ug/L	65	125
Acenaphthylene	208-96-8	0.1	0.03	ug/L	70	125
Acenaphthene	83-32-9	0.1	0.03	ug/L	65	125
Fluorene	86-73-7	0.1	0.03	ug/L	70	125
Phenanthrene	85-01-8	0.1	0.03	ug/L	70	125
Anthracene	120-12-7	0.1	0.03	ug/L	60	125
Fluoranthene	206-44-0	0.1	0.03	ug/L	75	125
Pyrene	129-00-0	0.1	0.03	ug/L	75	125
Benzo[a]anthracene	56-55-3	0.1	0.03	ug/L	70	125
Chrysene	218-01-9	0.1	0.03	ug/L	75	125
Benzo[b]fluoranthene	205-99-2	0.1	0.03	ug/L	70	125
Benzo[k]fluoranthene	207-08-9	0.1	0.03	ug/L	70	125
Benzo[a]pyrene	50-32-8	0.2	0.03	ug/L	55	125
Indeno[1,2,3-cd]pyrene	193-39-5	0.1	0.03	ug/L	65	125
Dibenz(a,h)anthracene	53-70-3	0.1	0.03	ug/L	65	130
Benzo[g,h,i]perylene	191-24-2	0.1	0.03	ug/L	65	125

Water	Liquid-Liquid Extraction (Continuous)	3520C
Water	Polychlorinated Biphenyls (PCBs) by Gas Chromatography	8082

Analyte Description	CAS Number	RL - Limit	MDL - Limit	Units	LCSREC - Recovery Low	LCSREC - Recovery High
Tetrachloro-m-xylene	877-09-8			ug/L		
DCB Decachlorobiphenyl	2051-24-3			ug/L		
PCB-1016	12674-11-2	0.5	0.045	ug/L	25	145
PCB-1016 Peak 1	STL00132	0.5	0.045	ug/L		
PCB-1016 Peak 2	STL00290	0.5	0.045	ug/L		

PCB-1016 Peak 3	STL00232	0.5	0.045	ug/L		
PCB-1016 Peak 4	STL00307	0.5	0.045	ug/L		
PCB-1016 Peak 5	STL00085	0.5	0.045	ug/L		
PCB-1221	11104-28-2	0.5	0.062	ug/L		
PCB-1221 Peak 1	STL00206	0.5	0.062	ug/L		
PCB-1221 Peak 2	STL00174	0.5	0.062	ug/L		
PCB-1221 Peak 3	STL00313	0.5	0.062	ug/L		
PCB-1232	11141-16-5	0.5	0.041	ug/L		
PCB-1232 Peak 1	STL00280	0.5	0.041	ug/L		
PCB-1232 Peak 2	STL00210	0.5	0.041	ug/L		
PCB-1232 Peak 3	STL00110	0.5	0.041	ug/L		
PCB-1232 Peak 4	STL00108	0.5	0.041	ug/L		
PCB-1232 Peak 5	STL00130	0.5	0.041	ug/L		
PCB-1242	53469-21-9	0.5	0.041	ug/L	44	127
PCB-1242 Peak 1	STL00059	0.5	0.041	ug/L		
PCB-1242 Peak 2	STL00276	0.5	0.041	ug/L		
PCB-1242 Peak 3	STL00020	0.5	0.041	ug/L		
PCB-1242 Peak 4	STL00075	0.5	0.041	ug/L		
PCB-1242 Peak 5	STL00148	0.5	0.041	ug/L		
PCB-1248	12672-29-6	0.5	0.071	ug/L		
PCB-1248 Peak 1	STL00039	0.5	0.071	ug/L		
PCB-1248 Peak 2	STL00268	0.5	0.071	ug/L		
PCB-1248 Peak 3	STL00294	0.5	0.071	ug/L		
PCB-1248 Peak 4	STL00264	0.5	0.071	ug/L		
PCB-1248 Peak 5	STL00218	0.5	0.071	ug/L		
PCB-1254	11097-69-1	0.5	0.044	ug/L		
PCB-1254 Peak 1	STL00292	0.5	0.044	ug/L		
PCB-1254 Peak 2	STL00297	0.5	0.044	ug/L		
PCB-1254 Peak 3	STL00022	0.5	0.044	ug/L		
PCB-1254 Peak 4	STL00042	0.5	0.044	ug/L		
PCB-1254 Peak 5	STL00067	0.5	0.044	ug/L		
PCB-1260	11096-82-5	0.5	0.039	ug/L	30	145
PCB-1260 Peak 1	STL00060	0.5	0.039	ug/L		
PCB-1260 Peak 2	STL00274	0.5	0.039	ug/L		
PCB-1260 Peak 3	STL00248	0.5	0.039	ug/L		
PCB-1260 Peak 4	STL00277	0.5	0.039	ug/L		
PCB-1260 Peak 5	STL00197	0.5	0.039	ug/L		

Water	Liquid-Liquid Extraction (Separatory Funnel)	3510C
Water	Metals (ICP/MS)	6020

Analyte Description	CAS Number	RL - Limit	MDL - Limit	Units	LCSREC - Recovery Low	LCSREC - Recovery High
Arsenic	7440-38-2	0.001	0.00075	mg/L	80	120
Barium	7440-39-3	0.0012	0.000054	mg/L	80	120
Cadmium	7440-43-9	0.0004	0.000028	mg/L	80	120
Chromium	7440-47-3	0.0004	0.00027	mg/L	80	120
Lead	7439-92-1	0.0004	0.000034	mg/L	80	120
Selenium	7782-49-2	0.001	0.00071	mg/L	80	120
Silver	7440-22-4	0.0004	0.00003	mg/L	80	120

Water	Preparation, Total Recoverable or Dissolved Metals	3005A
Water	Mercury (CVAA)	7470A

Analyte Description	CAS Number	RL - Limit	MDL - Limit	Units	LCSREC - Recovery Low	LCSREC - Recovery High
Mercury	7439-97-6	0.0002	0.000041	mg/L	80	120

Water	Preparation, Mercury	7470A_Prep
Water	Metals (ICP/MS)	6020

Analyte Description	CAS Number	RL - Limit	MDL - Limit	Units	LCSREC - Recovery Low	LCSREC - Recovery High
Lead	7439-92-1	0.0004	0.000034	mg/L	80	120

Water	Preparation, Total Recoverable or Dissolved Metals	3005A

LCSREC - Units	LCSRPD - Precision	LCSRPD - Units	MSREC - Recovery Low	MSREC - Recovery High	MSREC - Units	MSRPD - Precision	MSRPD - Units	SUREC - Recovery Low	SUREC - Recovery High	SUREC - Units
%		%			%		%	50	150	%
%	16	%	70	125	%	16	%			%
%	17	%	64	127	%	17	%			%

LCSREC - Units	LCSRPD - Precision	LCSRPD - Units	MSREC - Recovery Low	MSREC - Recovery High	MSREC - Units	MSRPD - Precision	MSRPD - Units	SUREC - Recovery Low	SUREC - Recovery High	SUREC - Units
%		%			%		%	50	150	%
%	25	%	50	150	%	35	%			%
%		%			%		%	50	150	%
		%								
		%								
		%								

LCSREC - Units	LCSRPD - Precision	LCSRPD - Units	MSREC - Recovery Low	MSREC - Recovery High	MSREC - Units	MSRPD - Precision	MSRPD - Units	SUREC - Recovery Low	SUREC - Recovery High	SUREC - Units
%		%			%		%	80	120	%
%		%			%		%	80	120	%
%		%			%		%	70	120	%
%	30	%	35	135	%	30	%			%
%	30	%	50	130	%	30	%			%
%	30	%	60	125	%	30	%			%
%	30	%	30	160	%	30	%			%
%	30	%	40	155	%	30	%			%
%	30	%	25	185	%	30	%			%
%	30	%	65	135	%	30	%			%
%	30	%	55	140	%	30	%			%
%	30	%	65	135	%	30	%			%
%	30	%	75	125	%	30	%			%
%	30	%	65	135	%	30	%			%
%	30	%	65	125	%	30	%			%
%	30	%	70	125	%	30	%			%
%	30	%	70	125	%	30	%			%
%	30	%	70	135	%	30	%			%
%	30	%	65	135	%	30	%			%

%	30	%	70	135	%	30	%			%
%	30	%	75	125	%	30	%			%
%	30	%	70	135	%	30	%			%
%	30	%	75	125	%	30	%			%
%	30	%	70	120	%	30	%			%
%	30	%	75	130	%	30	%			%
%	30	%	70	130	%	30	%			%
%	30	%	70	125	%	30	%			%
%	30	%	70	125	%	30	%			%
%	30	%	65	125	%	30	%			%
%	30	%	60	125	%	30	%			%
%	30	%	65	140	%	30	%			%
%	30	%	75	125	%	30	%			%
%	30	%	65	130	%	30	%			%
%	30	%	70	125	%	30	%			%
%	30	%	75	125	%	30	%			%
%	30	%	75	125	%	30	%			%
%	30	%	75	125	%	30	%			%
%	30	%	55	130	%	30	%			%
%	30	%	80	125	%	30	%			%
%	30	%	75	125	%	30	%			%
%	30	%	75	125	%	30	%			%
%	30	%	75	125	%	30	%			%
%	30	%	55	135	%	30	%			%
%	30	%	75	130	%	30	%			%
%	30	%	65	120	%	30	%			%
%	30	%	65	135	%	30	%			%
%	30	%	65	130	%	30	%			%
%	30	%	70	130	%	30	%			%
%	30	%	65	135	%	30	%			%
%	30	%	75	125	%	30	%			%
%	30	%	65	130	%	30	%			%
%	30	%	65	135	%	30	%			%
%	30	%	65	130	%	30	%			%
%	30	%	70	125	%	30	%			%
%	30	%	75	135	%	30	%			%
%	30	%	70	125	%	30	%			%
%	30	%	65	140	%	30	%			%
%	30	%	75	120	%	30	%			%
%	30	%	40	135	%	30	%			%
%	30	%	65	130	%	30	%			%

%	30	%	60	135	%	30	%			%
%	30	%	55	140	%	30	%			%
%	30	%	40	125	%	30	%			%
%		%			%		%	70	120	%
%		%			%		%	65	140	%
%	30	%	59	137	%	30	%			%

LCSREC - Units	LCSRPD - Precision	LCSRPD - Units	MSREC - Recovery Low	MSREC - Recovery High	MSREC - Units	MSRPD - Precision	MSRPD - Units	SUREC - Recovery Low	SUREC - Recovery High	SUREC - Units
%		%			%		%			%
%		%			%		%			%
%		%			%		%			%
%		%			%		%			%
%		%			%		%			%
%		%			%		%			%
%		%			%		%	38	141	%
%		%			%		%	42	140	%
%		%			%		%	42	151	%
%	26	%	64	129	%	26	%			%
%	27	%	65	125	%	27	%			%
%	30	%	48	148	%	30	%			%
%	28	%	69	129	%	28	%			%
%	27	%	65	130	%	27	%			%
%	31	%	68	128	%	31	%			%
%	28	%	65	125	%	28	%			%
%	27	%	73	123	%	27	%			%
%	36	%	61	121	%	36	%			%
%	31	%	54	134	%	31	%			%
%	27	%	64	124	%	27	%			%
%	26	%	71	126	%	26	%			%
%	31	%	66	136	%	31	%			%
%	31	%	63	143	%	31	%			%
%	30	%	68	128	%	30	%			%
%	29	%	59	139	%	29	%			%
%	30	%	57	142	%	30	%			%
%	28	%	57	142	%	28	%			%

LCSREC - Units	LCSRPD - Precision	LCSRPD - Units	MSREC - Recovery Low	MSREC - Recovery High	MSREC - Units	MSRPD - Precision	MSRPD - Units	SUREC - Recovery Low	SUREC - Recovery High	SUREC - Units
%		%			%		%	45	135	%
%		%			%		%	50	140	%
%	20	%	40	140	%	20	%			%
%		%			%		%			%
%		%			%		%			%
%		%			%		%			%
%		%			%		%			%
%		%			%		%			%
%		%			%		%			%
%		%			%		%			%
%		%			%		%			%
%		%			%		%			%
%		%			%		%			%
%	30	%	50	150	%	30	%			%
%		%			%		%			%
%		%			%		%			%
%		%			%		%			%
%		%			%		%			%
%		%			%		%			%
%	20	%	57	128	%	20	%			%
%		%			%		%			%
%		%			%		%			%
%		%			%		%			%
%		%			%		%			%
%		%			%		%			%
%		%			%		%			%
%		%			%		%			%
%		%			%		%			%
%		%			%		%			%
%		%			%		%			%
%		%			%		%			%
%		%			%		%			%
%	20	%	65	132	%	20	%			%
%		%			%		%			%
%		%			%		%			%
%		%			%		%			%
%		%			%		%			%
%		%			%		%			%
%	20	%	60	130	%	20	%			%
%		%			%		%			%

%		%			%		%			%
%		%			%		%			%
%		%			%		%			%
%		%			%		%			%

LCSREC - Units	LCSRPD - Precision	LCSRPD - Units	MSREC - Recovery Low	MSREC - Recovery High	MSREC - Units	MSRPD - Precision	MSRPD - Units	SUREC - Recovery Low	SUREC - Recovery High	SUREC - Units
%	20	%	80	120	%	20	%			
%	20	%	80	120	%	20	%			
%	20	%	80	120	%	20	%			
%	20	%	80	120	%	20	%			
%	20	%	80	120	%	20	%			
%	20	%	80	120	%	20	%			
%	20	%	75	120	%	20	%			

LCSREC - Units	LCSRPD - Precision	LCSRPD - Units	MSREC - Recovery Low	MSREC - Recovery High	MSREC - Units	MSRPD - Precision	MSRPD - Units	SUREC - Recovery Low	SUREC - Recovery High	SUREC - Units
%	20	%	80	120	%	20	%			

LCSREC - Units	LCSRPD - Precision	LCSRPD - Units	MSREC - Recovery Low	MSREC - Recovery High	MSREC - Units	MSRPD - Precision	MSRPD - Units	SUREC - Recovery Low	SUREC - Recovery High	SUREC - Units
%	20	%	80	120	%	20	%			

LCSREC - Units	LCSRPD - Precision	LCSRPD - Units	MSREC - Recovery Low	MSREC - Recovery High	MSREC - Units	MSRPD - Precision	MSRPD - Units	SUREC - Recovery Low	SUREC - Recovery High	SUREC - Units
%		%			%		%	50	150	%
%	27	%	70	140	%	27	%			%
%	27	%	66	125	%	27	%			%

LCSREC - Units	LCSRPD - Precision	LCSRPD - Units	MSREC - Recovery Low	MSREC - Recovery High	MSREC - Units	MSRPD - Precision	MSRPD - Units	SUREC - Recovery Low	SUREC - Recovery High	SUREC - Units
%		%			%		%	50	150	%
%	20	%	50	150	%	35	%			%

%		%			%		%	50	150	%
		%								
		%								
		%								

LCSREC - Units	LCSRPD - Precision	LCSRPD - Units	MSREC - Recovery Low	MSREC - Recovery High	MSREC - Units	MSRPD - Precision	MSRPD - Units	SUREC - Recovery Low	SUREC - Recovery High	SUREC - Units

LCSREC - Units	LCSRPD - Precision	LCSRPD - Units	MSREC - Recovery Low	MSREC - Recovery High	MSREC - Units	MSRPD - Precision	MSRPD - Units	SUREC - Recovery Low	SUREC - Recovery High	SUREC - Units
%	20	%	80	120	%	20	%			%
%	20	%	79	127	%	20	%			%
%		%			%		%	75	120	%
%	20	%	70	137	%	20	%			%
%	20	%	56	160	%	20	%			%
%	20	%	63	127	%	20	%			%
%	20	%	71	140	%	30	%			%
%	20	%	47	160	%	20	%			%
%	20	%	72	145	%	20	%			%
%	20	%	76	130	%	20	%			%
%		%			%		%	75	125	%
%	20	%	70	144	%	20	%			%
%	20	%	72	137	%	20	%			%
%	20	%	47	135	%	20	%			%
%	20	%	80	133	%	20	%			%
%	20	%	64	156	%	20	%			%
%	20	%	69	149	%	20	%			%
%	20	%	76	120	%	20	%			%
%	20	%	75	142	%	30	%			%
%		%			%		%	70	130	%
%	20	%	44	160	%	20	%			%
%	20	%	64	132	%	20	%			%
%	20	%	52	129	%	20	%			%
%	20	%	76	131	%	20	%			%
%		%			%		%	80	125	%
%	20	%	80	132	%	20	%			%
%	20	%	72	131	%	20	%			%
%	20	%	59	160	%	20	%			%

%	20	%	71	144	%	20	%			%
%	20	%	73	121	%	20	%			%
%	20	%	80	137	%	20	%			%
%	20	%	80	126	%	30	%			%
%	20	%	40	142	%	20	%			%
%	20	%	80	136	%	20	%			%
%	20	%	78	129	%	20	%			%
%	20	%	65	158	%	20	%			%
%	20	%	76	127	%	20	%			%
%	20	%	71	130	%	20	%			%
%	20	%	40	160	%	20	%			%
%	20	%	80	121	%	20	%			%
%		%			%		%	75	125	%
%	20	%	74	138	%	20	%			%
%	20	%	52	160	%	20	%			%
%	20	%	80	155	%	20	%			%
%	20	%	78	151	%	30	%			%
%	20	%	64	127	%	20	%			%
%	20	%	54	160	%	20	%			%
%	20	%	64	161	%	20	%			%
%	20	%	60	160	%	20	%			%
%	20	%	49	160	%	20	%			%
%	20	%	75	126	%	20	%			%
%	20	%	66	137	%	20	%			%
%	20	%	47	138	%	20	%			%
%	20	%	40	160	%	20	%			%
%	20	%	79	131	%	30	%			%
%	20	%	80	120	%	20	%			%
%	20	%	71	127	%	20	%			%
%	20	%	80	131	%	20	%			%
%	20	%	79	132	%	20	%			%
%	20	%	73	135	%	20	%			%
%	20	%	67	141	%	20	%			%
%	20	%	73	158	%	20	%			%
%	20	%	40	160	%	20	%			%
%	20	%	80	120	%	20	%			%
%		%			%		%			%
%	20	%	77	135	%	20	%			%

LCSREC - Units	LCSRPD - Precision	LCSRPD - Units	MSREC - Recovery Low	MSREC - Recovery High	MSREC - Units	MSRPD - Precision	MSRPD - Units	SUREC - Recovery Low	SUREC - Recovery High	SUREC - Units

LCSREC - Units	LCSRPD - Precision	LCSRPD - Units	MSREC - Recovery Low	MSREC - Recovery High	MSREC - Units	MSRPD - Precision	MSRPD - Units	SUREC - Recovery Low	SUREC - Recovery High	SUREC - Units
%		%			%		%			%
%		%			%		%			%
%		%			%		%			%
%		%			%		%			%
%		%			%		%			%
%		%			%		%			%
%		%			%		%	20	150	%
%	20	%	65	125	%	20	%			%
%	20	%	65	125	%	20	%			%
%	20	%	65	125	%	20	%			%
%	20	%	70	125	%	20	%			%
%	20	%	65	125	%	20	%			%
%	20	%	70	125	%	20	%			%
%	20	%	70	125	%	20	%			%
%	20	%	60	125	%	20	%			%
%	20	%	75	125	%	20	%			%
%	20	%	75	125	%	20	%			%
%	20	%	70	125	%	20	%			%
%	20	%	75	125	%	20	%			%
%	20	%	70	125	%	20	%			%
%	20	%	70	125	%	20	%			%
%	20	%	55	125	%	20	%			%
%	20	%	65	125	%	20	%			%
%	20	%	65	130	%	20	%			%
%	20	%	65	125	%	20	%			%

LCSREC - Units	LCSRPD - Precision	LCSRPD - Units	MSREC - Recovery Low	MSREC - Recovery High	MSREC - Units	MSRPD - Precision	MSRPD - Units	SUREC - Recovery Low	SUREC - Recovery High	SUREC - Units
%		%			%		%	60	150	%
%		%			%		%	40	135	%
%	27	%	25	145	%	27	%			%
%		%			%		%			%
%		%			%		%			%

%		%			%		%			%
%		%			%		%			%
%		%			%		%			%
%		%			%		%			%
%		%			%		%			%
%		%			%		%			%
%		%			%		%			%
%		%			%		%			%
%		%			%		%			%
%		%			%		%			%
%		%			%		%			%
%		%			%		%			%
%		%			%		%			%
%		%			%		%			%
%	27	%	44	127	%	27	%			%
%		%			%		%			%
%		%			%		%			%
%		%			%		%			%
%		%			%		%			%
%		%			%		%			%
%		%			%		%			%
%		%			%		%			%
%		%			%		%			%
%		%			%		%			%
%		%			%		%			%
%		%			%		%			%
%		%			%		%			%
%		%			%		%			%
%		%			%		%			%
%		%			%		%			%
%		%			%		%			%
%		%			%		%			%
%		%			%		%			%
%	22	%	30	145	%	22	%			%
%		%			%		%			%
%		%			%		%			%
%		%			%		%			%
%		%			%		%			%
%		%			%		%			%

LCSREC - Units	LCSRPD - Precision	LCSRPD - Units	MSREC - Recovery Low	MSREC - Recovery High	MSREC - Units	MSRPD - Precision	MSRPD - Units	SUREC - Recovery Low	SUREC - Recovery High	SUREC - Units
%	20	%	80	120	%	20	%			
%	20	%	80	120	%	20	%			
%	20	%	80	120	%	20	%			
%	20	%	80	120	%	20	%			
%	20	%	80	120	%	20	%			
%	20	%	80	120	%	20	%			
%	20	%	80	120	%	20	%			

LCSREC - Units	LCSRPD - Precision	LCSRPD - Units	MSREC - Recovery Low	MSREC - Recovery High	MSREC - Units	MSRPD - Precision	MSRPD - Units	SUREC - Recovery Low	SUREC - Recovery High	SUREC - Units
%	20	%	80	120	%	20	%			

LCSREC - Units	LCSRPD - Precision	LCSRPD - Units	MSREC - Recovery Low	MSREC - Recovery High	MSREC - Units	MSRPD - Precision	MSRPD - Units	SUREC - Recovery Low	SUREC - Recovery High	SUREC - Units
%	20	%	80	120	%	20	%			

APPENDIX B

Regenesis's Treatment Design

REGENESIS

December 20, 2010

Julie Sharp- Dahl
Bristol Environmental Services
111 W. 16th
Anchorage, AK 99503

RE: Proposal for Remediation using RegenOx and ORC Advanced at the Western Gas site

Dear Julie:

Thank you for the opportunity to technically evaluate this project. Below we have provided information related to the design and application of RegenOx™ and ORC Advanced® to treat sorbed- and dissolved-phase residual petroleum hydrocarbons within your defined target treatment area at the above-referenced site.

We have recommended 5 applications of RegenOx followed by ORC-Advanced. This is an estimate based upon saturated soil contaminant concentrations. You may find that you reach treatment objectives in 3 applications and you may find that more than 5 applications are necessary to remediate the saturated soil. We suggest that you begin to sample after the third application. This will give us a better idea of exactly how many ISCO injection events are necessary. Following the RegenOx injection we recommend that injection of ORC-Advanced to take care of the low level groundwater contamination.

Product Description

Detailed descriptions of RegenOx and ORC Advanced products can be found at the following website links: RegenOx and ORC Advanced.

Product Quantities and Cost*

RegenOx
1,450 lbs – Part A = 1,000 lbs Part B = 450 lbs
RegenOx Product Cost - $3,625 ($2.50/lb)

ORC Advanced
320 lbs
ORC Advanced Product Cost - $2,864 ($8.95/lb)

TOTAL COST - $6,489*

*The above cost does not include freight or applicable taxes. Please contact Regenesis customer service at 949.366.8000 or customerservice@regenesis.com or me at 916.409.9331.

Proposed Application Design

Our proposed treatment plan includes 5 applications of RegenOx chemical oxidation to be conducted approximately 1 to 2 weeks apart to reduce hydrocarbon mass in the 600 square foot area. The product should be injected into 8 points spaced approximately 8 to 9 feet on center and the injection should occur from 16 to 19 feet bgs. In order to maximize the distribution of the RegenOx the injection points should be offset by 4 feet between applications.

We recommend application of ORC Advanced approximately 2 weeks following the final RegenOx application. The ORC-Advanced should be injected into 8 points evenly spaced throughout the 600 square foot treatment area. In addition the injection should occur from 14 to 19 feet bgs. (Please note that the ORC-A can be injected at the same time as the RegenOx).

In order to ensure that the subsurface will accept this amount of RegenOx we suggest that you do a pre-aquifer injection test. This comprises of injecting 20 gallons of water over a three foot interval (16 to 19 feet bgs).

Application Guidance

We propose the use of direct push technology injection techniques to apply these products. Guidance for the mixing and application of ORC Advanced and RegenOx can be found at the following website links: ORC Advanced and RegenOx.

Groundwater Monitoring

We recommend collecting the following groundwater parameters prior to and during quarterly performance monitoring following completion of the injection activities: chemicals of concern, pH, dissolved oxygen, oxidation-reduction potential, specific conductance, chemical oxygen demand, biological oxygen demand, total and dissolved iron, and methane.

Regenesis appreciates the opportunity to present you with this proposal. If you need any additional information please feel free to contact your local Regenesis Sales Manager Jack Peabody at 925.944.5566 (jpeabody@regenesis.com) or me at 916.409.9331(bgriffiths@regenesis.com).

REGENESIS

Brittain Griffiths
Technical Manager – Western Region

attachments

REGENESIS

RegenOx

Design Summary Output

Regenesis Technical Support: USA (949) 366-8000

www.regenesis.com

Date:	4/30/2013
Site Name:	Western Gas
Treatment Area Location:	Dissolved Plume, Saturated Soil
Consultant/Contact:	Julie Sharp-Dahl

RegenOx Grid-Based Design Specifications - Direct Push Application

Western Gas

Dissolved Plume, Saturated Soil

Design Specifications	**Quantity**	**Units**
Number of Application Events	5	---
Application Frequency	1 to 2 weeks	---
Injection Point Spacing within row	8	ft on center
Injection Point Spacing between rows	10	ft on center
Number of Injection Points per Event	8	---
Total Injection Points (all applications)	40	---
Treatment Areal Extent	600	ft^2
Top of Treatment Interval	16	ft-bgs
Bottom of Treatment Interval	19	ft-bgs
Vertical Treatment Thickness	3	ft
Linear Footage to be Drilled	760	ft
Product Quantities	**Quantity**	**Units**
Total RegenOx (A&B all applications)	**1,450**	**lbs**
RegenOx Part A per Point	25	lbs
RegenOx Part A per Application	200	lbs
Total RegenOx Part A (all applications)	1,000	lbs
RegenOx Part B per Point	11	lbs
RegenOx Part B per Application	90	lbs
Total RegenOx Part B (all applications)	450	lbs
Total RegenOx Per Cubic Yard	20	lbs
Product Cost		
RegenOx Product Cost	**$3,625**	**not including tax/freight**
Field Mixing / Injection Ratios	**Value**	**Units**
% Solution	5%	% oxidant (Part A)
Mix Water Volume per Point	57	gallons
Mix Water Volume per Application	456	gallons
Total Mix Water Volume (All Applications)	2,278	gallons
RegenOx Injection Volume per Point	54	gallons
Total RegenOx Injection Volume (All Applications)	2,142	gallons

REGENESIS

RegenOx

Design Summary Output

SITE DATA - INPUT PARAMETERS		
Hydraulic Parameters	**Value**	**Units**
Soil Type	sand	---
Fraction Organic Carbon (foc)	0.002	g/g
Porosity	0.33	cm^3/cm^3
Effective Porosity	0.2	cm^3/cm^3
Pore Space Occupancy & Radius of Injection	**Value**	**Units**
Effective Pore Volume Filled	80%	%
Total Pore Volume Filled	48%	%
Radius of Injection (Theoretical Average)	3.9	ft
Saturated Soil Concentrations (sorbed mass)	**Concentration**	**Units**
Benzene	0.0	mg/kg
Toluene	0.0	mg/kg
Ethylbenzene	0.0	mg/kg
Xylenes	177.0	mg/kg
Tetrachloroethene (PCE)	0.0	mg/kg
Trichloroethene (TCE)	0.0	mg/kg
Naphthalene	0.0	mg/kg
Trimethylbenzenes	0.0	mg/kg
TPH-g	14160.0	mg/kg
TPH-d	0.0	mg/kg
MTBE	0.0	mg/kg
User added, add stoich. demand and Koc (see pull-c	0.0	mg/kg
Sorbed Phase Contaminant Mass	***2787.1***	***lbs***
Other Assumptions/Qualifications/Recommendations		
5 application is an estimate based on current saturated soil concentrations.		

REGENESIS

ORC ADVANCED
OXYGEN RELEASE COMPOUND

Design Summary Output

Regenesis Technical Support: USA (949) 366-8000

www.regenesis.com

Date:	4/30/2013
Site Name:	Western Gas
Location:	Dissolved Plume, Saturated Soil
Consultant (Firm/Name):	Mat Faust

ORC Advanced Grid Design Specifications

Western Gas

Dissolved Plume, Saturated Soil

Design Specifications	**Quantity**	**Units**
Treatment Area Size	600	ft^2
Depth to Top Treatment Interval	14	ft
Depth to Bottom Treatment Interval	19	ft
Vertical Treatment Thickness	5	ft
Number of Injection Points	8	---
Injection Point Spacing (within rows)	8	ft on center
Injection Point Spacing (between rows)	10	ft on center
Total Linear Drilling	152	ft
Product Quantities	**Quantity**	**Units**
ORC Advanced per Foot	8	lbs
ORC Advanced per Point	40	lbs
Total ORC Advanced	320	lbs
Field Mixing/Injection Ratios		**Units**
ORC Advanced Slurry %	30%	%
Mixing Water per Foot	2.2	gallons
Mixing Water per Point	11	gallons
Total Water for Mixing	90	gallons
Injection Slurry (Water + ORC-A) per Foot	2.6	gallons
Injection Slurry (Water + ORC-A) per Point	13	gallons
Total Injection Slurry (all Points)	104	gallons

REGENESIS

ORC ADVANCED
OXYGEN RELEASE COMPOUND

Design Summary Output

SITE DATA - INPUT PARAMETERS		
Hydraulic Parameters	**Value**	**Units**
Soil Type (sand, silt, gravel, clay, etc.)	sand, silty gravel	---
Porosity	0.33	cm^3/cm^3
Effective Porosity	0.2	cm^3/cm^3
Hydraulic Conductivity	25	ft/day
Hydraulic Gradient	0.005	ft/ft
Seepage Velocity	228.3	ft/yr
Groundwater Concentrations (dissolved mass)	**Concentration**	**Units**
Benzene	0.0	mg/L
Toluene	0.0	mg/L
Ethylbenzene	0.0	mg/L
Xylenes	0.0	mg/L
MTBE	0.0	mg/L
cis-1,2-DCE	0.0	mg/L
Vinyl Chloride	0.0	mg/L
Naphthalenes	0.0	mg/L
TMBs	0.3	mg/L
Reduced metals: Fe+2 and Mn+2	30.0	mg/L
Alternative Measures of Oxidant Demand	**Concentration**	**Units**
Total Petroleum Hydrocarbons (see pull-down for Ko	4.1	mg/L
Biological Oxygen Demand (BOD)	20.0	mg/L
Chemical Oxygen Demand (COD)	50.0	mg/L
Dissolved Phase Contaminant Mass	***6.4***	***lbs***

REGENESIS

Oxygen Release Compound (ORC®) Installation Instructions

(Direct-Injection Slurry Application)

SAFETY:

Pure ORC is shipped to you as a fine powder rated at -325 mesh (passes through a 44 micron screen). It is considered to be a mild oxidizer and as such should be handled with care while in the field. Field personnel should take precautions while applying the pure ORC. Typically, the operator should work upwind of the product as well as use appropriate safety equipment. These would include eye and respiratory protection, and gloves as deemed appropriate by exposure duration and field conditions.

Personnel operating the field equipment utilized during the installation process should have appropriate training, supervision and experience.

GENERAL GUIDELINES:

ORC may be installed in the contaminated saturated zone in the ground utilizing hand augered holes, Geoprobe® type hydraulic punch equipment, or hollow stem augers. This set of instructions is specific for Geoprobe equipment. Alternate instructions may be obtained from the Regenesis Technical Support Department.

For optimum results the ORC slurry installation should span the entire vertical contaminated saturated thickness, including the capillary fringe and “smear zone”.

Two general installation approaches are available. The first is to backfill only the probe hole with slurry. This is a simple approach, in that it is easy, straightforward, and the location of the ORC slurry is precisely known after installation. However, this method requires significantly more probe holes than the alternative, and may take more time for the completion of the remediation process. A separate set of instructions for this method utilizing Geoprobe equipment is available from Regenesis.

The second method is to inject the slurry through the probe holes into the contaminated saturated zone. This method requires fewer probe holes, is less disruptive to the site, and aids the spread of oxygen by spreading the ORC source material. However, it may be difficult to know the exact, final disposition of the ORC installed with this method. This is the method described in these instructions.

Note: It is important that the installation method and specific ORC slurry point location be established prior to field installation. It is also important that the ORC slurry volume and solids content for each drive point be predetermined. The Regenesis Technical Service Department is available to discuss these issues, and Helpful Hints at the end of these instructions offers relevant information. Regenesis also has available Technical Bulletins covering source treatments with ORC.

SPECIFIC INSTALLATION PROCEDURES

1. Identify the location of all underground structures, including utilities, tanks, distribution piping, sewers, drains, and landscape irrigation systems.
2. Identify surface and aerial impediments.
3. Adjust planned installation locations for all impediments and obstacles.
4. Pre-mark the installation grid point locations, noting any that have special depth requirements.
5. Set up the Geoprobe unit over each specific point, following manufacturer recommended procedures. Care should be taken to assure approximate vertical probe holes.
6. Penetrate surface pavement, if necessary, following standard Geoprobe procedures.
7. Drive the 1 1/2" (one-and-one-half inch) pre-probe (part #AT-148B) with the expendable tip (part #AT142B) to the desired maximum depth. Standard 1" (one inch) drive rods (part AT104B) should be used, after the pre-probe. (Hint: Pre-counted drive rods should be positioned prior to the installation driving procedure to assure the desired depth is reached.)
8. Disconnect the drive rods from the expendable tip, following standard Geoprobe procedures.
9. Mix the appropriate quantity of ORC slurry for the current drive point. (See separate "Directions for ORC® Slurry Mixing" and Helpful Hints). **Note: Do not mix more slurry than will be used within a 30 minute period.**
10. Set up and operate an appropriate slurry pump according to manufacturer's directions. Based on our experience, a Geoprobe model GS-1000 pump is recommended. Connect the pump to the probe grout pull cap (GS-1054) via a 1 inch diameter delivery hose. The hose is then attached to the 1" drive rod with its quick connector fitting. Upon confirmation of all connections add the ORC slurry to the pump hopper/tank.
11. Withdraw the pre-probe and drive stem 4' (four feet). (Also note Helpful Hints - Operations at end of instructions.)
12. Optional pretreatment step. (See Helpful Hints - Operations at end of instructions). Pump one to two gallons of tap water into the aquifer to enhance dispersion pathways from the probe hole.
13. Pump the predetermined quantity of ORC slurry for the depth interval being injected. Observe pump pressure levels for indications of slurry dispersion or refusal into the aquifer. (Increasing pressure indicates reduced acceptance of material by the aquifer).
14. Remove one 4' section of the 1" drive rod. The drive rod will contain slurry. This slurry should be returned to the ORC bucket for reuse.
15. Repeat steps 11, 13, and 14 until treatment of the entire affected thickness has been achieved. It is generally recommended that the procedure extend to the top of the capillary fringe/smear zone.
16. Install an appropriate seal, such as bentonite, above the ORC slurry through the entire vadose zone. This helps assure that the slurry stays in place and prevents contaminant migration from the surface. Depending on soil conditions and local regulations, a bentonite seal can be pumped through the slurry pump or added via chips or pellets after probe removal.
17. Remove and decontaminate the drive rods and pre-probe.

18. Finish the probe hole at surface as appropriate (concrete or asphalt cap, if necessary).
19. Move to the next probe point, repeating steps 5 through 18.

HELPFUL HINTS:

A. Physical characteristics

A1. Slurry

The ORC slurry is made using the dry ORC powder (rated at -325 mesh). It makes a smooth slurry, with a consistency that depends on the amount of water used.

A thick, but pumpable, slurry that approaches a paste can be made by using 65-67% solids. This material would normally be used for back-filling a bore or probe hole. It is especially useful where maximum density is desired such as where ground water is present in the hole or there are heaving sands.

Thinner slurries can be made by using more water. Typical solids for the thinner slurries content will range from 35% to 62%. Such slurries are useful for injecting through a probe or bore hole into the saturated aquifer.

As a rule, it is best to mix the first batch of slurry at the maximum solids content one would expect to use. It can then be thinned by adding additional water in small increments. By monitoring this process, the appropriate quantities of water for subsequent batches can be determined.

The slurry should be mixed at about the time it is expected to be used. It is best to not hold it for more than 30 minutes. Thinner slurries, especially, can experience a separation upon standing. All ORC slurries have a tendency to form cements when left standing. If a slurry begins to thicken too much, it should be mixed again and additional water added if necessary.

Care should be taken with slurry that may be left standing in a grout pump or hose. Problems can generally be avoided by periodically re-circulating the slurry through the pump and hose back into the pump's mixing or holding tank.

A2. Equipment

Most geotechnical grout pumping equipment has a holding tank with a capacity sufficient for injection.

When applying measured volumes of ORC slurry to probe holes, it is sometimes useful to know the volumes and content of the delivery system lines. The following information may be useful in this regard.

Geoprobe pump: At the end of a pump stroke virtually no deliverable slurry remains in the pump.

5/8" O.D. connecting hose (10 feet long):	0.2 gallons (26 fluid ounces).
Four foot (4') length of 1" drive rod:	.04 gallons (5 fluid ounces).
Three foot (3') length of 1 1/2" pre-probe:	.03 gallons (4 fluid ounces).

Cleaning and maintenance:

Pumping equipment and drive rods can be lightly cleaned by circulating clear water through them. Further cleaning and decontamination (if necessary due to subsurface conditions) should be performed according to the equipment supplier's standard procedures and local regulatory requirements.

B. Operating characteristics

B1. Operations - General

Judgment will be needed in the field when injecting ORC slurries. In general, it is relatively easy to inject ORC slurries into sandy soils, and this can usually be accomplished at very moderate pressures. Silts and clays require more pressure, and may accept less slurry.

Careful observation of pressure during slurry pumping is the best indication of the effectiveness of the slurry injection. To test the soil's ability to accept the slurry and to "precondition" the injection point for the slurry, it is sometimes useful to inject a small volume of plain water prior to the slurry. Normally, one-half (0.5) gallons to two (2) gallons would be appropriate.

During injection, increasing pressure and decreasing flow rate are signs of refusal by the soil matrix to accept the slurry. The site geologist should determine whether to increase pressure, and possibly fracture ("frac") the soil matrix to achieve ORC slurry installation in a tight site that has refused the slurry at lower pressures.

B2. Fill Volumes

Probe hole back-filling

Probe hole capacities:

Per 10' (Ten Foot) Length			
Theoretical		**Operating Volume**	
(Gallons/Fluid Ounces/Cubic Inches)		**(Gallons/Fluid Ounces)**	
Sand, Silts & Clay		**Sand**	**Silts & Clay**
1" Diameter	.41 gal/52 fl. oz./94.2 cu. in.	.61 gal/78 fl. oz.	.51 gal/65 fl. oz.
1 1/2" Diameter	.92 gal/117 fl. oz./212.0 cu. in.	1.38 gal/176 fl. oz.	1.15 gal/146 fl. oz.
2" Diameter	1.63 gal/209 fl. oz./376.8 cu. in.	2.44 gal/313 fl. oz.	2.04 gal/261 fl. oz.
2 1/4" Diameter	2.06 gal/264 fl. oz./476.9 cu. in	3.09 gal/396 fl. oz.	2.57 gal/330 fl. oz.

Note that the operating volumes include a 50% excess above the theoretical volume in sands and 25% in clays and silts. This is important to successful treatment. The additional material allows for a small degree of infiltration of the slurry into the surrounding soil and fractures, as well as hole diameter variability. It is important to assure that the entire contaminated saturated zone is treated (including the capillary fringe), since this is often the area of highest pollution concentration. Failure to treat this area due to improper installation can undermine an otherwise successful remediation effort.

For direct assistance or answers to any questions you may have regarding these instructions, contact Regenesis Technical Services at 949-366-8000.

REGENESIS, 2002
www.regenesis.com

REGENESIS

RegenOx™

CHEMICAL OXIDATION REDEFINED

RegenOx™ *In Situ* Chemical Oxidation Application Instructions

Using Direct-Push Injection (Step-by-Step Procedures)

RegenOx™ is the new generation of chemical oxidation. RegenOx™ is a proprietary (patent-applied-for) *in situ* chemical oxidation process using a solid oxidant complex (sodium percarbonate/catalytic formulation) and an activator complex (a composition of ferrous salt embedded in a micro-scale catalyst gel). RegenOx™ with its catalytic system has very high activity, capable of treating a very broad range of soil and groundwater contaminants including both petroleum hydrocarbons and chlorinated solvents.

Instructions

1) Prior to the installation of RegenOx™, any surface or overhead impediments should be identified as well as the location of all underground structures. Underground structures include but are not limited to utility lines; tanks; distribution piping; sewers; drains; and landscape irrigation systems. The planned installation locations should be adjusted to account for all impediments and obstacles. These considerations should be part of the SSHP or HASP.

2) Pre-mark the installation locations, noting any points that may have different vertical application requirements or total depth.

3) Set up the direct push unit over each point and follow the manufacturer standard operating procedures (SOP) for the direct push equipment. Care should be taken to assure that probe holes remain in the vertical.

4) For most applications, Regenesis suggests using 1.5-inch O.D./0.625-inch I.D drive rods. However, some applications may require the use of 2.125-inch O.D./1.5-inch I.D. or larger drive rods.

5) Advance drive rods through the surface pavement, as necessary, following SOP.

6) Push the drive rod assembly with an expendable tip to the desired maximum depth. Regenesis suggests pre-counting the number of drive rods needed to reach depth prior to starting injection activities.

7) After the drive rods have been pushed to the desired depth, the rod assembly should be withdrawn three to six inches. Then the expendable tip can be dropped from the drive rods, following SOP. If an injection tool was used instead of an expendable tip, the application of material can take place without any preliminary withdrawal of the rods.

8) In some cases, introduction of a large column of air prior to RegenOx™ application may be problematic because the air can block water flow to the treatment area. This is particularly the case in deep injections (>50 ft) with large diameter rods (>1.5-inch O.D.). To prevent the injection of air into the aquifer during RegenOx™ application, as well as to prevent problems associated with heaving sands, fill the drive rods with water, or the RegenOx™ mixture prior dropping the expendable tip or exposing the injection tool.

9) The RegenOx™ percent of the oxidizer in solution should range between 3% to 5%. Although solutions up to 8% may be used, this will likely increase the difficulty of injection due to reactivity. Solutions with greater than 8% oxidizer in solution will result in excess reaction and flocculation prior to injection and are not typically recommended

   Measure the appropriate quantity of RegenOx™ Oxidizer for one to four vertical foot of injection into a 55 gallon drum or mixing tank. The volume of water per injection location can be calculated from the following formula:

$$\frac{\text{RegenOx Oxidizer lbs/foot}}{(8.34\ \text{lbs/gal water})(\%\ \text{R}egenOx\_Oxidizer\ \text{solids})}\left[1-(\%\ \text{R}egenOx\_Oxidizer\ \text{solids})\right]$$

   Tighter formations (clays and silts), and even some fine sand formations will likely require higher oxidant percentages since less volume can be injected per location. The following are guides to various RegenOx™ mixing ratios based on the above equation.

   - to make a roughly 3% oxidant solution for every 10 lbs of oxidant and 10 lbs of activator (20 lbs total RegenOx™), use 38 gallons of water.
   - to make a roughly 4% oxidant solution for every 10 lbs of oxidant and 10 lbs of activator (20 lbs total RegenOx™), use 28 gallons of water.
   - to make a roughly 5% oxidant solution for every 10 lbs of oxidant and 10 lbs of activator (20 lbs total RegenOx™), use 22 gallons of water.

10) Pour the pre-measured quantity of RegenOx™ Oxidizer into the pre-measured volume of water to make the desired target % oxidant in solution. NOTE: always pour the Oxidizer into water, do not pour water into the Oxidizer. Mix the water and oxidant with a power drill and paint stirrer or other mechanical mixing device to ensure that the Oxidizer has dissolved in the water.

11) Pour the applicable quantity of the pre-mixed RegenOx™ Activator into the oxidant:water solution. Mix the Oxidant and Activator using a power drill paint stirrer or other mechanical mixing device for at least 5 minutes until a homogenous mixture is formed. After mixing the RegenOx™ mixture should be injected into the subsurface as soon as possible.

12) Do not mix more RegenOx™ material than will be used over roughly 1 to 4 feet of injection so as to minimize potential above ground reaction/flocculation prior to injection.

    Transfer the contents of the mixing tank to the pump using gravity feed or appropriate transfer pump. (See Section 9.2: Pump Selection) For some types of pumps, it may be desirable to perform a volume check prior to injecting RegenOx™

13) Connect the delivery hose to the pump outlet and the delivery sub-assembly. Circulate RegenOx™ though the hose and the delivery sub-assembly to displace air in the hose. NOTE: an appropriately sized pressure gauge should be placed between the pump outlet and the delivery sub-assembly in order to monitor application pump pressure and detect changes in aquifer backpressures during application.

14) Connect the sub-assembly to the drive rod. After confirming that all of the connections are secure, pump the RegenOx™ through the delivery system to displace the water/fluid in the rods.

15) Slowly withdraw the drive rods. Commonly RegenOx™ injection progress at 1-foot intervals. However, continuous injection while slowly withdrawing single lengths of drive rod (3 or 4 feet) is an acceptable option. The pre-determined volume of RegenOx™ should be pumped into the aquifer across the desired treatment interval.

16) Remove one section of the drive rod. The drive rod may contain some residual RegenOx™. Place the RegenOx™-filled rod in a clean, empty bucket and allow the RegenOx to drain. Eventually, the RegenOx™ should be returned to the RegenOx™ pump hopper for reuse.

17) Monitor for any indications of aquifer refusal. This is typically indicated by a spike in pressure as indicated or (in the case of shallow applications) RegenOx™ "surfacing" around the injection rods or previously installed injection points. At times backpressure caused by reaction off-gassing will impede the pumps delivery volume. This can be corrected by bleeding the pressure off using a pressure relief/bypass valve (placed inline between the pump discharge and the delivery sub-assembly) and then resume pumping. If aquifer acceptance appears to be low, as indicated by high back pressure, allow sufficient time for the aquifer to equilibrate prior to removing the drive rod.

18) Repeat steps 13 through 23 until treatment of the entire contaminated vertical zone has been achieved. It is recommended that the procedure extend to the top of the capillary fringe/smear zone, or to the top of the targeted treatment interval.

19) Install an appropriate seal, such as bentonite, above the RegenOx™ material through the entire vadose zone. Prior to emplacing the borehole seal, we recommend placing clean sand in the hole to the top of the RegenOx™ treatment zone (especially important in holes that stay open). Bentonite chips or granular bentonite should be placed immediately above the treatment zone, followed by a cement/bentonite grout to roughly 0.5 feet below ground surface. Quick-set concrete should then be used as a surface seal.

20) Remove and clean the drive rods as necessary.

21) Finish the borehole at the surface as appropriate (concrete or asphalt cap, as needed). We recommend a quick set concrete to provide a good surface seal with minimal set up time.

22) A proper borehole and surface seal assures that the RegenOx™ remains properly placed and prevents contaminant migration from the subsurface. Each borehole should be sealed immediately following RegenOx™ application to minimize RegenOx™ surfacing during the injection process. If RegenOx™ continues to "surface" up the direct push borehole, an appropriately sized (oversized) disposable drive tip or wood plug/stake can be used to plug the hole until the aquifer pressures equilibrates and the RegenOx™ stops surfacing. If wells are used for RegenOx™ injection the RegenOx™ injection wells and all nearby groundwater monitoring wells should be tightly capped to reduce potential for surfacing through nearby wells.

23) Periodically compare the pre- and post-injection volumes of RegenOx™ in the holding tank or pump hopper using the pre-marked volume levels. Volume level may not be present on all tanks or pump hoppers. In this case, volume level markings can be temporarily added using known amounts of water and a carpenter's grease pencil (Kiel crayon).

24) Move to the next probe point, repeating steps 8 through 29. We recommend that the next RegenOx™ injection point be as far a distance as possible within the treatment zone from the previous RegenOx™ injection point. This will further minimize RegenOx™ surfacing and short circuiting up an adjacent borehole. When possible, due to the high volumes of liquid being injected, working from the outside of the injection area towards the center will limit expansion of the plume.

Pump Selection

Regenesis has evaluated a number of pumps and many are capable of delivering RegenOx™ to the subsurface at a sufficient pressure and volumetric rate. However, even though a number of the evaluated pumps may be capable of delivering the RegenOx™ to the subsurface based on adequate pressures and delivery rates, each pump has its own set of practical issues that may make it more or less difficult to manage in a field setting.

In general, Regenesis strongly recommends using a pump with a pressure rating of 200 pounds per square inch (psi) in sandy soil settings, and 800 psi in silt, clay or weathered bedrock settings. Any pump under consideration should have a minimum delivery rate of 5 gallons per minute (gpm). A lower gpm rated pump may be used; however, they are not recommended due to the amount of time required to inject the volume of liquids typically associated with a RegenOx™ injection (i.e. 1,000 lbs of RegenOx™ [500 lbs Oxidant/500 lbs Activator] require roughly 1,100 gallons of water to make a 5% Oxidant solution).

Quite often diaphragm pumps are used for the delivery of chemical oxidants. Generally, these pumps operate pressures from 50-150 psi. Some of these pumps do not have the pressure head necessary to overcome the back pressure encountered in silt and clay lenses. In these cases the chemical oxidant thus ends up being delivered to the surrounding sands (the path of least resistance) and is not delivered to soil with residual adsorbed contamination. The use of a positive displacement pump such as a piston pump or a progressing cavity pump is may be superior because these pumps have the pressure necessary to overcome the resistance of low permeability soils. NOTE: be aware that application at pressures that are too high may over-consolidate the soil and minimize the direct contact of the oxidant. The key is to inject at a rate and pressure that maximizes the radius of influence without causing preferential flow. This can be achieved by injecting at the minimum pressure necessary to overcome the particular pressures associated with your site soil conditions.

Whether direct injection or wells are used, it is best to start by injecting RegenOx™ outside the contaminated area and spiral laterally inwards toward the source. Similarly, RegenOx™ should be applied starting vertically at the bottom elevation of contamination, through the layer of contamination, and a couple of feet above the layer of contamination. The reagents can be pushed out from the well bore with some water.

Pump Cleaning

For best results, flush all moving parts and hoses with clean water at the end of the day; flush the injection system with a mixture of water and biodegradable cleaner such as Simple Green.

For more information or technical assistance please call Regenesis at 949-366-8000

APPENDIX C

Bristol's Standard Operating Procedures
for Field Activities

BRISTOL ENVIRONMENTAL REMEDIATION SERVICES, LLC

SOIL SAMPLING

STANDARD OPERATING PROCEDURE BERS-01

Record of Changes

Revision No.	Date	Prepared by	Approved by
0	01/15/08	B. Allen	S. Ruth
1	10/14/09	L. Maserjian	P. Curl
2	02/17/10	L. Maserjian	B. Allen

SOIL SAMPLING

STANDARD OPERATING PROCEDURE

Summary: Soil samples may be collected using a variety of methods and equipment. The methods and equipment used are dependent on the depth of the desired sample, the type of the sample required, and the soil type. Manual techniques and equipment, such as hand augers, are usually used for surface or shallow, subsurface soil sampling. Power-operated equipment is usually associated with collecting deep samples, but this equipment can also be used for collecting shallow samples.

Soil samples collected for volatile organic compound (VOC) analysis are handled in a manner that minimizes the loss of contaminants due to volatilization and biodegradation. Where required to meet project objectives, field extraction and preservation with methanol are performed to ensure sample integrity and representativeness during sample handling and transport.

Health and Safety: Sampling activity should only be conducted in accordance with an approved Site Health and Safety Plan.

Personnel Qualifications: Sampling personnel will be trained and certified as hazardous site workers per Title 29 Code of Federal Regulations, Part 1910.120(e) [29 CFR 1910.120(e)]. If applicable, additional qualification requirements will be specified in the site Quality Assurance Project Plan (QAPP) and will be met.

Equipment and Materials: Prior to deployment in the field, the requisite sampling equipment and materials will be identified, secured, and inspected for signs of damage or potential contamination. Sampling equipment will be selected to preserve the chemical and physical integrity of the samples. Equipment selection will be based on the depth of the samples to be collected and, to a certain extent, the characteristics of the material being sampled. Equipment used for sampling trace contaminants should be constructed of inert materials, such as Teflon® or stainless steel. Ancillary equipment, such as auger flights, post hole diggers, etc., may be constructed of other materials if this equipment does not come in contact with the samples. However, plastic, chromium, galvanized, painted, or rusted equipment should not be used for routine soil sampling operations.

- Sample containers for collecting samples using the methanol extraction and preservation method must be prepared and weighed in advance by the laboratory performing the analysis. Required equipment may include disposable plastic syringes and a clean, sharp utility knife.
- Surface soil is generally classified as soil between the ground surface and 6 inches below ground surface (bgs). Surface soil sampling equipment typically consists of spoons, shovels, hand-augers, push tubes, and post-hole diggers.

- Subsurface soil is generally soil that is at least 6 inches bgs, and can be collected using manual or powered sampling devices. Manual sampling devices consist of hand augers, push tubes, and post-hole diggers. Powered devices typically consist of power augers; split-spoon samplers, which are driven with a drill rig drive-weight assembly or hydraulically pushed using drill rig hydraulics; continuous split-spoon samplers; specialized hydraulic cone penetrometer rigs; and/or backhoes.
- In addition to soil sampling equipment, sampling support equipment may include Global Positioning System (GPS) or survey equipment for locating sample points, organic vapor analyzer with a photoionization detector (PID), tape measures, survey stakes or flags, stainless steel buckets/bowls or disposable aluminum pie pans, canvas/plastic sheet, pre-cleaned sample containers, decontamination supplies and equipment, safety equipment, logbooks, camera, chain-of-custody forms, and supplies for sample labeling, packaging, and shipping.
- Sample containers will be of the type and size specified in the governing QAPP.

Field Screening

Field-screening samples are typically collected either before or concurrently with laboratory analytical samples. Field screening before sample collection can help guide the selection of the most appropriate location to collect a laboratory analytical sample. Collecting field-screening samples concurrently with laboratory samples can help establish a correlation between screening and analytical results at a particular site. Field screening is commonly performed using an organic vapor analyzer, such as a PID.

Headspace PID Screening

Headspace PID screening samples are collected by filling a resealable Ziploc® bag approximately one-third to one-half full of freshly exposed or uncovered soil, and immediately sealing the bag. The soil should be agitated and then allowed to warm for approximately five minutes to an hour, to allow the headspace vapors to develop. After the headspace vapors have developed, insert the tip of a calibrated PID into the void headspace of the bag, and record the highest reading.

In-Situ PID Screening

In some instances, where a limited volume of soil is available for field screening, such as soil cores from a direct-push rig or split spoon, using the headspace method may use up material that potentially could be used for a laboratory analytical sample. In these cases, field screening of the soil may be performed by making small divots approximately every 6 inches along the length of the core, and inserting the calibrated PID tip just above the freshly exposed divot, taking care not to touch the material. Record the highest reading at each location in the field logbook or field form.

Surface Soil Sampling

1. If a thick, matted root zone is encountered at or near the surface, remove it before collecting the sample.
2. Carefully remove the top layer of soil or debris to the desired sample depth with a pre-cleaned spade.
3. Using a pre-cleaned, stainless steel scoop, spoon, or trowel, remove and discard a thin layer of soil from the area that came in contact with the spade.
4. Collect samples following procedures described in the General Soil Sampling Procedures Section.

Subsurface Soil Sampling: Subsurface samples can be collected using hand or power augers, Geoprobes®, split-spoon samplers, or from backhoes.

- Augering is the most common method used to collect shallow subsurface samples. The auger is used to bore a hole to the desired sampling depth. VOC samples are generally collected directly from the sampling device. Non-VOC samples are collected after thorough mixing. If a core sample is required, the auger tip is replaced with a thin wall tube sampler, and the system is lowered into the borehole and driven to the required sample depth. The system is withdrawn and the core is collected from the thin wall tube sampler.
- Geoprobe sampling uses a direct-push system that employs percussion power to essentially "hammer" sampling equipment (Macro-Core® samplers) into the subsurface to extract soil for laboratory analysis. The advantage of using a Geoprobe is that there is a much smaller hole diameter and minimal soil cuttings. The Macro-Core sampler is a solid barrel that is pushed into the subsurface for collecting continuous core samples of unconsolidated materials at depth. The Macro-Core soil samples are collected in a 4 to 5 foot long Teflon, polyvinyl chloride (PVC), or polyethylene terephalate glycol (PETG) liner; the samples can be obtained by splitting the liner or capped to it to preserve the samples for future analysis.
- On underground storage tank or contaminated soil excavation sites, a backhoe bucket is commonly used for collecting soil for samples. When a trench or excavation is deeper than four feet, the bucket of the backhoe or excavator will be used to collect soil, so that personnel do not climb into an unprotected hole.
- Power hand augers are commonly used to aid in the collection of subsurface soil samples at depths where hand augering is impractical. This equipment is a sampling aid, and not a sampling device; the typical lower depth range available with these devices is 20 to 25 feet. The power auger is used to advance a hole to the required sampling depth, at which point a hand auger is usually used to collect the sample.
- Split-spoon sampling provides for the collection and extraction of undisturbed soil cores of 18 or 24 inches in length. A series of consecutive cores may be extracted to

give a complete soil column profile, or an auger may be used to drill down to the desired depth for sampling. The split spoon is then driven to the sampling depth through the bottom of the augered hole, and the core is extracted. When used in conjunction with drilling, split-spoon samplers are usually driven either inside a hollow-stem auger or inside an open borehole after the rotary or cable tool drilling equipment has been temporarily removed.

- When split spoon or Macro-Core sampling is performed to gain geologic information, sampling will be performed in accordance with SOP BERS-06 Borehole Logging.
- Continuous split-spoon samplers may be used to obtain five-foot-long, continuous samples, approximately 3 to 5 inches in diameter. These devices are placed inside a five-foot section of hollow-stem auger and advanced with the auger during drilling. As the auger advances, the central core of soil moves into the sampler.
- Cone Penetrometer Rigs use a standard split spoon that is modified with a releasable tip to keep the spoon closed during the sampling push. Upon arrival at the desired depth, the tip can be remotely released and the push continued. During the subsequent push, the released tip floats freely up the inside of the sample barrel as the soil core displaces it. Split-spoon soil samples, therefore can be collected without drilling by simply pushing the device to the desired depth. This technique is particularly beneficial at highly contaminated sites, because cuttings are not produced as with drill rigs. This results in limited investigation-derived waste (IDW) and minimal exposure to sampling personnel.

Sampling using a Hand Drill with an Auger Attachment

1. Attach the auger bit to a drill-rod extension, and attach the “T” handle to the drill rod.
2. Clear the area to be sampled of any surface debris (e.g., twigs, rocks, and litter). It is generally advisable to remove the first three to six inches of surface soil.
3. Begin augering, periodically removing the auger from the hole and depositing accumulated soils onto a plastic sheet spread near the hole.
4. After reaching the desired depth, slowly and carefully remove the auger from the boring.
5. Remove auger tip from drill rods and replace with a pre-cleaned, thin-wall tube sampler. Install the proper cutting tip.
6. Carefully lower the tube sampler down the borehole. Gradually force the tube sampler into the soil. Take care to avoid scraping the borehole sides. Avoid hammering the drill rods to facilitate coring as the vibrations may cause the boring walls to collapse.
7. Remove the tube sampler and unscrew the drill rods.
8. Remove the cutting tip and the core from the device.
9. Discard approximately 1 inch off the top of the core. Place the remaining core into a labeled sample container without mixing.

10. Collect samples following procedures described in the General Soil Sampling Procedures Section.
11. If another sample is to be collected in the same hole, but at a greater depth, reattach the auger bit to the drill and assembly and follow steps 3 through 10, making sure to decontaminate the auger and tube sampler between samples.
12. Abandon the hole according to applicable state regulations and corporate procedures.

Sampling using a Geoprobe with a Macro-Core Attachment

1. Clear the area to be sampled of any surface debris (e.g., twigs, rocks, and litter). It is generally advisable to remove the first three to six inches of surface soil.
2. Begin hammering the Geoprobe.
3. After hammering the depth of the Macro-Core (either 4 or 5 feet in length), slowly and carefully remove the Macro-Core from the hammer.
4. Open the Macro-Core tubing using a two-razor tool designed for the Macro-Core.
5. Use a pre-cleaned stainless steel spoon or knife to obtain soil from the core at selected locations, based on PID field screening.
6. Collect samples following procedures described in the General Soil Sampling Procedures Section. The Macro-Core is not reusable. Remove any soil on the core material, place the used core in a plastic trash bag (with as many used cores as will fit), tie the ends of the plastic bag, and dispose of it in a trash receptacle.
7. Abandon the hole according to applicable state regulations and corporate procedures.

Sampling with a Hand Auger

1. Insert the hand auger into the material to be sampled at a 0° to 45° angle from the horizon.
2. Rotate the auger once or twice to cut a core of material.
3. Slowly withdraw the auger, with the slot facing upward.
4. Collect samples following the procedures described in the General Soil Sampling Procedures Section.

Sampling with a Split Spoon

1. Assemble the sampler by aligning both sides of barrel, then screw the drive shoe on the bottom and the headpiece on top.
2. Place the sampler perpendicular to the material sampled.
3. Using a well ring, drive the tube. Do not drive the tube past the bottom of the head piece, or the sample may be compressed.

4. Record the length of the tube used to penetrate the material being sampled, and the number of blows required to obtain this depth in the site logbook or on field data sheets.
5. Withdraw the sampler and open by unscrewing the bit and head, and splitting the barrel. Record the amount of recovery and soil type on the boring log. If a split sample is required, use a clean stainless steel knife to divide the tube contents in half, lengthwise.
6. Without disturbing the core, collect samples following the procedures described in the General Soil Sampling Procedures Section.

Sampling from a Backhoe

1. If backhoe buckets are not cleaned in between sample locations, collect samples from material in the center of the bucket.
2. Prior to collecting samples from soil in the backhoe bucket, dress the surface with a stainless steel shovel, spatula, knife, or spoon, to remove at least six inches of surface layer of soil, which may have been smeared across the trench wall as the bucket passed.
3. Be aware of "sluff" material in the bucket that is not representative of the specified sample depth.
4. Collect samples following procedures described in the General Soil Sampling Procedures Section.

General Soil Sampling Procedures:

1. If the QAPP requires field screening for VOCs using a PID, follow the procedures described in the Field Screening Section.
2. Collect samples in appropriate containers in order of volatility, with the most volatile samples collected first. Containers should be either pre-labeled or labeled immediately after sample collection. Follow the procedures for collecting volatile samples described in the following section.
3. For non-VOC samples, place the material into the appropriate container.
4. If a composite non-VOC sample is required, place the material from the designated sampling intervals or locations into a mixing bowl, mix thoroughly, and collect the sample from the mixture into the appropriate container.
5. If non-VOC duplicate, split, duplicate, or other quality assurance/quality control (QA/QC) samples are required, collect twice the routine amount of sample material, mix thoroughly, and fill two identical sets of sample containers.
6. Fill sample containers to the top with measures taken to prevent soil from remaining in the lid threads prior to being sealed.
7. After sample containers are filled, immediately seal them, chill them, and process them for shipment to the laboratory.

Volatile Sample Collection – Sample Collection for Methanol or other Preservative Extraction

General procedures for all volatile sample collection:

- Soil samples for volatile analysis can be collected using any of the sampling methods described above.
- When collecting soil for volatile sample analysis, always submit a separate non-preserved sample for moisture analysis/dry weight calculation, unless already submitting non-volatile samples from the same location.
- Never composite VOC samples.
- If VOC duplicate, split, or other QA/QC samples are required, collect and containerize samples that are co-located, not composited.
- If VOC samples are required, transfer the sample into a labeled sample container with a stainless steel laboratory spoon, or equivalent, and secure the cap tightly.
- Avoid placing pebbles or other large particles into the sample. To the extent practical, the sample should consist of sand, silt, or clay, with care to avoid rocks or pebbles.
- Ensure that the threads on the sample container and cap are free of soil particles. Wipe with a clean brush or paper towel if needed. The sample container should be open for the shortest time possible to prevent evaporation of the methanol and surrogate solution.
- After soil is placed in methanol or other preservative, it should be gently agitated or swirled so that the soil is immersed in the preservative. Do not shake the sample, as it may cause undue volatilization.

The different methods of collecting volatile samples with field extraction, using methanol or another preservative, are described in general below. Refer to the project QAPP for site-specific information on specific soil and methanol volumes required for the appropriate analytical method:

<u>Measuring 10 grams of soil into a VOA vial containing methanol</u>:

1. "Zero" one 40-milliliter volatile organic analyte (VOA) vial containing 10 milliliters of methanol on a small scale.
2. Use a disposable scoop to collect soil.
3. Very gently, transfer the soil into the vial until 10 grams of soil is weighed. Try not to let any soil drop outside the sample container onto the scale. Immediately cap the vial.
4. Ensure that the methanol does not splash. If methanol splashes or spills from the sample container, discard the container and re-sample.
5. Record the tare weight onto the sample sheet or label.

6. Repeat the process for the second VOA vial containing methanol.
7. Place the samples in a protective sleeve and store on ice until delivery to the laboratory.

Using a sampling coring device to collect soil for VOC analysis:

1. Coring devices (for example, En Core® or Terracore®) are disposable, and are not to be reused after each sample.
2. Push the core sampler into freshly exposed soil until the sample chamber is filled. Most of these devices deliver approximately 5 grams of soil.
3. Once the core is filled with soil, retrieve the coring device from the soil.
4. Wipe all soil from outside of the sampler. The soil plug should be flush with the mouth of the sampler.
5. If the QAPP requires using a preservative (for example methanol or sodium bisulfate), use the following preservation procedure:
    a. Place the mouth of the sampler into a pre-tared VOA vial containing the appropriate preservative, and extrude the sample by pushing the plunger down. Immediately cap the VOA vial.
    b. Place the labeled sample in protective padding and on ice.
6. If the QAPP requires freezing the samples unpreserved, use the following procedure:
    a. Place the mouth of the sampler into a pre-tared VOA vial containing exactly 5 milliliters of deionized water, and extrude the sample by pushing the plunger down.
    b. Cap the VOA vial and be sure the soil is below the water level. Gently swirl the vial.
    c. Repeat the process to collect a second soil vial.
    d. Immediately place the labeled sample in protective padding and on ice.
    e. As soon as practical, freeze sample in a freezer or by placing in a cooler containing dry ice.
    f. When freezing the soil vials, it is recommended that the vials be placed at a 45° angle to reduce the likelihood of vial breakage due to freezing.
7. If the QAPP requires submitting unpreserved, unfrozen samples that were collected using an EnCore device, use the following procedure:
    a. Immediately place the cap on the open end of the core. Place the capped core inside the foil sample bag. Make sure that the sample bag is labeled.
    b. Place the sample bag on ice for shipment to the laboratory for analysis within 48 hours.

Quality Control:

The following procedures apply:

- Samples will be packaged, handled, and shipped in accordance with SOP BERS-03 *Sample Management Procedures.*
- Equipment will be operated and used in accordance with the manufacturer's instructions, unless otherwise specified in the site QAPP.
- Equipment examination activities should occur prior to field deployment, and they should be documented.
- An equipment rinsate blank is generally required per matrix, and for each sampling event, to evaluate the potential of cross contamination from sampling equipment. Equipment rinsate blanks will be collected by pouring analyte-free water over the decontaminated sampling equipment.
- Depending on the needs of the project, a field blank may be required per matrix and for each sampling event to evaluate whether contaminants have been introduced into the samples during the sampling process. Field blank samples will be obtained by pouring analyte-free water into a sampling container at the sampling point.

Interferences and Potential Problems:

There are two primary problem areas associated with soil sampling: cross-contamination and improper sample collection.

Cross-contamination can be eliminated or minimized through the use of dedicated sampling equipment. If this is not possible or practical, field personnel will decontaminate sampling equipment as described in the site Sampling and Analysis Plan (SAP). Improper techniques may include using contaminated equipment, disturbing the matrix, compacting the sample, and inadequate homogenization of the samples, any of which can produce non-representative samples.

To safeguard against collecting non-representative soil samples, the following guidelines and techniques should be adhered to during sampling:

- Samples for VOC analysis should be collected before other samples are collected, and should be transferred immediately from the sampling device into the sample container to reduce volatilization. Step-by-step instructions for field extraction and preservation with methanol are described above.
- Anytime a vertical or near vertical surface, such as that which is achieved when shovels or backhoes are used for subsurface sampling, the surface should be dressed to remove the outer smear zone. This is necessary to minimize the effects of cross contamination due to smearing of materials from other levels.

- It is extremely important that soil samples intended for non-VOC analyses be mixed as thoroughly as possible to ensure that each sample is representative of the material sampled. The most common method of mixing is referred to as quartering. Where required by the QAPP, quartering will be performed as follows:
  a. Divide the material in the sample pan into quarters and mix each quarter individually.
  b. Mix two quarters to form halves.
  c. Mix the two halves to form a homogenous matrix.
  d. Repeat this procedure until the sample is adequately mixed.
  e. If round bowls are used for sample mixing, stir the material in a circular fashion, reversing direction, and occasionally turning the material over.
- One trip blank per cooler is generally required when submitting samples for VOC analysis. Trip blanks are prepared and sealed by the laboratory. They are transported to the field and returned, unopened, to the laboratory in the same cooler as the samples collected for VOC analysis.
- Methanol blanks may also be required when soil samples designated for VOC analysis are preserved with methanol.
- Blanks will be collected at the frequency and locations specified in the site QAPP. Blanks will be analyzed for the same target analytes as the associated field samples. Each blank will be assigned a unique sample number, and submitted blind to the laboratory.

BRISTOL ENVIRONMENTAL REMEDIATION SERVICES, LLC

SAMPLE MANAGEMENT

STANDARD OPERATING PROCEDURE BERS-03

Record of Changes

Revision No.	Date	Prepared by	Approved by
0	01/15/08	B. Allen	S. Ruth
1	2/23/2010	M. Faust	B. Allen

SAMPLE MANAGEMENT

STANDARD OPERATING PROCEDURE

Method Summary: To ensure the quality and integrity of analytical data, samples will be managed in accordance with rigorous sample handling, shipping, and custody protocols at all times. Pertinent protocols will be determined prior to initiation of field sampling activity and will apply to sampling, transport, and analysis activities.

Health and Safety: Sampling activity should only be conducted in accordance with an approved Site Health and Safety Plan.

Interferences and Potential Problems: Improper sample management may result in a number of problems, including, but not limited to:

- Inability to collect samples during the field event due to lack of appropriate sample containers and/or preservatives.
- Contamination and/or loss of samples or sample constituents through improper storage and handling, tampering, or breakage.
- Inability to validate resulting data.
- Development of erroneous conclusions regarding site contamination based on inaccurate data and/or problems correlating data and sample locations at the site.
- Mishandling of residual sample material following analysis.

Personnel Qualifications: Sample management personnel will be trained and certified as hazardous site workers per Title 29 Code of Federal Regulations, Part 1910.120(e) [29 CFR 19 10.120(e)] and trained in applicable DOT sample shipping regulations of 49 CFR Part 172, Subpart H. If applicable, additional qualification requirements will be specified in the site-specific Quality Assurance Project Plan (QAPP) and met by designated personnel.

Equipment and Materials: Equipment selection will be based on the objectives of the sampling program and the analytes of concern. Prior to deployment in the field, the requisite sampling equipment and materials will be identified, secured, and inspected for signs of damage or potential contamination.

Sample Identification and Labeling: Sample identification and labeling protocols will follow the procedures specified in the governing program QAPP.

Each collected sample will be assigned a unique sample identification number. The designated sample number will be included on the sample label and referenced on associated sample tags, field logbooks, chain-of-custody forms, analysis request forms, and all data reports related to the samples.

To prevent misidentification of samples, the field team will affix legible labels to each sample container. The labels will be sufficiently durable, and an indelible pen will be used to record data on the labels, so that sample identification information remains legible even when wet. Markers should never be used for sample labeling, as they can be a source of volatile compounds and potential contamination of the sample. Additional labeling requirements will be presented in the site-specific QAPP.

Information that is generally included on the container label and/or sample tag includes:

- Sample identification number;
- Sample collector's name or initials;
- Date and time of sample collection;
- Chemical/physical preservatives used;
- Type of sample (composite, grab, filtered); and
- Analytical parameters requested

Sample Containers and Coolers: Sample containers will be selected, prepared, cleaned, and controlled in accordance with EPA Office of Solid Waste and Emergency Response (OSWER) Directive #9240.0-05A *Specifications and Guidance for Contaminant-Free Sample Containers* (EPA *540/R-93/05* 1, December 1992), and as specified in the governing program QAPP. In advance of each sampling event, the subcontract laboratory should prepare a complete set of precleaned sample containers.

Prior to field activity, field personnel will implement the following steps:

1. Check all sample containers against the specifications of the site-specific QAPP. Ensure that the sample containers and caps are in good condition and free of obvious contamination, constructed of the appropriate material (i.e., plastic or glass), contain appropriate preservative solutions, and will hold sufficient volume for planned analyses, if specified.
2. Verify that sample identification labels are properly affixed to each container.
3. Verify that an adequate quantity of each type and volume of sample container is available for the anticipated environmental and quality control samples. Verify that extra containers are readily available to field staff as contingency for damaged or potentially contaminated containers, and for collecting samples of opportunity.
4. Ensure that containers and coolers are stored in clean areas to prevent exposure to fuels, solvents, and other potential contaminants.

Sample Collection: Field personnel will collect samples as prescribed in the governing QAPP. Samples should be transferred in the field from the sampling equipment directly into

a container that has been specifically prepared for that sample (based on the analytes of concern, preservation requirements, and the type of analysis to be performed).

To minimize the potential for cross-contamination and loss of sample constituents, sample fractions should be collected and containerized in the order of volatilization sensitivity of the analytes of interest. The following sample collection order is recommended:

- Volatile organic compounds (VOCs)
- Purgeable organic carbon
- Purgeable organic halogens
- Total organic halogens
- Total organic carbon
- Extractable organic compounds
- Metals
- Phenols
- Cyanide
- Sulfate and chloride
- Turbidity
- Nitrate and ammonia
- Radionuclides
- Ignitability
- Corrosivity
- Reactivity

As the samples are being collected, or immediately thereafter, the field sampling team will document the date and time of sample collection, pertinent field information (e.g., sampling depth), and the identity of sampling personnel, on each container label. Additional detail on the sampling event may be documented in the site logbook as appropriate.

Sample Custody: BERS will ensure the integrity and security of all samples under their control, using a stringent chain-of-custody protocol. This will be supplemented as needed to meet all work assignment requirements.

During the sampling event, field personnel will prepare a chain-of-custody form documenting each sample collected as follows:

- Sample numbers, date and time of collection, sampling location, name of the person who collected the samples, preservatives used, and the analyses requested.
- Document each sample transfer on the custody sheet. Ensure that this form remains with the samples until they arrive at, and are processed by, the laboratory.
- When samples are relinquished to a commercial carrier for transport to the laboratory, sign the chain-of-custody form under "Relinquished By," enter the name of the carrier organization under "Received By," and document the date and time of transfer. Upon receipt of the samples, the laboratory sample custodian will similarly sign and date the chain-of-custody form.

Under no circumstance is there to be a break in custody.

Sample Packaging: Unless otherwise specified in the site-specific QAPP, field personnel will implement the following steps when packaging environmental samples for shipment:

- Tighten all sample lids. Verify that all containers are labeled and intact. Verify that all container labels are secure, legible, and complete.
- Bag samples individually in appropriate-sized plastic bags (e.g., Ziploc®) and seal. Up to 3 VOC vials may be packed together in container bags.
- Secure and tape the drain plug on the cooler with fiber or duct tape.
- Spread inert packing material (rubber foam, air pillows, or "bubble" wrap) in the bottom of the bag inside the cooler and place sample bags on top of the packing material.
- Include a temperature blank (a small container filled with water) to be used by the laboratory to determine the internal temperature of the cooler upon receipt at the laboratory.
- Place ice packs (e.g., blue ice) into cooler. If ice packs are unavailable, place ice into doubled heavy-duty polyethylene bags and seal with tape. Put double-bagged ice on top of, and in between, samples. Fill in remaining space with packing material.
- Place the chain-of-custody record into a plastic sealable bag (e.g., Ziploc), seal the bag, and tape it to the inside of the cooler lid.
- Close the cooler and tape the top of the cooler shut. Affix custody seals to the top and sides of the cooler, such that the cooler cannot be opened without breaking at least one seal.
- Mark the cooler with "This End Up" and arrows to indicate the proper upward position.
- Tape a label containing the name and address of the destination to the outside of the cooler.

Sample Scheduling, Delivery, and Holding Times: In work assignments where analytical services are procured from a subcontractor laboratory, the laboratory will be required to designate a point of contact (POC) for both normal business hours, and for emergency situations during off-hours. In addition, the laboratory will be required to designate a sample custodian, who will be notified by the BERS field sampling supervisor each time samples are shipped.

Unless otherwise approved, samples will be delivered to, and received by, the laboratory within 24 hours of collection.

Sample holding time tracking begins with the collection of samples, and continues until the analysis is complete. The site-specific QAPP will specify holding time requirements for each analyte of interest to the project.

Quality Control: No additional QC procedures apply.

Data Management and Records Management: Sampling records will be generated and maintained as prescribed in this procedure and the governing QA plans. Sampling data will be documented on field data sheets or in the logbooks.

(Intentionally blank)

BRISTOL ENVIRONMENTAL REMEDIATION SERVICES, LLC

FIELD MEASUREMENT AND TEST EQUIPMENT

STANDARD OPERATING PROCEDURE BERS-04

Record of Changes

Revision No.	Date	Prepared by	Approved by
0	01/15/09	B. Allen	L. Maserjian
1	2/5/10	M. Hannah	B. Allen

FIELD MEASUREMENT AND TEST EQUIPMENT

STANDARD OPERATING PROCEDURE

Summary: Various types of instruments are used to measure the physical and chemical characteristics of a sample in the field. In general, field measurement and test equipment (M&TE) are maintained and operated according to the manufacturer's instructions specific to each instrument. Field M&TE are inspected for function and damage on a regular basis and prior to each use. All findings are recorded in the appropriate logbook. Field M&TE are calibrated in accordance with the manufacturer's specifications. Calibrations are checked on a regular basis and prior to and after use in the field. When daily calibrations are required, calibrations and/or checks are performed at the beginning and end of the day, and the results are recorded in the field logbook. When daily calibrations are not required during field use, checks against appropriate standards are performed.

Health and Safety: Field activities will only be conducted in accordance with an approved Site Health and Safety Plan.

Interferences and Potential Problems: When multiple measurements are taken from the same sample material, the order in which the measurements are made becomes very important. Conductivity may be affected by temperature of the measured solution; therefore, temperature of the sample should be read first, so that appropriate adjustments can be made in accordance with the manufacturer's instructions.

Personnel Qualifications: Field personnel will be trained and certified as hazardous site workers per Title 29 Code of Federal Regulations, Part 1910.120(e) [29 CFR 1910.120(e)]. If applicable, additional qualification requirements will be specified in the site Quality Assurance Project Plan (QAPP).

Equipment and Materials: Prior to deployment in the field, the appropriate equipment and materials will be identified, secured, and inspected for signs of damage or potential contamination. Manufacturer's instructions and specifications for each instrument used will be maintained in the project files. Materials used for calibration of instrumentation, such as standard solutions, must be traceable to relevant, recognized performance standards.

Planning Considerations: Procedures used for the maintenance and use of field equipment, including those performed by subcontractors and suppliers, will be outlined preceding field activities and in accordance with the procedures outlined in this SOP. Equipment must be inspected prior to use in the field for damage and function. Calibration and maintenance of field equipment will be performed according the manufacturer's instructions for that particular instrument. The required frequency of calibration varies between instruments. While some instrumentation must be calibrated only annually or semi-annually, other instrumentation must be calibrated daily during use in the field. Instrumentation that does not require field calibration usually requires a check against a standard. Attention should be paid

to specific requirements for each instrument used in the field, and it is important to remember that the requirements for each instrument may differ.

Instructions for the Maintenance and Use of Field Equipment: Refer to the following sections for instructions on the proper calibration, maintenance, and use of field instrumentation used to measure physical/chemical properties of sample material:

Multi-parameter Water Quality Meter: Many water quality meters are capable of measuring several parameters, such as temperature, conductivity, pH, dissolved oxygen (DO), and oxygen reduction potential (ORP). The following sections provide general instructions for calibrating each parameter. The field personnel will adhere to the calibration instructions for the each instrument used.

Temperature

Temperature, defined as a measure of hotness or coldness on a defined scale, is measured using a thermometer. Three types of thermometers are commercially available: digital (thermocouple) thermistor; glass bulb, mercury-filled thermometer; and bi-metal strip dial indicator thermometer.

Calibration

Thermometers will be calibrated in accordance with the manufacturer's instructions or calibrated semi-annually against a National Institute of Standards and Technology (NIST)-certified thermometer. Thermistors should be checked against a mercury bulb thermometer in water prior to use, and should agree within ± 0.5° degree Celsius (°C).

Maintenance

All thermometers should be inspected regularly and prior to use for leaks, cracks, and function.

Use

Measurements should be made in situ, when possible. To measure the temperature of sample material, perform the following steps:

1. Clean the probe with deionized water, and immerse into the sample.
2. Swirl the thermometer in the sample.
3. Allow the thermometer to equilibrate with the sample.
4. Suspend the thermometer away from the sides and bottom to observe the reading.
5. In a logbook, record the reading to the nearest 0.5° C.
6. Report results to the nearest 0.5° C.

Conductivity: Conductivity, the quality or power of conducting or transmitting, is typically measured using the Wheatstone bridge meter. Conductivity is measured in millisiemens per centimeter (mS/cm) at 25° C. While the sample temperature may be lower, nearly all conductivity meters will convert specific conductance (which is not corrected for temperature) to conductivity.

Calibration

Conductivity will be calibrated in accordance with the manufacturer's instructions. During use in the field, checks against a one-point standard will be performed to ensure the accuracy of the meter, and results will be recorded in a field logbook. The following steps will be implemented both before and after use of the meter to measure the conductivity of sample material in the field:

1. Check and record the temperature of the standard solutions.
2. Rinse the probe with analyte-free water before immersing it in the standards solution.
3. Turn the probe on, immerse it in the standard solution, and record the results.
4. If the meter is not accurate to within ± 10% of the standards, correct the problem before proceeding.

Maintenance

All conductivity meters should be inspected regularly and prior to use for damage and function. Conductivity sensors may become fouled with minerals or other materials, and may require cleaning in the field. Cleaning is accomplished by passing a nylon brush along the sensor surface in a light scrubbing motion, until a metallic shine appears on the sensor. Follow up the cleaning with a fresh or deionized water rinse. DO NOT use a metal brush to clean the sensor surface.

Use

1. Collect the sample and record its temperature.
2. Correct the instrument's temperature adjustment to the temperature of the sample (if necessary).
3. Immerse the probe in the sample. Keep the probe away from the sides and bottom of the container, and ensure that the sensor is in full contact with the sample.
4. Record the results in a logbook.
5. Rinse the probe.
6. Report results to the nearest ten units for readings below 1,000 mS/cm at 25° C and the nearest one hundred units for readings above 1000 mS/cm at 25° C.

Hydrogen Ion Concentration (pH): The pH of a solution is defined as the negative logarithm of the effective hydrogen ion concentration in gram equivalents per liter. The pH is used to measure acidity and alkalinity on a scale ranging from 0 to 14, with 7 representing neutrality. Orion and YSI Water Quality Monitoring System meters are examples of commercially available meters used to measure the pH of liquid-state material.

Calibration

Any pH meter will be calibrated in accordance with the manufacturer's instructions. During use in the field, a two-point or three-point standard will be used to ensure the accuracy of the meter. Results will be recorded in a field logbook. The expected pH of the sample to be collected, estimated from either historical data or by using four-color pH paper, should fall between the two buffering points. Both prior to and after use in the field, the following procedures should be followed as a minimum:

1. Remove the meter from storage and allow it to equilibrate to ambient temperature.
2. Select either pH 4 and pH 7, or pH 7 and pH 10, as the appropriate standard solutions as described above.
3. Use a thermometer to determine the temperature of the buffering solutions, and record the temperature.
4. Rinse the probe with analyte-free water, and immerse it into the pH 7 buffer and set the meter to 7. If the solution temperature is not at 25°C, a table with corrected pH values can be found on the calibration solution bottle or in the operations manual.
5. Rinse the probe with analyte-free water and immerse it into the second buffer, and record the reading.
6. Rinse and store the probe in a container filled with analyte-free water.

Maintenance

All pH meters should be inspected for damage and function regularly and prior to use. During use, periodically check the calibration of the meter by rinsing it with analyte-free water and immersing it into the pH 7 buffer solution.

Use

Follow these steps when measuring the pH of a sample:

1. If measuring temperature, record temperature prior to measuring pH.
2. Immerse the probe in the sample, keeping it away from the sides and bottom of the container. Allow the probe to equilibrate with the sample material.
3. With the probe suspended away from the container surface, record the pH.
4. Rinse the probe with analyte-free water and store in a container filled with analyte-free

water until the next sample is ready.

5. Record results to the nearest 0.1 Standard Unit (SU).

Storage

After use, rinse the unit with fresh water or Alconox®, followed by fresh water, at contaminated sites. Leave a small amount (20mL) of pH 4 solution in the storage cup before sealing the unit in order to keep the pH sensor moist during storage.

Dissolved Oxygen (DO): The membrane/electrode (ME) is the most commonly used instrument for measuring the dissolved oxygen present in a sample.

Calibration

Calibrate the DO probe according to the manufacturer's instructions, either in air-saturated water, or in a water-saturated air environment.

Maintenance

The DO probe should be inspected regularly and prior to use for damage and function. The membrane of the DO meter should be inspected for air bubbles, holes, and dryness. If the membrane is dry, replace and soak it in analyte-free water prior to calibration of the meter. If the metallic sensor is discolored, or does not appear shiny, use the fine-grit sandpaper (supplied with the DO sensor replacement kit) and buff the metal surface in a circular pattern until the surface shines. Rinse the sensor with deionized water before installing a new membrane.

Use

When measuring DO in situ with a field probe, follow these steps:

1. Allow the DO reading to stabilize.
2. Read the dial to the nearest 0.1 mg/L, and record the measurement.

Oxygen Reduction Potential (ORP): ORP, also known as redox potential, is the tendency of a chemical species to acquire electrons and thereby be reduced. Each species has its own intrinsic reduction potential; the more positive the potential, the greater the species' affinity for electrons and tendency to be reduced.

Calibration

Calibrate the ORP probe according to the manufacturer's instructions in a standardized calibration solution. The ORP is affected by temperature. Refer to the calibration solution or operations manual to correct for temperature during calibration.

Maintenance

The ORP probe should be inspected regularly and prior to use for damage and function.

Use

When measuring ORP in situ with a field probe, follow these steps:

1. Immerse the probe in the sample, keeping it away from the sides and bottom of the container. Allow the probe to equilibrate with the sample material.
2. With the probe suspended away from the container surface, record the ORP to the nearest 1.0 millivolt.
3. Rinse the probe with analyte-free water and store in a container filled with analyte-free water until the next sample is ready. Do not store the unit in deionized water.

Turbidity Meter: A nephelometer/turbidmeter is used to measure the turbidity of a liquid sample by determining how much light can pass through it. The Hach® Turbidimeter is the most commonly used commercially available meter for measuring the turbidity of a sample. Turbidity is measured in nephelometric turbidity units (NTUs).

Calibration

Calibration of turbidity meters will be performed in accordance with manufacturer's instructions. Any turbidity meter must be calibrated at both the beginning and end of the day during use in the field, and results will be recorded in a field logbook. The following procedures will be used to calibrate a turbidity meter in the field:

1. Turn the meter "ON" and allow 2 minutes for the lamp to stabilize.
2. Rinse the sample cell with organic-free or deionized water.
3. To "zero" the calibration, fill the cell to the fill line with organic-free or deionized water and then cap the cell.
4. Use lens paper to wipe off excess water and streaks from the outside of the cell.
5. Open the cover and insert the cell (arrow to the front) into the unit and close the cover.
6. Press "Blank" and wait for the "light bulb" icon to go off. Record the reading.
7. Hach turbidity meters require calibration with known standards. Refer to the operations manual for information on calibrating the meter.
8. Using the Gelex Turbidity Standards, repeat steps 4, 5, and 6. Record all findings.

Maintenance

Turbidity meters should be inspected regularly and prior to use for damage and function. During use, periodic checks should be performed using the standards to ensure continued proper calibration of the instrument. If error codes appear on the unit display, refer to the owner's manual to resolve the error.

Use

Follow these steps to measure the turbidity of a sample:

1. Pour sample material into the cell to the fill line and replace the cap on the cell.
2. Wipe excess water and any streaks from the outside of the cell with lens paper.
3. Place the cell inside the measurement chamber with the arrow towards the front and close the cover.
4. Press "READ" and wait for the "light bulb" icon to turn off
5. Record the reading.
6. Empty the cell and rinse with organic or analyte-free water.

Quality Control: The following procedures apply:

- Equipment will be operated and used in accordance with the manufacturer's instructions, unless otherwise specified in the site-specific work plan or its equivalent.
- Equipment examination activities will occur prior to field deployment, and they should be documented.

Calculations and Data Reduction: Does not apply.

Data Management and Records Management: Equipment calibration and maintenance records will be generated and maintained as prescribed in the governing QAPPs.

(Intentionally blank)

BRISTOL ENVIRONMENTAL REMEDIATION SERVICES, LLC

EQUIPMENT DECONTAMINATION

STANDARD OPERATING PROCEDURE BERS-05

Record of Changes

Revision No.	Date	Prepared by	Approved by
1	10/14/09	B. Allen	L. Maserjian
2	2/23/10	L. Maserjian	B. Allen

EQUIPMENT DECONTAMINATION

STANDARD OPERATING PROCEDURE

Summary: Disposable tools and equipment should be used when possible. However, where non-disposable items are used, appropriate decontamination will be accomplished according to the type of equipment being used and the type of samples being collected. In general, field equipment will be decontaminated by means of the following steps:

1. Perform non-phosphate detergent and tap water wash, using a brush if necessary.
2. Perform tap-water rinse.

When sampling for trace organic compounds, the following step will be added:

3. Perform deionized/distilled water rinse.

Health and Safety: Field activities should only be conducted in accordance with an approved Site Health and Safety Plan. Decontamination hazards and precautions include the following:

- Hazardous substances may be incompatible with decontamination materials. For example, the decontamination solution may react with contaminants to produce heat, explosion, or toxic products. Also, vapors from decontamination solutions may pose a direct health hazard to workers by inhalation, contact, fire, or explosion. The Site Health and Safety Plan will provide procedures and identify responsibilities to ensure that incompatible materials are identified and segregated from each other.
- The Site Health and Safety Plan will specify the use of personal protective equipment (PPE) that is appropriate for both the contaminants of concern and the decontamination chemicals used. The PPE selection will take into account that decontamination materials may degrade protective clothing or equipment, and that some solvents can permeate protective clothing.
- Solvent rinsing operations will be performed in well-ventilated areas.
- Investigation-derived waste (IDW) generated from decontamination activities will be managed as prescribed in SOP BERS-09: *IDW Management.*
- Material Safety Data Sheets (MSDS) will be kept with all decontamination solvents or solutions as required by the Hazard Communication Standard.
- Phosphate-containing detergents will not be used in jurisdictions where they are banned.

Interferences and Potential Problems: Potential problems related to equipment decontamination can be eliminated by the use of appropriate materials, reagents, and techniques.

- The use of distilled and/or deionized water commonly available from commercial vendors may be acceptable for decontamination of sampling equipment.

- The use of an untreated potable water supply is not an acceptable substitute for tap water. Tap water may be used from any municipal or industrial water treatment system.
- If acids or solvents are utilized in decontamination, they raise health and safety and waste disposal concerns.
- Washing complex and sophisticated sampling equipment with acids or solvents can damage the equipment.
- If not used immediately, cleaned equipment will be stored to prevent recontamination.
- PVC and plastic items will not be rinsed with solvents.

Personnel Qualifications: Field personnel will be trained and certified as hazardous site workers per Title 29 Code of Federal Regulations, Part 1910.120(e) [29 CFR 1910.120(e)]. If applicable, additional qualification requirements will be specified in the site Quality Assurance Project Plan (QAPP).

Equipment Requirements: Prior to deployment in the field, the requisite sampling equipment and materials will be identified, secured, and inspected for signs of damage or potential contamination. Decontamination equipment, materials, and supplies are generally selected based on availability. Other considerations include the ease of decontaminating or disposing of the equipment.

The following standard materials and equipment are recommended for decontamination activities:

- Non-phosphate detergent.
- Tap water.
- Distilled/deionized water
- Pesticide grade solvent
- Long- and short-handled brushes
- Bottle brushes
- Drop cloth/plastic sheeting
- Paper towels
- Plastic or galvanized tubs or buckets
- Pressurized sprayers (H_20)
- Solvent sprayer with Teflon nozzle
- Aluminum foil
- Plastic sheeting

- PPE
- Trash bags
- Trash containers
- 55-gallon drums
- Metal/plastic buckets/containers for storage and disposal of decontamination solutions.

The appropriate materials and equipment will be selected as needed on a site-specific basis.

Planning Considerations: Equipment decontamination activities, including those performed by subcontractors and suppliers, will be planned in advance of field activities and in consultation with program health and safety personnel.

Decontamination: Depending on the nature of the work, field equipment requiring decontamination may include heavy equipment, downhole equipment, sampling equipment, and groundwater pumping equipment.

Heavy Equipment Decontamination: Heavy equipment includes the drilling rig and backhoe. Field personnel will implement the following steps to decontaminate heavy equipment:

1. Set up a decontamination pad that is large enough to fully contain the equipment to be cleaned. Use one or more layers of heavy plastic sheeting to cover the ground surface.
2. Spray areas of the equipment that may have been exposed to contaminated soils using steam or high-pressure sprayer and detergent. Be sure to spray down all surfaces, including the rear area of the undercarriage.
3. Rinse the equipment with potable water.
4. Remove equipment from the decontamination pad and allow to air dry.

Downhole Equipment Decontamination: Downhole equipment includes hollow-stem augers and drill pipes. Well casings and screens will be decontaminated as decribed under "Sampling Equipment". Field personnel will implement the following steps to decontaminate downhole equipment:

1. Set up a centralized decontamination area, if possible. This area should be set up to contain contaminated rinse waters, and to minimize the spread of airborne spray.
2. Set up a "clean" area upwind of the decontamination area to receive cleaned equipment for air drying. At minimum, clean plastic sheeting must be used to cover the ground, tables, or other surfaces where decontaminated equipment is to be placed.
3. Wearing the required PPE, use a high-pressure sprayer or steam unit and detergent to clean the contaminated equipment. Aim downward to avoid spraying outside the decontamination area. Be sure to spray inside corners and gaps. If necessary, use a brush to dislodge dirt or debris.

4. Rinse the equipment using potable water.
5. Remove the equipment from the decontamination area and place in the clean area to air dry.
6. Cover the equipment to prevent contamination if the equipment is not used immediately.
7. Collect all contaminated waters, plastic sheeting, and disposable gloves, boots, and clothing in the designated containers. Receptacles containing contaminated items must be properly labeled for disposal. Containerize liquids and solids separately.

Sampling Equipment Decontamination: Sampling equipment includes split spoon samplers, spatulas, compositing bowls, and other utensils that come into direct contact with samples.

Field personnel will collect disposable sampling equipment in the designated containers and dispose of them as prescribed in the Site Health and Safety Plan and SOP BERS-09: *IDW Management.* Field personnel will implement the following steps to decontaminate non-disposable equipment:

1. Set up a decontamination line on plastic sheeting. The decontamination line should progress from dirty to clean, and end with an area for drying decontaminated equipment. At minimum, use clean, plastic sheeting to cover the ground, tables, or other surfaces on which decontaminated equipment will be placed. Set up a containment system for collecting wash/rinse waste.
2. Wash the item thoroughly in a bucket of soapy water. Use a stiff-bristle brush to dislodge dirt or debris. Before washing, disassemble items that might trap contaminants internally. Do not re-assemble until decontamination is complete.
3. Rinse the item in potable water. Rinse water should be replaced as needed, generally when cloudy.
4. Allow to air dry.
5. Collect all contaminated waters, plastic sheeting, and disposable gloves, boots, and clothing in the designated containers. Receptacles containing contaminated items must be properly labeled for disposal. Liquids and solids must be drummed separately.

Groundwater Sampling Pumping Equipment Decontamination: Field personnel will implement the following steps to decontaminate sampling pumps:

1. Set up a decontamination area and a separate clean storage area using plastic sheeting to cover the ground, tables, and other porous surfaces where decontaminated equipment will be placed. Set up three clean containers of the appropriate size and shape for immersing the pump assembly. Fill the first container with dilute, non-foaming soapy water, and the second with potable water. Use the third container for waste discharge.

2. If decontaminating an electric submersible pump (e.g., Grundfos® Redi-Flo), remove the bottom screw plug to flush the cooling water. Replace this water with deionized water after the decontamination process is complete.
3. Set up the pump assembly in the same configuration as used for sampling. Submerge pump intake and all downhole wetted parts (tubing, piping, and foot valve) in the soapy water container. Place the discharge outlet in the waste container above the level of wastewater. Pump soapy water through the pump assembly until it discharges to the waste container.
4. Move the pump assembly to the rinse water container while leaving discharge outlet in the waste container. Ensure that all downhole wetted parts are immersed in the potable water rinse. Pump potable water through the pump assembly until it runs clear.
5. Pump a sufficient amount of analyte-free water through the hose to flush out the tap water, then purge with the pump in reverse mode. Rinse the outside of the pump using analyte-free water. Decontaminate the discharge outlet by hand following the steps for decontamination of sampling equipment.
6. Remove the decontaminated pump assembly to the clean area and allow to air-dry.
7. Cover intake and outtake orifices with aluminum foil to prevent the entry of airborne contaminants or particles.
8. Place pump in clean plastic bag.

Quality Control: The following procedures apply:

- Equipment will be operated and used in accordance with the manufacturer's instructions, unless otherwise specified in the site-specific work plan or its equivalent.
- Equipment examination activities should occur prior to field deployment, and should be documented.
- After decontamination activities, the field personnel should make a record of the equipment type, date, time, and method of decontamination in the field logbook.
- If sampling equipment requires the use of plastic tubing, dispose of it as contaminated. Replace with clean tubing before conducting additional sampling.

Calculations and Data Reduction: Does not apply.

Data Management and Records Management: Generate and maintain decontamination records as prescribed in the governing QAPPs.

(Intentionally blank)

BRISTOL ENVIRONMENTAL REMEDIATION SERVICES, LLC

BOREHOLE LOGGING

STANDARD OPERATING PROCEDURE BERS-06

Record of Changes

Revision No.	Date	Prepared by	Approved by
0	01/15/09	B. Allen	L. Maserjian
1	2/23/10	L. Maserjian	B. Allen
2	4/01/10	L. Kleppin	L. Maserjian

BOREHOLE LOGGING

STANDARD OPERATING PROCEDURE

Summary: Boreholes are typically advanced for the purposes of well or piezometer installation, subsurface soil sampling, or rock coring. Drill rigs (typically hollow-stem auger, air rotary, or cable tool), or direct-push technologies (i.e., Geoprobe®) are the most common methods for advancing borings. Because borehole logging typically involves the collection of subsurface soil samples to describe the geologic material, this procedure supplements what is presented in the downhole sampling portion of the SOP BERS-01: *Soil Sampling Procedures.*

To facilitate uniformity in description, boreholes are generally logged on the basis of their identifiable physical characteristics, rather than assigning interpreted depositional soil types or rock names (i.e., glacial till) that may represent the bias or experiences of the field geologist/engineer. This procedure uses the term soil to identify any unconsolidated geologic material, typically of sedimentary or volcanic origin. The term rock is used to describe any consolidated or indurated geologic material.

Health and Safety: Subsurface sampling should only be conducted in accordance with an approved Site Health and Safety Plan. Prior to the commencement of drilling activities, all boring locations must be verified free and clear of underground and overhead utilities.

Interferences and Potential Problems: Several factors can potentially result in problems in the logging and physical description of a soil boring, such as incomplete subsurface sampling, sample collection from unknown depth intervals, and inconsistent description of soil or rock types by two different geologists/engineers.

Field personnel will take the following considerations into account during soil boring activities:

- When collecting a subsurface soil sample with an apparatus such as a split-spoon, recovery of the entire sample interval is often incomplete. Even if the entire length of the split-spoon is driven, factors of soil-pore pressure, density, or texture often result in partial sample recovery. In these circumstances, it is important to note that the recovered portion of the sample is not necessarily representative of the entire sample interval.
- Depending on the project objectives, continuous subsurface sampling may not be specified in the work plan. Consequently, gaps are present in the borehole log regardless of sample recovery. It must be recognized that interpolating the geology between sample intervals contains an element of uncertainty, the magnitude of which is dependent of the heterogeneity of the geologic material.
- After advancing the drill bit to the sample depth interval, it is necessary to thoroughly remove all cuttings and loose material from the bottom of the hole prior to sampling. Failure to perform this task will result in the sampling of "sluff," i.e., soil material from unknown depth intervals. Sluff can often be identified by low-density, homogeneous material at the top of the sample interval, and is not representative of the undisturbed soil.

- When logging a borehole through the description of drill cuttings (as opposed to undisturbed sampling), it is important to recognize the lag time between when the drill bit cuts the soil or rock material, and when these cuttings appear at the surface. Accordingly, cuttings at the surface are not representative of the material being drilled at the bottom of the hole. The duration of the lag time is a function of the depth of the boring, and the method of drilling.
- Problems resulting from the inconsistent description of identical soil or rock types by two different geologists/engineers can be minimized through the use of standard soil/rock classification and grain size charts that reduce the subjective nature of the description. Effective communication between members of the field team will also minimize these problems.

Personnel Qualifications: Personnel will be trained and certified as hazardous site workers per Title 29 Code of Federal Regulations, Part 1910.120(e) [29 CFR 1910.120(e)]. If applicable, additional qualification requirements will be specified in the site Quality Assurance Project Plan (QAPP) and met.

Equipment and Materials: Prior to deployment in the field, the requisite drilling and downhole sampling equipment and materials shall be identified, secured, and inspected for signs of damage or potential contamination.

The types and quantities of equipment and supplies necessary to complete a boring depend on the drilling and sampling method as specified in the work plan. In general, the drilling subcontractor will provide the equipment and supplies necessary for the completion of the boring in compliance with applicable state and federal regulations.

This equipment includes a fully tooled drill rig and all supplies necessary to either complete a well in the boring, or abandon the hole. Such items may include: the drill rig and support vehicles, auger flights, steel casing, hydraulic jacks, welding supplies, well screen and casing, sand, bentonite chip and/or grout, water tanks, steam cleaner, cement, 55-gallon drums, pumps, well development supplies, surface monuments, shovels, and all tools necessary to complete the job. Although the drilling subcontractor typically provides these items, it is the responsibility of the project manager and project geologist/engineer to ensure that all required equipment and supplies are specified and present on site.

Bristol typically provides all items pertaining to health and safety, field screening, sampling, as well as descriptive logging of the borehole. The descriptive logging of a borehole requires certain reference materials to provide an accurate physical description of the geologic material, including: soil and rock classification charts, Munsell color chart, grain size chart, hand lens, scales, an assortment of pens and markers, field logbook, and the Boring Log Form.

Other materials related to health and safety, field screening, or sampling may include: personnel protective equipment, photoionization detector (PID), folding table, spoons, utility knife, shovel, water level indicator, tape measure (graduated in tenths of feet), survey stakes or flags,

laboratory-supplied sample containers, coolers, ice, decontamination supplies, camera, logbooks, chain-of-custody forms, and labeling, packaging, and shipping supplies.

Borehole Logging Procedures: Prior to the initiation of any drilling, sampling, or logging activities, it is necessary to organize and stage all equipment and materials at the borehole location. This may include the following:

1. Decontaminate the drill rig and all sampling equipment.
2. Ensure that utilities have been checked and cleared.
3. Ensure that the water supply is from an approved source.
4. Stage drums or containers for investigation-derived waste (IDW) near the borehole.
5. Establish a convenient area, preferably a solid platform or table, for use while examining geologic material or preparing samples.
6. Have all reference material (e.g., classification and color charts), logbooks, and boring log forms ready and accessible.
7. At the beginning of drilling activities, monitor for organic vapors with a PID in accordance with the SOP BERS-10: *General Air Monitoring and Sampling Procedures*. Air monitoring of the breathing zone around the borehole should continue intermittently (or as specified in the work plan) until total depth (TD) is attained.
8. During drilling activities, record any observations or unusual motions of the rig that may be a function of the hardness or density of the geologic materials. This includes rate and ease of penetration, bit chatter, and bouncing. Record these observations in the field logbook and/or the Boring Log Form.
9. Containerize soil and rock cuttings in open-top 55-gallon drums, or as specified for project IDW. Label each drum with the site name, date, location, depth intervals of contained material, and the nature of the material.
10. Collect subsurface samples at the depths and frequency specified in the site QAPP. For subsurface soil sampling, this will typically be accomplished by driving a split-spoon or pushing a sample sleeve with the drill rig. If a split-spoon sampler is used, drive the spoon using a 140-pound hammer, dropped 30 inches. Record the number of hammer blows (blow count) required to attain full penetration of the sampler (in a series of three, 6-inch intervals). Refer to American Society for Testing Materials (ASTM) Method Dl586 if needed.
11. Upon retrieval of the sample spoon/sleeve, collect all required samples prior to performing any descriptive logging activities. This is especially critical for sample aliquots designated for volatile organic analyses. These procedures are described in detail in the SOP BERS-01: *Soil Sampling Procedures.*
12. Describe the soil on the basis of the physical characteristics (i.e., texture) of the soil sample. Unless otherwise specified in the site QAPP, perform soil identification and description in accordance with the Unified Soil Classification System (USCS, ASTM

Method D2487). Fill material should be described in the same depth of detail as soil samples. Because visual determinations of grain size and grain size distribution (i.e., grading or sorting) are critical to the assignment of a USCS Group Symbol, a grain size reference card will be available to facilitate this estimation, and to provide consistency between different field geologists/engineers. Sample description are listed, in the following order:

1. **Soil Type** (USCS description)
2. **USCS Classification Symbol**
3. **Consistency or Relative Density** (based on blow count)
4. **Moisture Content** (dry, moist, wet)
5. **Color** (based on Munsell chart)
6. **Structures present**, if any (e.g., laminated, bedded)
7. **Additional modifying information** (e.g., grain sizes, particle shape, cementation) and observations or characteristics which may affect the engineering or chemical fate properties of the material (e.g., presence of fill, organics, or rooting)
8. **Odors, sheens, or visual evidence of contamination** (slight petroleum odor, strong solvent odor, etc.)

The following are examples of typical entries to be placed on the Boring Log Form and/or the field logbook:

- SANDY GRAVEL (GP) – medium dense, moist, gray (GLEY 6/N) subrounded to 2-inch maximum diameter, slight petroleum odor
- SILTY SAND (SM) – medium dense, wet, reddish brown (5YR 5/3) fine to medium grained
- SILTY CLAY (CL) – medium stiff, wet, dark greenish gray (GLEY 4/1) occasional fine-grained sand seams 3 inches thick
- GRAVELLY SANDY SILT (ML) – very stiff, moist, dark brown (7.5YR 3/2) abundant cobbles

  (Note: "very stiff" consistency describes silt that is plastic, i.e., like clay)
- SANDY SILT (ML) – medium dense, moist, yellow (10YR 7/6) fine-grained sand

  (Note: "medium dense" relative density describes silt that is non-plastic, i.e., like sand)

USCS Classification Symbol

Two-letter USCS codes should follow the USCS description. Do not use dual symbols such as CL-ML on field logs; use dual symbols when laboratory test results indicate that soil should be

classified as such, for example: If the results of a laboratory grain-size distribution test indicate there is 49 percent gravel, 40 percent sand, and 11 percent silt, the soil should be classified as: SANDY GRAVEL with SILT (GP-GM). If the results of an Atterberg Limit Test indicate that the soil has a plasticity index of 5 and a liquid limit of 20, the soil should be classified as: CLAYEY SILT (CL-ML).

Consistency and Relative Density

Consistency. An estimate of the consistency (if available, use standard penetration resistance as a guide) should accompany descriptions of all fine-grained soils (silts and clays where more than 50 percent of the material would pass the No. 200 sieve). The following consistency terminology should be used:

SPT N-value*	Consistency	Identification Procedure	Approximate Shear Strength (psf)
0 – 2	Very soft	Very easily penetrated several inches with fist	Less than 250
2 – 4	Soft	Easily penetrated several inches by thumb	250 – 500
4 – 8	Medium stiff	Penetrated several inches by thumb with moderate effort	500 – 1,000
8 – 15	Stiff	Readily indented by thumb, but penetrated only with great effort	1,000 – 2,000
15 – 30	Very stiff	Readily indented by thumb nail	2,000 – 4,000
greater than 30	Hard	Indented with difficulty by thumb nail	Greater than 4,000

Note: *SPT (standard penetration test) N-value is defined as the total number of blows required to drive the second and third 6 inches of penetration (the first 6 inches' blow count is also recorded), while driving a 2-inch-OD sampler with a 140-pound hammer falling from a height of 30 inches.

OD = outer diameter

psf = pounds per square inch

If laboratory strength tests are performed, the logs should be corrected if necessary.

Relative Density. Descriptions of all coarse-grained soil (sand and gravel where less than 50 percent of the material would pass the No. 200 sieve and 100 percent would pass the 3-inch size) should be accompanied by an estimate of the relative density. The following guideline terminology should be used:

SPT N-value*	Relative Density
Less than 4	Very loose
4-10	loose
10-30	Medium dense
30-50	Dense
Over 50	Very dense

Note: *SPT (standard penetration test) N-value is defined as the total number of blows required to drive the second and third 6 inches of penetration (the first 6 inches' blow count is also recorded), while driving a 2-inch outer diameter sampler with a 140-pound hammer falling from a height of 30 inches.

Moisture Content

Moisture content should be estimated using only the terminology described below. An estimate of the moisture content should be included immediately following the estimated consistency or relative density. The following terms should be used:

Description	Criteria
Dry	Absence of moisture, dry to the touch
Moist	Damp, but no visible water
Wet	Visible free water, usually below water table

Color

Color is correctly described by comparing the soil sample with a Munsell color chart and applying the correct designations and descriptions. If the Munsell color chart is not available, colors should be described using only red, green, yellow, purple, blue, black, brown, gray, white, or orange. Descriptions such as tan, buff, etc., should be avoided, though modifiers such as light or dark are acceptable. Dual color descriptions (red-brown) can also be used.

Structure

The structural characteristics of undisturbed samples often provide important information. Some of the more common terms are defined below and should be used when applicable.

- Stratified – Alternating layers of different types or colors of soil. If the layers are less than about ¼ inch thick, describe as laminated
- Fissured – Tendency to shear along definite fracture planes with little resistance
- Slickensided – Soils containing polished, glossy surfaces on soil particles or fractures
- Blocky – Easily broken into small angular lumps that resist further breakdown

- Lensed – Inclusion of small pockets of different texture, such as small pockets of sand scattered through a clay mass
- Angularity – Angularity can be described of the coarse grains as follows:

Description	Criteria
Angular	Particles have sharp edges and relatively plane sides with unpolished surfaces
Subangular	Particles are similar to angular description, but have rounded edges
Subrounded	Particles have nearly plane sides, but have well-rounded corners and edges
Rounded	Particles have smoothly curved sides and no edges

Additional Modifying Information

Other descriptive information that should be included, if applicable, are approximate percentages of clay, silt, sand, and gravel; average grain size and maximum size of particles; shape/angularity of coarse grains; general composition or mineralogical description of grains (e.g., granitic, micaceous, etc.) coatings on coarse grains; plasticity; organic content; cementation; and local or geologic name.

- Editorial comments and notes on drilling procedure or difficulty should follow the stratum identification.
- The term "grading" is often misused:

  Misuse – grading lighter
  Better – becomes light brown at 5 feet

  Misuse – grading sandier at 10 feet
  Better – grades to sand at (or below) 10 feet

Odors, Sheens, or Visual Evidence of Contamination

An odor description should always indicate the type of odor (e.g., petroleum, solvent, etc.) and may include modifiers such as strong or slight.

Bedrock Logging Procedure

Consolidated rock material will typically be described and logged through either drill cuttings or diamond coring. At the present time, there is not a unified system for classifying rock. Because there are numerous types of rock classification schemes in common use, the specific format to be used will be specified in the site QAPP, depending on the local geology and terminology. Formal stratigraphic nomenclature can also be used in addition to the rock name. Rock is classified as rock and not as soil, as it sometimes appears in the cuttings. Conversely, rock fragments or coarse gravel should not be referred to as "rock", unless larger than on cubic yard (see also definition of cobbles and gravel).

A systematic procedure is presented below for qualitatively evaluating the physical condition of a rock mass that was originally developed for logging rock encountered in boreholes; however, it is equally applicable to rocks exposed by natural processes or by excavation. A typical description is presented below, followed by discussions of the description.

Typical Descriptions:

- SANDSTONE, gray. little fractured, hard, moderately strong, moderately weathered.
- TALC SCHIST, yellow, thin-bedded, crushed, low hardness, weak, deep weathering, strike NW, dip 50 degrees NE.

Color. Use Munsell numerical designations to describe color names.

Bedding and Splitting. Rock in place varies in character from massive to thinly laminated or foliated. These planar features (layers) were either formed at the time of deposition (as in the case of sedimentary and some volcanic rocks), or may have resulted from forces acting on the rock at depth (as in the case of foliated metamorphic rocks, such as schist).

The following quantitative classification has been developed to describe layered rock.

Splitting Property	Stratification	Thickness
A. Massive	Very thick-bedded	Greater than 4.0 feet
B. Blocky	Thick-bedded	2.0 to 4.0 feet
C. Slabby	Thin-bedded	0.2 to 2.0 feet
D. Flaggy	Very thin-bedded	0.05 to 0.2 foot
E. Shaley or Platey	Laminated	0.01 to 0.05 foot
F. Papery	Thinly laminated	Less than 0.01 foot

Strike and Dip. Strike and dip of rock bedding planes are obtained from oriented rock cores or surface outcrops. Strike is the direction of a horizontal line on a bedding plane related to the points of the compass (e.g., N 30 degrees E). Dip is the vertical angle below a horizontal plane and the direction down dip related to the four quadrants of the compass (e.g., 45 degrees SE). Always note strike and dip on the field log if they are known or can be estimated.

Fracturing. Fractures include joints, faults, shears, and other more or less continuous ruptures in the rock. Joints are fractures (no relative movement of the rock on either side) and can be regular or irregular and discontinuous. Faults or shears are fractures on which movement has taken place. These features tend to reduce the overall mass hardness and strength of the rock. You should describe whether or not fractures contain open voids, or, if they are filled, describe the type of filling (e.g., clay, quartz, etc.)

Categories of fracturing are based on measurements of the lengths of recovered diamond drill core lengths and inspections of trench exposures.

Intensity	Size of Pieces (feet)
Crushed	Less than 0.05
Intensely fractured	0.05-01.
Closely fractured	0.1 – 0.5
Moderately fractured	0.5 – 1.0
Little fractured	1.0 – 4.0
Massive	Greater than 4.0

Hardness. Hardness and strength are interrelated in many ways, and the meaning of the term depends on how it is being used. Hardness is evaluated with a pocket knife on an unfractured specimen. When equating mass hardness, the degree of fracturing should always be considered, as follows:

- Soft – Reserved for plastic material alone
- Low Hardness – Can be gouged deeply or carved with a pocket knife
- Moderately Hard – Can be readily scratched by a knife blade, scratch leaves heavy trace of dust and is readily visible after the powder has been blown away
- Hard – Can be scratched with difficulty, scratch produces little powder and is often faintly visible
- Very Hard – Cannot be scratched with pocket knife, leaves a metallic streak

Strength. Evaluating the strength of rock in the field is usually a somewhat subjective activity in that it depends on both the observer's interpretation and on the way unfractured specimens respond to hammer blows. Consistency between observers can be evaluated through laboratory test results. Some categories of rock strength include:

- Plastic
- Friable – Crumbles by rubbing with fingers
- Weak – An unfractured specimen of such material will crumble under light hammer blows
- Moderately Strong – Specimen will withstand a few heavy ringing hammer blows before breaking

- Strong – Specimen will withstand a few heavy ringing hammer blows and usually yields large fragments
- Very Strong – Rock will resist heavy ringing hammer blows and with difficulty will yield only dust and small flying fragments

Weathering. Both hardness and strength are influenced by weathering, which usually decomposes or disintegrates the mineral constituents of a rock mass. Permeability is also an important control on the depth of weathering. Pre-existing fractures act as access for water and, therefore, as avenues along which the rate of weathering is accelerated.

Quality Control: Appropriate quality control measures for borehole logging may include the following:

- If more than one geologist/engineer is present on site, collaborate on the description of selected soil samples to ensure uniformity of description.
- Collect an assortment of soil samples representative of the variety of geologic materials, and send these samples to a geotechnical laboratory for analysis. In most cases, a grain size (sieve) analysis, followed by a hydrometer test to divide the fine fraction will be suitable. This test (ASTM Method D422) will provide a grain size distribution curve, and identify a USCS Group Symbol on the overall sample. This serves as a control check for the field geologist/engineer.

BRISTOL ENVIRONMENTAL REMEDIATION SERVICES, LLC

WATER LEVEL MEASUREMENT

STANDARD OPERATING PROCEDURE BERS-08

Record of Changes

Revision No.	Date	Prepared by	Approved by
0	01/15/08	B. Allen	S. Ruth
1	2/23/10	M. Faust	B. Allen

WATER LEVEL MEASUREMENT

STANDARD OPERATING PROCEDURE

Purpose and Scope: The purpose of this document is to provide sufficient and appropriate instructions for the determination of the depth-to-water and floating chemical product (i.e., gasoline, kerosene) in an open borehole, cased borehole, monitoring well, or piezometer.

Summary: Prior to measurement, water levels in piezometers and monitoring wells are allowed to stabilize for a minimum of 24 hours after well construction and development. A survey mark is placed on the casing for use as a reference point for measurement. The distance from water surface to reference point on well casing is measured at least twice and recorded.

Health and Safety: Field activities should only be conducted in accordance with an approved Site Health and Safety Plan.

Interferences and Potential Problems: Generally, water level measurements taken in boreholes, piezometers, or monitoring wells are used to construct water table or potentiometric surface maps, and to determine flow direction, as well as many other aquifer characteristics. Situations that may impact the accuracy of water level measurements include:

- The magnitude of the observed changes between wells appears too large;
- Atmospheric pressure changes;
- Aquifers that are tidally influenced;
- Aquifers affected by river stage, impoundments, and/or unlined ditches;
- Aquifers stressed by intermittent pumping of production wells;
- Aquifers being actively recharged due to precipitation event;
- Occurrence of pumping; and
- During storm events over a shallow aquifer where recharge is rapid.

Additional sources of error may include the following:

- The chalk used on steel tape may contaminate the well.
- Cascading water may obscure the water mark, or cause it to be inaccurate.
- Many types of electric sounders use metal indicators at 5-foot intervals around a conducting wire. To ensure accuracy, these intervals should be checked with a survey tape (preferably with units divided in hundredths of a foot).

- If there is product or oil present on the water, it can insulate the contacts of the probe on an electric sounder, or give false readings due to thickness of the oil. If this situation is suspected, it is recommended that interface probes be used to determine the thickness and density of the oil layer in order to determine the correct water level.
- Turbulence in the well and/or cascading water can make water level determination difficult with either an electric sounder or steel tape.

Personnel Qualifications: Field personnel will be trained and certified as hazardous site workers per Title 29 Code of Federal Regulations, Part 1910.120(e) [29 CFR 1910.120(e)]. If applicable, additional qualification requirements will be specified in the site-specific Quality Assurance Project Plan (QAPP) and will be met.

Equipment and Materials: Prior to deployment in the field, the requisite sampling equipment and materials will be identified, secured, and inspected for signs of damage or potential contamination.

There are a number of devices that can be used to measure water levels. The device must be capable of attaining an accuracy of 0.02 feet, and calibrated on a regular basis.

Field equipment for performing water level measurements include:

- Air monitoring equipment (e.g., photoionization detector [PID] or flame ionization detector [FID])
- Well depth measurement device
- Electronic water level indicator
- Metal tape measure
- Chalk
- Ruler
- Watch
- Logbook
- Paper towels
- Groundwater water level data forms
- pH meter (optional)
- Specific conductivity meter (optional)
- Thermometer (optional).

Site Preparation: The following steps will be followed before measurement activities are performed:

- Determine the extent of the sampling effort, the sampling methods to be employed, and the types and amounts of equipment and supplies needed.
- Obtain necessary sampling and monitoring equipment.
- Decontaminate or pre-clean equipment, and ensure that it is in working order.
- Perform a general site survey prior to site entry in accordance with the Site-Specific Health and Safety Plan.
- Identify and mark all sampling locations.

Water Level Measurement: A survey mark should be placed on the north side of the casing for use as a reference point for measurement. Generally, the reference point is marked on the top of the well casing, and is established at the time the well is surveyed. The measuring point should be documented in the site logbook and on a groundwater level data form. Every attempt should be made to notify future field personnel of such reference points in order to ensure comparable data and measurements.

Prior to measurement, water levels in piezometers and monitoring wells should be allowed to stabilize for a minimum of 24 hours after well construction/development. In low-yield situations, recovery may take longer. Measurements should be made to the closest 0.01 feet.

The following procedures will be followed to determine groundwater elevation:

1. Make sure that water level measuring equipment is in good operating condition.
2. To the extent known, measure wells in order of increasing contamination.
3. Clean all equipment entering the well.
4. Remove locking well cap, note well ID, time of day, elevation (top of casing) and date in site logbook or an appropriate groundwater level data form.
5. Remove well casing cap.
6. If required by site-specific conditions, monitor headspace of well with a PID or FID to determine presence of volatile organic compounds, and record in site logbook.
7. Lower electric water level measuring device or equivalent into the well until water surface is encountered.
8. Measure the distance from the water surface to the reference measuring point on the well casing or protective barrier post, and record in the site logbook. In addition, note that the water level measurement was from the top of the steel casing, the top of the PVC riser pipe, the ground surface, or some other position on the wellhead.
9. Groundwater level data should be documented as follows:
    - Logger Name – Person taking field notes;
    - Site Name;

- Date the water levels are measured;
- Location – Monitor well number and physical location;
- Time (24-hour clock) at which the water level measurement was recorded;
- Depth to Water – Water level measurement in feet, tenths, or hundredths of feet, depending on the equipment used. Two measurements are required to ensure accuracy;
- Comments – Any information the field personnel deems applicable may be included here;
- Measuring Point – Marked measuring point on PVC riser pipe, protective steel casing, or concrete pad surrounding well casing, from which all water level measurements for individual wells should be measured. This provides consistency in future water level measurements.

10. Measure total depth of well (at least twice to confirm measurement) and record in field logbook or on groundwater level data form.
11. Remove all downhole equipment; replace well casing cap and locking steel caps.
12. Rinse all downhole equipment and store for transport to next well.
13. Decontaminate all equipment.
14. Note any physical changes, such as erosion or cracks in protective concrete pad or variation in total depth of well, in field logbook and on groundwater level data form.

Quality Control: The following procedures apply:

- Equipment will be operated and used in accordance with the manufacturer's instructions, unless otherwise specified in the site-specific work plan or its equivalent.
- Equipment examination activities should occur prior to field deployment, and they should be documented.
- Each well should be tested at least twice in order to compare results.

Calculations and Data Reduction: Calculations and data reduction will be performed using the following equations and rules:

Groundwater elevation above mean sea level: $E_w = E - D$

where:

E_w = Elevation of water above mean sea level or local datum (feet or meters)

E = Elevation above sea level or local datum at point of measurement (feet or meters)

D = Depth to water (feet or meters)

BRISTOL ENVIRONMENTAL REMEDIATION SERVICES, LLC

FIELD DOCUMENTATION

STANDARD OPERATING PROCEDURE BERS-11

Record of Changes

Revision No.	Date	Prepared by	Approved by
0	01/05/10	L. Maserjian	B. Allen

FIELD DOCUMENTATION

STANDARD OPERATING PROCEDURE

Method Summary: To ensure the quality and integrity of field and analytical data, field activities will be documented in the project field notebook. In the event that more than one person is working on the site and performing different activities, more than one field notebook will be designated for the site. When the field notebook is filled, a new notebook will be started. Pertinent protocols for documenting field activities are provided below.

Notebook Cover: The cover of each field notebook will contain the following information:

- Job title
- Job number
- Name of company
- Name of personnel in charge of notebook
- Date of field activities covered in the notebook.

First Page of Each Day: The following information must be provided in the beginning of each day of work:

- Job title
- Names of all personnel on site
- Weather conditions
- Location, if multiple sites
- Health and Safety meeting notes.

Each Page of Notebook: The following information must be provided on each page of the field notebook:

- Date
- Initials or signature of person taking notes (bottom of page)
- Location, if you have changed during the day
- Page number, if not on the notebook.

Required General Information for Field Notebooks:

- Do not erase mistakes/errors – draw a line through the deletion and initial it.
- Do not leave pages blank. If a page is skipped, draw a diagonal line across the page and initial the line.
- Record persons arriving and leaving site (guests to site, clients, regulatory agency personnel).
- Record health and safety issues that arise (close calls or accidents should also be documented on required forms).
- Note photographs taken and direction in which photograph was taken.
- Take an overview photograph of site before digging/drilling, etc.
- Include a photograph of the site after it is restored (if applicable).

Required Documentation for Sample Collection Activities:

- Instrument name;
- Calibration record (when, by whom, results, gas type);
- Sampling location map with North arrow (field-screening and analytical samples);
- Sample ID, with description of soil material;
- Duplicate information;
- Sample time, each sample;
- Sample depth;
- List what analyses sample will be analyzed for;
- Field-screening measurements;
- Type of machinery used if not already recorded on field forms (Macro-Core sampler, split spoon, pumps, sampling meters);
- If Global Positioning System (GPS) is used, make note of where it was used;
- Delivery or pick-up information (airway bill #, Fed Ex tracking #, Fed Ex pick up information).

Required Documentation for Underground Storage Tank (UST)/Aboveground Storage Tank (AST) Removal Activities:

- UST or AST dimensions;
- Dimensions of tank excavations, depth to groundwater, and depth of excavation;

- Footage of fuel piping (how many feet from dispenser to tanks);
- Where vent lines, fill ports, dispensers and pipe runs are located;
- Location of piping joints;
- Amount of sludge/water removed from tanks prior to decommissioning;
- Amount of contaminated soil/media (cubic yards of stockpiles);
- Amount of contaminated soil or debris hauled from site (number of truckloads);
- Amount of clean fill brought to the site;
- Type of machinery used.

Required Documentation for Monitoring Well/Soil Boring Activities (This list does not include the documentation that will be provided on a boring log and groundwater sample collection form.):

- Always collect swing-tie measurements to monitoring wells (even if you have a GPS);
- If drillers add water during well installation, note how much was added;
- Well screen slot size;
- Well filter sand pack size;
- Depth of top and bottom of well screen;
- Total depth of well;
- Amount of well construction materials used for each well (e.g., bags of silica sand, concrete, amount of screened casing, and amount of blank casing);
- Location of sand filter pack, bentonite seal, and grout used;
- Amount of water removed during development (unless you are using a well development form);
- Drill rig type;
- Changes in level of the water table/ aquifer.

Interferences and Potential Problems: Improper documentation of field activities may result in a number of problems, including, but not limited to:

- Inability to find sample collection locations that is needed for maps or finding areas for further assessment/excavation;
- Inability to create an as-built map;
- Inability to legally support data due to poor documentation;

- Development of erroneous conclusions regarding site contamination based on inaccurate data and/or problems correlating data and sample locations at the site;
- Difficulty in writing thorough reports due to poor documentation.

BRISTOL ENVIRONMENTAL REMEDIATION SERVICES, LLC

Anchorage, Alaska

PHOTOIONIZATION DETECTOR

STANDARD OPERATING PROCEDURE BERS-16

Record of Changes

Revision No.	Date	Prepared by	Approved by
0	03/14/13	M. Faust	P. Yang

PHOTOIONIZATION DETECTOR

STANDARD OPERATING PROCEDURE

Summary: Photoionization detectors (PIDs) are devices used to screen for concentrations of volatile organic compound (VOC) vapors. The main components of the device include an UV lamp, a sensor detector, an ionization chamber, and a sampling pump. A PID works by drawing an air sample into the device with a sampling pump. The air sample is then bombarded with photons emitted by an UV lamp. The VOCs present in the air sample become ionized (positively charged) and produce an electric current. The PID then detects the current, and displays a numerical reading on a light-emitting diode (LED) screen. The numerical reading is proportionate to the magnitude of the current produced by the ionized VOCs. The greater the concentration of VOCs in the air sample, the greater the electrical current produced by photoionization, and the greater the reading displayed on the LED screen.

Photoionization detectors are used for a variety of applications in industries, including as safety devices to determine whether a work environment is safe. For environmental applications, PIDs are typically used to provide qualitative or semi-quantitative data about petroleum hydrocarbon contamination in soil. The following SOP is oriented towards environmental applications and provides information about field screening methods, calibration, and maintenance. This SOP is not necessarily model-specific, although much of the detail is oriented towards the MiniRAE Lite because this is Bristol's preferred PID model. If a different model is used, refer to your instrument's operating manual for model-specific procedures.

Health and Safety: Sampling activity should only be conducted in accordance with an approved Site Safety and Health Plan.

Personnel Qualifications: Sampling personnel will be trained and certified as hazardous site workers per Title 29 Code of Federal Regulations, Part 1910.120(e) [29 CFR 1910.120(e)]. If applicable, additional qualification requirements will be specified in the site Quality Assurance Project Plan and will be met.

Equipment and Materials: Prior to deployment in the field, the requisite PID, ancillary equipment, and materials will be identified, secured, and inspected for signs of damage or potential contamination.

- **PID –** This SOP is written using the MiniRAE Lite as the primary model, which is a product of RAE Systems, Inc.; however, most of the information is relevant to other PID units as well. **Note:** The PID should be fully charged, calibrated, and calibration checked prior to deployment in the field. Procedures for charging, calibration, and calibration checking are detailed below.
- **Calibration gas –** The standard calibration gas to be used is 100 parts per million (ppm) compressed isobutylene gas in a 34-liter compressed-gas steel cylinder. **Note:** Consider how the calibration gas will be transported to the job site. Shipping

- compressed gas requires special labeling and packaging procedures. Another option is to fill Tedlar bags with calibration gas and bring them into the field, which avoids the need to ship compressed gas.
- **Regulator –** Regulators are affixed to calibration gas cylinders to allow calibration gas to be released from the gas cylinder and drawn into the PID. Regulators come in two main varieties: fixed flow and on-demand. If the PID being calibrated utilizes a sampling pump, an on-demand regulator can be used and is preferred to economize the use of calibration gas. **Note:** Regulators can be male or female-threaded. Verify that the regulator matches the calibration gas cylinder prior to field deployment.
- **Tubing –** Tubing is used both during instrument calibration and during field screening activities. Tubing must be flexible, inert, and have the proper inner diameter to connect tightly to the PID inlet and moisture traps.
- **Moisture Traps –** Moisture traps are small plastic discs with inlet and outlet connectors and they contain 0.45-micrometer pore size filters. When attached with flexible tubing to the PID inlet, the traps can protect the PID sensor and lamp from moisture. Verify that the outlet connecter of the filter is attached to the PID inlet (using inert flexible tubing) prior to use. Spare moisture traps should be brought into the field, as they can wear out relatively quickly.
- **Battery and battery charger –** PIDs will typically be used with a rechargeable lithium ion battery. Spare lithium ion batteries and/or adapters for use with AA alkaline batteries may also be used. Lithium ion batteries may be charged using a charger stand or a travel charger. **Note:** Ensure that the AC/DC adaptor for the charger stand or travel charger is in the PID case prior to deployment to the field.
- **Maintenance kit –** A maintenance kit should be provided in the PID case, which includes: a bottle of methyl alcohol, latex finger cots, cotton swabs, and optical lens paper. The only maintenance that is typically performed in the field is cleaning the PID lamp. The cleaning procedures are presented below.
- **Manual –** A user's manual should always be included in the PID case brought into the field.
- **Calibration and Maintenance Log –** A fieldbook containing a calibration and maintenance log should be included in the PID case and should be updated each time the PID is calibrated and/or maintenance is performed.

Turning the Instrument On and Off: Procedures for powering on and off the specific PID model that is being used may be found in the user's manual for each model. Procedures for the MiniRAE Lite PID are as follows:

1. To turn on the PID, press and hold the middle button for approximately one second. The PID will go through the power-on procedures, including testing the sampling pump.

2. To turn off the PID, press and hold the middle button for approximately 7 seconds. The PID will beep repeatedly and the display will show a 5-second countdown as it powers off.

Charging the Battery: The PID must be charged prior to deployment in the field and at the end of each field day that it is used. Charging procedures for the specific PID model being used should be found in the user's manual for each model. For charging a MiniRAE Lite PID using a travel charger, procedures are as follows:

1. Leave the rechargeable lithium ion battery in the PID. Visually inspect the contacts on the bottom of the PID and on the travel charger. If they are dirty, wipe with a soft cloth (do not use solvents or other cleaners).

2. Plug the AC/DC adapter's round connector into the travel charger, and plug the adapter into an electrical outlet.

3. Attach the travel charger to the PID. This is accomplished by holding the travel charger firmly against the bottom of the PID in a position where the electrical contacts and attachment screws line up correctly, and then turn the attachment screws clockwise until they stop and the charger is firmly secured to the PID.

4. The PID will begin charging automatically. The green LED light on the travel charger will flash and the PID display screen will read "Charging…". When the PID is fully charged, the LED light will glow continuously and the PID display screen will read "Fully Charged".

Calibration Procedure: The required frequency with which calibration is performed may vary depending on the requirements of the job and the client. MiniRAE Lite PIDs can theoretically maintain a calibration for up to 30 days; however, most often they are recalibrated at the beginning of every field day. Refer to the Work Plan for the specific job to verify the calibration requirements.

Calibration is typically performed using the PID, a canister of 100-ppm compressed isobutylene gas, a regulator (either fixed flow or on-demand), and a short (approximately 6-inch) piece of inert flexible tubing that fits tightly on the regulator outlet and the PID inlet.

Standard procedure is for a 2-point calibration to be performed, including a fresh air/ zero-calibration and a span-gas calibration. Calibration procedures for the specific PID model that is being used may be found in the user's manual for each model. Calibration procedures for the MiniRAE Lite PID are as follows:

1. Turn the PID on. The display will show the home screen as "Ready… Start Sampling?". Press the middle button and the N/- button simultaneously for several seconds until the password screen is shown on the display. It is not standard

procedure to use a password for calibration, so you may bypass this prompt. Simply press the middle "Enter" button once to enter calibration mode.

2. First perform a zero calibration:

    a. Select "Zero Calibration" on the PID display. Press the Y/+ button ("Select").

    b. The next screen will display "Apply zero gas…". In a situation (for example, a site highly contaminated with petroleum) where the ambient air contains abundant fumes and it is impractical to move to an area of fresh air, it may be necessary to use a canister of compressed 0-ppm gas. For most situations, it is more practical to simply verify that the PID inlet is located in an area of relatively "fresh" air (for example, no obvious petroleum fumes from contaminated soil or vehicle traffic detectable to the human nose).

    c. Press the Y/+ ("Start") button and wait for the display screen to count down from 30 to 0. If performed correctly, the screen should display "Zero done! Reading = 0.0 ppm".

3. Next, perform a span-gas calibration:

    a. Select "Span Calibration" on the PID display. Press the Y/+ button ("Select"). The display screen should now indicate the type of calibration gas to be used (isobutylene) and the concentration of the gas (100 ppm).

    b. Attach the regulator (either an on-demand or 500 cubic centimeters (cc) per minute flow limiting regulator) to the 100-ppm compressed isobutylene gas canister. Connect the outlet of the regulator to the inlet of the PID using the short piece of inert flexible tubing. Press the Y/+ button ("Start") on the PID.

    c. The PID screen will then display a 30-second countdown. At the end of the 30 seconds, the screen will display the message "Span 1 is done!" and a concentration reading that should be very close (within 1.0 ppm) of the concentration of the span gas (100 ppm).

4. When the 2-point calibration is complete, press the middle button ("Back") and the screen will display "Updating settings". The display will return to the home screen within a few seconds.

5. Update the calibration log that is kept in the PID case with the date of calibration, zero calibration final reading, and span-gas final reading.

Calibration Check: A calibration check is sometimes referred to as a "bump test". On certain jobs, it may be acceptable to perform a calibration check at the beginning of each field day, rather than a calibration. Check the Work Plan to verify. Calibration checks may also be

conducted periodically throughout a field day to verify that the PID calibration has been maintained.

To perform a calibration check for a PID, apply calibration gas to a calibrated PID. Verify that the reading is within 10 percent (%) of the concentration of VOCs in the calibration gas. If the reading is outside of range, perform recalibration. If the inaccurate reading persists, the device may need to be taken in for maintenance prior to field use.

Lamp Selection: PIDs are outfitted with a UV lamp that breaks down VOCs into ions. Various lamps with different photon energies (measured in electron volts [eV]) are available for installation. To select the appropriate lamp for a particular job, it is important to know the ionization potential (IP) of the compound(s) targeted for detection. In order for the lamp to break down the compound so that it can be detected by the PID, the photon energy of the lamp must be greater than the IP of the target compound(s).

The most common PID lamps used have photon energies of 10.6 eV or 11.7 eV. A common application of a PID is for field screening soil samples for evidence of petroleum hydrocarbon contamination. In this application, a 10.6-eV lamp is appropriate, as most of the key contaminants of concern (COCs) associated with petroleum hydrocarbons (including benzene, toluene, ethylbenzene, and xylenes) have IPs that are below 10.6 eV. Lamps with higher photon energies (for example, 11.7 eV) should not be outfitted in a PID unless required to detect an important COC, as these lamps have shorter shelf lives and are less accurate for the relatively low IP COCs.

PID Field Screening: Field screening samples are typically collected either before or concurrently with laboratory analytical samples. Field screening before sample collection can help guide the selection of the most appropriate location from which to collect a laboratory analytical sample. Collecting field screening samples concurrently with laboratory samples can help establish a correlation between screening and analytical results at a particular site.

Two common methods of PID field screening are the headspace method and the in-situ method. Both are detailed below.

Headspace PID Screening: The headspace PID field screening method is the most accurate way to use a PID in the field and is typically required by the Work Plan for obtaining the highest quality field screening data.

1. For this method, field screening samples are collected by filling a resealable, ziptop, polyethylene bag approximately one-third to one-half full of freshly exposed or uncovered soil, and immediately sealing the bag. It is important that the soil be deposited into the bag as soon as practical after being exposed to ambient air, as the VOCs will immediately start volatizing, resulting in a low bias for the field screening result.

2. The bags should be agitated for approximately 15 seconds, and then allowed to warm for at least 10 minutes, but no more than one hour to allow the headspace vapors to develop. The soil must be warmed to at least 40º degrees Fahrenheit. The bags may be placed in a heated vehicle or simply in the sun if conditions are warm enough to achieve this temperature.

3. After the headspace vapors have developed, the bag should be agitated again for approximately 15 seconds. Insert the tip of a calibrated PID into the approximate midpoint of the void headspace of the bag, taking care to keep the opening as small as possible to minimize loss of VOCs. Measurement procedures for the specific PID model that is being used may be found in the user's manual. Procedures for the MiniRAE Lite PID are as follows:

    a. From the home screen (where the display reads: "Ready . . . Start sampling?"), press the Y/+ button ("Yes") to enter sampling mode. The sampling pump will engage and the display screen will show a concentration reading (initially should be close to 0.0 ppm).

    b. To prevent moisture or particles from being sucked into the PID and fouling the detector, a moisture trap is typically attached to the inlet of the PID using two short pieces of inert flexible tubing.

    c. Insert the tip of the flexible tubing into the headspace being measured and watch the PID display. If there are detectable compounds, the concentration reading will start climbing before eventually dropping again as these compounds are consumed by the ionizing lamp. Record the highest reading displayed (should occur within 5 to 10 seconds).

    d. If you are concerned that the highest reading was missed or forgotten, the PID records peak measurements. To view this value, press the N/- button ("→") twice to view the peak reading screen. Record this number, and then press the Y/+ button ("Clear"). Press the Y/+ button again ("Yes"), and then press the N/- ("→") three times to return to sampling mode. **Note:** It is important to clear the peak value because if the next peak value is lower, it will not be recorded.

In-Situ PID Screening: In some instances, where a limited volume of soil is available for field screening, such as soil cores from a direct-push rig or split spoon, using the headspace method may use up material that potentially could be used for a laboratory analytical sample. In these cases, field screening of the soil may be performed by making small divots approximately every 6 inches along the length of the core and inserting the calibrated PID tip just above the freshly exposed divot, taking care not to touch the material. Record the highest reading at each location in the field logbook or field form.

The in-situ method may be used by field personnel who want to quickly gather a large amount of qualitative field screening data in order to identify the zones with the highest level of contamination. Please note that VOC concentrations obtained using this method are typically lower and less accurate than concentrations obtained using the headspace field screening method. In-situ field screening results will not likely be accepted by regulatory agencies, and may need to be supplemented by targeted headspace field screening samples or laboratory analytical samples once the zones of contamination have been identified.

Interferences and Potential Problems: The most common PID problems that may occur in the field are detailed below, along with possible solutions:

Moisture Interference: Soil moisture may volatize during headspace development and present as moisture in the headspace. This moisture will commonly be ionized by the PID lamp and picked up by the detector, resulting in field screening results that are biased high. The best protection against moisture interference is to use a moisture trap. If you suspect a high bias as a result of moisture, record the concentration as displayed but make a note of the possible interference in the fieldbook. If a moisture trap is not used, it is possible for liquids to be sucked into the PID through the integrated porous metal filter and to damage the PID sensor and/or lamp. If this happens, the sensor and/or lamp may need to be cleaned or replaced.

Pump Failure: If the inlet port becomes blocked while the sampling pump is running, the PID will start beeping and the pump icon on the display will begin to flash. If this occurs, stop sampling (on a MiniRAE Lite, press the N/- button [“→”] and then the Y/+ button [“Yes”] when prompted to “Stop Sampling”) and ensure that the blockage has been cleared. The PID will have to be powered down, and then back on in order to reset the pump alarm.

Erratic Readings: If erratic readings occur, first recalibrate the instrument. If the problem persists, it may be that the sensor and/or lamp have become dirty or damaged. If in the field, try cleaning the lamp as described below under the maintenance section. Cleaning the sensor, or replacing the sensor or lamp, may be required but are not procedures that are typically undertaken in the field.

Inability to Zero between Measurements: It is commonly the case that the PID will not reach 0.0 ppm between field screening measurements. This may occur because of VOCs that have accumulated in the tubing or moisture trap. If the reading is relatively low (for example, below 1.0 ppm) it is usually fine to ignore it and consider it to be functionally zero. It may also be helpful to remove the tubing and moisture trap to allow them to air out for a few minutes. It is often necessary to replace the tubing and/or moisture trap if the elevated readings persist in fresh air. If the elevated readings persist after replacing the tubing and moisture trap, and you are relatively certain that the ambient air is clean, you may be able to solve the problem by repeating the zero point (fresh air) calibration procedure.

Maintenance: Only maintenance procedures that would typically be undertaken in the field are detailed here. Any procedures above and beyond those described here would typically be performed by in-house personnel in the controlled environment of the home office, or by outside instrument specialists. Anytime that a PID is dissembled, great care should be taken to avoid allowing dirt into the PID assembly. Make sure your hands and the work area are clean, and use the finger cots included in the maintenance kit on any finger that will come in contact with the inner workings of the PID. Do not disassemble outside if there is significant wind, to prevent grit from blowing into the inner workings of the PID.

Cleaning the Lamp Housing: For instructions specific to the PID that you are using, refer to the user's manual for each model. The following instructions are specific for the MiniRAE Lite PID: Gently unscrew the lamp housing cap, and remove the inlet probe/metal filter assembly. Pull the PID sensor straight out; some gentle rocking may be required. Clean the lamp window surface and lamp housing with methanol and cotton swabs. Hold the lamp up to the light at an angle to detect any remaining dirt. Repeat the wiping process until the lamp surface and housing are clean. Thoroughly dry the lamp housing and surface with optical lens paper. Reassemble the PID when it is completely dry.

Examining the PID Sensor: For instructions specific to the PID that you are using, refer to the user's manual for each model. The following instructions are specific for the MiniRAE Lite PID: Gently unscrew the lamp housing cap, and remove the inlet probe/metal filter assembly. Pull the PID sensor straight out; some gentle rocking may be required. Turn over the sensor unit to view the sensor electrodes. The electrodes should be flat and straight with no evidence of corrosion. If the sensor has become corroded or otherwise damaged, it will need to be replaced. If the sensor electrodes appear to be out of alignment, <u>gently</u> bend them until they are in alignment and do not touch the sensor housing. Reassemble the PID.

Clearing the Metal Filter: If it seems that the porous metal filter located within the sensor cover assembly has become clogged, possibly causing the sampling pump to stall and resulting in a pump failure, it may be possible to clear the filter. This will require a canister of compressed air (electronics duster) that is not typically included in the PID maintenance kit. Remove the inlet probe assembly and sensor cover assembly from the PID. Aim the compressed air at the <u>inner</u> side of the filter, and blow the compressed gas <u>backwards</u> through the filter. Replace the two assemblies and restart the sampling pump to see whether flow rates have improved.

APPENDIX D

Field Forms

Bristol
ENVIRONMENTAL
REMEDIATION SERVICES, LLC

Client:

Job No.

BOREHOLE ID:

Site Name:
Site Location:
Date Started: Completed:
Drill Rig: Drill Company:
Driller: Borehole Dia. (ft):
Logged By:
Company:
Sample Method: Total Depth:

Sketch Well Location:

Depth in Feet	Sample Interval	Blow Count	% Recovery	Sample ID	DESCRIPTION	PID (ppm)	WATER LEVEL	USCS	Estimated % Fines	Estimated % Sand	Estimated % Grave	REMARKS

APPENDIX E.2

E. Erickson LUST Site
Quinault Indian Reservation
Amanda Park, Washington

TABLE OF CONTENTS

SECTION **PAGE**

ACRONYMS AND ABBREVIATIONS ii
1.0 SITE-SPECIFIC INFORMATION 1
1.1 Site-Specific Description 1
1.2 Site-Specific History 1
2.0 PROGRAM MANAGEMENT 3
2.1 A5 – Site-Specific Problem Definition/Background 3
2.2 A6 – Site-Specific Project/Task Description and Schedule 4
2.2.1 Activity – Site Assessment 7
2.3 A7 – Site-Specific Quality Objectives and Criteria 10
2.3.1 Program Quality Objectives 10
2.3.2 Program Objectives 11
3.0 SITE-SPECIFIC DATA GENERATION AND ACQUISITION 13
3.1 B1 – Sampling Process Design 13
3.2 B2 – Sampling Methods 15
3.3 B3 – Sample Handling and Custody 16
3.4 B4 – Analytical Methods 17

TABLES

Table E.2-1 MTCA A Cleanup Levels for Soil 5
Table E.2-2 MTCA A Cleanup Levels for Groundwater 6
Table E.2-3 Soil Analytical Requirements 8
Table E.2-4 Groundwater Analytical Requirements 9
Table E.2-5 Soil Field Screening Criteria 14
Table E.2-6 Groundwater Field Parameters Criteria 15
Table E.2-7 Sample Identification System 17

FIGURES

Figure E.2-1 Site Location Map

Figure E.2-2 Site Map

Figure E.2-3 Former UST and 2013 Soil Confirmation Sample Locations

ACRONYMS AND ABBREVIATIONS

bgs	below ground surface
BTEX	benzene, toluene, ethylbenzene, and xylene
EPA	U.S. Environmental Protection Agency
MTCA	Model Toxic Control Act
PCS	petroleum-contaminated soil
RegenOx	chemical oxidizer
TO	task order
TPH	total petroleum hydrocarbons
UST	underground storage tank
VOA	volatile organic analysis
VOC	volatile organic compounds

1.0 SITE-SPECIFIC INFORMATION

1.1 SITE-SPECIFIC DESCRIPTION

The former A.E. Erickson site, EPA Facility No. 415007, is located within the Quinault Indian Reservation at milepost 135.8 of U. S. Highway 101 in Amanda Park, Washington (Figure E.2-1). The site is directly across the highway from the intersection of BIA Road 9205 with Highway 101. According to the EPA's *Performance Work Statement*, Market World Reality and Development, LLC presently owns the site.

1.2 SITE-SPECIFIC HISTORY

The site history is based on information that the EPA provided; it is contained in the July 2013 *Underground Storage Tank Decommissioning and Site Assessment* report.

The subject site is the location of a former shake mill that was owned by A.E. Erickson Enterprises, Inc. (A.E. Erickson). In May 1986, a Underground Storage Tank (UST) Notification Form submitted to the Washington State Department of Ecology indicated that one 5,000-gallon diesel UST was operating at the site, and that it had been installed in 1981. In 1993, A.E. Erickson filed for bankruptcy and the shake mill ceased operations. The EPA believes that the UST has not been used to store or dispense product since the shake mill closed. Shortly after A.E. Erickson filed for bankruptcy, Grays Harbor County seized the property due to tax delinquency. The property remained in the hands of the county until October 2007 when the property was sold in auction to Market World Reality and Development, LLC (Market World).

In September 2012, the EPA conducted an inspection of the out-of-use UST system. As a result of the inspection, a Field Notice of Non-Compliance was issued to Market World which required that the UST system be permanently closed in accordance with 40 CFR § 280.71. In May 2013, Market World removed the tank and performed the required site assessment. EPA personnel observed that the UST was completely filled

with water at the time of the removal, but noted that no regulated substance had been stored in the UST. Soils in the vicinity of the excavated UST emitted petroleum odors and were discolored. Since the excavated UST had no observable holes, any release was likely caused by spills and overfills related to bad housekeeping practices. Free product was encountered in shallow groundwater, approximately 7 feet below ground surface (bgs). Soil samples from the side walls of the tank excavation indicate the presence of petroleum contaminants above Model Toxic Control Act (MTCA) A cleanup levels. Although no groundwater sample was collected, visual evidence indicates groundwater contamination. No further work has been performed to characterize the degree and extent of contamination or to evaluate the threat posed to human health and the environment.

During the May 2013 tank closure, field personnel observed that shallow groundwater contacts petroleum contaminated soil, causing free product to accumulate in the water table.

2.0 PROGRAM MANAGEMENT

2.1 A5 – SITE-SPECIFIC PROBLEM DEFINITION/BACKGROUND

Petroleum contamination at the A. E. Erickson site is above MTCA A cleanup levels, specifically total petroleum hydrocarbons (TPH)-diesel and TPH-heavy oil in soil. The removed UST was surrounded by black-stained unconsolidated soil (top 2-4 inches) composed of sandy loam, gravelly sand, and clay; the soil smelled of petroleum. Results from soil confirmation sampling showed that TPH-diesel and TPH-heavy oil were present in site soil above MTCA A cleanup levels. Petroleum contaminants TPH-diesel and TPH-heavy oil were present at concentrations with TPH-diesel concentrations as high as 16,000 mg/kg, and TPH-heavy oil concentrations were as high as 4,000 mg/kg in two sidewall soil samples collected at 5.5-feet bgs. Groundwater was encountered at 7 feet bgs during the removal, but it is known to fluctuate during heavy rain events according to Dave Bingaman, director of the Quinault Nation Department of Natural Resources. The *Site Assessment Report* recommends the removal of petroleum-contaminated soil and further groundwater sampling.

Lunch Creek is a perennial water body that flows through the site and is a major tributary that forms the Raft River. The Raft River supports anadromous runs of coho salmon and steelhead trout, considered a vital food and cultural resource to the people of the Quinault Tribe; maintaining healthy fish runs in the waterways within the reservation is a top priority of the Tribal Council. Because the Lunch Creek elevation is several feet lower than the site's water table, petroleum-contaminated groundwater is likely discharging into Lunch Creek thus posing a risk to the fish runs in the Raft River.

The objective of the TO activities is to eventually close this LUST site. The TO calls for evacuating and removing petroleum-contaminated soil and collecting confirmation soil samples. After removal of the contaminated soil down to the soil-groundwater interface, a chemical oxidant will be added to the bottom of the excavation, and the excavation

backfilled. In addition, groundwater monitoring wells may be drilled, installed, developed, and sampled in the future to characterize groundwater quality and hydrology at the site.

ORC-Advanced, a Regenesis Remediation product, was selected for the first stage of treatment. ORC-Advanced accelerates naturally occurring aerobic bioremediation by supplying oxygen to the contaminated media.

2.2 A6 – Site-Specific Project/Task Description and Schedule

Site assessment and remediation are the primary tasks. Bristol will begin remediation by removing the petroleum-contaminated soil (PCS) source and applying a chemical oxidant onto the petroleum-hydrocarbon-impacted soil-groundwater interface. For the site assessment, Bristol will perform confirmation soil sampling and possibly future groundwater monitoring.

The analytical results for the soil confirmation samples from the excavation will be compared to the MTCA A Soil Cleanup Levels for Unrestricted Land Uses, presented on Table E.2-1.

Table E.2-1 MTCA A Cleanup Levels for Soil

Analyte	Method	MTCA A Cleanup Level (mg/kg)
TPH-gas[1]	NWTPH -Gx	100 (30)
TPH-diesel/heavy oil	NWTPH-Dx extended	2,000
Benzene	EPA SW8260	0.03
Toluene	EPA SW8260	7
Ethylbenzene	EPA SW8260	6
Xylenes	EPA SW8260	9

Notes:

The analytical laboratory will be TestAmerica in Tacoma, Washington.

[1]Cleanup level without benzene and total ETX is less than 1% is 100 mg/kg; the cleanup level for all other gasoline mixtures is 30 mg/kg.

EPA = U.S. Environmental Protection Agency
ETX = ethylbenzene, toluene, xylenes
kg = kilogram
mg = milligram
MTCA = Washington Department of Ecology Model Toxics Control Action
NWTPH = Northwest Total Petroleum Hydrocarbon
SW = EPA Solid Waste Method
TPH = total petroleum hydrocarbons

The analytical results for potential COCs in groundwater samples from monitoring wells will be compared to the MTCA A Groundwater Cleanup Levels presented on Table E.2-2.

Table E.2-2 MTCA A Cleanup Levels for Groundwater

Analyte	EPA Method	MTCA A Cleanup Level (mg/L)
Benzene	8260B	0.005
Toluene	8260B	1.0
Ethylbenzene	8260B	0.7
Naphthalene	8260B	0.16
Xylenes[a]	8260B	1.0[a]
1,2-Dibromoethane (EDB)	8011	0.00001
TPH-gas[1]	NWTPH-Gx	0.8 [b] (1.0)
TPH-diesel/heavy oil	NWTPH-Dx extended	0.5
Lead	6020	0.015

Notes:

[a]The cleanup level in this table is built on the assumption that xylene doesn't exceed the maximum allowed cleanup level for total petroleum hydrocarbons (TPH), and it is designed to prevent adverse aesthetic characteristics. This is a total value for all xylenes.

[b]The cleanup level is designed to protect groundwater from carcinogens so that it can be used as drinking water. Two cleanup levels are provided. The higher value assumes that no benzene is present in the groundwater sample. If any amount of benzene is detected in the groundwater sample, then the lower TPH cleanup level must be used.

EPA = U.S. Environmental Protection Agency

mg/L = milligrams per liter

MTCA = Washington Department of Ecology Model Toxics Control Act

NWTPH-Dx = Northwest Total Petroleum Hydrocarbon-diesel and heavy oil

NWTPH-Gx = Northwest Total Petroleum Hydrocarbon-gasoline

TestAmerica = TestAmerica Laboratories, Inc.

Bristol expects fieldwork to take approximately two calendar days, beginning in September 2014:

Date	Activity
September 2014	Mobilize and conduct field activities.
60 calendar days after completion of field work	Submit draft Report for EPA review.
15 working days after receipt of SME's comments on the draft Report	Submit final Report to EPA, the Quinault Natural Resources Director, and other Quinault Tribal agencies as requested by EPA.

2.2.1 Activity – Site Assessment

2.2.1.1 Soil Sampling and Analysis

Soil confirmation samples from the excavation will be collected and submitted for laboratory analysis once field screening indicates that the PCS has been removed, or until contract volumes of PCS have been removed. The soil samples will be collected from a backhoe if the excavation depth is 4 feet or deeper, in accordance with SOP BERS-01. Bristol will submit analytical samples from the locations with the highest PID readings, from each sidewall of the excavation, and from the bottom (even if wet) at the soil-groundwater interface of the excavation. If contract volumes are reached and the PCS has not been completely removed, soil samples will be collected from the excavation sidewalls from the area exhibiting the highest PID reading. Soil samples will be analyzed for TPH-gas, TPH-diesel/heavy oil, and benzene, toluene, ethylbenzene, and xylene (BTEX. Analytical methods, containers, sample preservation, and holding time requirements are described in Table E.2-3.

Table E.2-3 Soil Analytical Requirements

Contaminants of Concern	Analytical (Extraction) Method	Holding Time	Preservative	Sample Size and Container	QC Samples[1]
TPH-gas	NWTPH-Gx (5035)	14 days to analysis	Cool 4°C	One 40-mL VOA vial containing 10 mL methanol. Add 10 grams of soil to the VOA vial. One 4-oz. glass jar	4
TPH-diesel/heavy oil	NWTPH-Dx extended (3550)	14 days for extraction, 40 days for analysis	Cool 4°C	One 4-oz. glass jar	3
BTEX (low-level analysis)	EPA SW8260 (5035)	Freeze immediately after sampling; 14 days after collection	Keep frozen until delivered to laboratory	Two 40-mL VOA vials, each containing 10 mL deionized water. Add 10 grams of soil to each of the VOA vials. One 40-mL VOA vial containing 10 mL methanol. Add approximately 10 grams of soil to the VOA vial. One 4-oz. glass jar	4

Notes:

[1]QC will include one field duplicate at a rate of 1:10, an MS/MSD pair at a rate of 1:20 for environmental samples of the same matrix, and a trip blank to be included with coolers containing volatile samples (TPH-gas, BTEX).

°C = degrees Celsius
BTEX = benzene, toluene, ethylbenzene, xylenes
EPA = U.S. Environmental Protection Agency
mL = milliliter
NWTPH = Northwest Total Petroleum Hydrocarbon
oz. = ounce
VOA = volatile organic analysis

2.2.1.2 Groundwater Sampling and Analysis

Bristol will collect groundwater samples if the EPA decides, based on site assessment results, that groundwater monitoring wells should be installed in order to monitor the treatment's impact on petroleum-contaminated groundwater. Field personnel will use the low-flow technique of Puls and Barcelona (EPA, 1996) to collect water samples, as described in SOP BERS-02. Field parameters will be measured with a YSI meter and a

turbidimeter. Field parameters will include pH, dissolved oxygen, conductivity, temperature, and oxidation-reduction potential. Groundwater samples will be collected when these parameters stabilize. Groundwater samples will be analyzed for TPH-gas, TPH-diesel/heavy oil, BTEX and/or VOCs (to include BTEX) and potentially lead. Analytical methods, containers, sample preservation, and holding time requirements are described in Table E.2-4.

Table E.2-4 Groundwater Analytical Requirements

Contaminants of Concern	Analytical Method	Holding Time	Preservative	Sample Size and Container	QC Samples[1]
TPH-gas	NWTPH-Gx	14 days	HCl, cool 2-6°C	Three 40-mL glass VOA vials with teflon septum and screw top lids	4
TPH-diesel/heavy oil	NWTPH-Dx extended	14 days to extract, 40 days to analyze	HCl, cool 2-6°C	250 mL amber glass	3
VOCs	EPA SW8260	14 days	HCl, cool 2-6°C	Three 40-mL glass VOA vials with teflon septum and screw top lids	4
Ethylene dibromide (EDB)	EPA SW8011	Extract and analyze within 14 days	Sodium thiosulfate, cool 2-6°C	Three 40-mL glass VOA vials with teflon septum and screw top lids	4
Lead	EPA SW6020	6 months	HNO_3 to pH <2.0	500 mL poly bottle	3

[1]QC will include one field duplicate at a rate of 1:10, an MS/MSD pair at a rate of 1:20 for environmental samples of the same matrix, and a trip blank to be included with coolers containing volatile samples.

°C = degrees Celsius
EPA = U.S. Environmental Protection Agency
HCl = hydrochloric acid
mL = milliliter
NWTPH = Northwest Total Petroleum Hydrocarbon
TPH = total petroleum hydrocarbon
VOA = volatile organic analysis
VOC = volatile organic compound

2.2.1.4 Remedial Treatment

When the site was closed in May 2013, the PCS appeared to reach the water table. Since it is not feasible to excavate much of the PCS below the water table, the EPA decided to treat the soil-groundwater interface at the bottom of the excavation with a Regenesis product.

Regenesis was provided the limited site data, including the nominal aquifer soil and the highest analytical results from the 2013 soil confirmation samples. Based on site data, Regenesis recommended the applying 331 pounds of ORC-Advanced to the bottom of the excavation (estimated to be approximately 12 by 12 feet). ORC-Advanced will provide an oxygen source to help stimulate bioremediation for approximately 12 months. The pellets will be evenly distributed throughout the base of the excavation, and the bucket on the excavator will be used to mix the pellets with the top 3 to 4 feet of the aquifer.

Once the ORC-Advanced is applied to the bottom of the excavation, the excavation will be backfilled with clean soil, and compacted.

2.3 A7 – Site-Specific Quality Objectives and Criteria

2.3.1 Program Quality Objectives

The overall quality objective is to develop and implement the sampling, sample handling, and field measurement procedures that will provide data of the quantity and quality necessary for good decision making. In order to support specific decisions and regulatory actions, field and laboratory data must meet the quality requirements described in section 2.3.2. Soil analytical results and field data will be used to determine the extent of any remaining contamination. Data from any future groundwater sampling activities will be used to determine the nature and extent of groundwater contamination as well as monitor site cleanup.

2.3.2 Program Objectives

The objective is initiate cleanup by removing petroleum-contaminated soil from the site, and applying a chemical oxidant at the soil-groundwater interface to promote more rapid attention of the petroleum hydrocarbons in the groundwater. The ultimate goal is site closure.

Soil samples and future groundwater samples will be tested for suspected analytes:

- Total petroleum hydrocarbons (TPH) as gasoline and as diesel/heavy oil
- Benzene, toluene, ethylbenzene, total xylenes (BTEX)

Groundwater samples may need additional analysis for fuel additives like EDB and lead.

Action levels for these analytes are presented in Tables E.2-1 and E.2-2.

(Intentionally blank)

3.0 SITE-SPECIFIC DATA GENERATION AND ACQUISITION

Appendix D of the QAPP contains *Bristol Standard Operating Procedures,* guidance for field environmental measurement and sample collection. The procedures are described in detail to ensure that the data represents environmental conditions.

Bristol will obtain permission to conduct work on the A. E. Erickson site prior to mobilization. Bristol will coordinate with the property owner to establish the work zone upon arrival.

3.1 B1 – SAMPLING PROCESS DESIGN

Petroleum-contaminated soil will be evacuated and disposed off site in order to remove the source of the groundwater contamination. Soil confirmation samples will be collected from the four sidewalls and the bottom of the excavation (even if wet) when all of the PCS above MTCA A cleanup levels has been removed, or until contract capacity of PCS has been reached. Results from the soil confirmation samples will confirm that Bristol removed the PCS above cleanup levels or that contract volumes of PCS were insufficient; the results will provide information on the remaining concentration of TPH in the soil. Groundwater monitoring wells may be installed, depending on the results of the assessment and removal.

Personnel will field screen the soil during PCS removal, initially using a PID. Once PID responses drop to background, personnel will collect additional soil samples from the sidewalls and bottom of the excavation and use the Hanby Screening Method to test for TPH. If the Hanby results indicate all PCS has been removed, soil confirmation samples will be collected for laboratory analysis. If contract volumes of soil are reached and not all of the PCS has been removed, field personnel will collect soil confirmation samples from the excavation sidewalls and bottom to document the remaining PCS concentration.

Brisol will make every attempt to access the former tank area. If denied access or unexpected site conditions prevent access (e.g. heavy metal debris is scattered across the excavation area), the Bristol Project Manager will immediately contact the EPA SME, at which point the SME will obtain access for Bristol or the SME and the Bristol Project Manager will decide on an alternate approach.

If the EPA directs Bristol to install groundwater monitoring wells, the wells will be installed and sampled to determine the nature and extent of groundwater contamination. Wells containing free product will not be sampled.

Analytical results are critical to determining the extent of soil (and potentially groundwater) contamination. The field parameter data collected during groundwater sampling will help Bristol evaluate the impacted groundwater's natural attenuation processes. Table E.2-6 presents field parameters, stability criteria, and instruments that will be used for groundwater monitoring and sampling. Soil and groundwater sample analytical requirements are listed in Table E.2-3 and Table E.2-4, respectively. Soil screening criteria is presented in Table E.2-5. Instrument manuals are included in Appendix B of the QAPP.

Table E.2-5 Soil Field Screening Criteria

Field Parameters	Units	Instrument
Volatile organic vapors	ppm	MiniRAE Lite
TPH	ppm	Hanby

Notes:
ppm = parts per million

Table E.2-6 Groundwater Field Parameters Criteria

Field Parameter	Units	Criteria	Instrument
Turbidity	NTU	±10% for values greater than 1 NTU	Hach Turbidimeter, Model 2100P
Dissolved Oxygen	mg/L	± 10 %	YSI 556 Multi-Parameter Meter
Specific Conductance	µS/cm	± 3%	
Temperature	°C	± 3%	
pH	-	±0.1 pH units	
Oxidation Reduction Potential	mV	±10 feet	
Water Level	Feet	0.01 feet	Solinst Interface Probe Model 122

Notes:
°C = degrees Celsius
mL = milligrams per liter
NTU = nephelometric turbidity units
µS/cm = microsiemens per centimeter
mV = millivolts
pH = potential hydrogen

Groundwater elevation may vary since it is known to fluctuate during heavy rain events. The water level during the 2013 site assessment was approximately 7 feet bgs. If the water level is higher or lower, the depth and width of the excavation will change; the calculated chemical oxidation quantity (30 cubic yards) is based on an excavation depth of 7 feet bgs. All other variables should be within reasonable control as Bristol will use the same field technician and analytical laboratory historically used for EPA Region 10 work under this contract and the prior EPA contract (EP-W-07-104), in addition to standardized analytical, field screening, and sampling techniques. See Appendix D for more details.

3.2 B2 – Sampling Methods

Soil confirmation samples from the walls and bottom of the excavation will be collected in accordance with SOP BERS-01 after field screening indicates all PCS above MTCA A cleanup levels has been removed or contract quantities reached. Samples will be collected from the backhoe bucket, as it is anticipated that the excavation will be too deep for site personnel to enter. Samples will be collected as described under the section titled *General*

Soil Sampling Procedures in SOP BERS-01. Potential COCs, analytical methods to be used, and sample container requirements are summarized in Table E.2-3. Volatile samples will be collected first, using a Terra core sampler to collect and transfer 10 grams of soil to the sample containers for TPH-gas and BTEX analyses.

If the EPA decides at a later date to direct Bristol to install groundwater monitoring wells at this site, Bristol will collect groundwater samples from the wells using the low-flow technique of Puls and Barcelona (EPA, 1996) detailed in Appendix D, SOP BERS-02.

- Before purging and sampling, the well will be checked for the presence of free-product, and then the depth to groundwater from a mark on the top of the casing will be established with a water level meter to an accuracy of 0.01 foot. The equipment manual for the water level meter is contained in Appendix C.
- During purging, parameters will be measured with a YSI water quality instrument and a tubidimeter. Parameters to be measured and recorded include pH, dissolved oxygen, conductivity, temperature, oxidation-reduction potential, and turbidity. Equipment manuals for these instruments are contained in Appendix C.
- Once parameters stabilize within the limits presented in Table E.2-5, samples for laboratory analysis will be collected.
- Personnel collecting the samples will wear new, disposable nitrile gloves for each sample.
- Water samples will be collected using precleaned containers provided by the laboratory.

Potential COCs, analytical methods, and sample container requirements are summarized in Table E.2-4. Water samples to be analyzed for volatile organic compounds (VOCs) will be containerized before all other water samples.

3.3 B3 – Sample Handling and Custody

Sample handling and custody procedures are described in SOP BERS-03 in Appendix D of the QAPP, and the TestAmerica Laboratory Quality Assurance Manual in Appendix A of the QAPP. Additional information is included in Section 3.3 of the QAPP.

Samples will be identified using the system described in Table E.2-7.

Table E.2-7 Sample Identification System

Sample ID Component	Example	Note
Project Identifier	AEE	Former A.E. Erickson LUST Site
Sample Type	EX	Excavation
	MW	Monitoring Well
Loc ID	N/S/E/W/EB	North, South, East, West Sidewalls; Excavation Bottom
	1	Monitoring Well
Date	YYYYMMDD	-

Note:
A soil sample collected on September 15, 2014 from the eastern wall of the excavation at the A.E. Erickson LUST Site would have the identifier of AEE-EX-E-20140915, and a groundwater sample from the first groundwater well installed and sampled would have the identifier of AEE-MW-1-20140915.

3.4 B4 – Analytical Methods

Analytical methods, containers, sample preservation, and holding time requirements are described in Table E.2-3 and E.2-4 ("Soil Analytical Requirements" and "Groundwater Analytical Requirements").

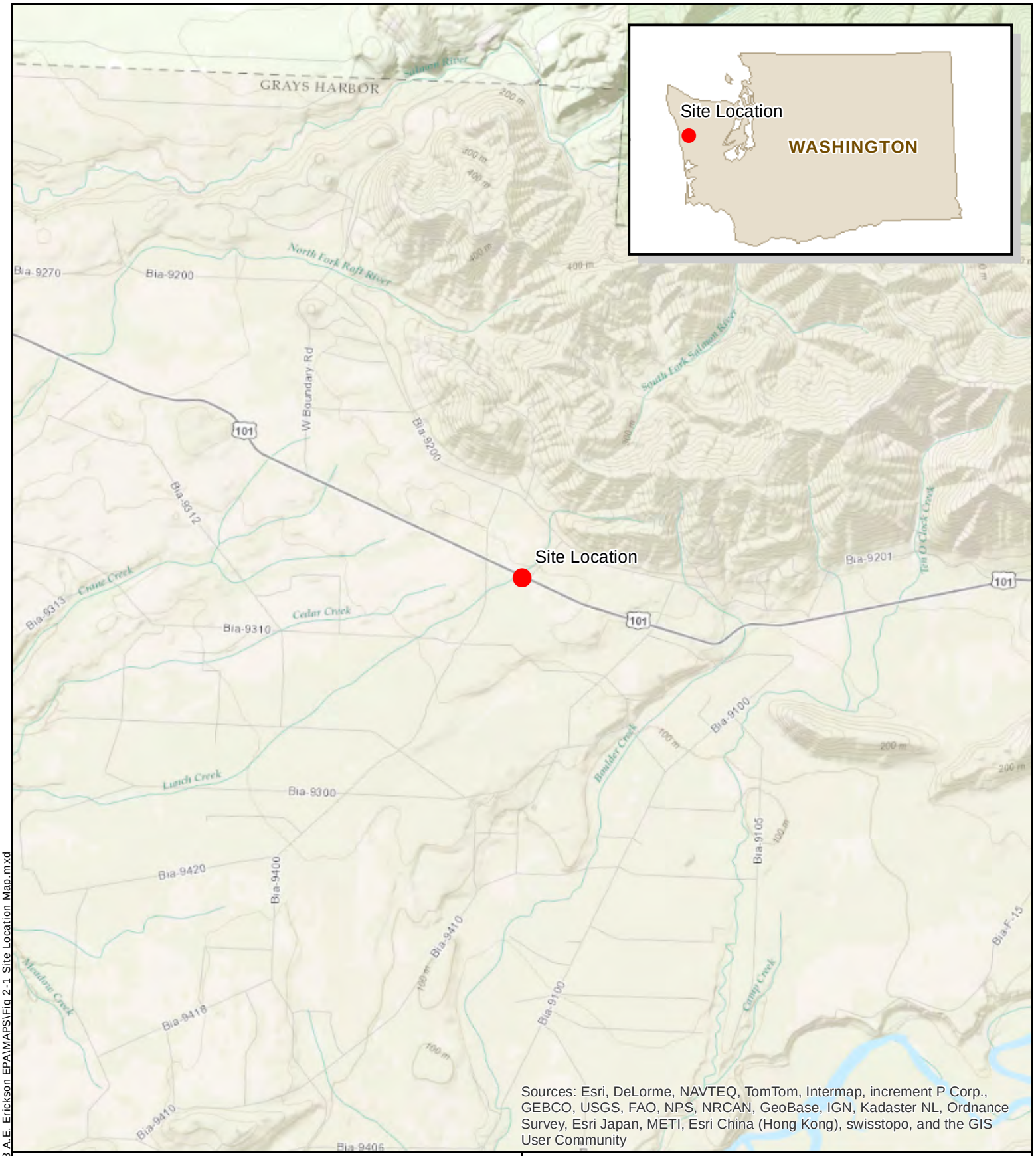
Field parameters to be measured are presented in Tables E.2-5 and E.2-6 for soil and groundwater, respectively. Procedures for measuring the groundwater field parameters are included in SOPs BERS-01 and BERS-04 which are included in Appendix D of the QAPP.

The operations and maintenance manuals for the MiniRAE Lite PID meter, the YSI 556Multi-Parameter Meter, the Hach turbidity meter, and the Solinst Interface Probe Model 122 are included in Appendix C. The instructions for performing the Hanby field screening method are also included in Appendix C.

Copies of these manuals and instructions are maintained in the Bristol Anchorage, Alaska office, and will also accompany the instruments and the Hanby test kit into the field.

(Intentionally blank)

FIGURES



N
W E
S

0 1
Miles

Figure E.2-1
EPA 1009 A.E. Erickson
Milepost 135.8, U.S. Highway 101
Amanda Park, WA
Site Location Map

Bristol
ENVIRONMENTAL REMEDIATION SERVICES, LLC
Phone (907)563-0013 Fax (907)563-6713

DATUM: NAD 83	DATE 06-10-14	SHEET
PROJECTION: SP WA S FT	DWN. EM	1
Project No.	SCALE 1:5,280	of
34150038	APPRVD. JSD	1

Document Path: G:\Jobs\34150038 A.E. Erickson EPA\MAPS\Fig 2-1 Site Location Map.mxd

N
W E
S

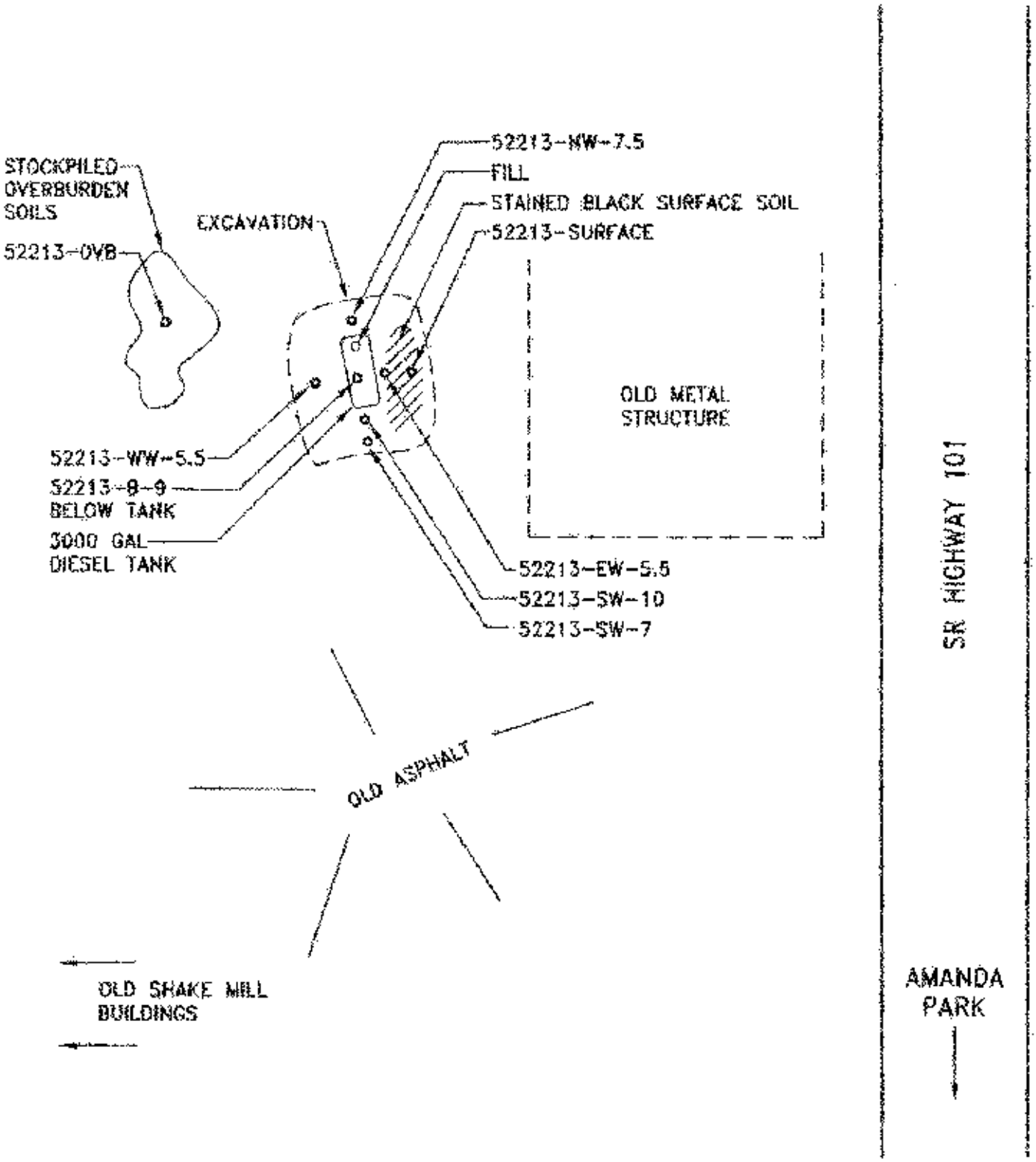
0 25 50
Feet

Figure E.2-2
EPA 1009 A.E. Erickson
Milepost 135.8, U.S. Highway 101
Amanda Park, WA
Site Map

Bristol
ENVIRONMENTAL REMEDIATION SERVICES, LLC
Phone (907)563-0013 Fax (907)563-6713

DATUM: NAD 83	DATE 06-10-14	SHEET
PROJECTION: SP WA S FT	DWN. EM	1
Project No. 34150038	SCALE 1:5,280	of
	APPRVD. JSD	1

Document Path: G:\Jobs\34150038 A.E. Erickson EPA\MAPS\Fig 2-2 Site Map.mxd



Source: DLH Environmental Consulting
5/22/2013

Figure E.2-3
EPA 1009 A.E. Erickson
Milepost 135.8, U.S. Highway 101
Amanda Park, WA
Former UST and 2013 Soil Confirmation Sample Locations

Bristol
ENVIRONMENTAL REMEDIATION SERVICES, LLC
Phone (907)563-0013 Fax (907)563-6713

DATUM: NAD 83	DATE 06-10-14	SHEET
PROJECTION: SP WA S FT	DWN. EM	1
Project No. 34150038	SCALE NTS	of
	APPRVD. JSD	1

Document Path: G:\Jobs\34150038 A.E. Erickson EPA\MAPS\fig 3 Former UST 2013 soil samples.mxd