PRE-DESIGN INVESTIGATION PROJECT PLAN

FINAL

DEFENSE NATIONAL STOCKPILE CENTER SCOTIA DEPOT SITE
GLENVILLE, NEW YORK

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LIST OF ACRONYMS

AHA	Activity Hazard Analyses
AMC	Army Material Command
ARARs	Applicable or relevant and appropriate requirements
CERCLA	Comprehensive Environmental Response, Compensation and Liability Act
COC	contaminants of concern
CONDEC	Consolidated Diesel Electric Company
CSM	conceptual site model
CSS	Contract Compliance Screening
CVOCs	chlorinated volatile organic compounds
DCE	dichloroethene
DLA/DNSC	Defense Logistics Agency/Defense National Stockpile Center
DoD	Department of Defense
DQOs	data quality objectives
EDD	electronic data deliverable
EDMS	Environmental Data Management System
ELAP	Environmental Laboratory Accreditation Program
ESI	Expanded Site Investigation
fbgs	feet below ground surface
FOL	field operations leader
FS	Feasibility Study
FSP	Field Sampling Plan
ft	feet/foot
GSA	US General Services Agency
HSP	Health and Safety Plan
IDW	investigation-derived waste
IRM	interim remedial measure
LCS	Laboratory Control Samples
MCL	maximum contaminant level
MDL	Method Detection Limit
MS/MSD	matrix spikes/matrix spike duplicates
NAE	New England District
NAN	New York District
NAPL	non-aqueous phase liquid
NAVD	North American Vertical Datum
NCR	non-conformance report
NELAP	National Environmental Laboratory Accreditation Program
NYSDEC	New York State Department of Environmental Conservation
NYSDOH	New York State Department of Health
PBC	performance-based contract
PCE	tetrachloroethene
PCOCs	potential contaminants of concern
PRB	permeable reactive barrier
QAPP	Quality Assurance Project Plan

QA/QC	Quality Assurance/Quality Control
QAO	quality assurance officer
RI	Remedial Investigation
RL	reporting limit
SCDA	Schenectady County Development Agency
SEDD	staged electronic data deliverable
Site	Former Defense National Stockpile Center Scotia Depot
SOP	Standard Operating Procedure
SOW	Scope of Work
Stone	Stone Environmental, Inc.
SVI	soil vapor intrusion
TAL	Target Analyte List
TCA	trichloroethane
TCE	trichloroethene
TCL	Target Compound List
TIC	tentatively identified compound
µg/Kg	micrograms per Kilogram
USACE	United States Army Corps of Engineers
USEPA	US Environmental Protection Agency
VOC	volatile organic compound
XML	extensible markup language
ZVI	zero-valent iron

1. PROJECT OVERVIEW

This Pre-Design Project Plan (Project Plan) has been prepared by Stone Environmental, Inc. (Stone) to address planned pre-design activities at and near the former Defense National Stockpile Center Scotia Depot Site (the "Site") in Glenville, New York (Figure 1). Stone Environmental will complete this work under a performance-based contract with the United States Army Corps of Engineers (USACE), New England District (NAE), which will be completed under an Interagency Agreement with the General Services Administration (GSA) as part of the New York State Department of Environmental Conservation (NYSDEC) State Superfund Program. The ultimate goal of the USACE for this Task Order is to collect sufficient data to support the design and cost estimate for the remedy prescribed in the May 2010 Record of Decision (ROD) for the project.

1.1. Purpose of the Project Plan

This Project Plan provides for additional field activities, a treatability study, and a pre-design investigation for the remedy for the project prescribed in the ROD, in order to support the Scope of Work issued by USACE NAE on 13 December 2012. The ROD prescribes the installation of a Permeable Reactive Barrier (PRB) to intercept significantly-contaminated groundwater before it reaches the site boundary. The prescribed PRB involves direct injection of zero-valent iron (ZVI) that will destroy dissolved trichloroethene (TCE) that is present in groundwater that comes in contact with it, and thereby will reduce the offsite flux of TCE in groundwater.

The objectives of the project activities are to:

1. Perform Remedial Design (RD) pre-design investigations to verify the nature and extent of contamination described in the *Expanded Site Investigation Report* (NYSDEC / GeoLogic, 2007) and the *Feasibility Study (FS) Report* (Malcolm Pirnie, 2010), including:
    a. Additional investigation to verify and further define the horizontal and vertical extent of the TCE plume;
    b. Focused investigations to verify the location and dimensions of the selected remedy; and
    c. Investigations of the vapor intrusion pathway for onsite buildings.
2. Collect data necessary to support the design of the selected remedy in the ROD (NYSDEC, 2010).

The objective of the pre-design investigation is to document the nature and extent of contamination related to the Site and to determine the best location for the selected remedy. The purpose of the treatability study is to determine the parameters required for the selected remedy to address the contamination.

1.2. Scope of Work

The scope of work for the pre-design activities covered by this Project Plan includes the following categories of tasks:

- Project Planning
- Historical Data Analysis
- Field Investigations
- Laboratory Analysis
- Sample Tracking and Data Management

- Evaluation of Remedial Alternatives
- Management and Meetings
- Reporting

1.3. Identification of Participatory and Regulatory Organizations

The Site is included in NYSDEC's State Superfund Program (Site ID 44702), and the ROD governing the remediation of the Site has been executed between NYSDEC and the Department of the Army. The project will therefore be conducted in accordance with the ROD and NYSDEC regulations. The Site is also included in the U.S. Environmental Protection Agency's (USEPA) Comprehensive Environmental Response, Liability and Compensation Act (CERCLA) Program (ID No. NYN000204534) as amended by the 1986 Superfund Amendments and Reauthorization Act (SARA), and therefore the project is also being conducted in accordance with CERCLA and the National Contingency Plan to the maximum extent practical, and pursuant to *Environmental Quality - Formerly Used Defense Sites (FUDS) Program Policy* (ER 200-3-1), dated 16 March 2004. Applicable or relevant and appropriate requirements (ARARs) such as New York State and USEPA regulations will be incorporated into the pre-design investigation and treatability study. It is the policy of the Department of the Army to ensure that all activities conducted on sites are protective of human health and the environment and to meet (or waive) the substantive provisions of permitting regulations that are ARARs. A complete list of participatory and regulatory organizations and their contacts is included in Subsection 2.2.

1.4. Supporting Documents

The development of this Project Plan relied on the elements of a Conceptual Site Model (CSM) included in the *Final Expanded Site Investigation (ESI) Report* (NYSDEC / GeoLogic, 2007) and *Final Feasibility Study Report* (Malcolm Pirnie, 2010). The *Final ESI Report* and *Final FS Report* present a detailed review of historical uses, past operations, and prior environmental investigations for the project, along with a summary of Site geology, hydrogeology, contaminant sources and fate and transport processes, potential receptors, and exposure pathways based on current and future property uses. The CSM is referenced in different sections of this Project Plan and its source documents should be consulted for a complete presentation of Site history, prior uses, and contaminant and subsurface conditions.

In support of this Project Plan, the following documents are included as appendices:

- Field Sampling Plan (FSP), prepared in accordance with *Engineer Manual 200-1-3*, which includes the Quality Assurance Project Plan (QAPP), prepared in accordance with *Uniform Federal Policy for Quality Assurance Project Plans* (DTIC ADA 427785) (Appendix A); and
- The combined Health and Safety Plan (HSP), which includes emergency and contingency information, as well as Activity Hazard Analyses (AHA), prepared in accordance with *Engineer Manual 385-1-1* (Appendix B)

It should be noted that, consistent with USACE policy, Stone has made every effort to eliminate redundancy between the Project Plan and its supporting documents. This effort is intended to streamline USACE and stakeholder review of the Project Plan as well as reduce potential errors and conserve resources. To this end, a number of tables are cross-referenced between documents, primarily between the Project Plan and FSP, rather than being reproduced in their entirety in both documents.

The Project Plan focuses on project background information, data gaps and overall sampling rationale, the overall scope of work (SOW), project schedule, and project personnel and their responsibilities. The FSP focuses on detailed descriptions of sampling objectives, locations, frequency, designations, equipment and procedures, and handling and analysis. The QAPP focuses on data quality objectives (DQOs) and the measures in place to document that DQOs are achieved. Finally, the combined HSP documents the planning and procedures in place to perform all work safely.

2. PROJECT TEAM AND ORGANIZATION

2.1. Project Planning

Project planning includes the overall project management activities and the development of the FSP, QAPP, and HSP. Project management will include keeping stakeholders informed of project status, existing or potential problems, and any changes that may be required to prudently manage the project and meet the needs of the project stakeholders and decision makers. Monthly status reports will be prepared and circulated to all stakeholders, which will summarize activities completed during the previous month, activities planned for the upcoming month, updates to the project schedule, and problems encountered and the resolution. Additionally, during the field program, weekly progress updates will be provided to all stakeholders via e-mail, which will include recently obtained field or laboratory data, field observations, and field decisions made along with the rationale for those decisions. For critical field decisions, conference calls will be held with all interested stakeholders (including USACE and NYSDEC) before field decisions are made. The background and relevant data will be summarized and recommendations presented during the call. Documentation of the decision will be circulated to all parties following each critical decision conference call, including the rationale for the decision. Many of these critical field decisions are expected to be time critical, therefore timely conference calls and prompt decisions will be necessary.

2.2. Project Team

2.2.1. US Army Corps of Engineers

The project is being performed for USACE, New England District (NAE), on behalf of USACE, New York District (NAN), at the request of the United States General Services Administration (GSA), the property owner. USACE NAE and NAN will provide execution and technical oversight as well as administrative support and review. The contact information for USACE team members is provided in Table 1.

Table 1: USACE Team Members Contact Information

Title	Name	Phone Number	Email Address
Project Manager	Gregory Goepfert	917-790-8235	Gregory.J.Goepfert@usace.army.mil
Engineering Technical Lead	Dean Brammer, C.H.M.M.	978-318-8144	Dean.D.Brammer@usace.army.mil
Project Hydrogeologist	Ken Heim, Ph.D.	978-318-8650	Kenneth.J.Heim@usace.army.mil
Project Chemist	David Lubianez	978-318-8311	David.J.Lubianez@usace.army.mil
Innovative Technologies	Dan Groher, P.E.	978-318-8404	Daniel.M.Groher@usace.army.mil
Health & Safety Officer	Kane Turmelle	978-318-8661	Kane.Turmelle@usace.army.mil
Human Health Risk Assessor	Cynthia A. Colquitt	978-318-8042	Cynthia.A.Colquitt@usace.army.mil

2.2.2. State of New York

The project is being performed under the regulatory oversight of NYSDEC. Contact information for the NYSDEC Project Manager is:

Jason M. Pelton

New York State Department of Environmental Conservation
625 Broadway
Albany, NY 12233

518-402-9814
jmpelton@gw.dec.state.ny.us

The project is also subject to the regulatory oversight of NYSDOH. Contact information for the NYSDOH Project Manager is:

Maureen Schuck
New York State Department of Health
Bureau of Environmental Exposure Investigation
Empire State Plaza, Corning Tower, Room 1787
Albany, NY 12237

518-402-7860
mer10@health.state.ny.us

2.2.3. Other Stakeholders

The GSA contacts for the project are:

David Baker
U.S. General Services Administration
Design and Construction Division, Northeast and Caribbean Region
26 Federal Plaza 1655
New York, NY 10278-0004

(212) 577-7920
david.baker@gsa.gov

Kenneth Simmons
Construction Control Representative, Albany Office U.S. General Services Administration
1 Clinton Square, Leo O'Brien Federal Building, Room 826
Albany, NY 12207-2335

(518) 378-8861
kenneth.simmons@gsa.gov

The Site is primarily located on property owned by the Defense Logistics Agency/Defense National Stockpile Center (DLA/DNSC). The contact is:

Lisa Sanders
Administrative Assistant
DLA/DNSC-MOSC
Route 5, Building #12
Scotia, NY 12302-9463

(518) 370-3347
lisa.sanders@dla.mil

Portions of the Site are located in Scotia Industrial Park, Inc., owned by the Galesi Corporation. The contact is:

David Ahl
Galesi Corporation
695 Rotterdam Industrial Park, Building 6
Schenectady, New York 12306

(518) 356-4445
dahl@galesi.com

The EPA Region 2 contact for the Site is:

Jeanette Dadusc
US EPA Region 2, Federal Facilities Program
290 Broadway, 21st Floor
New York, NY 10007-1866

(212) 637-4050
r2fedfac@epamail.epa.gov

2.2.4. Stone Environmental

Stone is the lead contractor that will be performing the pre-design investigation and treatability study field activities. Key Stone personnel for tasks performed for the pre-design investigation and treatability study field operations and their specific responsibilities are discussed below. Contact information for Stone team members is provided in Table 2.

Table 2: Stone Environmental, Inc. Team Members Contact Information

Title	Name	Responsibilities	Mobile Phone	Email Address
Program Manager	Seth Pitkin	Program management, technical review and support	802-249-8700	spitkin@stone-env.com
Project Manager	Joseph Schmidl, P.G.	Project management, technical hydrogeologic interpretation and review	802-229-5377	jschmidl@stone-env.com
Field Operations Leader	David Linari	Data collection	802-578-5817	dlinari@stone-env.com
Site Safety Officer	David Linari	Site safety	802-578-5817	dlinari@stone-env.com
Lead Chemist	Michael Rossi	Laboratory management	802-229-2194	mrossi@stone-env.com
QA Officer	Kim Watson	Data quality	802-249-7753	kwatson@stone-env.com
Health & Safety Officer	Kim Watson	Health and safety	802-249-7753	kwatson@stone-env.com

2.2.4.1. Program Manager

The Program Manager is responsible for overall management of contract and task order requirements with USACE. He will assure that the schedule is being met and that necessary company resources are available for

performance of project tasks. He will also provide final editorial review for all technical documents submitted for this project.

2.2.4.2. Project Manager (PM)

The PM organizes the assigned project staff and initiates project planning and implementation activities at the Task Order level. The PM controls the budget and schedule, ensures that contract requirements are met, and is responsible for management of all field activities (including subcontractors), data management and interpretation, coordination with NAE and NAN, and other stakeholders, and preparation of project deliverables. The PM is responsible for ensuring that all project activities conform to USACE requirements and specifications, field data acquisition meets the DQOs, and each form/report is reviewed for accuracy and content.

2.2.4.3. Field Operations Leader (FOL)

The FOL will report directly to the PM and is responsible for desktop planning, equipment procurement, scheduling, subcontracting, and mobilization for the field program. The FOL also has overall responsibility for completion of all field activities in accordance with this FSP. Specific responsibilities of the FOL include:

- Coordinating activities in the field;
- Assigning specific duties to field team members;
- Mobilizing and demobilizing the field team, including the onsite laboratory and drilling equipment, as well as Stone subcontractors;
- Directing the activities of subcontractors in the field;
- Resolving any logistical problems that could potentially hinder field activities, such as equipment malfunctions or availability, or weather-dependent working conditions; and
- Coordinating with the QAO (see below) in implementing field quality control (QC) including issuance and tracking of measurement and test equipment; the proper labeling, handling, storage, shipping, and chain-of-custody procedures used at the time of sampling; and control and collection of all field documentation.

2.2.4.4. Quality Assurance Officer (QAO)

The QAO is responsible for the project quality assurance/quality control (QA/QC) in accordance with the FSP and appropriate management guidance. The QAO will ensure that the project field activity readiness review is conducted; approve variances during field activities before work continues; approve, evaluate, and document the disposition of non-conformance reports (NCRs). The QAO will also oversee and approve any required project training, audit/surveillance plans, and subsequently supervise these activities. The QAO will be responsible for coordinating the analytical services, the acquisition and delivery of sample bottles to the site, and validation of the analytical data.

2.2.4.5. Health & Safety Officer (HSO)

The HSO is responsible for ensuring that health and safety procedures designed to protect personnel are maintained throughout the field activities conducted for this project. This will be accomplished by strict adherence to the project-specific HSP and compliance with all NAE safety and health guidance and Stone's

corporate health and safety plan. The HSO will also be responsible for the coordination and communication of health and safety issues for field personnel.

2.2.4.6. Site Safety Officer (SSO)

All field personnel are responsible for implementing the safety requirements specified in the HSP for field activities. However, one person will be designated to serve as the SSO. The SSO will be onsite during all Stone activities. The SSO is responsible for enforcing the requirements of the HSP once work begins. The SSO has the authority to correct, immediately, all situations where noncompliance with the HSP is noted and to stop work in cases where an immediate danger is perceived. A full description of SSO responsibilities will be outlined in the HSP.

2.2.4.7. Field Staff

The field staff, who will be made up of Stone staff and also possibly Watermark field staff (see Subsection 2.2.4.8.1) report directly to the FOL. The Stone personnel responsible for operating drill rigs will hold applicable licenses. The responsibilities of the field team include:

- Collecting samples, conducting field measurements, and decontaminating sampling equipment according to documented procedures stated in the FSP;
- Ensuring that field instruments are properly operated, calibrated, and maintained, and that adequate documentation is kept for all instruments;
- Collecting the required QC samples and thoroughly documenting QC sample collection;
- Ensuring that field documentation and data are complete and accurate;
- Operating and maintaining drilling equipment in accordance with documented procedures stated in the FSP;
- Analyzing samples in the onsite laboratory in accordance with documented procedures stated in the FSP;
- Ensuring that all health and safety protocols are followed for Stone field activities, in accordance with the project specific HSP; and
- Communicating any non-conformance or potential data quality issues to the FOL and QAO.

2.2.4.8. Subcontractors

Stone has or will retain subcontractors in order to complete the requirements of the project. A description of these subcontractors and their responsibilities is presented below. The subcontractors are as follows:

2.2.4.8.1. Field Staff

Additional field staff may be provided by Watermark Environmental Inc. (Watermark) from their Lowell or Hanover, MA offices. Watermark is headquartered at 175 Cabot Street, Lowell, MA 01854.

2.2.4.8.2. Remediation Design Contractor

Remediation design services will be provided by:

Larry Pannell
Watermark Environmental, Inc.

175 Cabot Street
Lowell, MA 01854

2.2.4.8.3. Laboratory

Fixed laboratory services are currently anticipated to be provided by:

Michele Kersey, Project Manager
TestAmerica Laboratories - Savannah
5102 LaRoche Avenue
Savannah, GA 31404

912-354-7858

Christopher Anderson, Project Manager
Alpha Analytical Laboratories
8 Walkup Drive
Westborough, MA 01581

508-898-9220

Phil Dennis, Senior Manager
SiREM Laboratory
130 Research Lane, Suite 2
Guelph, Ontario, Canada N1G 5G3

519-822-2265 ext. 238

2.2.4.8.4. Subsurface Utility Clearance Contractor

A subsurface utility clearance contractor will be engaged to perform utility clearance as necessary at each soil boring location, prior to drilling activities. Utility clearance services will be provided by:

Michael Goodfellow
New York Leak Detection, Inc.
PO Box 269
Jamesville, NY 13078

315-469-4601

2.2.4.8.5. Surveying Contractor

A New York-licensed surveyor will be used to survey monitoring well locations and elevations.

Jim Cook
C.T. Male, Inc.
50 Century Hill Drive
Latham, NY 12110

518-786-7605

3. REVIEW OF BACKGROUND DOCUMENTS

The following section provides an overview of the background and setting for the project, based on Stone's review of available background documents.

3.1. Property Identification

The Scotia Naval Depot is adjacent to the north side of New York State (NYS) Route 5 in the Town of Glenville, Schenectady County, New York (see Figure 1: Locus Map). The Site and adjacent properties are zoned for commercial use. The Mohawk River is located approximately 1,500 feet west-southwest of the Scotia Naval Depot and represents the major drainage feature in Schenectady County (see Figure 2: Site Map). The Site overlies an USEPA designated Sole Source Aquifer referred to as the Schenectady or Great Flats Aquifer system. Relative to a series of four aquifer protection zones established to protect municipal water supplies relying on the aquifer system, the Site lies in Zone III, or the General Aquifer Recharge Area (see Figure 2: Site Map).

Portions of the original Scotia Naval Depot have been subdivided and sold since 1972 by the United States Government. The Site now consists of several large privately held parcels in addition to a portion of land still administered by the GSA. The private parcels contain a variety of industrial tenants; while the GSA leases its remaining portion to the DLA/DNSC and the Navy. The disposition of land at the original Scotia Naval Depot is as follows:

- In 1972, approximately 77 acres were transferred to Schenectady Industrial Development; now known as Scotia Industrial Park, Inc. (a.k.a., Galesi Corporation);
- In June 1985, approximately 126 acres were sold to SCDA and became Scotia Glenville Industrial Park. Also in June 1985, approximately 27 acres were sold to the Galesi Corporation and were added to Corporations Park;
- In 1988, approximately 36 acres were separated to the Navy and the Depot retained the remainder of the GSA administered land as the DLA;
- In July 1997, GSA sold approximately 11.5 acres of the Depot to SCDA; and
- Currently, the Navy occupies approximately 14.2 acres and the DLA/DNSC occupies approximately 59.7 acres. Figure 2 shows the approximate boundaries of the Defense National Stockpile Center Scotia Depot when established in 1942, and the approximate current property boundaries. The Site is currently owned by the GSA and operated by the DNSC.

The areas surrounding the Site are largely residential with commercial / industrial land use.

3.2. Property History

The Scotia Depot was built in 1942 and1943 and was commissioned as a United States Navy facility on March 30, 1943. It served as a storage and supply depot for naval forces along the Atlantic coast and Europe, and as a storage and distribution point for National Stockpile materials. The parcel originally consisted of approximately 337 acres. The facility mostly stored large items such as boilers, turbines and reduction gears and was the home of the Navy's Landing Craft Maintenance and Battle Damage Program and the Navy's Automotive and Handling Equipment Spare Parts Program. Employment peaked in 1945 at 2,342 personnel. On January 1, 1960 the Navy turned the facility over to the GSA.

During the period between early 1966 and approximately 1973, USACE / Army Material Command (AMC) leased buildings from the Navy for the fabrication and storage of vehicles as well as other military equipment. GSA records indicate that these included the Larc V Amphibious Lighter. These operations were predominantly conducted by the AMC contractor, Consolidated Diesel Electric Company (CONDEC). In early 1968 through 1971 inspections at the CONDEC operation indicated the continuing presence of fire and safety hazards. These included the improper storage of flammables and paints, and the "dangerous air contaminants created or released by open surface dip tank operations."

Additionally, between 1967 and 1969, the GSA and the Navy leased two buildings to the United States Army / Defense Supply Agency. The agreement indicated these buildings were used for the "preservation and rail loading of trucks; storage of trucks and ten 6 × 6 AIC vehicles."

In response to the detection of TCE at concentrations less than 1 part per billion (the New York State Drinking Water Standard is 5 parts per billion (ppb)) in the Town of Rotterdam and City of Schenectady well fields, NYSDOH performed private water supply sampling in 1991. The private water supply sampling included residences located on NYS Route 5 in the Town of Glenville and hydraulically downgradient of the Site. Chlorinated volatile organic compounds (CVOCs), including TCE, 1,1,1-trichloroethane (1,1,1-TCA), and tetrachloroethene (PCE), were detected in groundwater collected in some of the residential wells. Consistent with the groundwater contamination at the Site, TCE was the primary CVOC detected in the residential well water samples (at concentrations up to 320 ppb). Following a recommendation by the NYSDOH to connect to public water, the homes on NYS Route 5 were subsequently connected to public water provided by the Town of Glenville. Although the drinking water standard was never exceeded in the City of Schenectady and the Town of Rotterdam municipal water supply wells, increased groundwater quality monitoring was initiated following the identification of the contamination.

Subsequent to the NYSDOH residential groundwater sampling, six subsurface investigations were completed to identify the possible source of TCE in the residential wells and possibly the Town of Rotterdam and City of Schenectady municipal well fields and to delineate the extent of the TCE groundwater plume. The investigations were completed between 1995 and 2007 and focused on the assemblage of properties comprising the former 337-acre Defense National Stockpile Center Scotia Depot. During the investigations, two areas thought to represent possible TCE source areas, including a former burn pit and the Sacandaga Road Landfill (see Figure 2) were evaluated.

Data suggested that although these areas may be contributing to minor amounts of groundwater contamination, they do not represent TCE source areas. Instead, investigation data indicated that TCE disposal occurred in the northeast corner of the property.

In addition and unrelated to the investigations related to the TCE groundwater plume, a site assessment was completed by the Defense National Stockpile Center in advance of transferring unused portions of the property back to the GSA. Based on the site assessment, soil with metals present above background concentrations was excavated from two former metals and ore stockpile areas and two drywells were removed as part of interim remedial measures (IRMs) in 2005.

3.3. Selected Remedy

The major components of the selected remedy prescribed in the ROD for the Site include the following:

The installation of a PRB will include the direct injection of a ZVI PRB to reduce the off-site flux of groundwater TCE contamination. Based on the current CSM and the interpreted orientation and distribution of

groundwater CVOC contamination, the PRB will be located on the adjacent downgradient property and in the central portion of Parcel 29.-3-16.41, (see Figure 2: Site Map). The PRB will be constructed by directly injecting ZVI into the aquifer. TCE that contacts the ZVI as the groundwater flows through the PRB will be destroyed. It is expected that the PRB will continue to treat the CVOC groundwater contamination for up to 30 years.

The elements of the selected remedy are as follows:

1. A remedial design program will be implemented to provide the details necessary for the construction, operation, maintenance, and monitoring of the remedial program. The pre-design study will be completed to verify the exact location and dimensions of the PRB.
2. Over an approximately 250-foot width of the CVOC plume (see Figure 3) and using multiple borings, ZVI will be injected from approximately 65 feet bgs (average depth of the water table) to an approximate average depth of 105 feet below ground surface. Based on the assumed 250-foot length, the PRB will contain approximately 250 to 300 tons of ZVI, depending on the barrier thickness (to be determined during the pre-design study). The location and dimensions of the PRB may change depending on the outcome of the remedial design program. Since this remedy requires no permanent aboveground structures, the disturbed treatment area will be restored to pre-existing conditions following ZVI injection.
3. Imposition of an institutional control in the form of an environmental easement that will require:
    a. Compliance with an approved Site Management Plan;
    b. Restricting the use of groundwater as a source of potable or process water, without necessary water quality treatment as determined by NYSDOH; and
    c. The property owner, or designated representative, to complete and submit to NYSDEC a periodic certification of institutional and engineering controls.
4. The property owner, or designated representative, will provide a periodic certification of institutional and engineering controls, prepared and submitted by a professional engineer or such other expert acceptable to NYSDEC, until NYSDEC notifies the property owner in writing that this certification is no longer needed. This submittal will:
    a. Contain certification that the institutional controls and engineering controls put in place are still in place and are either unchanged from the previous certification or are compliant with NYSDEC-approved modifications;
    b. Allow NYSDEC access to the Site; and
    c. State that nothing has occurred that will impair the ability of the control to protect public health or the environment, or constitute a violation or failure to comply with the Site Management Plan unless otherwise approved by NYSDEC.
5. The operation of the components of the remedy will continue until the remedial objectives have been achieved, or until NYSDEC determines that continued operation is technically impracticable or not feasible.
6. Since the remedy results in untreated hazardous waste remaining at the Site, a Site Management Plan is required, which will include the following institutional and engineering controls:

a. Long-term groundwater quality monitoring from wells upgradient, downgradient, and in the vicinity of the PRB to evaluate the effectiveness of the remedy and to verify the extent of the dissolved-phase CVOC plume;
b. Continued implementation of a vapor intrusion monitoring program, and if necessary, installation of air mitigation systems for buildings located above the TCE and carbon tetrachloride plumes;
c. Identification of any use restrictions on the Site;
d. Provisions for the continued proper operation and maintenance of the components of the remedy, and
e. Provisions for any new structures in the area of the groundwater contamination to include sub-slab construction that allows for installation and operation of mitigation systems.

The long-term monitoring program will allow the effectiveness of the groundwater CVOC concentration reduction to be monitored and will be a component of the long-term management for the Site.

3.4. Conceptual Site Model

3.4.1. Hydrogeology

The Site overlies the Great Flats Aquifer system. The Great Flats Aquifer is an EPA-Designated Sole Source Aquifer that is adjacent and subjacent to the Mohawk River over a distance of approximately 12 miles in Schenectady County. The aquifer is within the Mohawk Lowlands Physiographic province which includes the Mohawk Valley and the uplands to the north (Glenville hills) and the south (Rotterdam hills). At its widest point near the Village of Scotia and the Town of Rotterdam (just west of the City of Schenectady), the aquifer is approximately 3 miles wide. The deepest portion of the aquifer is near the Site, where the sand and gravel is approximately 200 feet thick. A total of five municipalities rely on the Great Flats Aquifer system as a source of drinking water. These include the City of Schenectady, Village of Scotia, and the Towns of Glenville, Niskayuna, and Rotterdam. The Scotia Naval Depot is located approximately 1,500 feet south of the Village of Scotia well field and approximately 1.25 miles north of the Town of Rotterdam and City of Schenectady well fields. On an average day, these three well fields withdraw greater than 20 million gallons of water from the Great Flats Aquifer system (NYSDEC / GeoLogic, 2007).

The unconsolidated deposits beneath the Site include ice-proximal end moraine and esker gravel units which vary in thickness from less than a foot to more than 50 feet, and overlie basal till, which appears to act as an aquitard. There are several sub-facies with lateral and vertical variation in grain size present. These ice-proximal deposits filled the bedrock ravine beneath the Site and also appear to extend close to the ground surface beneath the apparent Source Area at the Site (NYSDEC / GeoLogic, 2007).

In most locations, the Great Flats Aquifer is predominantly sand and gravel with an average thickness of approximately 100 feet. The material has commonly been classified as a glacial outwash deposit although the sand and gravel was deposited in a fluvial environment. Specifically, the coarse sand and gravel was transported and deposited nearly 10,000 years ago when there was a catastrophic release of glacial lake water down the Mohawk Valley from Glacial Lake Albany. Within the sand and gravel, there are occasional, localized fine sand and silty clay lenses within the sand and gravel deposit. There have also been documented zones of entirely coarse gravel and cobbles within the deposit (NYSDEC / GeoLogic, 2007). Estimates of

hydraulic conductivity of the unconsolidated deposits beneath the Site are relatively high, ranging from 0.1 to 100 centimeters per second. The sand and gravel is underlain by a dense glacial till deposit.

The Mohawk River represents the regional groundwater discharge point. Groundwater beneath the Site occurs at a depth of approximately 65 to 70 feet below ground surface and flows from east-northeast to west-southwest beneath the majority of the Site toward the Mohawk River. Although the New York State Canal does not have a major impact on groundwater flow beneath the Site, in the area near Canal Lock 8, the operation of the lock has a direct influence on both surface water flow and groundwater flow. During the navigation season (approximately April to November), the gates at Lock 8 are used to maintain an approximate 12-ft head differential between the upper pool and the lower pool. During the non-navigation season (November to April), the gates at Lock 8 are removed and there is less than a 2-ft head differential between the upper and lower pools. This situation tends to stabilize the groundwater elevations along the southeastern boundary of the Site (NYSDEC / GeoLogic, 2007).

In addition, USACE has suggested that there may be a seasonal fluctuation in groundwater flow direction at the eastern end of the groundwater plume, particularly in the vicinity of monitoring well MW-22. Specifically, seasonal fluctuations in TCE concentrations in well MW-22 suggest that groundwater flow may shift southward seasonally, increasing TCE concentrations. The existing monitoring well network is insufficient to shed further light on this phenomenon, due to a lack of additional monitoring points between existing wells MW-22 and B-1 and B-2.

3.4.2. Contaminant Distribution

In developing the CSM, the observations regarding Site background and environmental setting each have implications for understanding the nature and extent of contamination and potential remedial strategies. The following text summarizes the CSM.

The Site comprises a linear, east-west trending TCE groundwater plume and an east-west trending carbon tetrachloride groundwater plume. On the DLA/DNSC property, the highest TCE concentrations (up to 800 ppb) have consistently been detected in monitoring well GEP-2. Offsite, TCE has been consistently detected in out-of-service residential water supply wells at concentrations ranging from 50 to 100 ppb. Further offsite, trace levels (less than 1 ppb) of TCE have periodically been detected in the City of Schenectady and the Town of Rotterdam municipal well fields. The carbon tetrachloride plume is located offset and to the north of the TCE plume and appears to extend offsite (NYSDEC / GeoLogic, 2007).

The highest soil TCE concentrations do not directly overly the area where the highest TCE groundwater concentrations were detected. This may be explained by the Site stratigraphy, the vadose zone being nearly 70 feet thick, and the downward migration and transport of the TCE following its initial release. In particular, occasional fine grained sand and silt lenses are present in the overburden deposits beneath the Site. As the contamination migrated under gravity flow downward through nearly 70 feet of unsaturated glaciofluvial deposits, the low-permeability fine sand and silt lenses may have caused lateral migration of the contaminants down the dip/slope of the low-permeability lenses until reaching the boundary or the margins of the low-permeability lens. Once beyond the low-permeability lens, the contamination would flow vertically downward until reaching the water table. Once the contamination reached the water table, it would migrate laterally as dissolved phase in the direction of groundwater flow (NYSDEC / GeoLogic, 2007). If migrating as a dense, non-aqueous phase liquid (DNAPL), it would continue to migrate downward in a stepwise fashion, ultimately reaching the top of the till layer. This step-wise migration of contamination may have led to a complicated

plume structure; the key objective of the pre-design investigation is to determine the plume location and structure, in order to optimize the location of the proposed ZVI PRB.

It is expected that not all groundwater from within the TCE and carbon tetrachloride plumes discharge into the Mohawk River. Instead, it is likely that some fraction of the plume underflows down-valley beneath the Mohawk River. As the plume migrates down-valley as underflow, the concentrations decrease through dilution and dispersion processes. Groundwater pumping at the municipal well fields, in particular the Town of Rotterdam well field, may capture some portion of the remaining plumes. This may be the reason for the periodic detection of trace levels of TCE below the drinking water standard in the Rotterdam well field (NYSDEC / GeoLogic, 2007).

Based on the indoor air monitoring program, the presence of TCE at concentrations of approximately 50 ppb in offsite groundwater does not influence indoor air quality at homes that directly overlie or that are near the TCE groundwater plume. Low levels of carbon tetrachloride in a groundwater plume offset to the west of the TCE groundwater plume, however, are influencing both sub-slab and indoor air quality. Due to the presence of carbon tetrachloride in both sub-slab and indoor air samples, two property owners were contacted regarding the installation of sub-slab mitigation systems. Although both property owners declined, follow-up indoor air monitoring will be completed to assess conditions over time (NYSDEC / GeoLogic, 2007). The ROD calls for continued soil vapor intrusion monitoring; see Subsection 5.12 for further details.

4. TECHNICAL APPROACH

4.1. Objectives of the Pre-Design Investigation / Treatability Study Field Program

As previously described in Subsection 1.1, the objectives of the pre-design investigation field program are to:

1. Perform Remedial Design (RD) pre-design investigations to verify the nature and extent of contamination described in the *Expanded Site Investigation Report* (NYSDEC/GeoLogic, 2007) and the *Feasibility Study (FS) Report* (Malcolm Pirnie, 2010), including:
    a. Additional investigation to verify and further define the horizontal and vertical extent of the TCE plume;
    b. Focused investigations to verify the location and dimensions of the selected remedy; and,
    c. Investigations of the vapor intrusion pathway for onsite buildings.
2. Collect the data necessary to support the design of the selected remedy in the ROD (NYSDEC, 2010).

4.2. Pre-Design Investigation / Treatability Study Field Task Development

The tasks described in the Project Plan are designed to eliminate the identified data gaps and collect the data necessary to support the design of the selected remedy for the project. The list of field tasks has been developed in an iterative fashion, with input and approval from USACE and Stone, through the following steps:

1. Initial SOW tasks were included in the *SOW*, dated 4 October 2010;
2. Subsequent discussions between USACE and Stone resulted in revisions to the *SOW*, dated 30 October 2012, 30 November 2012, and 7 and 13 December 2012.

4.3. Pre-Design Investigation / Treatability Study Field Task Summary

Using the above process, the pre-design investigation / treatability study field tasks were developed to address the data gaps identified in the *SOW*. Table 3 summarizes the six pre-design investigation and one treatability study field tasks objectives, additional purposes, and methods. Section 2 of the FSP provides detailed descriptions of the field task methodologies. Data quality objectives (DQOs), which further detail the objectives for each field task, are described in Subsection 6.1 of this Project Plan, as well as in QAPP Worksheet #16.

Table 3: Field Tasks and Methods

Field Task	Objectives	Additional Purpose(s)	Data Collection Task and Methods
Collection and analysis of groundwater samples from the Site and vicinity	Obtain current CVOC concentrations in groundwater.	Provide additional groundwater quality data for siting vertical profiling locations.	Tasks: Monitoring well sampling and analyses. Methods: Low-flow groundwater sampling, water level measurement, sample location, sample analysis, investigation-derived waste (IDW) characterization and disposal.
Synoptic measurement of groundwater elevations	Obtain synoptic groundwater elevation data.	Provide additional understanding of groundwater flow.	Tasks: Water level measurements. Methods: Water level measurement, sample location.
Vertical groundwater profiling of the dissolved groundwater plume	Determine vertical and horizontal extent of the dissolved plume. Determine current groundwater contaminant concentrations.	Determine aquifer stratigraphy and physical and chemical properties.	Tasks: Vertical groundwater profiling. Methods: Utility clearance, vertical groundwater profiling, sample location, sample analysis, IDW characterization and disposal.
Collection and analysis of geotechnical soil samples	Provide aquifer geotechnical data.	Provide aquifer characteristics for remedy design.	Tasks: Subsurface soil sampling and analyses. Methods: Utility clearance, soil boring / soil sampling, sample location, sample analysis, IDW characterization and disposal.
Installation and development of overburden groundwater monitoring wells	Obtain current CVOC concentrations in groundwater from locations currently lacking groundwater monitoring wells.	Provide additional long-term groundwater monitoring locations. Determine aquifer stratigraphy.	Tasks: Monitoring well installation, sampling, and analyses. Methods: Utility clearance, soil boring / monitoring well installation, monitoring well development, low-flow groundwater sampling, sample location, sample analysis, IDW characterization and disposal.
Hydraulic conductivity testing of monitoring wells	Obtain aquifer hydraulic data to support remedy design.	Provide additional understanding of contaminant transport pathways. Provide plume treatability data.	Tasks: Hydraulic conductivity testing of monitoring wells. Methods: Hydraulic conductivity testing.
Treatability testing (bench-scale column tests) of soil and groundwater	Obtain remedy design parameters.	Provide additional understanding of contaminant transport pathways.	Tasks: Collection and bench-scale column tests of soil and groundwater samples. Methods: Utility clearance, soil boring / soil sampling, low-flow groundwater sampling, sample location, sample analysis, IDW characterization and disposal.
Collection and analysis of soil vapor / indoor air samples	Obtain current soil vapor and indoor air CVOC concentrations from nearby onsite receptors.	Evaluate potential human health risk via the SVI pathway.	Tasks: Soil vapor / indoor air sampling and analysis. Methods: Sub-slab soil vapor sampling, indoor air sampling sample location, sample analysis.

4.4. Chemical Analysis Program

4.4.1. Sample Analysis

Groundwater, soil, soil vapor, and indoor air samples will be collected and analyzed by a fixed laboratory as part of the pre-design investigation / treatability study field program described in Subsection 4.3 of the Project Plan. Groundwater samples will be collected from monitoring wells, and, representative samples of water purged from the wells during sampling—investigation-derived waste (IDW)—will be collected and sent to the fixed laboratory for analysis. Soil samples will include soil from soil borings, and samples will be collected from soil IDW (e.g., drill cuttings) and sent to the laboratory for analysis.

Groundwater samples will be submitted to an onsite laboratory for select CVOC screening, and others will be submitted to a fixed, offsite laboratory. The samples will be analyzed for the parameters and by the analytical methods listed below and in Table 4 of the FSP, found in FSP Subsection 2.9. Quality Assurance/Quality Control (QA/QC) samples comprising trip blanks, equipment blanks, field duplicates, and matrix spikes/matrix spike duplicates (MS/MSD) associated with the samples will also be collected and submitted for analysis as indicated in Table 5 of the FSP and described in detail in QAPP Worksheets #12, 15, 20, 28.

4.4.2. Laboratory Methods and Reporting

Analytical methods procured through Stone's onsite laboratory or through a subcontracted laboratory under this contract are included in the descriptions of each pre-design investigation / treatability study field task in Section 2 of the FSP, and the analytical methods are fully summarized, including proposed numbers of samples and QA/QC samples, in Table 4 of the FSP. A summary of the reporting limits for each analytical method is included in QAPP Worksheet #15. Details regarding each analytical method (i.e., DQOs, QA/QC acceptance criteria, etc.) are described in detail in QAPP Worksheets #12, 20 and 28. Data reporting is described in Subsection 2.9.4.1 of the FSP.

5. WORK BREAKDOWN STRUCTURE AND BUDGET

This section presents the proposed approach and the assumptions used in developing the proposed pricing for the project. Stone's approach and assumptions are provided in accordance with the task structure in the *SOW*. The period of performance for this project is from 31 December 2012 until 30 September 2013. The investigations may be conducted in phases depending on available funding.

5.1. Task 1: Project Management / Status Reports

Stone will provide overall management of the project under this task in accordance with the *SOW*. The following subtasks apply to the management of the project.

5.1.1. Task 1A: Status Reports

Stone will provide a Monthly Status Report (MSR) by the end of the first week of every month. The project duration is 9 months; therefore nine (9) MSRs will be required.

5.1.2. Task 1B: Invoicing

Stone will submit invoices by the end of the first week of every month. The project duration is 9 months; therefore ten (10) invoices will be required, with one additional invoice to account for retainage.

5.1.3. Task 1C: Coordination

Stone will develop and maintain a resource-loaded schedule that supports the technical approach and outlines the due dates and cost expenditure percentages for all tasks and payable deliverables. The schedule shall be in conformance with task durations included in the *SOW*.

5.1.4. Task 1D: Project Meetings

Stone and Watermark will participate in up to four (4) meetings with USACE NAE, USACE NAN, and possibly GSA and NYSDEC. Two (2) of these meetings will be held in the vicinity of the Site and two (2) meetings will be held at the USACE NAE office in Concord, Massachusetts. The assumptions listed below were made for this task and the associated pricing:

- The Stone Project Manager (PM) will attend all four meetings;
- The Stone Program Manager will attend two of the meetings, one (1) at the Site and one (1) in Concord, Massachusetts;
- The Watermark Senior Environmental Engineer will attend all four meetings;
- Meetings will not exceed four (4) hours in duration; and
- Effort is included for the preparation of meeting presentation materials.
- Effort and expenses are included for round-trip travel to each meeting.

5.2. Task 2: Site-Specific Plans

The following planning subtasks apply to the project.

5.2.1. Task 2A: Overall Project Plan

Stone will develop a Pre-Remedial Design Investigation Project Plan (Project Plan), and update the document as needed. The Project Plan will include the following elements:

- Project Overview;
- Project Team and Organization;
- Review of Background Documents;
- Technical Approach;
- Work Breakdown Structure and Budget (cost to complete the work assignment) including necessary procedures, inspections, any lower-tiered subcontractor costs, and deliverables;
- Quality Management Plan;
- Communications Plan; and
- Project Schedule.

5.2.2. Task 2B: Quality Assurance Project Plan / Field Sampling Plan

Stone will prepare a Field Sampling Plan (FSP) that addresses all field sampling and related activities including those related to sampling, drilling, monitoring well installation, IDW characterization and disposal, and any field data gathering activities. Estimated costs for the IDW characterization and disposal will be included. The FSP shall include a QAPP prepared in accordance with *Uniform Federal Policy for Quality Assurance Project Plans* (UFP-QAPP).

Field activities and contract deliverables related to acquiring and reporting of chemical data for this investigation will be in accordance with EM200-1-3.

5.2.3. Task 2C: Health and Safety Plan / Emergency Contingency Plan

Stone will prepare a site-specific Health and Safety Plan (HASP) that specifies employee training, protective equipment, medical surveillance requirements, standard operating procedures, and a contingency plan. The HASP will include the following components:

- Accident Prevention Plan;
- Activity Hazard Analysis; and
- Accident Reporting.

5.3. Task 3 (Optional): Community Involvement

The following technical support for community involvement tasks apply to the project.

- USACE NAN will be the lead for community involvement;
- Stone and Watermark will attend one public meeting and prepare associated presentation materials in the vicinity of the Site; and
- Stone will develop one Fact Sheet for the Site.

5.4. Task 4: Pre-Design Investigation

The following technical pre-design investigation tasks apply to the project.

5.4.1. Task 4A: Site-Wide Groundwater Sampling/Analysis

Stone will collect a baseline round of groundwater samples from 26 monitoring wells at the locations shown in Table 1 of the *SOW*. The following assumptions listed below were made for this task and the associated pricing:

- Each monitoring well will be sampled in accordance with the EPA Region I low-stress sampling procedures;
- Up to six wells can be sampled per team, per 10-hour day;
- The groundwater samples will be analyzed in TestAmerica's Department of Defense (DoD)-, National Environmental Laboratory Accreditation Program (NELAP)-, and NYSDOH-certified laboratory for volatile organic compounds (VOCs) that includes all the contaminants of concern for the Site;
- The groundwater samples will be analyzed, via EPA Method 8260B, using QA/QC protocols that follow EPA SW846 800 methodologies;
- Groundwater analytical results will be provided with a 7-day turnaround time; and,
- Approximately one-half day mobilization to and demobilization from the Site.

5.4.2. Task 4B: Synoptic Groundwater Elevation Measurement

In addition to the water level data collected from the sampled monitoring wells listed in Task 4A above, Stone will collect groundwater levels from 13 additional monitoring well locations listed in Table 2 of the *SOW*. The following assumption was made for this task and the associated pricing:

- Mobilization and demobilization for this task will be accounted for under Task 4A;
- Equipment rental for this task will be accounted for under Task 4A; and
- The groundwater elevation measurements at the additional 13 wells can be made in approximately 3 hours.

5.4.3. Task 4C (Optional): Preliminary Groundwater Monitoring

Stone team subcontractor AMEC will perform preliminary groundwater modeling to evaluate potential remedy performance, in advance of Task 5. The following assumptions were made for this task and the associated pricing:

- Groundwater modeling data for this task will be the groundwater analytical and elevation data collected under Tasks 4A and 4B; and
- Groundwater modeling for this task will be performed by AMEC, using a screening level, 3-dimensional, numerical flow and reactive transport model; and
- The groundwater model results will be provided in a Letter Report.

5.5. Task 5: Focused PRB Investigations

The following technical investigation tasks apply to the project, and are dependent on the findings of the Pre-Design Investigation.

5.5.1. Task 5A: Transect Vertical Groundwater Profiling

Stone will advance up to two (2) transects of vertical groundwater profiles perpendicular to the groundwater flow path across the longitudinal the axis of the estimated TCE plume. The locations of the proposed transects are depicted in Figure 1. The assumptions listed below were made for this task and the associated pricing.

- The first transect will be advanced at the approximate proposed location of the ZVI PRB and will consist of up to six (6) locations.
- The second transect of three (3) profiles is proposed to be located further downgradient in the vicinity of where the estimated TCE plume crosses the property line (upgradient of monitoring wells MW-6 and MW-9).
- The groundwater profiles will be spaced approximately 125 feet apart along each transect. The actual distance between borings will be based on field data results.
- A tenth groundwater profile is proposed to be located along the approximate axis of the TCE plume between MW-13 and MW-6/MW-9, with final location to be determined based on the results of the nine previous profiles.
- The proposed drilling method is a hybrid approach that includes using ODEX methods to advance 6-inch casing to the water table (approximately 65 to 70 feet bgs). The groundwater profiling tool (Waterloo$^{APS\ TM}$) will then be lowered into the casing and driven out of the casing using a second heavy Geoprobe rig and direct push technology or equivalent; two profiling trucks will be utilized to increase drilling production and reduce the duration of the drilling program.
- Upon refusal, the groundwater profiler will be withdrawn from the hole and slim-bore, mud-rotary methods will be used to advance 3-inch casing will be further advanced to the refusal depth using the mud rotary technique. The groundwater profiler will then be reinserted into the casing and the driving and sampling process continued as before.
- Groundwater samples will be collected every five (5) feet below the water table, adjusted as necessary to avoid low-permeability layers. Additional samples may also be collected at intervals where a stratigraphic change is observed (via the Index of Hydraulic Conductivity testing that is integral to the Waterloo$^{APS\ TM}$).
- Stone assumes that no more than three (3) of the locations will be profiled to the estimated depth of the till layer (approximately 170 feet bgs) and will take approximately 3 rig-days each to complete. Stone assumes that the remaining seven (7) profiles will be advanced to approximately 110 feet bgs) and will take approximately 2 rig-days to complete.
- The criteria for terminating a groundwater profile will be two consecutive groundwater profile samples that are non-detect for CVOCs of interest, below a series of detections at shallower depths with each decision to be approved by CENAE.
- Based on the above assumptions, Stone will collect an estimated 12 to 20 groundwater profile samples per location, depending on total depth. Stone estimates that a total of 24 groundwater samples will be collected per day, between the two drill rigs.
- During the collection of each groundwater profile sample, water quality parameters will be recorded including dissolved oxygen (DO), oxidation/reduction potential (ORP), pH and specific

conductance (SC). Samples will be collected upon three consecutive readings within 10 percent difference. The groundwater profile samples will be collected for CVOC screening onsite by Stone's mobile laboratory.

- The maximum vertical sample spacing will be 5 feet, until the vertical extent of the TCE plume is delineated, adjusted as necessary to avoid low-permeability layers. During advancement of the groundwater profiler, a continuous index of hydraulic conductivity will also be recorded.
- Based on the above technical approach, approximately 23 rig-days will be required to advance the ten proposed profiles to the estimated depths (approximately 110 to 170 ft bgs), during an approximate three- (3) week period;
- Approximately one-half day mobilization to and demobilization from the Former Scotia Naval Depot, and approximately one day setup for the drill rigs and mobile laboratory following arrival.

5.5.2. Task 5B (Optional - Executed): Geotechnical Sampling

Stone will perform geotechnical sampling to provide design criteria that will be used to determine the specifications and the cost estimate for the implementation of the ZVI PRB. The following assumptions listed below were made for this task and the associated pricing:

- Stone will collect geotechnical samples at up to four (4) soil boring locations, which will also be used to install overburden monitoring wells under Subtask 5E;
- The proposed drilling method is mud-rotary to advance 6-inch casing, and use of a piston-type soil coring sampler, performed solely using the mud rotary rig (these drilling costs are included under Subtask 5E);
- A hydrogeologist will log soil during the drilling process, and screen soil headspace using a photoionization detector (PID), in order to facilitate geotechnical sample selection;
- A total of five (5) soil samples will be collected from five (5) ft intervals from depths within the anticipated vertical span of the ZVI PRB, based on the vertical extent and distribution of the groundwater TCE plume. An experienced hydrogeologist will examine and identify the soil sample immediately upon collection. The soil samples will be archived in glass jar;
- One soil sample collected at each soil boring location (a total of five (5)) will be submitted for geotechnical analyses, including :
  - Laboratory sieve analysis by ASTM Method D422;
  - Bulk density analysis by ASTM Method D2937; and,
  - Effective porosity analysis by ASTM Method D7263; and,
- The labor for this subtask is limited to sample collection and handling.

5.5.3. Task 5C (Optional - Executed): Non-Transect Vertical Groundwater Profiling

Following Subtask 5A, and based upon the near real-time results of the focused investigation, Stone will advance up to five (5) groundwater profiles within the estimated TCE plume. The locations of the proposed locations are depicted in Figure 2, but are subject to change. The assumptions listed below were made for this task and the associated pricing.

- The proposed drilling/groundwater profiling methods are the same as those described under Subtask 5A, as will groundwater profile sample collection rates;
- Stone assumes that the five profiles will be advanced to approximately 110 feet bgs and will take approximately 2 rig-days to complete, during an approximate one- (1) week period;
- Approximately one-half day mobilization to and demobilization from the Site, and approximately one day setup for the drill rig following arrival; the cost of the mobilization is $9,460.

5.5.4. Task 5D: Mobile Laboratory Analysis of Groundwater Profile Samples

Stone will provide near real-time groundwater analytical results, by screening groundwater profile samples collected during Tasks 5A and 5B in an onsite, mobile laboratory. The following assumptions listed below were made for this task and the associated pricing:

- The groundwater profile samples will be analyzed in Stone's DoD- and NELAP-certified onsite, mobile laboratory for a reduced CVOC list that includes all the contaminants of concern for the Site;
  - Tetrachloroethene,
  - 1,1,1-Trichloroethane,
  - Trichloroethene;
  - Carbon tetrachloride,
  - Chloroform;
  - 1,1-Dichloroethene;
  - Cis-1,2-Dichlroethene;
  - Trans-1,2-Dichlroethene;
  - 1,1-Dichloroethane; and,
  - Vinyl chloride;
- The onsite laboratory is capable of analyzing up to 30 groundwater profile samples per day;
- The groundwater profile samples will be analyzed via EPA Method 8260C (modified), using QA/QC protocols that follow EPA SW846 800 methodologies (this analysis is considered a screening analysis);
- Approximately 10% of the groundwater profile samples will be split and analyzed separately for confirmation in TestAmerica's DoD-, NELAP-, and NYSDOH-certified fixed laboratory for TCL VOCs via EPA Method 8260B, that includes all the contaminants of concern for the Site and,
- The groundwater analytical results will be reported in the Pre-Design Sampling Report (Task 7A).

5.5.5. Task 5E (Optional - Executed): Monitoring Well Installation

Following, and based in part upon the results of the groundwater profiling investigation, Stone will advance and complete up to four (4) soil borings as monitoring wells to provide additional long-term groundwater contaminant and elevation data locations. The depths and screen intervals for the monitoring wells will be

determined based on evaluation of the data from the vertical groundwater profiling (Task 5A and Optional Task 5B [if executed]). The following assumptions listed below were made for this task and the associated pricing:

- A single screened interval of no more than 5 ft will be installed at each location. If additional screened intervals are required (based on the distribution of TCE plume cores) the scope will be modified following completion of the profiling tasks;
- The proposed drilling method is mud rotary; using the same drill rig deployed during Task 5B. The drilling will advance 6-inch casing to the bottom of the screen interval. A hydrogeologist will log soil samples from the proposed screen interval during the drilling process, and screen soil headspace using a PID;
- The monitoring wells will be constructed of polyvinyl chloride screen and riser;
- Based on the above technical approach, approximately 3 rig-days will be required to advance each soil boring and install a monitoring well; and
- Approximately one-half day mobilization to and demobilization from the Site ($1,400), and approximately one day setup for the drill rig following arrival.

5.5.6. Task 5F (Optional - Executed): Hydraulic Conductivity Testing

Stone will conduct hydraulic conductivity testing on up to four (4) wells to determine formation hydraulic conductivity, to facilitate estimation of groundwater flow velocity across the proposed location of the PRB. The following assumptions listed below were made for this task and the associated pricing:

- Stone will utilize a pneumatic slug for the hydraulic conductivity testing;
- The hydraulic conductivity testing for the wells network will take approximately one-half (½) day per well (total of two (2) days effort for the four (4) wells); and,
- The results of the hydraulic conductivity testing will be reported in the Pre-Design Sampling Report (Task 7A).

5.5.7. Task 5G (Optional): Groundwater Modeling

Stone team subcontractor AMEC will perform groundwater modeling to predict the fate of the plume downgradient of the proposed PRB placement. This model will be used to predict the fate of the plume downgradient of the proposed PRB and finalize the design placement of the PRB. The following assumptions listed below were made for this task and the associated pricing:

- Stone will be the primary contact with CENAE;
- Groundwater modeling for this task will be performed by AMEC, and the modeling approach will build on the preliminary model developed under Task 4C and focus on iterative optimization of the PRB design;
- The model will be a 3-dimensional, numerical flow and reactive transport model; and,
- The results of the groundwater modeling will be reported in the Pre-Design Sampling Report (Task 7A).

5.6. Task 6: Treatability Study

The following tasks apply to the focused investigations to verify the location and dimensions of the prescribed ZVI PRB.

5.6.1. Task 6A: Treatability Study Testing

Watermark will be the lead for a treatability study to develop sizing and operations criteria that are used in the ZVI PRB design and cost estimate. The following assumptions listed below were made for this task and the associated pricing:

- Stone will be the primary contact with USACE NAE.
- Stone will work with Watermark to procure a test facility and equipment (SiREM of Guelph, Ontario, Canada);
- Bench-scale treatability tests (column tests) will be performed to determine the applicability of particular reactive treatment systems for the project;
- Laboratory column iron reactivity tests will be performed on groundwater samples collected from the project area to quantify the degradation rates of volatile organic compounds and metals in the presence of iron;
- The column test will also identify any potential for precipitation or clogging of the iron as a treatment medium and will also provide design data for the final design of a ZVI PRB;
- Six groundwater samples will be collected from the groundwater monitoring well locations or temporary well points in the vicinity of the proposed ZVI PRB using low-flow methods, and submitted for geochemical analyses, including:
    - Total organic carbon /total dissolved carbon by EPA Method 9060;
    - Total dissolved solids / total suspended solids by methods SM 2540C and 2540D, respectively;
    - Oil & grease by EPA Method 1664A;
    - pH by EPA Method 9040B;
    - Hardness by method SM 2340C;
    - Ferrous and ferric iron by ASTM Method D3872-05 (2011)and manganous and manganic manganese (EPA Method 1638, Modified);
    - Nitrate / nitrite by EPA Method 300.0;
    - Sulfate / sulfite by EPA Method 300.0;
    - Total metals, including chromium, iron, magnesium, manganese, silicon, and titanium by EPA Methods 6010B;
    - Hexavalent and trivalent chromium by EPA Method 7196A; and,
    - Carbonate by EPA Method 2320B;
- The samples will be collected during the same mobilization as work performed under either Subtask 4A, 5A, or Optional Task 5E, and will not incur an additional mobilization; and,

- The results of the treatability testing will be reported in the Pre-Design Sampling Report (Task 6B).

5.6.2. Task 6B: Treatability Study Laboratory Report

Watermark will be the lead for the compilation of the bench-scale treatability testing performed during Subtask 6A. The following assumptions listed below were made for this task and the associated pricing:

- Stone will be the primary contact with USACE NAE;
- Reporting will be limited to a review of the laboratory report and preparation for and participation in a conference call with USACE to discuss the results and potential issues identified by the testing that may affect full-scale implementation and cost.

5.7. Task 7: Pre-Design Sampling Report

Stone will be the lead for the preparation of the Client Draft, Draft, and Final Pre-Design Sampling Report in accordance with USEPA *Guidance for Conducting Remedial Investigations and Feasibility Studies under CERCLA* (EPA/540/G-89/004).

The following clarifications and assumptions are associated with this task:

- Stone will be the primary contact with CENAE;
- Up to 15 figures (assumed to be prepared using computer-aided design [CAD] and/or geographic information system [GIS] software) will be completed to support the conceptual design of remedial alternatives;
- The Client Draft Pre-Design Sampling Report will be reviewed concurrently by USACE NAE, USACE NAN, GSA and CX;
- The Pre-Design Sampling Report will include the following components
    - A summary of pre-design characterization activities;
    - The findings of the pre-design study;
    - Recommended locations and dimensions of the ZVI PRB;
    - Estimated construction costs;
    - Tabulated groundwater and soil data, including aquifer testing results and the calculations used to determine aquifer parameters;
    - Soil boring logs and monitoring well construction logs;
    - Geologic cross-sections, a groundwater contour map, updated location of the TCE plume, and groundwater flow and contaminant migration;
    - A land access study for the proposed location of the ZVI PRB; and
    - Calculations estimating how long the proposed PRB design would be effective.

5.8. Task 8 (Optional): Additional Vertical Groundwater Profiling Locations

Should additional pre-design investigation groundwater profiles be required, beyond the fifteen (15) included under Tasks 5A and 5B, Stone will advance an additional groundwater profile to provide additional data to verify and refine the CSM. The following clarifications and assumptions are associated with this task:

- Groundwater profiling will be performed in the same manner described under Task 5A;
- Approximately one-half day mobilization to and demobilization from the Former Scotia Naval Depot, and approximately one day setup for the drill rig following arrival; the cost of the mobilization ($9,460) would only apply to the first additional location, assuming all additional vertical profiles are performed during the same mobilization and,
- This optional task may be executed up to five (5) times (the Cost Estimate shows the cost for the first additional vertical profile location and includes the mobilization cost; additional locations would cost $10,162.65).

5.9. Task 9 (Optional): Additional Monitoring Well Locations

Should additional pre-design investigation monitoring wells be required beyond the five (5) included under Task 5E, Stone will install a monitoring well to supplement plume definition in order to provide additional data to verify and refine the CSM. The following clarifications and assumptions are associated with this task:

- Monitoring wells will be installed at additional groundwater profiling locations that are performed under Optional Task 8;
- Monitoring well installation will be performed in the same manner described under Task 5E;
- Approximately one-half day mobilization to and demobilization from the Former Scotia Naval Depot, and approximately one day setup for the drill rig following arrival; the cost of the mobilization ($1,400) would only apply to the first additional well, assuming all additional wells are installed during the same mobilization and,
- This optional task may be executed up to four (4) times (the Cost Estimate shows the cost for the first additional well and includes the mobilization cost; additional wells would cost $16,747.64).

5.10. Task 10 (Optional): Additional Monitoring Well Sampling

Should additional pre-design investigation groundwater sampling be required at any monitoring wells installed under Optional Task 9, Stone will collect groundwater samples from a monitoring well to supplement plume definition, in order to provide additional data to verify and refine the CSM. The following clarifications and assumptions are associated with this task:

- Monitoring well groundwater sampling will be performed in the same manner described under Task 4A; and,
- This optional task may be executed up to four (4) times.

5.11. Task 11 (Optional): Additional Hydraulic Conductivity Testing

Should additional pre-design hydraulic conductivity testing be required beyond the four (4) wells included under Task 5F, Stone will perform hydraulic conductivity testing in a monitoring well to supplement plume definition, in order to provide additional data to verify and refine the CSM. The following clarifications and assumptions are associated with this task:

- Hydraulic conductivity testing will be performed in the same manner described under Task 5F;
- Hydraulic conductivity testing will be performed using the same setup and during the same mobilization described under Task 5F; and,

- This optional task may be executed up to four (4) times.

5.12. Task 12 (Optional - Executed): Soil Vapor Intrusion Investigation

Stone will complete a SVI Investigation at selected onsite buildings and offsite residences at the Former Scotia Navy Depot, to evaluate current human health risks. Based on USEPA, DoD, NYSDEC, and NYDOH SVI guidance documents, in order to evaluate current human health risks, Stone proposes to collect synchronous sub-slab soil vapor, indoor air, and outdoor air samples.

5.12.1. Task 12A: Soil Vapor Intrusion Study Planning

Stone will conduct a pre-sampling site inspection of the on-site buildings over the estimated TCE and carbon tetrachloride plumes, in preparation for SVI sampling, and prepare an SVI Investigation Project Plan. The following clarifications and assumptions are associated with this task:

- The inspection will include Buildings 201, 202, 203, and 204 and up to four (4) off-site residential locations, to identify the appropriate sampling locations;
- Based on NYSDOH SVI guidance (*Guidance for Evaluating Soil Vapor Intrusion in the State of New York*, October 2006), Stone will collect sub-slab and indoor air samples to evaluate the possibility of SVI;
- Rooms where indoor air sampling will be conducted will be inspected to ensure that no internal sources of the contaminants are present;
- Stone will accurately complete the Exhibit G-3 Building Evaluation Form from the DoD SVI Handbook and NYSDOH Guidance, Appendix B, Indoor Air Quality Questionnaire Building Inventory following the inspection;
- Stone will also perform the SVI study in accordance with the following guidance documents:
  - *Tri-Services Environmental Risk Assessment Workgroup, DoD Vapor Intrusion Handbook*, (January 2009);
  - USEPA's current *Guidance for Evaluating the Vapor Intrusion to Indoor Air Pathway from Groundwater and Soils* (November 2002), and,
- Using the site inspection results and other data, Stone will prepare a client draft, draft, and final SVI Investigation Project Plan for the SVI Investigation, including proposed number, types, and locations of SVI samples, per the description of samples included in the SOW.

5.12.2. Task 12B: Vapor Intrusion Sampling, Onsite

Stone will collect up to thirty-one (31) SVI samples under conservative conditions (e.g., windows and doors closed, heating, ventilating, and/or air conditioning in operation). The following assumptions are associated with this task:

- Up to thirty-one (21) samples will be collected from the six buildings and submitted for analysis:
  - Two to three (2 to 3) indoor air samples from each 200-block building on the ground or sub-grade floor (total of ten (10) samples, plus one duplicate sample);
  - Two to three (2 to 3) sub-slab samples from each 200- and 400-block building, located near the estimated plume areas (total of fourteen (14) samples plus one duplicate sample);

  - Two (2) outdoor ambient air samples (will serve for all four 200- and two 400-block buildings) (total of two (2)); and;
  - One (1) trip blank sample;
- Sub-slab soil vapor and indoor air samples will be collected from the basement or the lowest occupied floor of each building;
- The sub-slab samples and indoor air samples will be collected concurrently from the 200-block buildings over an 8-hr period;
- The SVI samples will be analyzed via EPA Toxic Organic (TO) Method 15. Selected ion monitoring (SIM) analysis shall be performed for carbon tetrachloride and TCE, and their breakdown products;
- The laboratory selected to perform the analysis will meet Method T0-15 laboratory certification requirements specified by the NYSDOH and be accredited for Method T0-15 according to the DoD Environmental Laboratory Accreditation Program (ELAP); and
- Sample reporting limits will be sufficient to meet NYSDOH and EPA screening levels for carbon tetrachloride and TCE, and their breakdown products.

5.12.3. Task 12C: Vapor Intrusion Sampling, Offsite

GSA was unable to obtain any access to offsite residential properties, so Stone will not collect SVI samples at offsite locations identified in the ROD.

5.12.4. Task 12D: Vapor Intrusion Investigation Report and Data Deliverable

Stone will prepare a report summarizing the results of the SVI Investigation. The following assumptions are associated with this task:

- Stone will prepare Draft, Draft Final, and Final versions. Stone will prepare response to comment packages to the draft and draft final versions of the report and modify the versions to reflect the comments received.
- Current NYSDEC, DoD, and EPA guidance will be used to evaluate and interpret the results.
- Stone will provide the SVI sampling data in a suitable electronic format.

5.12.5. Task 12E: Additional Vapor Intrusion Sampling

Additional air and sub-slab samples may be identified during the SVI Planning (Optional Task 12A). Stone has provided a per sample cost for an indoor air sample, ambient air sample, and sub-slab sample. The following assumptions are associated with this task:

- The per sample costs include additional labor, installation/equipment, and laboratory analytical costs.
- The costs assume the samples will be collected during the same mobilization as those collected during Tasks 12B and 12C.

5.13. Task 13: Project Closeout

Stone will perform the necessary activities to close out the work assignment in accordance with contract requirements. The following assumptions are associated with this task:

- Electronic submission of working files;
- Packaging and returning documents to the government; and,
- Archiving files in accordance with Federal Record Center.

5.14. Payment Plan (Schedule)

The project will be invoiced on a milestone/percent complete basis. Each milestone and associated percent complete is shown in Table 1 below. Each milestone will be triggered by a defined deliverable, as shown in Table 1.

Table 4: Payment Schedule

Task/Milestone	Milestone Deliverables	Associated % Complete	Milestone Payment
1 Project Management / Status Reports			
1A / 1B / 1C Status Reports / Invoicing / Project Coordination			
Submittal of Monthly Status Report	Monthly Status Reports (10% per month for 10 months)	10%	$2,460.78/Month
	Total	**100%**	**$24,607.78**
1D Project Meetings			
First Onsite Meeting	First Onsite Meeting Submit Draft Minutes	33%	$6,345.22
Second Onsite Meeting	Second Onsite Meeting Submit Draft Minutes	33%	$6,345.22
First Offsite Meeting	First Offsite Meeting Submit Final Minutes	17%	$3,268.75
Second Offsite Meeting	Second Offsite Meeting Submit Final Minutes	17%	$3,268.75
	Total	**100%**	**$ 19,227.93**
2 Site-Specific Plans			
2A / 2B / 2C Overall Project Plan / Field Sampling Plan / Quality Assurance Project Plan / Health and Safety Plan			
Submittal of Draft Project Plans	Client Draft PP / FSP / QAPP / HASP	60%	$13,959.38
Submittal of Final Project Plans	Final PP / FSP / QAPP / HASP & all associated electronic files	30%	$6,979.69
Approval of Final Project Plans	CENAE acceptance e-mail stating that milestone is complete	10%	$2,326.56
	Total	**100%**	**$23,265.64**

Task/Milestone	Milestone Deliverables	Associated % Complete	Milestone Payment
3 Community Involvement (Optional)			
Prepare for and Attend one Public Meeting	Attend one Public Meeting	35%	$3,463.09
Draft Public Meeting Minutes	Client Draft Public Meeting Minutes	10%	$989.45
Final Public Meeting Minutes	Final Public Meeting Minutes & all associated electronic files	5%	$494.73
Approval of Final Public Meeting Minutes	CENAE acceptance e-mail stating that milestone is complete	5%	$494.73
Submittal of Draft Fact Sheet	Client Draft Fact Sheet	20%	$1,978.91
Submittal of Final Fact Sheet	Final Fact Sheet	15%	$1,484.18
Approval of Final Fact Sheet	CENAE acceptance e-mail stating that milestone is complete	10%	$989.45
	Total	**100%**	**$$9,894.53**
4 Site-Wide Groundwater Sampling			
4A / 4B Groundwater Sampling / Synoptic Measurements			
Field Work	Completion of Field Work	60%	$14,076.80
Submittal of Draft Field Trip Report	Client Draft Field Trip Report	20%	$4,692.27
Submittal of Final Field Trip Report	Final Field Trip Report & all associated electronic files	10%	$4,692.27
Approval of Final Field Trip Report	CENAE acceptance e-mail stating that milestone is complete	10%	$2,346.13
	Total	**100%**	**$23,461.34**
4C Preliminary Groundwater Modeling (Optional)			
Submittal of Client Draft Groundwater Model	Client Draft Groundwater Model	60%	$26,033.30
Submittal of Final Groundwater Model	Final Groundwater Model & all associated electronic files	30%	$13,016.65
Approval of Final Groundwater Model	CENAE acceptance e-mail stating that milestone is complete	10%	$4,338.88
	Total	**100%**	**$43,388.84**

Task/Milestone	Milestone Deliverables	Associated % Complete	Milestone Payment
5A / 5D Transect Vertical Profiling/Mobile Laboratory Analysis			
Field Work	Completion of Field Work	60%	$138,452.90
Submittal of Draft Field Trip Report	Client Draft Field Trip Report	20%	$46,150.97
Submittal of Final Field Trip Report	Final Field Trip Report & all associated electronic files	10%	$23,075.48
Approval of Final Field Trip Report	CENAE acceptance e-mail stating that milestone is complete	10%	$23,075.48
	Total	**100%**	**$230,754.83**
5B Geotechnical Sampling (Optional - Executed)			
Field Work	Completion of Field Work	60%	$6,038.03
Submittal of Draft Field Trip Report	Client Draft Field Trip Report	20%	$2,012.68
Submittal of Final Field Trip Report	Final Field Trip Report & all associated electronic files	10%	$1,006.34
Approval of Final Field Trip Report	CENAE acceptance e-mail stating that milestone is complete	10%	$1,006.34
	Total	**100%**	**$10,063.39**
5C Non-Transect Vertical Profiling (Optional - Executed)			
Field Work	Completion of Field Work	60%	$51,971.71
Submittal of Draft Field Trip Report	Client Draft Field Trip Report	20%	$17,323.90
Submittal of Final Field Trip Report	Final Field Trip Report & all associated electronic files	10%	$17,323.90
Approval of Final Field Trip Report	CENAE acceptance e-mail stating that milestone is complete	10%	$8,661.95
	Total	**100%**	**$86,619.51**
5E Monitoring Well Installation (Optional - Executed)			
Field Work	Completion of Field Work	60%	$32,040.22
Submittal of Draft Field Trip Report	Client Draft Field Trip Report	20%	$10,680.07
Submittal of Final Field Trip Report	Final Field Trip Report & all associated electronic files	10%	$5,340.04
Approval of Final Field Trip Report	CENAE acceptance e-mail stating that milestone is complete	10%	$5,340.04
	Total	**100%**	**$53,400.36**

Task/Milestone	Milestone Deliverables	Associated % Complete	Milestone Payment
5F Hydraulic Conductivity Testing (Optional - Executed)			
Field Work	Completion of Field Work	60%	$3,701.63
Submittal of Draft Field Trip Report	Client Draft Field Trip Report	20%	$1,233.88
Submittal of Final Field Trip Report	Final Field Trip Report & all associated electronic files	10%	$616.94
Approval of Final Field Trip Report	CENAE acceptance e-mail stating that milestone is complete	10%	$616.94
	Total	**100%**	**$6,169.39**
5G Groundwater Modeling (Optional)			
Submittal of Client Draft Groundwater Model	Client Draft Groundwater Model	60%	$19,779.34
Submittal of Final Groundwater Model	Final Groundwater Model & all associated electronic files	30%	$9,889.67
Approval of Final Groundwater Model	CENAE acceptance e-mail stating that milestone is complete	10%	$3,296.56
Attend Meeting	**Total**	**100%**	**$32,965.57**
6 Treatability Study			
6A Treatability Study Testing			
Submittal of Client Draft Groundwater Model	Submittal of Client Draft Groundwater Model	60%	$19,779.34
Submittal of Final Groundwater Model	Submittal of Final Groundwater Model	30%	$9,889.67
Approval of Final Groundwater Model	CENAE acceptance e-mail stating that milestone is complete	10%	$3,296.56
	Total	**100%**	**$50,969.19**
6B Treatability Study Laboratory Report			
Review/Submittal of Treatability Study Laboratory Report	Schedule Conference Call for Treatability Study Laboratory Report	50%	$1,252.15
Conference Call for Treatability Study Laboratory Report	Conference Call for Treatability Study Laboratory Report	30%	$751.29
Minutes for Treatability Study Laboratory Report Conference Call	Minutes for Treatability Study Laboratory Report Conference Call & all associated electronic files	10%	$250.43
Approval of Final Treatability Study Laboratory Report Conference Call Minutes	CENAE acceptance e-mail stating that milestone is complete	10%	$250.43
	Total	**100%**	**$2,504.29**

Task/Milestone	Milestone Deliverables	Associated % Complete	Milestone Payment
7 Pre-Design Investigation Report			
Submittal of Client Draft Pre-Design Investigation Report	Client Draft Pre-Design Investigation Report	50%	$19,976.39
Submittal of Draft Pre-Design Investigation Report	Draft Pre-Design Investigation Report	25%	$9,988.19
Submittal of Final Pre-Design Investigation Report	Final Pre-Design Investigation Report & all associated electronic files	15%	$5,992.92
Approval of Final Pre-Design Investigation Report	CENAE acceptance e-mail stating that milestone is complete	10%	$3,995.28
	Total	**100%**	**$39,952.77**
8 Additional Vertical Profiling Locations (Optional)			
Field Work	Completion of Field Work	60%	$16,895.19
Submittal of Draft Field Trip Report	Client Draft Field Trip Report	20%	$5,631.73
Submittal of Final Field Trip Report	Final Field Trip Report & all associated electronic files	10%	$2,815.87
Approval of Final Field Trip Report	CENAE acceptance e-mail stating that milestone is complete	10%	$2,815.87
	Total	**100%**	**$28,158.65**
9 Additional Monitoring Well Locations (Optional)			
Field Work	Completion of Field Work	60%	$10,888.58
Submittal of Draft Field Trip Report	Client Draft Field Trip Report	20%	$3,629.53
Submittal of Final Field Trip Report	Final Field Trip Report & all associated electronic files	10%	$1,814.76
Approval of Final Field Trip Report	CENAE acceptance e-mail stating that milestone is complete	10%	$1,814.76
	Total	**100%**	**$18,147.64**
10 Additional Monitoring Well Sampling (Optional)			
Field Work	Completion of Field Work	60%	$1,275.94
Submittal of Draft Field Trip Report	Client Draft Field Trip Report	20%	$425.31
Submittal of Final Field Trip Report	Final Field Trip Report & all associated electronic files	10%	$212.66
Approval of Final Field Trip Report	CENAE acceptance e-mail stating that milestone is complete	10%	$212.66
	Total	**100%**	**$2,126.57**

Task/Milestone	Milestone Deliverables	Associated % Complete	Milestone Payment
11 Additional Hydraulic Conductivity Testing (Optional)			
Field Work	Completion of Field Work	60%	$584.35
Submittal of Draft Field Trip Report	Client Draft Field Trip Report	20%	$194.78
Submittal of Final Field Trip Report	Final Field Trip Report & all associated electronic files	10%	$97.39
Approval of Final Field Trip Report	CENAE acceptance e-mail stating that milestone is complete	10%	$97.39
	Total	**100%**	**$973.91**
12 Soil Vapor Intrusion Study (Optional - Executed)			
12A Soil Vapor Intrusion Study Planning (Optional - Executed)			
Submittal of Draft FSP / QAPP / HASP	Client Draft FSP / QAPP / HASP	60%	$10,725.86
Submittal of Final FSP / QAPP / HASP	Final FSP / QAPP / HASP & all associated electronic files	30%	$5,362.93
Approval of Final FSP / QAPP / HASP	CENAE acceptance e-mail stating that milestone is complete	10%	$1,787.64
	Total	**100%**	**$17,876.43**
12B Soil Vapor Intrusion Sampling, Onsite (Optional - Executed)			
Field Work	Completion of Field Work	60%	$12,465.28
Submittal of Draft Field Trip Report	Client Draft Field Trip Report	20%	$4,155.09
Submittal of Final Field Trip Report	Final Field Trip Report & all associated electronic files	10%	$2,077.55
Approval of Final Field Trip Report	CENAE acceptance e-mail stating that milestone is complete	10%	$2,077.55
	Total	**100%**	**$20,775.46**
12C Soil Vapor Intrusion Sampling, Offsite (Optional - Executed)			
Field Work	Completion of Field Work	60%	$4,384.08
Submittal of Draft Field Trip Report	Client Draft Field Trip Report	20%	$1,461.36
Submittal of Final Field Trip Report	Final Field Trip Report & all associated electronic files	10%	$730.68
Approval of Final Field Trip Report	CENAE acceptance e-mail stating that milestone is complete	10%	$730.68
	Total	**100%**	**$7,306.80**

Task/Milestone	Milestone Deliverables		Associated % Complete	Milestone Payment
12D Soil Vapor Intrusion Investigation Report (Optional - Executed)				
Submittal of Client Draft Pre-Design Investigation Report	Client Draft Pre-Design Investigation Report		60%	$5,873.86
Submittal of Draft Pre-Design Investigation Report	Draft Pre-Design Investigation Report		20%	$2,936.93
Submittal of Final Pre-Design Investigation Report	Final Pre-Design Investigation Report		10%	$1,762.16
Approval of Final Pre-Design Investigation Report	CENAE acceptance e-mail stating that milestone is complete		10%	$1,174.77
		Total	**100%**	**$11,747.71**
12E Additional Soil Vapor Intrusion Sampling (Optional)				
Field Work	Completion of Field Work		60%	$810.10
Submittal of Draft Field Trip Report	Client Draft Field Trip Report		20%	$270.03
Submittal of Final Field Trip Report	Final Field Trip Report & all associated electronic files		10%	$135.02
Approval of Final Field Trip Report	CENAE acceptance e-mail stating that milestone is complete		10%	$135.02
		Total	**100%**	**$1,350.17**
13 Project Closeout				
Submittal of all associated electronic files	CENAE acceptance e-mail stating that milestone is complete		100%	$1,747.83
		Total	**100%**	**$1,747.83**
Total Project Value				**$767,456.53**

6. QUALITY MANAGEMENT PLAN

6.1. Data Quality Objectives

Achieving the data quality objectives (DQOs) for the pre-design investigation and treatability study field tasks will enable proper data interpretation, confident updating of the CSM, and a sound technical basis from which to evaluate remedy options for the project. A summary of the task-specific DQOs for each field task is provided in Table 5.

Table 5: Summary of Task-Specific Data Quality Objectives.

Task	Data Quality Objective(s)
Collection and analysis of groundwater samples from the Site and vicinity	Identify appropriate monitoring wells for sampling. Perform groundwater sampling in accordance with low-flow methodology to obtain representative groundwater samples. Achieve analytical detection limit objectives.
Synoptic measurement of groundwater elevations	Identify appropriate monitoring wells for measurement. Achieve measurement reporting limit objectives.
Vertical groundwater profiling of the dissolved groundwater plume	Delineate the extent of the dissolved CVOC plume source in three dimensions. Characterize the chemical and physical properties of the aquifer. Achieve analytical detection limit objectives.
Collection and analysis of geotechnical soil samples	Characterize the physical properties of the aquifer. Achieve analytical detection limit objectives.
Installation of overburden groundwater monitoring wells	Install and develop overburden monitoring wells at appropriate screened intervals to collect representative groundwater samples.
Hydraulic conductivity testing of monitoring wells	Collect representative hydraulic conductivity data to characterize the physical properties of the aquifer.
Groundwater modeling	Synthesize physical and chemical aquifer data to characterize the properties of the aquifer.
Treatability testing (bench-scale column tests) of soil and groundwater	Characterize the physical and chemical properties of the aquifer. Characterize the treatability of the dissolved CVOC plume. Achieve analytical detection limit objectives.
Collection and analysis of soil vapor / indoor air samples	Identify appropriate potential SVI receptors for soil vapor / indoor air sampling. Perform soil vapor / indoor air sampling in accordance with USEPA, DoD, NYSDEC, and NYSDOH guidance to obtain representative samples.

QAPP Worksheets #11, 12, and 20 provide greater detail regarding task and analytical DQOs for each task. These details have been incorporated into the descriptions of the procedures for the field tasks described in Subsections 2.1 through 2.14 of the FSP.

6.2. Quality Control

Appropriate documentation of chemical quality control shall be provided whenever sampling or analysis for chemical constituents is required in order to meet the *SOW* objectives. Quality control for traditional soils, geophysical, or geotechnical testing shall also be included. The laboratories to be used by Stone will be certified by NELAP or an equivalent agency. Stone's onsite laboratory shall meet the requirements of EPA and NYSDOH requirements. Stone shall also comply with all requirements of the *DoD Quality Systems Manual*, Final Version 3, dated January 2006, or more recent version.

6.2.1. Field Quality Control Audits

Field records will be reviewed by the field operations leader (FOL) to ensure that:

- Logbooks and standardized forms have been filled out completely and that the information recorded accurately reflects the activities that were performed.
- Records are legible and in accordance with good recordkeeping practices, e.g., entries are signed and dated, data are not obliterated, and changes are initialed, dated, and explained.
- Sample collection, handling, preservation, and storage procedures were conducted in accordance with the FSP and any deviations were documented and approved by the appropriate personnel.

6.2.2. Laboratory Quality Control Audits

Prior to being released as final, laboratory data will proceed through a tiered review process. Data verification starts with the analyst, who performs a 100% review of the data to ensure the work was done correctly the first time.

Following the completion of the initial verification by the analyst performing the data reduction, a systematic check of the data will be performed by an experienced peer or supervisor.

Laboratory data reduction procedures will be performed according to the following protocol: Information related to analysis will be documented in controlled laboratory logbooks, instrument printouts, or other approved forms. All entries that are not generated by an automated data system will be made neatly and legibly in permanent, waterproof ink. Information will not be erased or obliterated. Corrections will be made by drawing a single line through the error and entering the correct information adjacent to the strikeout. All changes will be initialed, dated, and, if appropriate, accompanied by a brief explanation. Unused pages or portions of pages will be crossed out to prevent future data entry. Analytical laboratory records will be reviewed by the Laboratory Quality Assurance (QA) Coordinator periodically, to verify adherence to documentation requirements.

6.2.3. Field and Analytical Quality Control

6.2.3.1. Field Quality Control

All field procedures performed throughout the pre-design investigation and treatability study will be performed pursuant to all Standard Operating Procedures (SOPs) and/or USACE-approved methods included in the FSP. All monitoring equipment used onsite will be maintained, calibrated, and operated as specified in the FSP.

Field QC samples will be collected during sampling to assess the accuracy and precision of the analytical data. These samples include trip blanks, equipment blanks, field duplicates, and matrix spike/matrix spike duplicates (MS/MSDs). The field QC samples and the acceptance criteria for these samples are summarized in Table 6.

Table 6: Summary of Field Quality Assurance Samples

QA Sample	Description	Acceptance Criteria
Trip Blank	Trip blanks will be included with each shipment of sample vials delivered to the Site. Trip blanks associated with aqueous CVOC samples will originate in the laboratories (onsite and offsite) and will be prepared by filling two 40-mL sample vials with laboratory deionized water and sealing the vials with septum-lined caps (allowing no headspace). Trip blanks will accompany the sample bottles to the sampling sites and will remain (unopened) in the shipping container until the sample bottles are delivered to the respective laboratories..	Analytes in the trip blanks, if detected, should be less than one-half the laboratory reporting limit.
Equipment Blank	Equipment blanks will be prepared by routing analyte-free water (provided by the laboratory) through or over non-dedicated sampling equipment after equipment decontamination and before field sample collection. Equipment blanks will be collected for samples collected with non-dedicated equipment (at a frequency of one per ten or less samples) and will be analyzed for the same VOC compound list as the primary samples.	Analytes in the equipment blanks, if detected, should be less than one-half the laboratory reporting limit.
Field Duplicate	Field duplicates will be collected at a frequency of one field duplicate for every 10 or less samples submitted for CVOC analyses. Sample containers for aqueous field duplicates will be filled consecutively.	Field duplicate relative percent differences (RPDs) are considered acceptable if they are within +/- 30% for aqueous samples and within +/- 50% for solid samples.
MS/MSD	MS/MSD samples will be collected at a frequency of one for every 20 or less samples submitted for VOC analyses. For those samples designated as MS/MSDs and replicates, sufficient additional volume will be provided.	Acceptance criteria for the percent recoveries of spiked compounds and RPDs for the duplicate analyses will be specified in the QAPP

6.2.3.2. Analytical Quality Control

The selected analytical laboratories will have QC programs in place to ensure the reliability and validity of the analysis performed at the laboratories. All analytical procedures will be documented in writing as SOPs and each SOP will include the minimum requirements for the procedure. In general, the QC requirements include the following:

- Blanks (method, reagent/preparation, instrument)
- MS/MSDs
- Surrogate spikes
- Laboratory Control Samples (LCSs)
- Internal standard areas and retention times (GC/MS analysis)

The criteria by which these QC samples will be evaluated are specified in QAPP Worksheet # 12.

In order to demonstrate the accuracy of the onsite laboratory, three performance evaluation (PE) samples will be analyzed at the beginning of the field program. The PE samples will be purchased from Environmental Resource Associates (ERA). Two PE standards will be purchased: one with a concentration between 1 and 100 µg/L (low-range), and the second between 100 and 500 µg/L (medium-range). The PE samples will include the limited analytes listed for onsite laboratory reporting listed in Subsection 2.9 of the FSP. The PE samples

provided by ERA have performance limits published by the vendor. ERA will issue an acceptance criteria summary which is based on the lot number and the results from various labs.

6.2.4. Data Review and Validation

The completeness goal for the project is that more than 90% of the data must be usable (not qualified as "rejected"), with the exception of serial dilutions where the result for one dilution is rejected and replaced by that of another. Acceptable limits for laboratory quality control tests will be described in the laboratory-specific SOPs that will be part of the QAPP.

6.2.4.1. Fixed Laboratory Automated Data Review

One EDD will be provided by the fixed laboratory as a staged electronic data deliverable (SEDD) file in accordance with USACE requirements as described below.

Each fixed laboratory will report data using the SEDD format. The minimum requirement for the laboratories is the delivery of a SEDD Stage 2a deliverable, a PDF document file of the laboratories' final data report, and a hard copy of the laboratories' final data report. The laboratories will provide any supplemental information (e.g., chromatograms, instrument calibrations) upon request.

Once the SEDD electronic data files have been received from the laboratory, Stone will electronically review the files to check project data quality requirements using USACE Automated Data Review (ADR) software. The ADR Software will be provided by Laboratory Data Consultants Inc. through USACE. Stone will develop a comprehensive ADR project library file for all of the methods to be analyzed on the project. The project library files will accurately reflect all of the analytical quality requirements as documented in the final project QAPP and addendums and will be provided prior to field sampling. The project library file will be provided to the laboratory for use in screening the EDD submittals.

Each laboratory is required to check the integrity of the SEDD using the SEDD checker tool and check the SEDD file against the ADR project library using the Contract Compliance Screening (CCS) tool contained in ADR (Laboratory Version). EDD non-conformances generated by ADR for which the laboratory is responsible will be corrected at the laboratory. A new SEDD file will be resubmitted and reviewed again. The laboratory shall address via a brief explanation in the non-conformance log any non-conformance that is not within their control. The final SEDD extensible markup language (XML) file and final laboratory non-conformance report will be provided to USACE's chemist for review.

Stone will perform a data review on the SEDD XML file by importing the SEDD files directly into ADR. The files will be reviewed utilizing the ADR (Consultant Version) software to check for compliance using the same ADR Project Library used by the laboratory. The ADR reviewed files will be imported into Stone's copy of the USACE Environmental Data Management System (EDMS). Stone will provide USACE with:

1. The reviewed data from ADR in standard ADR format (A1 through A5) as .txt files; and
2. The USACE EDMS generated database file(s) in *.mdb format incorporating current chemistry data and corresponding field data via the USACE ftp web site, or some other mutually agreed upon means.

The database file will be submitted to USACE after any subsequent updates. All postings to the USACE ftp web site will be accompanied by notification of said posting to a USACE project point of contact.

All electronic data submitted by the laboratory is required to be error free, and in complete agreement with the hard copy data. Data files are to be delivered both by e-mail and on high-density CD accompanying the hard copy data reports. The disk must be submitted with a transmittal letter from the laboratory that certifies that the file is in agreement with hard copy data reports and has been found to be free of errors using the latest version of the ADR evaluation software provided to the laboratory. The laboratory will correct any errors identified by Stone or USACE.

Each laboratory is required to check the integrity of the SEDD using the Contract Compliance Screening (CCS) tool contained in ADR (Laboratory Version) against the ADR Project Library. EDD non-conformances generated by ADR that the laboratory is responsible for will be corrected at the laboratory and a new SEDD file will be resubmitted and reviewed again. The laboratory will address via a brief explanation in the non-conformance log any non-conformance that is not within their control. The data evaluation for this project will conform to the requirements summarized in Chapter 2 of EM 200 1 6 (USACE, 1997), as defined in the ADR project library. The data evaluation will include the following:

1. Review of chain-of-custody documents to verify sample identities.
1. Review of sample log-in documents to verify any potential problems with custody seals, container integrity, sample preservation, labeling, etc.
2. Review of field rinsate blank data to ascertain any potential problems with container contamination, preservative contamination, sampling equipment contamination, or cross-contamination between samples during transport.
3. Review of trip blank data to identify any potential problems with sample container contamination, preservative contamination, laboratory reagent water contamination, or cross-contamination between samples during transport.
4. Review of method blank data to determine the presence of any sources of contamination in the analytical process due to laboratory contamination.
5. Review the matrix spike (MS) data to evaluate the potential for matrix effects and as a measure of analytical accuracy. MS recoveries will be compared against laboratory acceptance criteria and the ADR project library acceptance criteria to determine if they are within or outside of warning and control limits for percent recoveries.
6. Review of matrix spike/matrix spike duplicate (MS/MSD) data to evaluate sample homogeneity and as a measure of analytical precision. MS/MSD data will be compared to laboratory acceptance criteria and the ADR project library acceptance criteria for the maximum relative percent deviation (RPD).
7. Review of any laboratory control sample and laboratory control sample duplicate (LCS/LCSD) data (if available) as a measure of analytical accuracy and precision. LCS/LCSD recoveries will be compared against laboratory acceptance criteria and the ADR project library to determine if they are within or outside of warning and control limits for percent recoveries.
8. Review of LCS/LCSD data (if available) as a measure of analytical precision. LCS/LCSD data will be compared to laboratory acceptance criteria for the maximum RPD.
9. Standard reference material (SRM) or Laboratory Control Sample (LCS) data (if available) as a measure of analytical accuracy. SRM and LCS data will be compared to the certified acceptable ranges of analytical values.

10. Review of sample and sample duplicate data (if available) as a measure of sample homogeneity and as a measure of analytical precision. Sample and sample duplicate data will be compared against the laboratory acceptance criteria and the ADR project library for the maximum RPD.
11. Review of surrogate recovery data to access extraction efficiency, effectiveness of sample introduction, and possible loss during cleanup activities. Surrogate recoveries will be compared to laboratory acceptance criteria and the ADR project library to determine if they are within or outside of acceptable limits.
12. Review of sample dates, extraction/digestion dates, and analysis dates to determine if maximum method-specific holding times were met or exceeded.
13. The following items would typically not be reviewed under a SEDD stage 2a data validation effort: instrument tunes, standard curves, internal standard recoveries, system performance check compound results, continuing calibration results, inter-element correction check results, laboratory notebook pages, calculations, etc. However, supporting data (i.e. calibration curves, instrument tuning information) will be stored at the laboratory for a minimum of three years.

6.2.4.2. Data Review by Onsite Laboratory

Following the completion of the initial verification by the analyst performing the data reduction and reporting in the field, a systematic check of the data will be performed by an experienced peer or supervisor. This check will be performed to ensure that initial review has been completed correctly and thoroughly and will include a review of:

- Adherence to the requested analytical method SOP;
- Correct interpretation of chromatograms, mass spectra, etc.;
- Correctness of numerical input when computer programs are used (checked randomly);
- Correct identification and quantification of constituents with appropriate qualifiers;
- Numerical correctness of calculations and formulas (checked randomly);
- Acceptability of QC data;
- Documentation that instruments were operating according to method specifications (calibrations, performance checks, etc.);
- Documentation of dilution factors, standard concentrations, etc.; and
- Sample holding time assessment.

6.2.5. Reporting on Quality Control

6.2.5.1. Field Corrective Action

Corrective action in the field may be needed when sampling procedures and/or field analytical procedures require modification due to unexpected conditions. The field team may identify the need for corrective action. The FOL will approve the corrective action and notify the PM. The PM, in consultation with the QAO, will approve the corrective measure. The FOL will ensure that the corrective measure is implemented by the field team.

Additions to the proposed FSP (i.e., more samples, additional sampling locations other than those specified in the FSP, etc.) are not considered corrective actions. However, these changes will be discussed with USACE and NYSDEC during the weekly reports and before being implemented.

Corrective actions will be implemented and documented in the field record book or on a corrective action form. Documentation will include:

- A description of the circumstances that initiated the corrective action;
- The action taken in response;
- The final resolution; and
- Any necessary approvals.

No field crew member will initiate corrective action without prior review and authorization from the FOL.

6.2.5.2. Laboratory Corrective Action

Corrective action in the laboratory may occur prior to, during, and after initial analyses. A number of conditions such as broken sample containers, multiple phases, low/high pH readings, and potentially high concentration samples may be identified during sample log in or analysis. Following consultation with laboratory analysts and supervisory personnel, it may be necessary for the laboratory QA coordinator to approve the implementation of corrective action. If the non-conformance causes failure to achieve project objectives, the QAO or PM will be notified.

Laboratory corrective actions are performed prior to release of the data from the laboratory. The corrective action will be documented in both the laboratory's corrective action files, and in the narrative data report sent from the laboratory to the QAO. If the corrective action does not rectify the situation, the laboratory will contact the QAO, who will determine the action to be taken and inform the appropriate personnel.

7. COMMUNICATIONS PLAN

Project stakeholders include the USACE NAE / NAN, NYSDEC, NYSDOH, GSA, USEPA, DLA/DNSC and the Galesi Corporation. Clear communications at critical points in this project, verbally and in writing, will help to address stakeholders' concerns in a timely manner and advance the project. Outlined below are the protocols for:

- Monthly Status Reports;
- Submittal, review, and approval of deliverables;
- Maintenance of project repository and administrative record;
- Regulatory involvement;
- USACE Electronic Chemistry Data Deliverables.

7.1. Status Reports

Stone will issue Monthly Status Reports. Each status report will describe in bulleted format:

- Work that was completed in the previous month;
- Work that will be completed prior to the next monthly status report;
- Percent complete for each task; and,
- Any issues or comments related thereto.

7.2. Document Submittal, Review, and Approval Process

Table 7 outlines the general level of stakeholder involvement concerning each deliverable required by the *SOW* which is consistent with CERCLA/NCP and the NYSDEC regulations.

Table 7: Deliverables

Stakeholder	**Distribution**			
	Site Plans (PP, FSP, QAPP, HASP)	*Treatability Study Report*	*Pre-Investigation Design Report*	*SVI Investigation Report (Optional)*
U.S. Army Corps of Engineers New England District 696 Virginia Road Concord, MA 01742-2751 Attn: Dean Brammer	Electronic (Client Draft) 1 paper[1] and 1 CD[2] (Final)	Electronic (Client Draft) 1 paper[1] and 1 CD[2] (Final)	Electronic (Client Draft) 1 paper[1] and 1 CD[2] (Final)	Electronic (Client Draft) 1 paper[1] and 1 CD[2] (Final)
U.S. Army Corps of Engineers New York District CENAN-PP-E, Room 1811 26 Federal Plaza New York, NY 10278 Attn: Greg Goepfert	Electronic (Client Draft) 1 paper[1] and 1 CD[2] (Final)	Electronic (Client Draft) 1 paper[1] and 1 CD[2] (Final)	Electronic (Client Draft) 1 paper[1] and 1 CD[2] (Final)	Electronic (Client Draft) 1 paper[1] and 1 CD[2] (Final)

Stakeholder	Distribution			
General Services Administration Design and Construction Division, Northeast and Caribbean Region 26 Federal Plaza New York, NY 10278 Attn: David Baker	Electronic (Client Draft) 1 paper and 1 CD (Final)	Electronic (Client Draft) 1 paper and 1 CD (Final)	Electronic (Client Draft) 1 paper and 1 CD (Final)	Electronic (Client Draft) 1 paper and 1 CD (Final)
New York State Department of Environmental Conservation 625 Broadway, 12th Floor Albany, NY 12233-7017 Attn: Jason Pelton	Electronic[4] (Client Draft) 1 paper and 1 CD[3,4] (Final)	Electronic[4] (Client Draft) 1 paper and 1 CD[3,4] (Final)	Electronic[4] (Client Draft) 1 paper and 1 CD[3,4] (Final)	Electronic[4] (Client Draft) 1 paper and 1 CD[3,4] (Final)
New York State Department of Health Flannigan Square 547 River Street Troy, NY 12180 Attn: Maureen Schuck	--	1 paper and 1 CD[3] (Final)	Electronic (Client Draft) 1 paper and 1 CD[3] (Final)	Electronic (Client Draft) 1 paper and 1 CD[3] (Final)
Galesi Corporation 695 Rotterdam Industrial Park Building 6 Schenectady, NY 12306 Attn: David Ahl	--	--	--	1 paper and 1 CD (Final)
US EPA Region 2 Federal Facilities Program 290 Broadway, 21st Floor New York, NY 10007-1866 Attn: Jeanette Dadusc	--	--	--	1 paper and 1 CD (Final)

1 - Each paper report shall have a CD with the report in *.PDF format appended.

2 - The additional CDs for the USACE shall have both Word/Excel files and *.PDF files.

3 - Compact Disk being sent to NYDEC and NYDOH shall include the report and associated data in *.PDF format.

4 - Electronic document submissions to NYDEC will be accordance with 6 NYCRR 375-1.11(a).

Stone will submit documents as follows:

- Documents will be submitted as Client Draft and Final versions:
  - Project Plan / FSP / QAPP / HASP;
  - Site Walk / Meeting Minutes;
  - Trip Report for Implementing the Pre-Design Investigation Project Plan;
  - Pre-Design Investigation Report.
- There will be a twenty one (21) day client comment period following submittal of the Client Draft version;
- Response to Comments (RTC) packages will be prepared within twelve (12) business days following receipt of comments;
- There will be a ten (10) business day Comment Resolution period following the Client Draft RTC package;

- All reports will contain an Executive Summary;
- All documents will be submitted to the USACE NAE / NAN electronically and in hard copy. Stone will provide electronic submittals on computer CD-ROM which will include all working files. CD-ROMs will be clearly labeled with the file name and description in an orderly fashion. All working files will be provided in native format (Microsoft Word, Microsoft Excel, shape files, .dwg files, etc.). In addition, the electronic version will be submitted as one consolidated file in PDF format (Adobe Acrobat, most current version);
- Documents will be submitted in hard copy via overnight mail to the addresses in the project distribution list; and
- One copy of the final documents and other "Project-related information", on CD-ROM and hard copy, will be placed in the project repository.

All documents will be distributed with a transmittal letter from Stone. The letter of transmittal will include a certification that the submission has been subjected to Stone's own review and coordination procedures to ensure: (a) completeness for each discipline commensurate with the level of effort required for that submission, (b) elimination of conflicts, errors and omissions, and (c) overall professional and technical accuracy of the submission. Stone will also coordinate with the USACE NAE / NAN PMs for the number of copies to provide, since quantities vary depending on the type of deliverable. All project documents will include the following:

- Project Name – Former Defense National Stockpile Center Scotia Depot Site Project.
- CENAE Project Number – 397651; and,
- Current CERCLA Phase – Pre-Remedial.

7.3. Project Repository and Administrative Record

As part of project management activities, Stone will provide electronic documents to the Project Repository on behalf of USACE NAN. The Project Repository is the Schenectady County Public Library, located at 99 Clinton Street, Schenectady, New York (518-388-4500). These important resources enable all stakeholders to have complete and accurate information about the Site.

The Project Repository is USACE NAN property. The repository includes pertinent documentation and data necessary for project reviews, and to provide a clear record of the RD approach to support final decisions and phase completion. Stone will update documentation related to the project, in paper and electronic form, on a monthly basis to provide a complete record of project related information.

The Administrative Record will be updated by providing the USACE NAE electronic copies of documents. "Project-related information" includes all documents including critical e-mails generated during the course of this project. These documents are expected to be maintained in both electronic and paper copies.

7.3.1. PDF Standards

Stone will prepare all electronic documents according to the following specifications:

- Adobe PDF Version - The PDF file shall be in PDF Version 1.4, or later which is compatible with Acrobat 5.0;

- PDF Image and Searchable Text Conversion containing a bitmapped image of the original, and a hidden layer of searchable text. The conversion process will involve scanning the hardcopy original, performing Optical Character Recognition (OCR) to capture the text of the document, and distilling the two layers into a PDF searchable image file. The PDF searchable image files will be as legible as the original;
- Indexing - Stone will ensure that the PDF/Searchable Image files will be able to be indexed for full-text retrieval by any search engine capable of indexing PDF files; and
- Bookmarks - Stone will bookmark all major section headings and table of contents according to the document table of contents to provide quick access to specific areas of the PDF document in order to have retrieval of chapters, sections, and appendices one click away.

7.3.2. Document Security

Each PDF file will have the following document security settings applied. The Master Password will be "DERP-FUDS PIRS":

- Security Method: Adobe Standard Security;
- Encryption Level: 128-bit RC4 (Acrobat 4.0);
- User Password: No;
- Master Password: Yes;
- Printing: Fully Allowed;
- Changing the Document: Not Allowed;
- Copying Content or Extraction: Allowed;
- Authoring Comments and Form Fields: Not Allowed;
- Form Field Filling or Signing: Not Allowed;
- Content Accessibility Enabled: Allowed; and
- Document Assembly: Not Allowed.

7.3.3. Document Options

- Fast Web Viewing. The PDF file will be saved to optimize the file for Fast Web Viewing; and
- Background PDF Downloading. The PDF will be created to permit background PDF downloading.

7.3.4. Document Open Options

- Initial View: Bookmarks and Page;
- Page Number: 1;
- Magnification: Fit in Window;
- Window Options: Resize Window to initial page, and Display Title File Naming Convention. Each PDF file will be individually named by the list below. Between each item there will be an underscore and no spaces;

- File Extension will be “pdf”;

7.3.5. Scanning Standards

Stone will scan all pages according to the following:

Document Type	Color Type	Scanning Resolution(dpi)
Black & white text	Binary	300
Black & white drawing/map	Binary	150
Grayscale photo/drawing/map	Black & white photo 16 Shades of grey	150
Color Photo/drawing/map	Color Photo 256 colors	150

- Image Orientation: All pages will be at the correct orientation, i.e. text will be readable without rotating the pages; and
- De-skewing. If necessary, pages will be de-skewed to ensure correct page orientation.

7.3.6. Procedure for Retention and Digitization of Critical Emails

E-mails used in the decision making process are considered critical e-mails and must be both retained in the hardcopy project file and sent to the FUDS File email address that is listed in the previous section. Examples of critical e-mails include, but are not limited to the following:

- Review comments from regulators or the public on project documents;
- Approval/disapproval notices from regulators regarding project decisions;
- Meeting minutes; and
- E-mails describing situations that affect project direction/progression.

7.4. Regulatory Involvement

With the direction and approval of the USACE NAN PM, Stone will provide the necessary support to initiate, schedule, and address all regulatory involvement activities of the project (e.g., organizing discussions with regulators concerning site response objectives and completion requirements, obtaining regulator comments on site documents and appropriately addressing them, and obtaining written documentation, as necessary, for all of the sites identified in the PWS). Generally, the USACE NAN PM, or other USACE designee, shall attend and represent the USACE at all meetings with the regulators. With the approval of the USACE NAN PM, Stone may also informally discuss issues with regulators and provide an after-action report to the USACE NAN PM. Stone will not contact regulators without prior approval of the CENAE PM. The USACE will be the signature authority for all regulatory agreements and documentation.

7.5. Electronic Chemistry Data Deliverables

Stone's laboratory will report data using the Staged Electronic Data Deliverables (SEDD) format. At a minimum, the laboratory will produce a SEDD Stage 2a deliverable, a pdf document file of the laboratory's final data report, and a hard copy of the laboratory's final data report. The laboratory will make available any supplemental information (e.g. chromatograms, instrument calibrations) upon request.

Once the SEDD electronic data files have been received from the laboratory, Stone will electronically review the files to check project data quality requirements using the USACE Automated Data Review (ADR) software. USACE will provide the ADR Software to Stone. Stone will develop a comprehensive ADR Project Library file for all of the methods to be analyzed on the project. The Project Library files will accurately reflect all of the analytical quality and will be provided to the USACE for approval prior to field sampling. The Project Library File will be provided to the laboratory for use in screening the SEDD submittals.

The laboratory will be required to check the integrity of the SEDD using the SEDD checker tool and check the SEDD file against the ADR Project Library using the Contract Compliance Screening (CCS) tool contained in ADR (Laboratory Version). SEDD non-conformances generated by ADR for which the laboratory is responsible shall be corrected at the laboratory and a new SEDD file will be resubmitted and reviewed again. The laboratory will address, through a brief explanation in a Non-Conformance Log, any non-conformance that is not within their control. The final SEDD extensible markup language (XML) file and final laboratory non-conformance report will be provided to the USACE's chemist for review.

Stone will perform a data review on the SEDD XML file through the SEDD Parser converting it to ADR files. Watermark will then review the ADR files utilizing the ADR software to check for compliance using the same ADR Project Library used by the laboratory. Stone will import the ADR reviewed files into Watermark's copy of the USACE Environmental Data Management System (EDMS). Watermark will provide USACE:

- The reviewed data from ADR in Standard ADR format (A1 through A5) as .txt files; and
- The USACE EDMS generated database file(s) in *.mdb format incorporating current chemistry data and corresponding field data via the USACE ftp web site, or some other mutually agreed upon means.

Stone will re-submit the database file to the USACE after any subsequent updates. All postings to the USACE ftp web site shall be accompanied by notification to the ETL.

All electronic data submitted by the laboratory will be error-free, and in complete agreement with the hardcopy data. Data files will be delivered both by e-mail and on CD-ROM accompanying the hardcopy data reports. The disk will be submitted with a transmittal letter from the laboratory that certifies that the file is in agreement with hardcopy data reports and has been found to be free of errors using the latest version of the ADR evaluation software provided to the laboratory. The laboratory will correct any errors identified by Watermark or USACE.

8. PROJECT SCHEDULE

8.1. Project Schedule

The proposed schedule for the pre-design investigation and treatability study fieldwork and deliverables is based on the schedule requirements from Section 8 of the *SOW*, and is summarized in Table 8.

Table 8: Proposed Pre-Design Investigation and Treatability Study Schedule

Task	Proposed Date(s)
Kickoff Meeting (Task 1)	9 January 2013
Client Draft PP / FSP / QAPP / HASP (Task 2)	18 January 2013
Final PP / FSP / QAPP / HASP (Task 2)	10 February 2013
Field Mobilization (Subtasks 4A, 4B, 5A, and 5D)	4February 2013
Field Activities (Subtasks 5B, 5C, 5E, and/or 5F)	26 February 2013
Bench Testing (Subtask 6A)	1 March 2013
Client Draft Trip Report (Tasks 4 and 5)	21 March 2013
Treatability Study Conference Call (Subtask 6B)	13 March 2013
Final Trip Report (Tasks 4 and 5)	18 May 2013
Client Draft Pre-Design Investigation Report (Task 7)	21 May 2013
Final Pre-Design Investigation Report (Task 7)	30 July 2013
SVI Investigation Work Plan (Subtask 12A)	1 March 2013
SVI Investigation Field Work – Round 1 (Subtasks 12B, 12C, and/or 12D)	1 April 2013
SVI Investigation Field Work – Round 2 (Subtasks 12B and/or 12D)	reserved
SVI Investigation Report (Subtask 12E)	3 September 2013

9. REFERENCES

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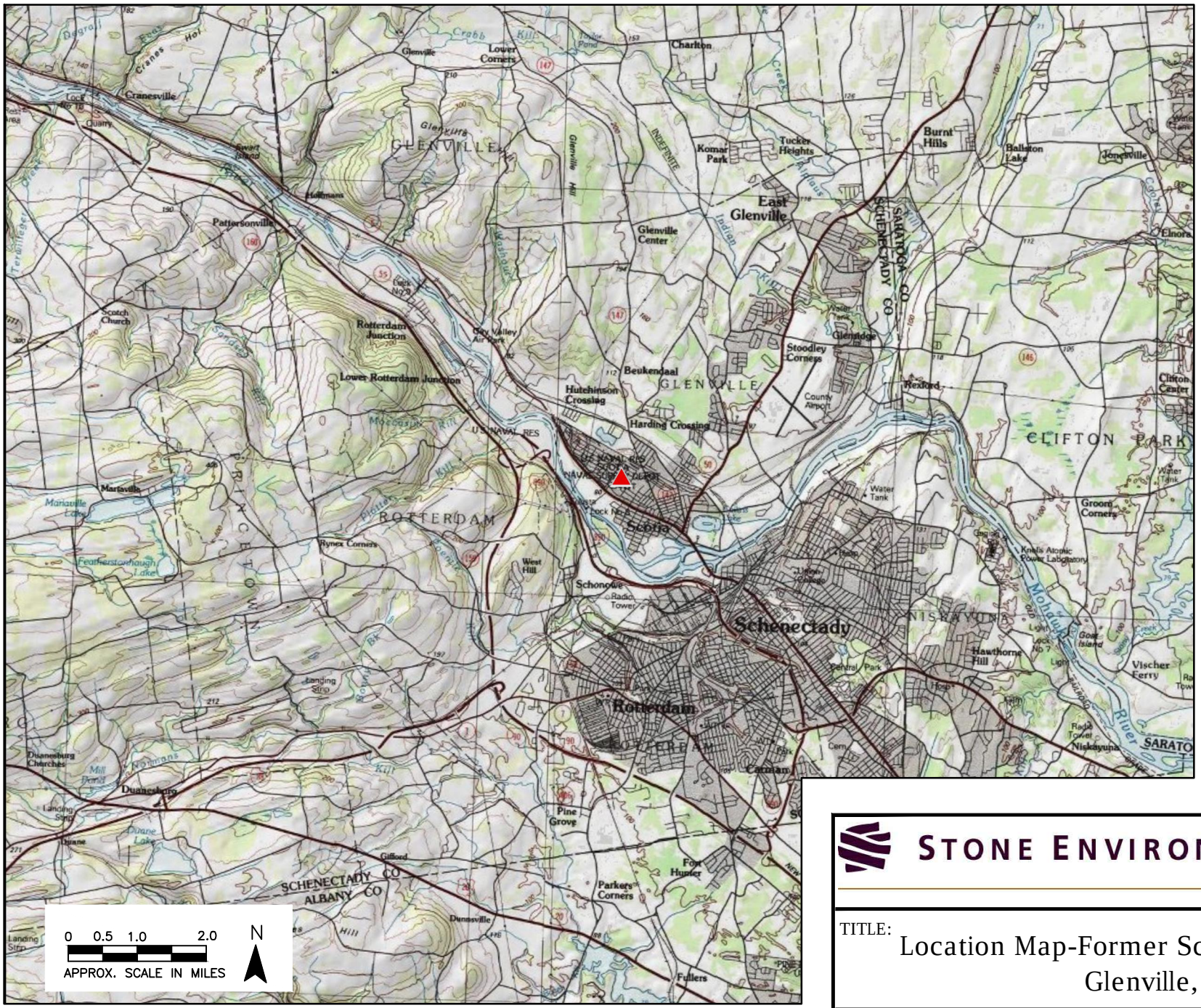
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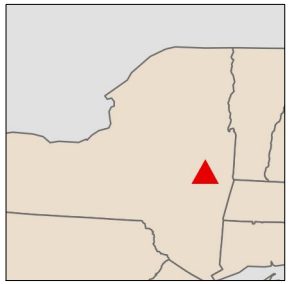
FIGURES

Figure 1 Location Map

Figure 2 Site Map

Figure 3 Remedy Map





DATUMS

Coord. System: N.Y. State Plane
Horizontal Datum: NAD 1983
Vertical Datum: NAVD 1988

Note:
Source- 2011 National Geographic Society, i-cubed

LEGEND

▲ Scotia Naval Depot

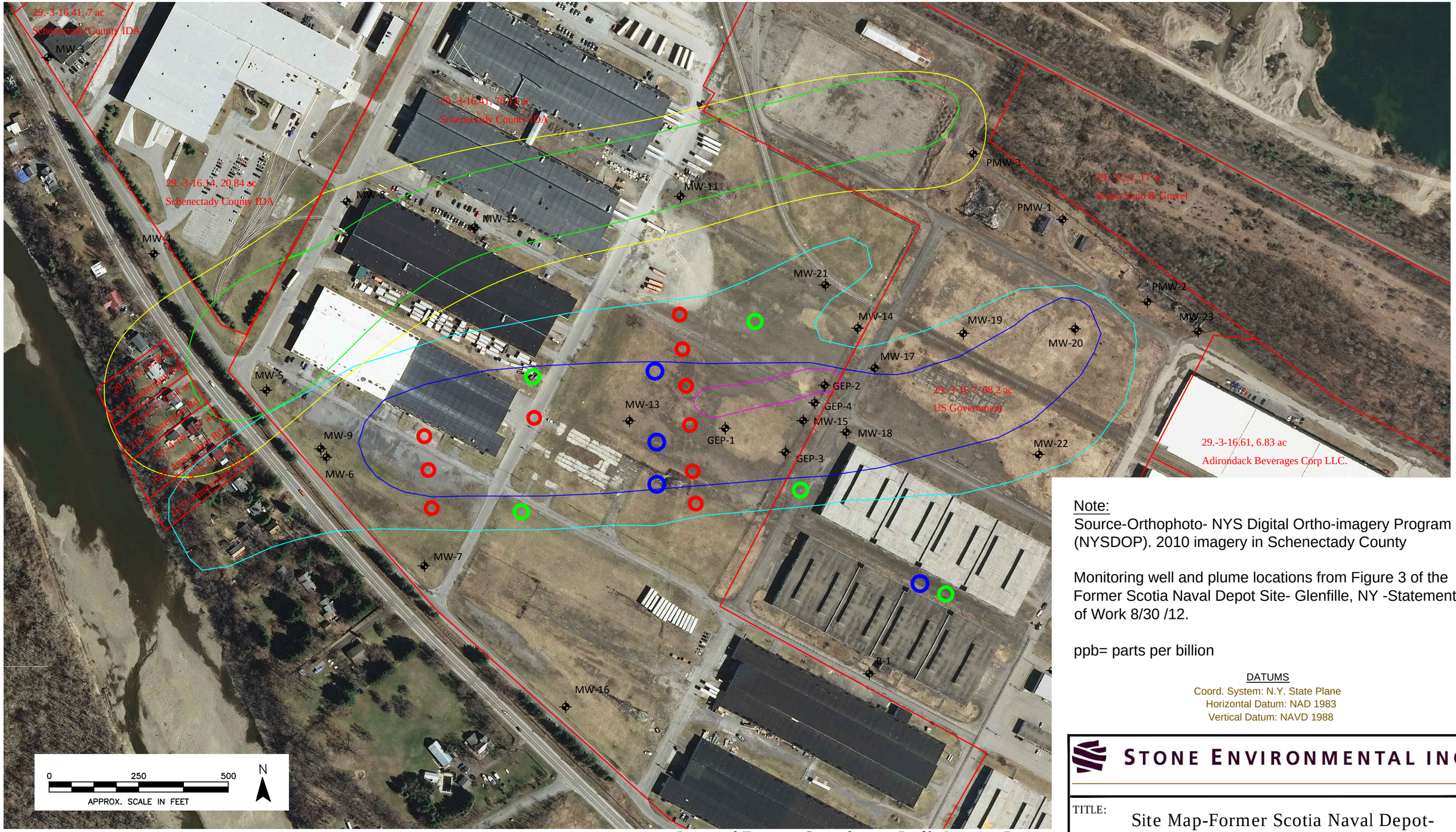
STONE ENVIRONMENTAL INC

TITLE: Location Map-Former Scotia Naval Depot-Glenville, NY

PROJECT: Former Scotia Naval Depot

FIGURE NO.: 1

DATE: 1/4/13 DRAWN BY: DJL CHECKED BY: JS



LEGEND

- Monitoring Well
- Parcel Boundaries with ID#

June 2005 Carbon Tetrachloride Plume

- 1 ppb
- 5 ppb

June 2005 Trichloroethylene Groundwater Plume

- 10 ppb
- 100 ppb
- 500 ppb

- Proposed Transect Groundwater Profile Location Boring
- Proposed Non-Transect Groundwater Profile Location (subject to change based on the results of the transect groundwater profile results)
- Proposed Monitoring Well Location (subject to change based on the results of the groundwater profile results)

Note:

Source-Orthophoto- NYS Digital Ortho-imagery Program (NYSDOP). 2010 imagery in Schenectady County

Monitoring well and plume locations from Figure 3 of the Former Scotia Naval Depot Site- Glenfille, NY -Statement of Work 8/30 /12.

ppb= parts per billion

DATUMS

Coord. System: N.Y. State Plane
Horizontal Datum: NAD 1983
Vertical Datum: NAVD 1988

STONE ENVIRONMENTAL INC

TITLE: Site Map-Former Scotia Naval Depot-Glenville, NY

PROJECT: Former Scotia Naval Depot

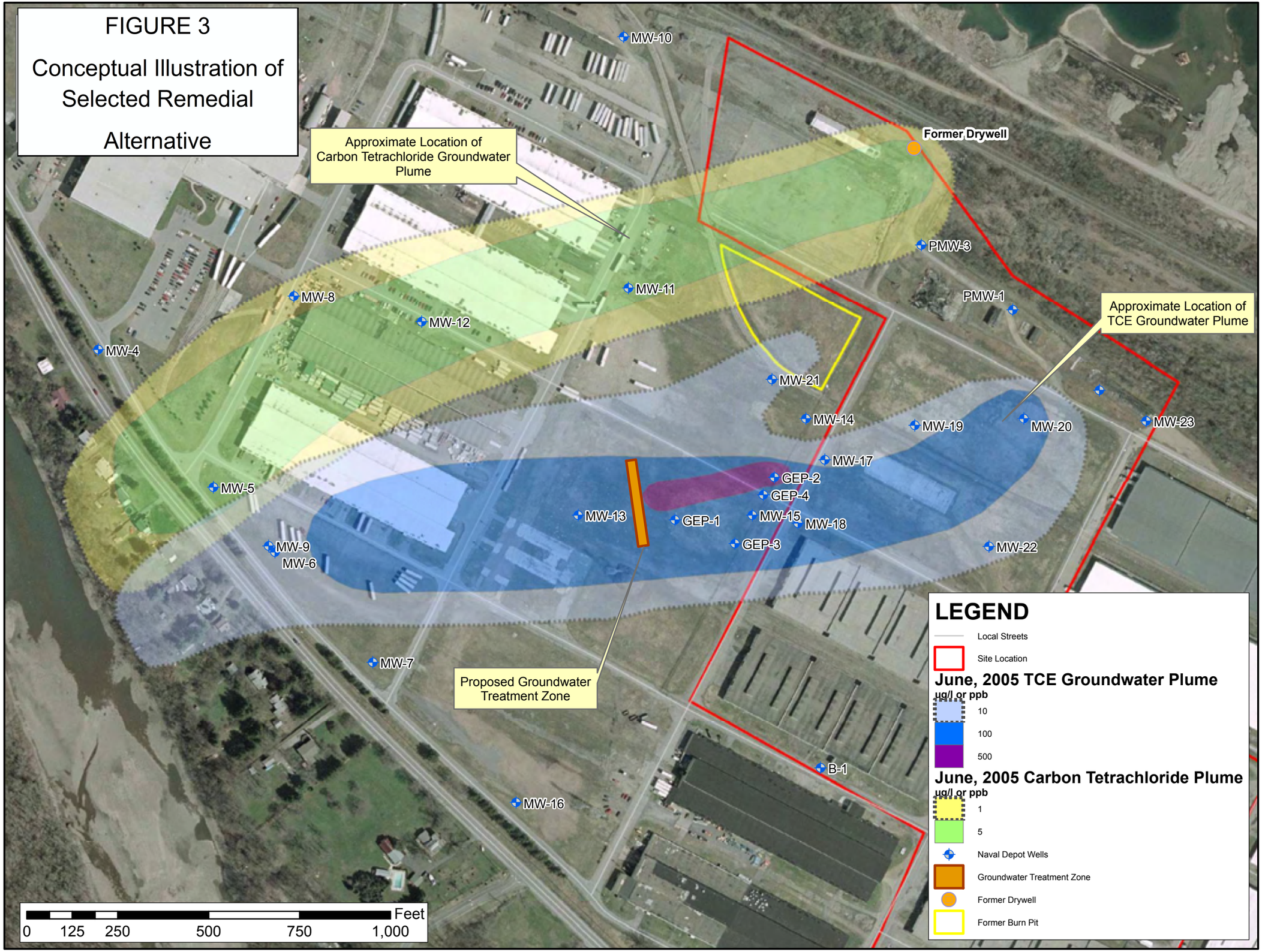
FIGURE NO.: 2

DATE: 1/14/13 DRAWN BY: DJL CHECKED BY: JS

D:\Proj-10\NX\2305-4 USACE NAE FUDS 39 Contract\07_ScotiaNavyReport\Data\autocad

FIGURE 3

Conceptual Illustration of Selected Remedial Alternative



New York State
Department of Environmental Conservation

Division of Environmental Remediation

Map Details

Created in ArcGIS 9.2

Created by J. Pelton

Date of Last Revision: 1/22/2010

UNAUTHORIZED DUPLICATION
IS A VIOLATION OF APPLICABLE LAWS

Defense National Stockpile Center Scotia Depot Site
Site # 4-47-023

Schenectady County
Town of Glenville

DEC Contacts:
J. Pelton

DOH Contact:
M. Schuck

Spring 2004
Aerial Photography

N
W E
S

North American Datum 1983
UTM Zone 18N

APPENDICES

APPENDIX A: FIELD SAMPLING PLAN / QUALITY ASSURANCE PROJECT PLAN

FIELD SAMPLING PLAN

FINAL

DEFENSE NATIONAL STOCKPILE CENTER
SCOTIA DEPOT SITE
GLENVILLE, NEW YORK

CERCLIS Number: NYN000204534
P2 Project Number: 397651

Contract Number: W912WJ-11-D-0001
Stone Project ID: 102305-R-C
April 2013

Prepared for:

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COMMITMENT TO IMPLEMENT THE ABOVE SAMPLING AND ANALYSIS PLAN		
Joseph Schmidl, PG, CWS		
Contractor's Project/Task Manager (print)	Signature	Date
Kim Watson		
Contractor's QC Manager (print)	Signature	Date
Other as Appropriate/Affiliation* (print)	Signature	Date
Other as Appropriate/Affiliation* (print)	Signature	Date
Other as Appropriate/Affiliation* (print)	Signature	Date

* Commitment signature is required for any ancillary sampling, analytical, or data assessment support provided by a contactor or subcontractor. For example, the Contractor's laboratory QC manager or director should sign the title page if analytical services are provided.

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LIST OF ACRONYMS

AHA	Activity Hazard Analyses
ARARs	Applicable or relevant and appropriate requirements
BTOC	below the top of the well casing
CERCLA	Comprehensive Environmental Response, Compensation and Liability Act
cm	centimeters
CoC	chain of custody
COC	contaminants of concern
CSM	conceptual site model
CSS	Contract Compliance Screening
CVOCs	chlorinated volatile organic compounds
DCE	dichloroethene
DLA	Defense Logistics Agency
DNAPL	dense non-aqueous phase liquid
DNSC	Defense National Stockpile Center
DO	dissolved oxygen
DoD	Department of Defense
DPT	direct-push technology
DQOs	data quality objectives
DNSC	Defense National Stockpile Center
EDD	electronic data deliverable
EDMS	Environmental Data Management System
eV	electron Volt
fbgs	feet below ground surface
FEP	fluorinated ethyl propylene
FID	flame ionization detector
FOL	field operations leader
FS	Feasibility Study
FSP	Field Sampling Plan
ft	feet/foot
GC/MS	Gas Chromatography/Mass Spectrometry
GPR	ground-penetrating radar
GSA	US General Services Agency
HDPE	high-density polyethylene
HHRA	Human Health Risk Assessment
ID	identification
IDW	investigation-derived waste
LCS	Laboratory Control Samples
MCL	maximum contaminant level
MDL	Method Detection Limit
mg/L	milligrams per liter
mg/Kg	milligrams per kilogram
MHz	megaHertz

mL	milliliter
MS/MSD	matrix spikes/matrix spike duplicates
mV	millivolt
NAPL	non-aqueous phase liquid
NAVD	North American Vertical Datum of 1988
NCR	non-conformance report
NELAP	National Environmental Laboratory Accreditation Program
NTU	nephelometric turbidity units
NYSDEC	New York Department of Environmental Conservation
NYSDOH	New York State Department of Health
ORP	oxygen-reduction potential
PBC	performance-based contract
PCE	tetrachloroethene
PCOCs	potential contaminants of concern
pH	log of hydrogen ion concentration
PID	photoionization detector
PPE	personal protection equipment
ppm v/v	parts per million by volume
PRB	permeable reactive barrier
PRG	Preliminary Remediation Goal
QAPP	Quality Assurance Project Plan
QA/QC	Quality Assurance/Quality Control
QAO	quality assurance officer
RAO	Remedial Action Objective
RBC	Risk-Based Concentration
RL	reporting limit
RSL	regional screening level
SEDD	staged electronic data deliverable
ORP	oxygen reduction potential
SIM	Selective Ion Monitoring
SOP	Standard Operating Procedure
SOW	Scope of Work
SPME	Solid Phase Microextraction
sq. ft	square feet
HASP	Health and Safety Plan
Stone	Stone Environmental, Inc.
TAL	Target Analyte List
TBD	to be determined
TCA	trichloroethane
TCE	trichloroethene
TCL	Target Compound List
TIC	tentatively identified compound
µg/Kg	micrograms per Kilogram
USACE	United States Army Corps of Engineers

USCS	Unified Soil Classification System
USEPA	US Environmental Protection Agency
VOA	volatile organic analyte
VOC	volatile organic compound
Project Plan	Pre-Design Investigation Project Plan
XML	extensible markup language
ZVI	zero-valent iron

1. INTRODUCTION

This Field Sampling Plan (FSP) has been prepared by Stone Environmental, Inc. (Stone) as a component of the *Pre-Design Investigation Project Plan* (Project Plan) to support planned pre-design activities at and near the former Defense National Stockpile Center Scotia Depot Site (the "site") in Glenville, New York (Figure 1). Stone Environmental will complete this work under a performance-based contract with the United States Army Corps of Engineers (USACE), New York District (NAN) and New England District (NAE), which will be completed under an Interagency Agreement as part of the New York State Department Environmental Conservation (NYSDEC) State Superfund Program. The ultimate goal of the USACE for this Task Order is to collect sufficient data to support the design and cost estimate for the remedy prescribed in the Record of Decision (ROD) for the project.

1.1. Project Organization

The project is being performed for USACE, New England District. USACE will provide execution and technical oversight as well as administrative support and review. The project is being performed under the regulatory oversight of the NYSDEC. Other stakeholders in the project include the United States Environmental Protection Agency (US EPA), the New York State Department of Health (NYSDOH), the U.S. General Services Administration (GSA), the Defense Logistics Agency/Defense National Stockpile Center (DLA/DNSC) and the Galeski Corporation. Stone will perform the work with the assistance of subcontractors for remedy design (Watermark Environmental, Inc.), fixed laboratory analysis (TestAmerica, Alpha Analytical, SiREM), subsurface utility location (New York Leak Detection, Inc.), and surveying (C.T. Male, Inc.). The details of the project organization structure are provided in Section 2 of the Project Plan.

1.2. Format of Sampling and Analysis Plan

Per Task 4 of the *Scope of Work (SOW)*, issued by USACE on October 4, 2012 and subsequently modified on October 30, November 30, and December 7and 13, 2012, this FSP has been prepared in accordance with the guidelines provided in *Requirements for the Preparation of Sampling and Analysis Plans*(EM 200-1-3), dated February 1, 2001, as well as Table 2-4 and Appendix B of US EPA's *Guidance for Conducting Remedial Investigations and Feasibility Studies Under CERCLA* (EPA/540/G-89/004), dated October 1988. The FSP also contains a Quality Assurance Project Plan (QAPP) in Appendix A.

1.2.1. Field Sampling Plan

The FSP has been prepared to include the elements described in Appendix B of US EPA's *Guidance for Conducting Remedial Investigations and Feasibility Studies Under CERCLA* (EPA/540/G-89/004), dated October 1988. The Section 2.0 of the FSP is divided into subsections for each specific method/procedure, wherein each of the FSP elements is described in detail.

1.2.1.1. Site Background

Please refer to Section 3 of the Project Plan for site background information. This summary includes a description of the Site and surrounding areas and a discussion of known and suspected contaminant sources, probable transport pathways, and other information about the project,. The analysis also includes descriptions of specific data gaps and ways in which sampling is designed to fill those gaps, found in Project Plan Section

3, and is reiterated in the initial subsection for each field sampling task, in an effort to help orient the sampling team in the field.

1.2.1.2. Sampling Objectives

The specific objectives of each sampling method/procedure that describe the intended uses of data are clearly and succinctly stated in each subsection of the FSP.

1.2.1.3. Sample Location and Frequency

This subsection of each sampling method/procedure described in the FSP identifies each sample matrix to be collected and the constituents to be analyzed. The number of samples to be collected is indicated in each subsection. The appropriate number of replicates and blanks is clearly identified in Table 4 in Subsection 2.9. A figure is referenced to show the locations of existing or proposed sample locations.

1.2.1.4. Sampling Equipment and Procedures

For each sampling method/procedure, a step-by-step description, cross-referenced to the appropriate Stone Standard Operating Procedure (SOP), has been provided, in order to enable the field team to gather data that will meet the data quality objectives (DQOs). A list of the equipment to be used and its material composition, along with decontamination procedures, is also included.

1.2.1.5. Sample Designation

A master sample numbering system has been established for this project, and is described in Subsection 2.8. The sample designation includes the sample or well number, the sampling round, the sample matrix (e.g., surface soil, groundwater, soil boring), and an identifier for the project.

1.2.1.6. Sample Handling and Analysis

The discussion of sample handling and analysis for the pre-design investigation tasks is included in Subsection 2.9. This subsection include a table that identifies the number and type of samples to be collected, Quality Assurance / Quality Control (QA/QC) sample requirements, sample preservation methods, types of sample containers, shipping requirements, and holding times. This subsection also cross-references applicable portions of the QAPP.

1.2.2. Quality Assurance Project Plan

Per Task 2B of the *SOW*, the QAPP has been prepared in accordance with *Uniform Federal Policy for Quality Assurance Project Plans* (DTIC ADA 427785), dated March 2005. Field sampling Standard Operating Procedures (SOPs) are provided in Appendix B and Laboratory SOPs are provided in Appendix C of this document.

1.2.3. Health and Safety

Stone has produced and implemented a written safety and health program meeting the requirements of all applicable federal, state, and local laws and regulations, and approved by USACE's contracting officer. Stone will ensure that its subcontractors, suppliers, and support personnel follow all safety and health provisions established in the approved Health and Safety Plan (HASP) and Activity and Hazard Analyses (AHA), found in Appendix B of the Project Plan. USACE reserves the right to stop work under this contract for any health

and safety violations at no additional cost to USACE. Once USACE verifies that corrective action has been implemented, Stone will be able to continue performance under the contract. The HASP contains the following elements: project description and contaminant characterization, safety and health hazard(s) assessment and risk analysis, organization and responsibilities of safety and health staff, site-specific training and medical surveillance parameters, personal protective equipment (PPE) and decontamination facilities with documented procedures to be used for same, monitoring and sampling requirements, safety and health work precautions and procedures, site control measures, onsite first aid and emergency equipment, emergency response plans and contingency procedures (onsite and offsite), logs, reports, and record keeping.

1.2.4. Pre-Design Investigation Field Task Summary

Eight pre-design investigation field tasks were developed to achieve the objectives of the pre-design investigation identified in Subsection 4.1 of the Project Plan. Table 1 summarizes the eight pre-design investigation tasks, and provides a breakdown of the sampling methods / procedures required to accomplish each task.

Table 1: Summary of Pre-Design Investigation Field Tasks, Data Quality Objectives, and Sampling Methods

Pre-Design Investigation Field Task	Data Quality Objective(s)	Sampling Methods/Procedures Required
Collection and analysis of groundwater samples from the Site and vicinity	Obtain current chlorinated volatile organic compound (CVOC) concentrations in groundwater, for use in evaluating potential impact to nearby receptors.	Low-flow groundwater sampling, water level measurement, sample location, sample analysis, investigation-derived waste (IDW) characterization and disposal.
Synoptic measurement of groundwater elevations	Obtain synoptic groundwater elevation data.	Water level measurement, sample location.
Vertical groundwater profiling of the dissolved groundwater plume	Determine vertical and horizontal extent of the dissolved plume. Determine current groundwater contaminant concentrations.	Utility clearance, vertical groundwater profiling, sample location, sample analysis, IDW characterization and disposal.
Collection and analysis of geotechnical soil samples	Provide aquifer geotechnical data.	Utility clearance, soil boring / soil sampling, sample location, sample analysis, IDW characterization and disposal.
Installation and development of overburden groundwater monitoring wells	Obtain current CVOC concentrations in groundwater from locations currently lacking groundwater monitoring wells.	Utility clearance, soil boring / monitoring well installation, monitoring well development, low-flow groundwater sampling, sample location, sample analysis, IDW characterization and disposal.
Hydraulic conductivity testing of monitoring wells	Obtain aquifer hydraulic data to support remedy design.	Hydraulic conductivity testing.
Treatability testing (bench-scale column tests) of soil and groundwater	Obtain remedy design parameters.	Utility clearance, soil boring / soil sampling, low-flow groundwater sampling, sample location, sample analysis, IDW characterization and disposal.
Collection and analysis of soil vapor / indoor air samples	Obtain current soil vapor and indoor air CVOC concentrations from nearby onsite receptors.	Sub-slab soil vapor sampling, indoor air sampling sample location, sample analysis.

The sampling methods/procedures required to perform the pre-design investigation field tasks are identified in the third column of Table 1, and are listed below in Table 2, along with a reference to their subsection in the FSP. Please note that pre-design investigation field tasks for sub-slab soil vapor sampling and indoor air sampling are not included in this FSP, and will be included in a separate FSP produced under Task 12, should Task 12 be executed.

Table 2: Summary of Pre-Design Investigation Field Task Sampling Methods/Procedures and Field Sampling Plan Location

Pre-Design Investigation Field Task Methods/Procedures	Field Sampling Plan Location
Utility Clearance	Subsection 2.1
Soil Boring and Subsurface Soil Sampling	Subsection 2.2
Monitoring Well Installation and Development	Subsection 2.3
Water Level Measurement	Subsection 2.4
Low-Flow Monitoring Well Sampling	Subsection 2.5
Vertical Groundwater Profiling	Subsection 2.6
Hydraulic Conductivity Testing	Subsection 2.7
Sample Identification	Subsection 2.8
Sample Handling and Analysis	Subsection 2.9
Sample Location Survey	Subsection 2.10
Groundwater Bench Testing	Subsection 2.11
Management of Investigation-Derived Waste	Subsection 2.12
Maintenance and Decontamination of Field Equipment	Subsection 2.13
Soil Vapor /Indoor Air Sampling	Subsection 2.14

2. FIELD SAMPLING TASKS

2.1. Utility Clearance

2.1.1. Site Background

Stone will advance an estimated 19 soil borings at the Site under Task 5 for the following purposes:

- Stone will advance up to 10 soil borings to support vertical groundwater profiling along transects under Subtask 5A;
- Stone may advance up to 5 soil borings to support vertical groundwater profiling not along transects under Subtask 5C (Optional); and,
- Stone may advance up to 4 soil borings to support collection of subsurface soil samples under subtask 5B (Optional) and the installation of overburden groundwater monitoring wells under Subtask 5E (Optional).

A component of these activities will require clearing potential soil boring locations for underground utilities.

2.1.2. Sampling Objectives

The objective of this procedure is to assure that soil borings advanced for vertical profiling and soil boring activities are conducted in areas free of underground utilities.

The successful achievement of this objective will result in no damage to subsurface utilities or injury to drilling equipment or personnel during subsurface investigations at the Site. The data analyses from this sampling method/procedure will be generated as soon as possible after data collection so that the information can be used with the re-processed geophysical data, aerial photo interpretation, bedrock outcrop data and groundwater profiling data to select the locations for new monitoring wells.

2.1.3. Sample Location and Frequency

The following areas will be subject to utility clearance as part of the SOW investigation:

- A 30-ft radius surrounding the locations of 15 proposed vertical groundwater profiling locations, depicted in Figure 3; and,
- A 30-ft radius surrounding the locations of four soil borings for proposed overburden groundwater monitoring wells, depicted in Figure 3.

2.1.4. Sampling Equipment and Procedures

Geophysical investigations will be performed by Stone subcontractor New York Leak Detection, Inc., using electromagnetic (EM) induction, ground penetrating radar (GPR) and precision utility location. Detail regarding surface geophysical techniques and procedures are provided in the text below.

2.1.4.1. Sampling Equipment

- Profiler EMP-400 electromagnetic induction sensor.

- Sensors and Software GPR system, equipped with a 250 megaHertz (MHz) antenna (for 0- to 30-ft depths) and 500 MHz antenna (for 0- to 6-ft depths).
- Radiodetection RD4000 series precision electromagnetic pipe and cable locator.

2.1.4.2. Sampling Procedure

1. Available as-built drawings and subsurface utility information from the property owners (e.g., Galesi Corporation and the DLA/DNSC) will be reviewed by Stone and the geophysics subcontractor prior to the location survey.
2. The survey areas will be cleared of moveable obstructions and brush prior to the survey.
3. A local 5-ft grid will be established, centered on the locations of the proposed soil borings.
4. The corners of the survey grid will be surveyed using GPS and noted on the Clearance Summary Sheets.
5. EM data and GPR data will be acquired along survey lines spaced no more than 5-feet apart in the accessible portions of the survey area.
6. The PUL equipment will used in passive mode to sweep the area of interest for utilities carrying live electrical current. If possible, connections are made to nearby utilities to determine if they enter the area of interest.
7. The data will be processed in the field and the interpretation will be marked on the ground at the Site and marked on the Clearance Summary Sheets for transmittal to the client at the end of each work day.

2.2. Soil Boring and Subsurface Soil Sampling

2.2.1. Site Background

2.2.1.1. Source Area Investigation

Stone will advance an estimated 19 soil borings at the Site under Task 5 for the following purposes:

- Stone will advance up to 10 soil borings to support vertical groundwater profiling along transects under Subtask 5A;
- Stone may advance up to 5 soil borings to support vertical groundwater profiling not along transects under Subtask 5C (Optional);
- Stone may advance additional soil borings to support vertical groundwater profiling under Task 8 (Optional);
- Stone may advance up to 4 soil borings to support collection of subsurface soil samples under subtask 5B (Optional) and the installation of overburden groundwater monitoring wells under Subtask 5E (Optional); and,
- Stone may advance additional soil borings to support the installation of overburden groundwater monitoring wells under Task 9 (Optional).

2.2.2. Sampling Objectives

The objective of this procedure is to advance soil borings for vertical profiling, monitoring well installation, and subsurface soil sampling activities in a manner that representative samples of soil can be obtained.

The soil borings advanced under these tasks should meet industry standards (e.g., are vertical, minimize heave) and facilitate the collection of representative subsurface soil samples at the Site. Soil characterization data from this sampling method/procedure will be used to characterize subsurface materials for the design of new monitoring wells, In addition, geotechnical analytical results of subsurface soil samples will be used to provide design criteria that will be used to determine the specifications and the cost estimate for the implementation of the zero-valent iron (ZVI) permeable reactive barrier (PRB).

2.2.3. Sample Location and Frequency

The locations of 10 proposed vertical groundwater profiling locations called for under Subtask 5A, five proposed vertical groundwater profiling locations called for under Subtask 5C (Optional), and the locations of the four soil borings for proposed overburden groundwater monitoring wells under Subtask 5E (Optional) and subsurface soil samples under Subtask 5B (Optional) are depicted on Figure 3.

Under Subtask 5B, a total of five (5) subsurface soil samples will be collected from five (5) ft intervals from depths within the anticipated vertical and horizontal extents of the ZVI PRB, based on the extent and distribution of the groundwater TCE plume determined using vertical groundwater profiling under Subtasks 5A and 5C. It is likely that samples will be collected from more than one interval of certain soil borings. The samples will be submitted for geotechnical analyses, including:

- Laboratory sieve analysis by ASTM Method D422;
- Bulk density analysis by ASTM Method D2937; and,
- Effective porosity analysis by ASTM Method D7263.

2.2.4. Sampling Equipment and Procedures

2.2.4.1. Soil Borings

The proposed drilling method is slim bore mud-rotary to advance casing, performed using the mud rotary rig. Soil borings will be advanced in accordance with SOP SEI-6.1.6.

For Subtasks 5A and 5C (Optional) and Task 8 (Optional), the proposed drilling will be performed using a combination of ODEX, direct push, and slim bore mud-rotary methods. At each profiling location, 5- or 6-inch casing will be advanced to 63 ft bgs at each location using ODEX methodology. The groundwater profiling tool (WaterlooAPS TM) will then be driven using a Series 7800 Geoprobe rig using direct-push technology.

Upon groundwater profiling tool (Waterloo$^{APS\ TM}$) refusal, the groundwater profiler will be withdrawn from the hole and 3-inch casing will be advanced to the refusal depth using the mud rotary technique. The groundwater profiler will then be reinserted into the casing and the driving and sampling process continued as before. In order to accomplish this, the mud rotary rig will be swapped with the Geoprobe rig, in order to maintain production at both soil boring locations. Details regarding the vertical groundwater profiling task are included in Subsection 2.6.

For Subtask 5E (Optional) and Task 9 (Optional), soil borings for the installation of monitoring wells, the proposed drilling method is mud-rotary to advance 6-inch casing, performed using the mud rotary rig. As part

of Subtask 5B (Optional), subsurface soil samples will be obtained during the soil boring process using a piston-type soil coring sampler. See Subsection 2.2.4.2 for a description of subsurface soil sampling equipment and procedures.

Under the provisions of NYSDEC's guidance, Section 3.3 (e) 1. i., drill cuttings and other soil generated onsite during the installation of soil borings are presumed to be contaminated until proven otherwise. Such cuttings and spoils must be stored on protective sheeting and covered with protective sheeting if cuttings remain on ground at the end of the day. Cuttings will be containerized and characterized, and disposed of in accordance with NYSDEC guidance, as described in Subsection 2.12.

2.2.4.2. Subsurface Soil Sampling

2.2.4.2.1. Sampling Equipment

- Geoprobe® 7800-series unit or rotary drilling rig
- DT22 dual-tube sampling tool (2¼-inch diameter) or 2-inch split spoon
- Photoionization Detector (PID) with an 11.7 electron Volt (eV) lamp
- Appropriate soil sample containers (e.g., ziplock plastic bags, wide mouth glass or high-density polyethylene (HDPE) jars).
- Field notebook, sample collection form, or other acceptable medium for recording field notes. Ballpoint, indelible pens.
- Shipping materials.
- Sample containers (see QAPP Worksheet #19 for sample container requirements).
- Sample Labels.
- Chain of Custody forms.
- Measuring tape.
- As necessary or as described in the HASP/AHA appropriate personal protection equipment (PPE) such as gloves.
- Rubber mallet and/or other appropriate hand tools.
- Plastic sheeting for laying out soil samples for characterization.

2.2.4.2.2. Subsurface Soil Sampling Procedure

Soil samples will be collected utilizing direct-push technology (DPT) per Stone SOP SEI-6.1.6.

1. Pre-clear all drilling locations by calling DigSafe (888-DIG-SAFE) for subsurface utility clearance at least 72 hours prior to drilling activities. In addition, a subsurface utility location contractor will be used to clear the soil boring locations (see Subsection 2.1 for details).
2. The DT22 dual-tube sampling tool or split spoon will be advanced though each 5-ft or 2-ft interval and retrieved.

3. An experienced geologist or other qualified geoscience professional will log the soils as per Stone SOP SEI-6.46.0, using the Unified Soil Classification System (USCS), noting the following characteristics:
    - Particle size distribution
    - Gradation
    - Plasticity
    - Dry strength
    - Color
    - Odor
    - Soil structure
    - Moisture content
    - Consistency
    - Cementation
4. Screen soil samples for volatile organic compounds (VOCs) with a PID in accordance with Stone SOP SEI-5.29.0. The PID will be calibrated daily to 100 ppm v/v isobutylene span gas and to zero using ambient air and a carbon filter.
5. The sample description and soil headspace screening results will be recorded in a field logbook.
6. Following sample description and field screening, analytical samples will be collected, in accordance with Stone SOP SEI-6.45.0. The sample will be homogenized, and sample aliquots will be placed into appropriate sample containers.
7. Excess sample material will be collected into appropriate storage containers, pending IDW determination (see Subsection 2.12).

2.3. Monitoring Well Installation and Development

2.3.1. Site Background

Subtask 5E (Optional) and Task 9 (Optional) calls for Stone, based in part upon the results of the groundwater profiling investigation, to advance and complete up to four (4) soil borings as monitoring wells to provide additional long-term groundwater contaminant and elevation data locations. The depths and screen intervals for the monitoring wells will be determined based on evaluation of the data from the vertical groundwater profiling (Task 5A and Task 5C (Optional), if executed). The proposed drilling method is slim bore mud-rotary to advance 6-inch casing, performed using the mud rotary rig (see Subsection 2.2 for further details).

2.3.2. Sampling Objectives

The objective of this procedure is to install additional overburden monitoring wells to provide additional locations to collect representative groundwater samples.

The wells installed under these tasks should meet industry standards, and allow the collection of representative groundwater samples. The standard used for this procedure is based on ASTM D5092.

2.3.3. Sample Location and Frequency

The locations of the four soil borings for proposed overburden groundwater monitoring wells under Subtask 5D (Optional) are depicted on Figure 3.

2.3.4. Sampling Equipment and Procedures

2.3.4.1. Sampling Equipment

- 6-inch steel casing
- Mud rotary drill rig
- Stainless steel trowel
- 2-inch diameter stainless steel well screen and riser (screen slot size to be determined based on soil data)
- 2-inch diameter, Schedule 40, threaded, polyvinyl chloride (PVC) well screen and riser (screen slot size to be determined based on soil data)
- Silica sand well filter pack (sand size to be determined based on formation parameters)
- Bentonite chips or pellets
- Bentonite powder
- Cement
- Protective casing
- YSI 600XL multi-parameter water quality meter, or equivalent.
- LaMotte Model 2020e turbidity meter, or equivalent.
- Whale pump and tubing
- Surge block and line
- 5-gallon pail
- Well Construction Log form

2.3.4.2. Sampling Procedure

2.3.4.2.1. Monitoring Well Installation

Monitoring wells will be installed according to SOP SEI-6.4.5.

1. Pre-clear all drilling locations by calling DigSafe (888-DIG-SAFE) for subsurface utility clearance at least 72 hours prior to drilling activities. In addition, a subsurface utility location contractor will be used to clear the soil boring locations (see Subsection 2.11 for details).
2. Advance drilling equipment (casing) to the desired depth, according to Stone SOP 6.1.6 (see Subsection 2.5). Replace drilling mud with potable water.

3. Using soil grain size data obtained from each soil boring, select appropriate sand filter pack and well screen sizes in accordance with ASTM 5092D.
4. Withdraw drill rods and plugs, and set screen/riser in the casing/auger.
5. Groundwater monitoring wells will be constructed of 2-inch diameter, Schedule 40, threaded, PVC well screen and riser, with 5 ft screens. No glue shall be used in the assembly of the well screen/riser.
6. Pour filter sand into the annulus between the borehole wall and well screen/riser, to a depth 1 ft above the top of the well screen. Add sand approximately 1 ft at a time, and withdraw casing/augers to match, to prevent binding the well screen/riser in the casing/auger. Account for the greater volume of the borehole after casing/auger removal by withdrawing less casing/auger length than depth of sand added, to maintain a consistent sand filter pack (e.g., do not raise bottom of casing/auger above the level of sand). The movement of sand into the sand pack should be monitored using a weighted tape measure; as the casing/auger is withdrawn, the level of sand in the casing/auger should drop.
7. Pour bentonite chips and/or pellets to the top of the filter pack, to a depth 3 ft above the top of the well screen (2-ft thick layer). If not emplaced below the water table, hydrate the chips/pellets using potable water.
8. Tremie (below the water table) / pump (above the water table) cement/bentonite grout into the annulus between the borehole wall and well riser below the water table, to a depth of 2 ft below ground surface. The grout mix should be 6 gallons of water per 94-lb bag of Type I Portland cement, with no more than 3 to 8% by dry weight bentonite powder.
9. Depending upon the well location and its immediate surroundings, install either a flush-mount waterproof well cover, or a steel protective casing with 2- to 3-ft stick up above ground surface.
10. A 1-foot by 1- foot by 8-inch thick concrete pad level with the ground surface will be placed at each well completed with a well guard. Wire mesh will be incorporated into the concrete pad for added strength. The concrete pad will be beveled at a minimum slope of 1 inch from the casing to the pad perimeter. Well completion will include a lockable well cap.
11. Mark a measurement point clearly with a permanent mark or notch on the top of the well riser (not the well protective casing) for surveying and water level measurement
12. Secure the monitoring well with a locking interior or exterior cap.
13. Prime and paint well protective casings using exterior-grade primer and paint. The well number will be painted on the outside of the guard in block characters. The locking well cap will also be labeled (inside and outside) with the well number and depth using a permanent marker such as a paint pen. Where possible, wells installed in vulnerable locations will be further protected with 2 to 3 steel and concrete bollards to protect them from traffic, snow plows, etc.

2.3.4.2.2. Well Development

1. Allow at least 24 hours following well installation before well development, to prevent mobilization of grout, etc. prior to its setting up.

2. Calibrate water quality instruments in accordance with Stone SOPs SEI-5.26.0, SEI-5.28.0, and SEI-5.30.0.
3. Develop each new well by pumping and mechanical surging, for a minimum of three well volumes. Use development pumping rates sufficient to stress the well and to reduce potential damage to the near-bore aquifer resulting from drilling. The use of acids, dispersing agents or explosives is prohibited.
4. Screen development purge water with a PID, and containerize the purge water (see Subsection 2.12).
5. Begin development until development water appears clear to the naked eye.
6. Begin monitoring well development at 5-minute intervals using a water quality meter with a flow through cell until pH, specific conductance and turbidity are stable for three successive measurements. Stability of parameters is defined as follows:
    - +/- 0.2 pH units;
    - <10% change in specific conductance; and,
    - <+/- 10% difference in turbidity between two consecutive readings or <5 NTU.
7. Record the well development procedures and results (e.g., pumping rates, water level drawdown and recovery rates, air monitoring readings, and color of discharged water) on the Well Development Form.

2.4. Water Level Measurement

2.4.1. Site Background

As part of Subtask Number 4B, a synoptic round of groundwater measurements will be made from approximately 39 monitoring wells at the Site.

2.4.2. Sampling Objectives

The objective of this procedure is to accurately measure the depth to water in overburden monitoring wells or NAPL monitoring wells.

The measurements obtained as part of these tasks should meet industry standards, namely, ASTM D4750, and be accurate to 0.01 ft.

2.4.3. Sample Location and Frequency

The water level measurements will be obtained from up to 39 groundwater monitoring wells across the Site, depicted on Figure 2, and summarized in Table 3.

Table 3: Monitoring Water Wells Proposed for Water Level Measurement and Sampling

Well ID	Screened interval (feet below ground surface)	Water Level Measurement	Groundwater Sampling
B-1	48 – 68	Yes	Yes
B-2	44.7 – 64.2	Yes	Yes

Well ID	Screened interval (feet below ground surface)	Water Level Measurement	Groundwater Sampling
B-3	47.5 – 67.5	Yes	Yes
B-6	Not available	Yes	No
GEP-1	59.6 – 74.6	Yes	Yes
GEP-2	60.6 – 75.6	Yes	Yes
GEP-3	59.6 – 74.6	Yes	Yes
GEP-4	60.15 – 75.15	Yes	Yes
MW-1	Not available	Yes	No
MW-2	66.3 – 76.3	Yes	No
MW-3	65 – 75	Yes	No
MW-4	63.8 – 73.8	Yes	No
MW-5	62.5 – 72.5	Yes	Yes
MW-6	58.5 – 68.5	Yes	Yes
MW-7	61 – 71	Yes	Yes
MW-8 (destroyed)	66 – 76	Yes	Yes
MW-9	110 – 120	Yes	Yes
MW-10	65 – 80	Yes	No
MW-11	65 – 80	Yes	Yes
MW-12	65 – 80	Yes	Yes
MW-13	65 – 80	Yes	Yes
MW-14	65 – 80	Yes	Yes
MW-15	65 – 80	Yes	Yes
MW-16	55 – 70	Yes	Yes
MW-17	60 – 75	Yes	Yes
MW-18	60 – 75	Yes	Yes
MW-19	62 – 77	Yes	Yes
MW-20	63 – 78	Yes	Yes
MW-21	57 – 72	Yes	Yes
MW-22	63 – 78	Yes	Yes
MW-23	63 – 78	Yes	Yes
PMW-1	63 – 83	Yes	No
PMW-2	64 – 84	Yes	No
PMW-3	60.5 – 80.5	Yes	Yes
PMW-7 (Offsite)	14.8 – 34.8	Yes	No
S-1D (Offsite)	Not available	Yes	No

2.4.4. Sampling Equipment and Procedures

2.4.4.1. Sampling Equipment

- Map of location(s) of well(s) to be measured.
- Keys to locked wells.
- Proper protective clothing (if necessary or as designated in the protocol or QAPP).
- Field notebook or other acceptable recording medium.
- Water level measuring device (electronic water indicator, interface probe, or other device).
- Sampling gloves.
- Decontamination materials, as necessary.
- Clean (new) polyethylene/plastic sheeting or other material on which well cap and other equipment may be placed.

2.4.4.2. Sampling Procedure

The procedures listed below are consistent with Stone SOP SEI-6.2.6.

1. Locate wells on the Site map.
2. Unlock and remove the well guard if one is present, then remove the well cap. Be careful to prevent the inside of the well cap from touching the ground in order to prevent possible contamination. Clean, poly/plastic sheeting (or other appropriate material) may be spread on the ground around the well to set the cap on.
3. Determine the established measuring point, which should be marked on the well casing with indelible ink. If no mark exists, establish a measuring point at the highest point on the rim of the well casing and mark it with indelible ink. The measuring point must be on the well casing itself and not on a separate well guard, since the well guard may shift due to ground surface settling or frost heaves. If a pump head assembly is dedicated to the well, a mark should be established on the head assembly (near the access port).
4. Prior to inserting anything down the well, be sure to don clean sampling gloves and to decontaminate the measuring device.
5. Whenever possible, use an electric water level indicator to measure the water level. The water indicator consists of a graduated electric cable with a weighted probe at the end. The probe consists of an open electric connection; when the probe come in contact with the water, the connection is closed and a current travels through the wire, causing a signal to be activated. The signal may be an audible alarm or a light that is turned on. Lower the probe into the well slowly until the signal is noted. Put the graduated wire against the measurement point, then raise and lower the probe with decreasing increments to determine precisely where the water surface is.
6. Record in the field notebook or appropriate form the water level based on the graduation of the tape; typically this is to the nearest $1/100^{th}$ of a foot. If a non-aqueous phase liquid (NAPL) is known or expected to be present, the measurements shall be made with an interface probe. The

probe is lowered slowly into the well until a liquid is encountered. The probe signifies a low conductance liquid such as NAPL with a continuous tone. The probe is then slowly lowered deeper into the well to determine the position of the NAPL/water interface. The presence of a high conductance fluid such as water is indicated by an intermittent tone.

7. Other measuring devices, such as a measuring tape or a string, may be used to make approximate water level measurements, but they should be used only if an electronic water indicator is not available.
8. Note the water level, and the NAPL level if applicable, in a field notebook or other acceptable medium as a measurement below the top of the well casing (BTOC). Also note the time of the water level measurement.
9. Decontaminate the measuring instrument after each measurement in accordance with SOP SEI-5.1.5.
10. The sequence of water level measurements shall be from least likely contaminated to most likely contaminated sampling points, or if contamination levels are unknown, from upgradient to downgradient locations, in order to reduce the potential for cross-contamination.
11. Once the water level is measured, and the measurement recorded, the well cap is then set back on the well casing, and the locking well guard re-locked (if applicable). If the lock or guard needs lubrication, be sure to take appropriate steps to prevent any possible contamination of the site, well, or well casing.

2.5. Low-Flow Groundwater Sampling

2.5.1. Site Background

As part of Subtask 4A, groundwater samples will be collected from approximately 26 monitoring wells at the Site. As part of Task 9 (Optional), up to four additional monitoring wells on the Site may be sampled.

As part of Subtask 6A, groundwater samples will be collected for geochemical analyses and for use in bench-scale treatability testing to establish the specifications of the proposed ZVI PRB.

2.5.2. Sampling Objectives

The objective of this procedure is to collect representative groundwater samples from overburden monitoring wells.

The samples collected should be representative of local groundwater and achieve the analytical objectives (groundwater Project Action Limits [PALs]) identified in QAPP Worksheet #15. The standard used for the groundwater sampling procedure is USEPA's *Low-Stress (low-flow) Purging and Sampling Procedure for the Collection of Groundwater Samples from Monitoring Wells* (EQASOP-GW-001), dated January 19, 2010. The low-flow sampling procedure is also described in Stone SOP SEI-6.34.1.

2.5.3. Sample Location and Frequency

Groundwater samples will be collected from up to 26 groundwater monitoring wells (and up to four additional monitoring wells if Task 9 (Optional) is executed) for one or more of the following reasons:

- Under Subtask 4A, obtain representative VOC results from approximately 26 monitoring wells to support trend analysis and evaluation of potential impact to nearby receptors. These samples will be analyzed in TestAmerica's Department of Defense (DoD)-, National Environmental Laboratory Accreditation Program (NELAP)-, and NYSDOH-certified fixed laboratory for Target Compound List (TCL) VOCs via USEPA Method 8260B; and,
- Under Subtask 6A, obtain current geochemical groundwater data from up to five intervals / locations within to support the evaluation of the treatability of the dissolved groundwater plume. These samples will be:
    - Total organic carbon /total dissolved carbon by EPA Method 9060;
    - Total dissolved solids / total suspended solids by methods SM 2540C and 2540D, respectively;
    - Oil & grease by EPA Method 1664A;
    - pH by EPA Method 9040B;
    - Hardness by method SM 2340C;
    - Ferrous and ferric iron by ASTM Method D3872-05 (2011)and manganous and manganic manganese (EPA Method 1638, Modified);
    - Nitrate / nitrite and Sulfate / sulfite by EPA Method 300.0;
    - Total metals, including chromium, iron, magnesium, manganese, silicon, and titanium by EPA Methods 6010B;
    - Hexavalent and trivalent chromium by EPA Method 7196A; and,
    - Carbonate by EPA Method 2320B.

The monitoring wells proposed for sampling under Subtask 4A are listed in Table 3 (see Subsection 2.4.3) and depicted on Figure 2. The sampling program for the 26 wells will be performed over an approximately 3-day period.

The monitoring wells proposed for sampling under Subtask 6A will be determined following the vertical groundwater profiling program, based on the extent and distribution of the groundwater TCE plume determined using vertical groundwater profiling under Subtasks 5A and 5C.

Information regarding the laboratory requirements for groundwater VOCs, and geochemical samples regarding detection limits, precision and accuracy are summarized in Table 4 and discussed in Subsection 2.9 and the QAPP (Appendix A).

2.5.4. Sampling Equipment and Procedures

2.5.4.1. Sampling Equipment

- Adjustable rate, submersible pump (e.g., bladder pump constructed of stainless steel or Teflon)
- Fluorinated ethyl propylene (FEP) tubing, sized appropriate to pump, ¼- or ⅜-inch inner diameter
- Water level indicator or oil/water interface probe, as appropriate
- 1-liter graduated cylinder

- Power source for pump, as appropriate
- Water quality meter for pH, ORP, DO, specific conductance, and temperature, equipped with a flow-through cell
- LaMotte turbidimeter
- 5-gallon pail
- Low-Flow Groundwater Sampling Log form
- Sample containers (see QAPP Worksheet #19 for sample container requirements).

2.5.4.2. Sampling Procedure - Groundwater

Groundwater samples will be collected according to the following procedure, per Stone SOP SEI-6-34.0.

1. Calibrate water quality instruments in accordance with Stone SOPs SEI-5.26.0, SEI-5.28.0, and SEI-5.30.0. Equipment will be calibrated prior to use each day and the calibration will be checked at the end of use on that day to document calibration shifts during use. The Standard Hydrogen Electrode conversion will be performed for final ORP measurements in accordance with the manufacturer's documentation by adding 200 milliVolt (mV).
2. Check for security damage or evidence of tampering on each well, record pertinent observations.
3. Remove well cap and immediately measure VOCs at the rim of the well with a PID or flame ionization detector (FID) instrument and record the reading in the logbook.
4. If the well casing does not have a reference point (usually a V-cut or indelible mark in the well casing), make one. Describe its location and record the date of the mark in the logbook.
5. Check newly constructed wells for the presence of NAPL before the initial sampling round using an oil/water interface probe, in accordance with SOP SEI-6.2.6.
6. Lower pump and any safety cables, tubing and/or electrical lines slowly (to minimize disturbance) into the well to the midpoint of the zone to be sampled. In most instances, the pump will be positioned in the middle of the well screen. For cases where the well screen straddles the water table, the pump should be position 2 feet above the bottom of the well.
7. Before starting pump, measure water level. If a recording pressure transducer is used, initialize starting condition. After recording the height of the water column in the well, determine the volume in gallons by the following formula:

   $$\text{Volume (gallons)} = (\text{Height of water column (ft)} \times \pi \times (\text{Radius (ft)})^2) \times 7.4805 \text{ gallons/ft}^3$$

8. Start 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 drawdown (less than 0.3 feet). If the minimal draw-down that can be achieved is greater than 0.3 feet, continue purging until indicator field parameters stabilize.
9. Discharge purge water into 5-gallon buckets, pending IDW determination (see Subsection 2.13)
10. Monitor and record water level and pumping rate every 3 to 5 minutes (or as appropriate) during purging. 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 fifteen minutes of pumping in order to help minimize purging time. During pump start-up, drawdown may exceed the 0.3 feet target and then "recover' as pump flow adjustments are made.

11. Using the appropriate volumetric container (one that is scalable relative to the volume to be purged), measure the volume of water purged at the same interval as field parameters are recorded (Section 4.3.4). If water is or believed to be contaminated, purge water must be containerized as specified in the Project Plan. Purge volume calculations should utilize stabilized drawdown value, not the initial drawdown. Do not allow the water level to fall to the intake level (if static water level is above the well screen, avoid lowering the water level into the screen). The final purge volume should be greater than the stabilized drawdown volume plus the extraction tubing volume. This may not be possible in wells with low recharge rates.
12. Wells with low recharge rates may require the use of special pumps capable of attaining very low pumping rates (bladder, peristaltic) and/or the use of special techniques. If the recharge rate of the well is lower than the pump's minimum extraction rate, then continue to purge until the drawdown is near (but not below) the top of the well screen. Once the drawdown is within six (6) inches of the top of the well screen, discontinue purging. The well should be sampled as soon as the water level has recovered sufficiently (i.e., 75%) to collect the appropriate volume needed for all anticipated samples (the intake should not be moved during this recovery period). In this case only, samples may be collected even if the well was not purged for four (4) hours and the indicated field parameters did not stabilize.
13. Monitor indicator field parameters (pH, temperature, specific conductance, ORP, and DO) during purging using a multi-parameter water quality meter equipped with a flow-through cell system. Turbidity will also be monitored, but using an aliquot of groundwater in a separate LaMotte turbidity meter. The parameters will be monitored for stabilization prior to collection of the sample. Stabilization is achieved when three consecutive readings, taken at 3- to 5-minute intervals, are within the following limits:
    - Turbidity ± 5 NTU for readings >1 nephelometric turbidity units (NTU) or <1 NTU
    - Dissolved oxygen (DO) (± 10% for values greater than 2 milligrams per liter (mg/L); 0.5 mg/L for values less than 2 mg/L)
    - Specific conductance ± 3%
    - Temperature ± 3%
    - pH (± 0.1)
    - ORP (± 10 mV)
    - Drawdown (no more than 0.3 feet)
14. The low-flow guidance suggests a maximum drawdown of 0.3 feet. The hydraulic head in the interval will be carefully monitored and a purge rate used that will minimize drawdown. If head continues to decline, the rate of decline will be calculated. Once this rate has stabilized for three consecutive readings, this criterion will have been satisfied.

15. Upon stabilization, collect water samples for laboratory analyses from a sample port located in front of the flow-through-cell (use a by-pass assembly or disconnect cell to obtain the sample).
16. VOC samples should be collected first and directly into the sample containers. Sample water should be allowed to fill sample containers gently down the inside of the container with minimal turbulence. For samples collected for VOC analysis, use pre-preserved 40-mL glass VOA vials with Teflon septa, ensuring no headspace in the closed sample container.
17. During purging and sampling, the tubing should remain filled with water so as to minimize possible changes in water chemistry upon contact with the atmosphere. If the pump tubing is not completely filled to the sampling point, use one of the following procedures to collect samples:
    a. Add a clamp, connector (Teflon or stainless steel), or valve to constrict the sampling end of tubing; or
    b. Insert small diameter Teflon tubing into water filled portion of pump tubing, allowing the end to protrude beyond the end of the pump tubing, collect samples from small diameter tubing.
18. Secure the well.
19. At a minimum, well purging times and/or volumes, water level in the well before and after pumping, calculation of one well volume, weather conditions (temperature, cloud cover, wind direction and recent precipitation events), and sample collection time will be recorded for each sampled well on the field forms provided with Stone SOP SEI-6-34.0.

2.6. Vertical Groundwater Profiling

2.6.1.1. Site Background

Stone will perform vertical groundwater profiling at the Site under Task 5 for the following purposes:

- Stone will perform up to 10 vertical groundwater profiles along transects under Subtask 5A;
- Stone may perform up to 5 vertical groundwater profiles not along transects under Subtask 5C (Optional).

2.6.2. Sampling Objectives

The objective of this procedure is to obtain vertical groundwater profiles in the vicinity of the plume, in order to optimize the location of the proposed ZVI PRB.

The vertical groundwater profiles under these tasks should meet industry standards and achieve the analytical objectives (groundwater Project Action Limits), listed in QAPP Worksheet #15. Refer to Subsection 2.9.4.2 for a description of how the fixed laboratory split samples will be used to document the achievement of the sampling objectives for the mobile laboratory screening samples.

2.6.3. Sample Location and Frequency

The locations of 10 proposed vertical groundwater profiling locations called for under Subtask 5A and five proposed vertical groundwater profiling locations called for under Subtask 5C (Optional) are depicted on Figure 3.

The groundwater samples will be screened in Stone's DoD- and NELAP-certified onsite laboratory in near-real time for a reduced list of CVOCs via EPA Method 8260C (modified) that includes all the contaminants of concern for the Site. These data will be considered screening data, as Stone's onsite laboratory lacks NYSDOH certification. For this reason, approximately 10% of the groundwater samples will be split and analyzed separately in TestAmerica's DoD-, NELAP-, and NYSDOH-certified laboratory for TCL VOCs via EPA Method 8260B, that includes all the contaminants of concern for the Site. Samples to be split for fixed laboratory confirmation analysis will represent the range of concentrations detected in the screening samples, and from various depths within the aquifer. The majority of the split samples will be collected and analyzed early in the vertical groundwater profiling program, in order to provide early assessment of data quality.

For the Task 5A vertical groundwater profiling task, an estimated 130 groundwater samples will be collected and screened, resulting in a total of 13 split samples for fixed laboratory analysis. During the first two days of vertical groundwater profiling, two profiles to approximately 170 ft bgs will be advanced near the center of the plume. Split samples will be collected from alternating intervals from each location, providing a wide range of samples to select split samples from, based on the near-real time screening results. Four samples will be submitted from the first day of sampling, to be analyzed with a 2-day turnaround time, and four samples will be submitted from the second day of sampling, to be analyzed with a 1-day turnaround time. This will provide eight of the 13 confirmation sample analytical results within 4 days of the beginning of the program.

2.6.4. Sampling Equipment and Procedures

2.6.4.1. Sampling Equipment

Profiling Equipment

- 1¾-inch Diameter Profiler Tips
- Stainless Steel (SS) Profiler Screens
- SS Allen Screws
- 5 ft × 1¾-inch Drill Rod
- 2 ft × 1¾-inch Drill Rod
- Swivel pull
- Geoprobe Pull Clamp for 1¾-inch Rod
- Drive Caps
- Slip Caps
- ⅛-inch SS Tubing
- SS Caps for SS Tubing
- Extra ⅛-inch SS Compression Fittings
- SS mesh for tips
- 1½-inch Geoprobe × 1¾-inch Rope crossover

KPRO Testing Box and Related Equipment

- String Pot
  - Com Cable
- Quick Clamp
- Compressed N_2 Tank
- Dual phase regulators
- Pressure Vessel
- Pressure Vessel quick connects
- ⅛-inch swage to quick connect
- KPRO Box
  - Com Cable
  - Power Cable
- Regulated Power Supply
- 1-Liter Graduated Cylinder (plastic)
- Field Computer

- Serial Port Card, PCMCIA
- Power Adapter
- Color Printer
- Printer Paper
- 5-gallon Pails for Purge Water/Decon
- Water Quality Sonde
  - Com Cable
  - Flow Cell
  - extra D-cell Batteries (Troll)
  - power cable (YSI)
  - Calibration Standards
  - DO membrane maintenance kit (Troll)
- Field Book/Field Forms
- Swage Fittings

Gas Drive Pump Assembly Equipment

- 2.4-ft × 1¾-inch female – female drill rod
- male – male pin
- male – female pin
- Springs
- Nylon Bushings
- Pump Bottom
- Pump Body
- Pump Top
- Reed Valves
- O-rings for Pump Body
- O-rings for Pump Bottom
- ¼-inch SS Tubing
- ¼-inch NPT male to ¼-inch compression fitting
- ¼-inch SS Ferrules

Sample Collection Equipment

- Quatro-rings for Sample Jig
- 40mL VOA Vials Vial Labels
- Silicon tubing for Peristaltic Pump
- Coolers and Ice

Decontamination Equipment

- Alconox
- Squirt Bottles
- Garden Spray Bottle (3-gallon)
- Plastic Sheeting
- Contractor Garbage Bags

Auxiliary Equipment

- 100-ft Engineers Tape
- Electrical Tape
- Ratchet Straps and Bungee Cords
- Teflon Tape
- Colored Zip Ties
- Halogen Work Lights
- Paper Towels
- Ground Fault Circuit Interrupt
- Extension Cords
- Land line for Circuit panel
- Surge Protector

Personal Protective Equipment/Safety Equipment

- Nitrile Gloves
- Cotton liner Gloves
- Leather Work Gloves
- Safety Glasses
- Hardhats
- Hearing Protection
- Tyvek™ Suits
- Emergency Eyewash
- First Aid Kit
- Compressed Air Horn
- Spill Kit
- Spare absorbent Pads
- Fire Extinguisher
- Flashlight

Hand Tools and Power Equipment

- Pipe Wrenches
- Open Ended Wrenches
- Screw Drivers, Regular and Phillips
- Impact Wrench
- Allen Wrenches, Standard and Metric
- Crescent Wrench
- Socket Set
- 9/16-inch Socket, Open Ended
- Taps for Profiler Tip Screws
- Tap Handle
- Torx Bits, T-25
- Picks, Full Set
- Pliers, Standard, Needle Nose, and Channel Lock
- Vise Grips
- Wire Strippers
- Electrical Multi-meter
- Soldering Iron
- Tin Snips
- Files
- Utility Knife
- Tubing Cutter
- Driller's Hammer, 3-lb
- Butane Lighter
- Electric Drill
- Drill Bits, Full Set
- Carbide Bit for Reaming SS Tubing
- Angle Grinder

Additional Winter Gear

- Pipe Insulation
- Heat Tape
- Propane Tanks
- Dryer Vent Hose
- Propane Heaters
- Kerosene Heaters

2.6.4.2. Sampling Procedure

Vertical Groundwater Profile samples will be collected according to the following procedure, per Stone SOP SEI-6.43.3. Due to the depth to groundwater being in excess of the suction limit Stone will utilize a stainless steel gas drive pump to purge groundwater and collect samples. Three drill rigs will be utilized to advance the vertical profiling tool. The first rig will be a truck-mounted ODEX rig; the second rig will be a truck mounted slim-bore mud-rotary rig. The third rig will be a Geoprobe® 7800-series track-mounted direct push rig.

1. The ODEX rig will set up on the first vertical groundwater profiling location and advance a 5- or 6-inch casing, using ODEX drilling techniques, to 60 feet below ground surface (ft bgs).
2. While ODEX rig advances casing at the first vertical groundwater profiling location two Stone data acquisition trucks will be set up with all necessary equipment to perform vertical groundwater profiling as proposed in Task 5A of the Project Plan. Details on equipment setup, tip and pump assembly, and calibration can be found in SEI SOP-6.43.3 sections 4.3, 4.4.2, and 4.5, respectively.
3. 1¾-inch diameter profiling rods will be threaded together in 20-foot sections at each of first two vertical groundwater profiling locations and placed on metal saw-horses in the vicinity of the borehole such that the wire-line of the mud-rotary or ODEX or mud rotary rig can easily access the rods. Enough 20-foot sections of profiling rods will be assembled to reach the bottom of the borehole when lowered through the mud-rotary casing.
4. Three pieces of 20-foot long ⅛-inch stainless steel tubing will be strung through each 20-foot section of profiling rod, capping the ends to ensure the tubing is not clogged.
5. Once the ODEX rig advances 5- or 6-inch casing to 60 ft bgs, the 20-foot sections of profiling rod, containing three pieces of ⅛-inch diameter stainless steel tubing, will be raised into a vertical position over the casing. A swivel pull and wire-line will be used to raise profiling rods. The lead section of profiling rod will have the profiling tip and gas drive pump assembly securely tightened to the profiling rod. Ensure a section of bicycle tire tube is secured around the sampling ports to prevent drilling fluids from entering the sample train.
6. Lower profiling rod into the 6-inch ODEX casing until approximately 1 foot is sticking above the top of the mud-rotary casing.
7. Secure profiling rod to the top of the ODEX casing using a clamp.
8. Unthread swivel pull from the lowered section of profiling rod and thread onto another 20-foot section of profiling rod. Use wire-line to raise this section of profiling rod into a vertical position such that there is approximately 2 feet of space between the male threads of the rods within the ODEX casing and the female threads of the rods hanging from the wire-line.
9. Connect and tighten three stainless steel tubes within hanging profile rod to three stainless steel tubes protruding from rods that have been lowered into mud-rotary casing. It does not matter which of the hanging tubes are attached to the tubes protruding from the clamped rods.
10. Lower hanging rods onto clamped rods and thread together and tighten with pipe wrenches.

11. Raise profiling rod a few inches so that the entire drill string is supported by the wire-line. Remove clamp and lower profiling rods so that approximately one foot is sticking above the top of the mud-rotary casing.
12. Repeat steps 5 through 12 until the profiling rods are resting on the bottom of the borehole.
13. Attach the harness to the stainless steel tubes sticking up out of the top of the profiler rod at the drive platform. The shortest stainless steel tube will be the nitrogen line (red harness piece), the next longest tube will be the sample line (yellow harness piece), and the longest tube will be the KPRO effluent line (blue harness piece).
14. The Geoprobe® rig will then advance the vertical groundwater profiling tool to the first sample interval according to Subsection 4.6 of SEI SOP-6.43.3. The profiling tool shall be driven at least 1 foot out of the mud-rotary casing prior to collecting a groundwater sample to prevent influences of drilling fluids.
15. Once the vertical profiling tool is positioned at the first sample interval on the first vertical groundwater profile the ODEX rig will relocate to the second vertical groundwater profiling location and repeat steps 1 and 5 through 14.
16. While the mud-rotary rig advances casing at the second vertical groundwater profiling location the Geoprobe® will move onto the first vertical groundwater profiling location and, following either a successful or unsuccessful sample attempt, will advance the vertical groundwater profiling tool according to Subsection 4.6 of SEI SOP-6.43.3. Sample intervals will be determined by changes in the index of hydraulic conductivity (I_k) and will not exceed 5 feet, unless low hydraulic conductivity strata are encountered that are thicker than 5 feet. If changes in I_k occur frequently (e.g., every 0.5 feet), Stone field staff will exercise professional judgment on where it is appropriate to collect a groundwater sample within each 5 foot interval.
17. Upon reaching a sample collection depth at either location groundwater samples will be collected utilizing a gas drive pump according to Subsections 4.7.3 and 4.7.4 of SEI SOP-6.43.3.
18. During groundwater purging monitor indicator field parameters (pH, temperature, specific conductance, ORP, and DO) using a multi-parameter water quality meter equipped with a flow-through cell system. The parameters will be monitored for stabilization prior to collection of the sample. Stabilization is achieved when three consecutive readings, taken after each nitrogen purge cycle, are within 10% of the previous value for each parameter. Indicator field parameters will be recorded on both paper and electronic forms.
19. Following successful sample collection advance the vertical groundwater profiling tool, collecting groundwater samples every five feet, adding profiling rod as necessary, according to Subsections 4.7.3 and 4.7.4 of SEI SOP-6.43.3.
20. Upon refusal of the profiling rods, the mud-rotary rig will be used to trip-out rods in 20-foot sections. The harness tubing will be disconnected from the down-hole tubing.

21. Use the swivel pull and wire-line to raise profiling rods, securely clamp rods 1 foot below the joint where the rods are to be unthreaded. Lower the clamp onto the top of the mud-rotary casing. Make sure the rods do not slip.
22. Unthread the top 20-feet of rod and use wire-line to place back on saw-horses.
23. Repeat steps 21 and 22 until all profiling rods are tripped out.
24. The mud-rotary rig will be used to advance the 3-inch casing to approximately the depth at which the profiling rods were refused or through strata which caused the profiling tool to be refused. Two mud tubs will be utilized to reduce the amount of time required to relocate between profiling locations.
25. While the mud-rotary rig advances casing the profile tooling that has just been tripped-out will be decontaminated and rebuilt. Clean water will be pumped through all stainless tubing followed by a nitrogen purge to remove water and prevent freezing issues. The profiling tip and gas drive pump will be decontaminated and rebuilt according to respective Subsections 4.91 and 4.4.2 of SEI SOP-6.43.3. Drive rods will be flushed of drilling mud using a pressure-washer. Rinsate will be captured within appropriate containers.
26. Once mud-rotary casing has been advanced to the desired depth the profiling tool will be re-inserted into the casing and lowered to the bottom of the borehole following steps 3 through 11 and sampling/tooling advancement can commence following steps 13 through 25 until the target depth is reached at each vertical groundwater profiling location. **Note:** steps 14 through 16 may vary depending on where the mud-rotary rig is required to advance 3-inch casing and lower/raise proofing rods.
27. Upon completion of a vertical groundwater profiling location the steps outlined above will be repeated for a new vertical groundwater profiling location until all locations are completed.

Vertical groundwater profiling will continue until there are two consecutive samples that are free of analytes or the till is encountered. Based on prior environmental assessment of the Site, the till unit is expected to be at approximately 170 ft below ground surface.

2.7. Hydraulic Conductivity Testing

2.7.1. Site Background

Under Subtask 5F (Optional), Stone will conduct hydraulic conductivity testing on up to four (4) monitoring wells.

2.7.2. Sampling Objectives

The objective of hydraulic conductivity testing is to determine formation hydraulic conductivity, to facilitate estimation of groundwater flow velocity across the proposed location of the PRB.

2.7.3. Sample Location and Frequency

The monitoring wells proposed for hydraulic conductivity testing under Subtask 5F will be determined following the vertical groundwater profiling program, based on the extent and distribution of the groundwater TCE plume determined using vertical groundwater profiling under Subtasks 5A and 5C.

2.7.4. Sampling Equipment and Procedures

2.7.4.1. Sampling Equipment

1. Water marker.
2. Distilled water or other decontamination equipment, as necessary.
3. Rubbish can and trash bags for storage of dedicated pumps if removal is necessary.
4. Bailer or slug bar (any cylindrical bar dense enough to sink in water) with outside diameter small enough to fit into the well and sufficient cable or line to allow complete submersion below the static level in the well. If a datalogger is used, the bailer or bar must fit in the well with the transducer cable in place. Disposable bailers should be used if slug tests are conducted at multiple sites to prevent cross contamination.

   Alternatively, a pneumatic slug may be utilized for wells which are screened fully below the water table. Pneumatic slugs offer the advantages of larger slug volumes for measurements in high-hydraulic conductivity aquifers, instantaneous slug removal, and no dripping back into the well (which can affect initial measurements).
5. Optional: An automated data logging system with a pressure transducer. A list of specific equipment required for use of the Troll SP4000 system is located in SOP 5.22.2.
6. Clock, watch, or other timepiece.
7. Field notebook with slug test forms or other acceptable recording medium.

2.7.4.2. Sampling Procedure

1. Measure the water level in the well, following standard operating procedures for water level measurement (see Subsection 2.4). This is the equilibrium water level. Note the time of the measurement.
2. Check water level against documented screened interval for the well. If the equilibrium water level is at or above the top of the well screen, then both a falling head test and a rising head test may be performed on the well. However, if the equilibrium water level is below the top of the screen, then only a rising head test will be valid.
3. If the well is equipped with a dedicated pump and associated tubing that will interfere with the transducer, cable, and bailer/slugbar, remove the pump assembly and coil it into a container lined with a new trash bag. Avoid letting the pump assembly contact the ground or any other potentially contaminated surfaces.
4. Decontaminate the pressure transducer and approximately 5 feet of cable by rinsing with deionized/distilled water and/or Alconox as appropriate.

5. Insert the transducer into the well. Gently lower it to the bottom of the well, then raise it slightly off the bottom. Note that the transducer has a response range, so that it is only accurate within a range of water depths. If the bottom of the well is outside the response range, raise the transducer until it is within range. Secure the transducer so that it will not move over the course of the test. Note the time at which the transducer is set in place.
6. Connect the transducer cable to the datalogger.
7. If a rising head test will be performed, carefully lower the bailer or slug bar into the well, making sure to raise and lower the bailer so that it completely fills with water.
8. Monitor the water level until it returns to equilibrium. Document the water level and time at which the water level reaches equilibrium.
9. Set up the datalogger according to the manufacturer's instructions.

2.7.4.2.1. Rising Head Slug Test

In a rising head slug test, the water level in the well is caused to drop suddenly, either by bailing a volume of water from the well or by pulling a slug bar from the well. If a falling head test has just been completed, a rising head test can be done immediately afterward, as long as the water level has returned to equilibrium.

1. Start the datalogger and quickly remove the bailer or slug bar from the well. Make sure that the transducer is not pulled up with the slug, and, if a bailer is used, pull it completely out of the well to make sure that water does not leak back into the well. Empty water away from well.
2. Take a water level reading and record the time. Repeat this every couple of minutes until equilibrium is reached. In very permeable soils, the water level can recover in less than a minute, but it is good to collect data for at least three minutes to make sure the test is completed. Stop the test after the water level has returned to equilibrium.
3. Take a final water level reading and record the time.

2.7.4.2.2. Falling Head Slug Test

In a falling head test, the water level in the well is caused to rise quickly, either by inserting a slug bar into the well or by pouring water into the well. If water is used, it should be distilled or deionized so that it will not interfere with any water samples that might be collected from the well. If a slug bar is used, care must be taken that it does not touch the transducer during the course of the slug test. One way to ensure that this does not happen is to tie off the slug bar line before starting the test, so that the bar cannot go far enough into the well to touch the transducer.

1. Begin the test after all equipment is in place and the water level has returned to equilibrium. First start the datalogger, then immediately drop the slug bar or pour the water into the well.
2. Take a water level reading and record the time. Repeat this every couple of minutes until equilibrium is reached. In very permeable soils, the water level can recover in less than a minute, but it is good to collect data for at least three minutes to make sure the test is completed. Stop the test after the water level has returned to equilibrium.
3. Take a final water level reading and record the time.

4. Download the test data from according to the manufacturer's instructions. Print a hard copy of the datalogger data as soon as is practical after the test is completed. Sign or initial, date, and write the study number on each page of the printout, and retain these print-outs as raw data. The total number of pages for each test should be documented on the first page of each test print-out.

2.8. Sample Identification

2.8.1. Site Background

As part of *SOW* Tasks 4 through 6, samples will be collected for laboratory analysis. Each of these activities will require identifying prior to sample handling and analysis.

2.8.2. Sampling Objectives

The objective of this procedure is to accurately assign each sample with a unique identifier.

The identifiers assigned as part of these tasks should allow for the accurate tracking of sample location, depth, and matrix.

2.8.3. Sample Location and Frequency

The sample locations for each area investigated as part of the project are depicted in Figure 2 and 3. Each sample collected as part of this investigation will be assigned a unique identifier.

2.8.4. Sampling Equipment and Procedures

2.8.5. Sampling Equipment

1. Sample labels
2. Indelible pen
3. Clear tape
4. Chain-of-Custody Form

2.8.5.1. Sampling Procedures

Each sample will be assigned a unique sample identifier shown on the COC and container labels.

The first set of characters will be the alphanumeric location identification (ID) which will identify the sample location. Samples from existing wells will use the most recent sample name for that well. All other locations will be assigned an individual consecutive number preceded by a two letter code for the type of sample:

"OB" for an overburden soil sample location.

"WP" for a Waterloo$^{APS\ TM}$ profiler location.

"MW" for a monitoring well location.

"SV" for a soil vapor sample location.

"AR" for an air sample location.

The location ID code will be followed by a dash (e.g., "OB1-").

The second set of characters will identify the depth interval from which the sample was collected. The actual depth will be used as rounded to the nearest 0.1 foot. The depth interval shall be the depth of the sample in ft bgs. For surface water samples the depth will be the depth below the water surface. If the sample interval sequence is not valid or is unknown, an "X" will be used. The interval code will also be followed by a dash.

The last character(s) will be a letter(s) identifying the sample matrix:

"GW" Groundwater sample

"S" Soil sample

"A" Air sample

"D" Field duplicate

"EB" Equipment Blank

"TB" Trip Blank

"MS" Matrix spike sample

"MSD" Matrix spike duplicate sample

"IDW" Investigation Derived Waste sample

Examples of sample IDs are as follows:

Sample OB1-5.0-S is a soil sample from the first investigation location OB1. The soil was collected beginning from a depth of 5.0 fbgs.

Equipment blanks will be identified as "EB" in relation to the sample location tested immediately prior to their collection/submittal to the onsite laboratory. For instance OB1-X-EB identifies an equipment blank collected after the testing of location OB1.

Trip blanks will be identified as "TB" followed by the date that they sent to the lab with the associated samples. For multiple trip blanks sent on the same day, sequential alphabetic letters were added to the identification; e.g. TB-091609A, TB-091609B, etc.

2.9. Sample Handling and Analysis

2.9.1. Site Background

As part of pre-design investigation Task Numbers 1 through 6, samples will be collected for laboratory analysis. Each of these activities will require sample handling and analysis.

2.9.2. Sampling Objectives

The objective of this procedure is to identify and manage:

1. The types of environmental samples collected;
2. The analytical methods that will be used and a listing of their analyte detection limits against the appropriate enforcement standards; and

3. The types and quantities of QC samples that will be used to evaluate the quality (precision and accuracy) of the data.

2.9.3. Sample Location and Frequency

To meet the DQOs that are stated for each of the various pre-design investigation tasks in Subsections 2.1 through 2.7, the following sampling and analytical criteria have been selected and are shown on Table 4. Table 4 summarizes the types of media being sampled, the analytical methods and the type and quantity of QC samples that will be collected as part of this work. Two types of laboratories will be used for this work, an onsite laboratory and a fixed laboratory.

Table 4: Laboratory Analytical Methods and Expected Numbers of QC Samples

Matrix	Sample Source/Type	Parameter	Analytical Method	Type of Lab.	# Field Samples	QC Samples			Total Samples
						Duplicate	Field/Trip Blanks	MS/MSD	
Water	Monitoring Wells	VOCs	EPA 8260B	Fixed	26 (30)	3	3/3	2	37 (41)
		TOC / TDC	EPA 9060	Fixed	5	1	0	0	6
		TDS / TSS	SM 2540C / 2540D	Fixed	5	1	0	0	6
		Oil & Grease	EPA 1664A	Fixed	5	1	0	0	6
		pH	EPA 9040B	Fixed	5	1	0	0	6
		Hardness	SM 2340C	Fixed	5	1	0	0	6
		Ferrous and Ferric Iron	ASTM D3872-05 (2011)	Fixed	5	1	0	0	6
		Manganous and Manganic Manganese	EPA 1638, Modified	Fixed	5	1	0	0	6
		Nitrate / Nitrite / Sulfate / Sulfite	EPA 300.0	Fixed	5	1	0	0	6
		Total Metals [1]	EPA 6010B	Fixed	8	1	0	0	6
		Hexavalent and Trivalent Chromium	EPA 7196A	Fixed	50	1	0	0	6
		Carbonate	EPA 2320B	Fixed	5	1	0	0	6
		Treatability Testing		Fixed	2	0	0/1	0	3
	Groundwater Profiler	CVOCs	EPA 8260C (modified)	Onsite	170	17	17/17	12	233
		VOCs	EPA 8260B	Fixed	17	2	0/4	2	25
	IDW	VOCs	EPA 8260B	Fixed	TBD [2]	0	0	0	TBD [2]

Table 4: Laboratory Analytical Methods and Expected Numbers of QC Samples

Matrix	Sample Source/Type	Parameter	Analytical Method	Type of Lab.	# Field Samples	QC Samples			Total Samples
						Duplicate	Field/Trip Blanks	MS/MSD	
Soil	Subsurface Soil	Grain Size	ASTM D422	Fixed	5	1	0	0	6
		Bulk Density	ASTM D2937	Fixed	5	1	0	0	6
		Effective Porosity	ASTM D7263	Fixed	5	1	0	0	6
	IDW	VOCs	TBD [2]	Fixed	TBD [2]	0	0	0	TBD [2]
Air	Soil Vapor	VOCs	TO-15	Fixed	14	1	0		15
	Ambient	VOCs	TO-15	Fixed	2	0	0		2
	Indoor Air	VOCs	TO-15	Fixed	10	1	1		12

Number in parentheses indicates the number of samples if all optional tasks are executed by USACE.

Note 1: The list of metals analytes includes chromium, iron, magnesium, manganese, silicon, and titanium.

Note 2: IDW is investigation derived waste samples for characterizing waste for offsite transport and disposal. The number of samples is to be determined pending the volume of water and soil generated, and type of analyses required.

2.9.4. Sampling Equipment and Procedures

Sample handling and transportation procedures will be conducted in accordance with Stone SOP SEI-6.16.4, and are summarized in QAPP Worksheets #26 and 27.

Samples will be maintained under chain of custody (CoC) in accordance with Stone SOP SEI-4.2.7. The samples will be kept in the possession of the sampler until they can be securely packaged. The sampler will seal the samples in a sturdy shipping box or other suitable container. The sampler will complete the CoC form, and insert the CoC form into a ziplock bag attached to the lid inside of the shipping container such that the form can be filled out and signed each time the samples change possession, from sampler, to courier, to laboratory, as appropriate. Details are included in are summarized in QAPP Worksheets #26, 27, and 29.

Data will be handled in accordance with Stone SOP SEI-4.5.11.

2.9.4.1. Data Reporting

Non-detect results for all parameters will be reported at the laboratory's RL. Any detection between the MDL and the RL will be qualified with a "J" estimated value.

Analytical data deliverables will be provided by the fixed laboratory within the contracted turnaround time from date of sample receipt at the laboratory, and by the onsite laboratory within 24 hours from the time of sample receipt.

The hard copy analytical data package will be equivalent to a Level 3A Data Package. Data deliverables for this package include: case narrative, final results with surrogate standard recoveries, MS/MSD results, Laboratory Control Samples (LCS), and blank summary results.

For the fixed laboratory, one electronic data deliverable (EDD) file will be generated as a staged EDD (SEDD) for processing per USACE procedures. The onsite laboratory will provide an EDD in a suitable Excel format that can be easily manipulated by the project team. All electronic data submitted by the contract laboratories are required to be error-free and in complete agreement with the hard copy data. The electronic CD of the data must be submitted with a transmittal letter from the laboratories that certifies that the file is in agreement with hard copy data reports and has been found to be free of errors.

The hard copy data package will include the information summarized below:

- Cover page including title of report, name and location of laboratory, contact information, name and location of any subcontractor laboratories, contract number unique ID for the report, client name and address, project name and site location, statement of authenticity and signature/title of person releasing report, and total number of report pages;
- Table of contents;
- Case narrative (see description below);
- Cross reference of field sample IDs and laboratory IDs with associated test methods;
- Method numbers and summary;
- Chain-of-custody documentation including shipping documents;
- Sample receipt checklist;
- Telephone conversation and e-mail records for the project;

- Dates of sample extraction and analysis;
- Sample results for all neat and diluted runs, including units, matrix, qualifiers, dilution factors, and percent solids;
- Sample preparation information, including batch numbers;
- Raw data for initial and continuing calibrations;
- Method detection limits and method/practical quantitation limits;
- Results, true values, and/or acceptance limits for MS/MSDs, method or preparation/calibration blanks, LCSs, internal standard areas, surrogate spikes, interference checks, and serial dilutions.

The case narrative will include the client name, project name and number, date of issuance, and a discussion of any deviations from analytical strategy (e.g., samples received but not analyzed), sample receipt issues (e.g., incorrect preservatives, air bubbles in volatile organic analyte (VOA) vials), missed holding times, definition of data qualifiers or flags, identification of manually-integrated data, technical problems, and QC failures or non-conformances and associated corrective actions. The report will be signed by the Laboratory Project Manager.

2.9.4.2. Onsite Laboratory

The onsite laboratory will be used to provide near-real time screening CVOC analytical data to support the delineation of the vertical and horizontal extent of CVOC contamination in the plume.

The onsite laboratory will analyze groundwater samples for CVOCs using a Solid Phase Microextraction (SPME) sample preparation technique followed by analyses on a Hewlett Packard 5890 Series II equipped with two injection ports, two capillary columns and Mass Spectrometer and Flame Ionization detectors (MS and FID, respectively). This configuration allows the instrument to analyze two samples simultaneously. The primary purpose of the FID method is to allow samples to be run at higher dilutions concurrently with the analyses being conducted at the MS system.

Onsite Laboratory Analysis will be performed in accordance with the following Stone SOPs.

- SEI-10.1.6 “Determination of Aromatic and Chlorinated Volatile Organics and Lightweight Petroleum Hydrocarbons (Typical Range C_4-C_{14}) Compounds using Solid Phase Microextraction (SPME) and A Gas Chromatograph in Soil and Water Samples (ASTM Method D6520. Modified SW846 Methods 8021/8015)
- SEI-10.15.9 “The Determination of Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS) (SW846 US EPA Method 8260C)

Unrestricted copies of these methods are provided in Attachment C, along with 2008 Method Detection Limit Studies for both methods. For the onsite laboratory analyses, results will be reported strictly to the reporting limits (RL). The RL by SW846 Selective Ion Monitoring (SIM) Method 8260B modified with SPME are 2 µg/L for water. The GC/FID method will only be used for dilutions, of which, the RL will be 10 µg/L for water. The field laboratory, using Method 8260B modified with SPME, will provide data only for a limited list of primary contaminants of potential concern (COPC): carbon tetrachloride, PCE; TCE; PCA; 1,1,1-TCA; cis-1,2-DCE; 1,1-DCE; and chloroform.

The onsite laboratory will provide screening data only for the limited list of chlorinated volatile organic compounds (CVOCs)/COPC listed above which are the Site-related compounds. By limiting the compound list, the field analyses will be quicker, and the relevant field decisions can therefore be made more effectively.

However, the onsite laboratory will also report, as tentatively identified compounds (TICs), non-target compounds that are encountered in the groundwater samples. The onsite laboratory uses a SIM mass spectrometer program, as opposed to a full-scan mass spectrometer program; therefore, the spectral information is limited to the ions selected for the SIM method. The selected ions are typically the same as the primary and qualifier ions that are recommended in the EPA's SW846 8260 method. The non-target compounds that can be identified by the onsite lab are listed in the onsite laboratory's SOP for Method 8260. Exceptions to this include the four ketone compounds, which are not in the calibration mix and will not be reported as TICs. Worksheet #15 of the QAPP summarizes the onsite laboratory CVOC target analytes, detection/reporting limits and regulatory limits.

The CVOC screening data will be compared to the fixed laboratory split samples using the same criteria used for field duplicate samples, namely, comparison of relative percent differences (RPDs) between each pair of samples. The results will be considered acceptable if the RPDs are within ±30%. If the results exceed the ±30% RPD, Stone will investigate the reasons for the exceedances, utilizing the methods included in QAPP Worksheet #32.

Data reporting schedules and requirements for the onsite laboratory are covered in Subsection 7.5 of the Project Plan.

2.9.4.3. Fixed Laboratory

A fixed laboratory will be used for all other analyses conducted as part of this work, as shown on Table 4. The fixed laboratory will be used for two purposes:

1. To provide VOC analytical results for groundwater samples to extend the trend analysis of the existing groundwater analytical data set;
2. To provide geotechnical analytical results for subsurface soil samples to evaluate the parameters of the aquifer, to support the design of the ZVI PRB;
3. To provide geochemistry analytical results for groundwater samples to evaluate the parameters of the plume; and
4. To characterize IDW for disposal.

TestAmerica Laboratory of Savannah, GA will be the primary fixed lab used for these analyses. TestAmerica is NYSDEC-, NELAP-, and DoD-accredited for the analyses required for this project. The fixed laboratory SOPs are contained in Attachment C. Quality control samples such as duplicates, matrix spikes, field blanks and trip blanks will be prepared and analyzed. TestAmerica will subcontract the analyses of manganous and manganic manganese to Brooks Rand Laboratories and others. Data reporting schedules and requirements for the fixed laboratory are covered in Section 7.5 of the Project Plan.

In addition, SiREM Laboratory of Guelph, Ontario, Canada will perform bench-scale treatability testing to provide geochemistry analytical results for groundwater samples to evaluate the parameters of the plume. Data reporting schedules and requirements for the fixed laboratory are covered in Section 7.5 of the Project Plan.

Worksheet #15 of the QAPP summarizes the fixed laboratory VOC target analytes and TAL Metals analytes, detection/reporting limits and regulatory limits for groundwater samples, as well as the fixed laboratory detection/reporting limits for the soil and remaining groundwater sample analyses, for which there are no regulatory limits.

2.10. Sample Location Survey

2.10.1. Site Background

Stone will advance an estimated 15 soil borings at the Site under Subtasks 5A and 5C (Optional) to support vertical groundwater profiling along transects and off transects, respectively. As part of Subtask 5E (Optional), up to 4 new overburden monitoring wells may be installed at the Site. A component of these activities will require accurate measurements of the sample locations / monitoring wells.

2.10.2. Sampling Objectives

The objective of this procedure is to accurately measure the location and elevation of soil borings and overburden monitoring wells.

The measurements obtained as part of these tasks should meet industry standards, namely, ASTM D4750, and be vertically accurate to 0.01 ft.

2.10.3. Sample Location and Frequency

The following locations will be subject to location and elevation surveying as part of the pre-design investigation:

- Up to 15 soil borings at the Site under Subtasks 5A and 5C (Optional), depicted on Figure 3; and
- Up to four newly-installed overburden groundwater monitoring wells at the Site, depicted on Figure 3.

2.10.4. Sampling Equipment and Procedures

2.10.4.1. Sampling Equipment

1. Level.
2. Tripod.
3. Stadia rod.
4. Field forms or other appropriate media.

2.10.4.2. Sampling Procedure

The following procedure describes the method to determine relative field elevations per Stone SOP SEI-6.23.1. All map coordinates for this project are referenced to the horizontal datum: New York State Plane Coordinate System Northing and Easting (coordinates in feet), North American Datum of 1983 (NAD83); and the North American Vertical Datum of 1988 (NAVD88).

The northing and easting of each sample location (borings, wells, etc.) will be determined to an accuracy of ± 0.1 foot. The ground surface elevation will be measured to the nearest 0.01 foot and reported to the nearest 0.1 foot referenced to the NAVD88 datum (in feet) using existing on-Site benchmark elevations provided by USACE. Top-of-casing elevations will be measured to the nearest 0.01 foot. All surveying measurements will be performed by a New York-licensed surveyor after all sampling locations are determined and will meet the USACE positional data quality requirements (EM 1110-1-1005, 2007 version (USACE, 2007)).

1. Set up a table in the field notebook or on field data sheets with columns labeled as point location, backshot (+), foreshot (-), and elevation.
2. Set the tripod up so as to have direct line-of-sight to as many points to be measured as possible, in order to minimize set-ups.
3. Level the instrument according to the manufacturer's instructions.
4. The stadia rod is to be held vertically over the measuring point of a piezometer (monitoring well) or on a point of some known elevation. Note that this measuring point is the same point from which water levels in the piezometers (monitoring wells) are to be measured. If there is no mark, one should be made in black indelible ink. Be very careful to keep dirt or other debris from falling into the well. It may be necessary to cover the well with a plastic baggie for protection.
5. With the instrument leveled, read the measurement on the stadia at the middle cross hair. Record this measurement as the backshot (+).
6. Move the stadia rod to the next position beyond the instrument roughly the same distance as the backshot. This is designated as a turning point (could be the next piezometer or monitoring well).
7. Pivot the level around being careful not to disturb the tripod. Read the stadia rod and record as the foreshot (-).
8. Move the instrument with its tripod and level, roughly the same distance beyond the stadia rod as before (this can be done by pacing).
9. Pivot the stadia rod being careful not to change the elevation.
10. Read the measurement on the stadia rod at the turning point. Record as the backshot.
11. Repeat steps 6 through 10 working around the field to close the loop until the last foreshot is made at the original starting point.

2.11. Groundwater Bench Testing

2.11.1. Site Background

Under Subtask 6A, Stone will conduct bench-scale treatability testing on groundwater samples from the vicinity of the proposed ZVI PRB (see Subsection 2.3 for groundwater sample collection details).

2.11.2. Sampling Objectives

The objective of this procedure is to perform bench-scale treatability testing on representative groundwater samples from the vicinity of the proposed ZVI PRB. The data obtained from this testing will be used to develop design parameters for the proposed ZVI PRB.

2.11.3. Sample Location and Frequency

The monitoring well proposed for sampling under Subtask 6A will be determined following the vertical groundwater profiling program, based on the extent and distribution of the groundwater TCE plume determined using vertical groundwater profiling under Subtasks 5A and 5C.

2.11.4. Sampling Equipment and Procedures

SiREM laboratories (SiREM) will perform a laboratory column bench-scale study (Study) using two (2) side by side ZVI columns. The Study will determine the potential for ZVI to degrade CVOCs in the groundwater at the Scotia former Naval Depot. The Study will use two identical columns packed with ZVI from two different manufactures. This will allow the Study to determine the effectiveness of the treatment technology and the manufacturer's product.

The study will provide site specific degradation rates for CVOCs for groundwater flowing through a ZVI PRB. The degradation rates will be used to calculate the required residence time in the PRB, which in turn, will allow for the calculation of the required PRB thickness. The PRB thickness will be calculated using the calculated residence time and the field-determined groundwater flow rate. The groundwater flow rate will be determined through field pneumatic slug testing and further refined using a hydraulic pulse interference test. The Study will also provide data that will help determine longevity of the PRB and possible fouling or passivation over time, which may require additional operations and maintenance to regenerate the PRB.

2.11.4.1. Sampling Equipment

1. Groundwater samples.
2. Plexiglas® test columns, with sampling ports.
3. ZVI test material.

2.11.4.2. Sampling Procedure

Bench-scale treatability testing will be performed in accordance with SiREM SOP Si-1725-110212. Groundwater will be collected in 1-gallon sample bottles provided by the laboratory. The samples will be collected using low-flow methods and the sample bottles will be filled in such a manner that the bottles have no headspace. Six gallons of water will be collected for each column, for a total of 12 gallons to be used for the Study. The groundwater sampling procedure is discussed Subsection 2.3.

At the beginning of the Study (time zero), SiREM will analyze the groundwater samples for CVOCs, dissolved hydrocarbon gases (DHGs), redox potential (Eh), pH, anions, cations, and alkalinity. Once these results are received and reviewed, the Study will begin.

The test columns will be constructed of Plexiglas® with a length of 1.6 feet (ft) and an internal diameter of 1.5 inches. Seven sampling ports will be positioned along the length of the column at distances of 0.08, 0.16, 0.33, 0.50, 0.66, 1.00, and 1.31 ft from the inlet end. Sampling ports will be used frequently (as described in Table 1) throughout the study to collect samples for analysis. Each sampling port will be constructed of a nylon Swagelok® fitting (0.063 in) secured into the side of the column. Within each fitting, a 16-gauge needle (Fischer Scientific) will be tightly secured to allow for sampling from the center axis of the column. To prevent solid materials from entering the needle, glass wool will be placed in the needle. Each sample port will be fitted with a Luer-Lok™ fitting, such that a glass syringe can be attached to the port to collect a sample. When not in operation, the ports will be sealed by Luer-Lok™. Both the top and bottom of the column will be sealed by rubber O-ring seals and contain fine (500 micron) mesh to inhibit solid material from entering the influent and effluent hose.

The ZVI material will be packed in vertical lift sections to assure a homogenized mixture. Values of bulk density, porosity, and pore volume will be determined gravimetrically. The Study will be performed at ambient groundwater temperature.

The column will operate for a period of six weeks. The Site groundwater will be supplied to the influent end of the columns at a constant rate using a laboratory pump. The flow velocity is estimated to be 1.6 feet per day, which will provide a total of 40 pore volumes over the six week period. This flow rate may be adjusted based on field data or scaled off of following the Study.

Water samples will be collected from the column influent, effluent, and individual ports for analysis of CVOCs, DHGs, Eh and pH on a weekly basis. Anions (including chloride, nitrate, and sulfate) will be collected from the column influent, effluent, and individual ports at three sampling events. Other analyses will include cations (as listed b) and alkalinity (as milligrams carbonate/L) on samples collected from the column influent and effluent at the start and end of the study to help predict possible mineral precipitation. Table 5 provides a summary of the sampling parameters and frequency.

Total Aluminum (Al)
Total Antimony (Sb)
Total Arsenic (As)
Total Barium (Ba)
Total Beryllium (Be)
Total Bismuth (Bi)
Total Boron (B)
Total Cadmium (Cd)
Total Calcium (Ca)
Total Chromium (Cr)
Total Cobalt (Co)
Total Copper (Cu)
Total Iron (Fe)
Total Lead (Pb)
Total Lithium (Li)
Total Magnesium (Mg)
Total Manganese (Mn)
Total Molybdenum (Mo)
Total Nickel (Ni)
Total Phosphorus (P)
Total Potassium (K)
Total Silicon (Si)
Total Selenium (Se)
Total Silver (Ag)
Total Sodium (Na)
Total Strontium (Sr)
Total Tellurium (Te)
Total Thallium (Tl)
Total Tin (Sn)
Total Titanium (Ti)
Total Tungsten (W)
Total Uranium (U)
Total Vanadium (V)
Total Zinc (Zn)
Total Zirconium (Zr)

Table 5: Bench-Scale Column Study Sampling and Analyses

Analyte	Number of Sampling events*	Number of Samples (Influent/Effluent)	Number of Samples (ports)	Total number of analyses per column
CVOCs and DHGs	7	2	7	55
Anions	3	2	7	28
Cations and Alkalinity	2	2	0	3
Eh / pH	7	2	7	55

*at time zero only the influent groundwater reservoir will be sampled.

Aqueous sampling will be conducted using a 1 milliliter (mL) glass syringe. For CCOC analyses, a 1.0 mL sample will be collected from the sampling port and transferred into a 10 mL auto sampler vial containing 5.0 mL of acidified deionized water (pH approximately 2). The water is acidified to inhibit microbial activity in the auto sampler vial. The vial will be sealed with an inert Teflon®-coated septum and aluminum crimp cap for immediate automated injection of 3 mL of headspace onto the GC. For anion analysis, a 0.5 mL sample will be taken from the sampling port and transferred into a 1 mL Eppendorf tube. The sample will be diluted in deionized water and a 25 µL sample will be injected into the ion chromatograph.

Eh will be determined using a combination silver / silver chloride reference electrode with a platinum button and a Corning 313 meter. The electrode is standardized with an Eh standard (Thermo Electron Corporation). Millivolt (mV) readings are converted to Eh, using the electrode reading and the standard potential of the silver / silver chloride electrode at a given temperature. The pH measurements are made using a combination pH/reference electrode and an Oakton™ meter, standardized with the pH buffer 7 and the appropriate buffer of either 4 or 10. A 3.0 mL sample will be collected from the sampling ports with a glass syringe and analyzed immediately for Eh and then pH.

Samples for inorganic parameters (cations and alkalinity) will be sent to a commercial laboratory, Maxxam Analytics Inc., Mississauga, Ontario. Cation analyses will be performed using inductively coupled plasma atomic emission spectroscopy via EPA Method 200.7. Alkalinity (as mg carbonate/L) in water will be determined by automated colorimetry via EPA Method 310.2.

Once groundwater samples are received by the laboratory, initial sample results will be available in 2 to 3 weeks. Once these results are available and reviewed, the Study will begin. The study itself will span approximately 6 weeks. Bi-weekly interim results and updates will be provided by SiREM. Final results will be available 2 weeks after completion of the test. A draft report will be submitted 3 weeks following receipt of the final results and the final report will be submitted 3 weeks following comments provided on the draft report. In total, the Study will be approximately 16 weeks.

2.12. Management of Investigation-Derived Waste

2.12.1. Site Background

As part of pre-design investigation Subtask 4A, groundwater samples will be collected from approximately 26 monitoring wells at the Site which will generate purge water. As part of pre-design investigation Subtasks 5A, 5C (Optional), and 5E (Optional), soil borings will be advanced at the Site which will generate soil cuttings. Under Subtask 5E (Optional), and overburden monitoring wells may be installed at the Site, which will generate soil cuttings, and development and purge water. As part of Subtask 6A, groundwater samples will be collected, which will generate purge water. Tasks 4, 5, and 6 will each generate some amount of decontamination fluids. Therefore, these tasks will require managing IDW.

2.12.2. Sampling Objectives

There are four types of IDW will be generated during the pre-design investigation:

1. Solid waste suitable for disposal at a certified solid waste landfill (non-hazardous);
2. Solid wastes containing hazardous constituents at concentrations above Remediation Regulation standards for residential direct exposure;
3. Liquid waste suitable for discharge to the ground surface; and

4. Liquid waste generated from investigation activities such as decontamination, monitoring well development and sampling.

Management of IDW, described in more detail below, will follow the requirements outlined in NYSDEC's *Technical Guidance for Site Investigation and Remediation* (DER-10) (NYSDEC, 2010).

2.12.3. Sample Location and Frequency

Stone will be responsible for the proper management of all IDW; however the USACE will be considered the "generator" of any IDW considered hazardous waste and will therefore need to sign any required manifests. The point of contact responsible for IDW management is Stone PM, Joseph Schmidl. IDW will be disposed of within 30 days of completion of that phase of the field investigation site work (including receipt of laboratory analysis).

Any IDW determined to be hazardous will be containerized and labeled in accordance with Resource Conservation and Recovery Act and NYSDEC hazardous waste regulations. All non-hazardous waste IDW storage containers will be labeled with the following information: class of IDW; source area; date of generation; and generator name, address and phone number. To the extent possible based on observations and field screening results, IDW will be segregated according to potential level of contamination (i.e., heavily- versus lightly-contaminated).

2.12.3.1.1. Sampling and Procedures for Solid Waste

Solid IDW for this program is anticipated to include sampling equipment (e.g., disposable valves, fittings and sample tubing used to collect samples), personal protective equipment (PPE), and soil/cuttings from soil borings. The plastic, glass, paper, and PPE IDW will be double bagged in plastic trash bags and placed into a dumpster for eventual disposal at a lined landfill.

The monitoring well drilling, development, and sampling will create approximately 10 cubic yards of solid IDW. Under the provisions of NYSDEC's guidance, Section 3.3 (e) 1. i., drill cuttings and other soil generated onsite during the installation of soil borings are presumed to be contaminated until proven otherwise. Such cuttings and spoils must be stored on protective sheeting and covered with protective sheeting if cuttings remain on ground at the end of the day. However, the soil cuttings from unsaturated soils above the plume will be considered non-hazardous unless PID screening results indicate the presence of VOCs; in such cases, the soils will be segregated and characterized for disposal as IDW.

The solid IDW will be temporarily stored in drums or roll-offs at the Site adjacent to the drilling locations. Laboratory analysis performed during vertical groundwater profiling will be used to characterize the soil IDW to determine if on- or offsite disposal is appropriate. If no groundwater VOC concentrations exceed NYSDEC GA Groundwater Standards, the soils will be considered eligible for onsite disposal. Onsite disposal will be performed only if VOC analytical data all meet NYSDEC Remediation Regulation standards for residential direct exposure. A minimum of one characterization sample will be collected. If characterization data indicate NYSDEC residential direct exposure standards are not exceeded, the soil IDW will be used to backfill the borehole. Soil IDW cannot be used to backfill soil borings completed as monitoring wells, and soil cuttings from these locations will be containerized, characterized, and disposed of offsite, as appropriate. If NYSDEC Remediation Regulation standards for residential direct exposure are exceeded by the soil IDW, the IDW will be containerized, characterized, and ultimately disposed of based on results of characterization sampling for VOCs. The determination will be based on results of analysis once a representative amount of soils has

accumulated. A temporary USEPA ID number will be obtained for the Site to be used for the transport and disposal of solid wastes containing contaminants in excess of acceptable levels for onsite disposal. The offsite transport and disposal will be conducted by a qualified contractor in accordance with state and federal regulations.

2.12.3.1.2. Sampling and Procedures for Liquid Waste

Liquid IDW for this program may include purge water from sampling activities, equipment and field rinse water, and decontamination water. Monitoring well drilling, development, and sampling may create hundreds of gallons of liquid IDW. Purge and well development water IDW will be containerized and temporarily stored in drums, polyethylene tanks, and/or frac-tanks at the Site. Laboratory analysis will be performed to characterize the liquid IDW to determine if on- or offsite disposal is appropriate. Drums will be stored on polyethylene sheets with a wooden frame to create secondary containment, and will be covered with polyethylene sheeting. Drums will be filled to allow for 10% ullage, to prevent splitting should the drums freeze.

As permitted under NYSDEC guidance, Section 3.3 (e) 5 ii (2) (A), onsite disposal will be performed only if VOC analytical results meet NYSDEC GA Groundwater Standards. A minimum of one characterization sample will be collected. Liquid IDW which does not exceed NYSDEC GA Groundwater Standards will be dispersed onto the ground surface without treatment near the location of the source of the liquid IDW and in such a manner as to prevent erosion and to prevent the water from entering surface water or storm drains.

2.13. Maintenance and Decontamination of Field Equipment

2.13.1. Site Background

As part of each pre-design investigation task, field equipment will be used, and will come in contact with potentially-contaminated media. Therefore, field equipment will require maintenance and decontamination.

2.13.2. Sampling Objectives

The objective of this procedure is to maintain and decontaminate field equipment, so that it can be relied upon to collect high-quality data.

Proper maintenance and decontamination of field equipment is an essential quality control measure and a safety precaution to ensure sampling equipment is in proper working condition and to remove any chemical or material contamination. The procedure prevents cross-contamination between sampling stations and helps maintain a clean working environment for the safety of all field personnel. Equipment decontamination helps prevent sampling bias.

2.13.3. Sample Location and Frequency

Field equipment, such as water quality meters and water level indicators, used in all parts of the pre-design investigation will require maintenance and decontamination. Equipment maintenance will be performed as necessary; equipment decontamination will be performed before equipment use, between sampling locations/intervals, and at the conclusion of the pre-design investigation.

2.13.4. Sampling Equipment and Procedures

2.13.4.1. Sampling Equipment

- Proper protective clothing (if necessary or as designated in the protocol or QAPP).
- Plastic buckets or tubs.
- Spray bottles.
- Brushes.
- Field notebook or other acceptable recording medium.
- Operating Manuals.
- Calibration standards.
- Decontamination materials, as necessary.
- Clean (new) polyethylene/plastic sheeting or other material on which decontaminated equipment may be placed to dry.

2.13.4.2. Sampling Procedures

2.13.4.3. Equipment Calibration

The procedures listed below are consistent with Stone SOP SEI-6.19.2.

1. Equipment calibration is instrument-specific, and must be performed according to the manufacturer's instructions.
2. Instrument calibration must be performed at the beginning of each work day, checked at mid-day or whenever it is suspected that the calibration may have drifted, and at the end of each work day.
3. Equipment calibration will be recorded in a field

2.13.4.4. Equipment Maintenance

The procedures listed below are consistent with Stone SOP SEI-5.1.5.

1. Records will be maintained of all inspection, maintenance, testing, and calibrating operations. These records will contain, at a minimum:
    a. Dates of operation.
    b. Whether maintenance was routine or non-routine.
    c. Whether maintenance followed written SOPs.
2. If non-routine repairs are performed, at a minimum, the following will be documented in addition to the items in 2 above:
    a. Nature of the defect.
    b. How and when the defect was discovered.

c. Any remedial or correction action taken in response to the defect.

3. Inspection, maintenance, testing and calibration efforts will be documented in the field logbook. The records shall include:
    a. The name of the item of equipment;
    b. The manufacturer's name, type identification, and serial number or other unique identification;
    c. Date received and date place in service (if available);
    d. Current location; where appropriate;
    e. Condition when received (e.g., new, used, reconditioned);
    f. Copy of the manufacturer's instructions,
    g. Dates and results of calibrations and/or verifications and date of the next calibration and/or verification;
    h. Details of maintenance carried out to date and planned for the future; and,
    i. History of any damage, malfunction, modification or repair
    j. Equipment which is malfunctioning and has been taken out of service, it shall be tagged as such.

2.13.4.5. Equipment Decontamination

The procedures listed below are consistent with Stone SOP SEI-5.1.5.

1. The following procedure applies to decontamination of hand-held sampling devices used for collecting samples for trace organic or inorganic constituent:
    a. Clean the device with tap water and soap, using a brush if necessary to remove particulate matter and surface films.
    b. Rinse thoroughly with tap water.
    c. Rinse thoroughly with analyte- or organic-free water.
    d. If necessary and appropriate, rinse thoroughly with solvent. Do not solvent-rinse polyvinyl chloride or plastic items.
    e. Rinse thoroughly with organic/analyte free water, or allow equipment to dry completely.
    f. Remove the equipment from the decontamination area. Equipment stored overnight should be wrapped in aluminum foil and covered with clean, unused plastic.
2. The following procedure is applies to decontamination of heavy equipment to be used for soil borings, etc.:
    a. Wipe/scrape the equipment to remove gross contamination directly into IDW containers.
    b. Thoroughly clean the equipment using high pressure steam.
    c. Rinse thoroughly with tap water.
    d. Remove the equipment from the decontamination area.

2.14. Soil Vapor / Indoor Air Sampling

2.14.1. Site Background

Stone will complete a SVI Investigation at selected onsite buildings at the Former Scotia Navy Depot, to evaluate current human health risks. Based on USEPA, Department of Defense (DoD), NYSDEC and NYSDoH SVI guidance documents, in order to evaluate current human health risks, Stone proposes to collect synchronous sub-slab soil vapor, indoor air, and ambient outdoor air samples.

2.14.2. Sampling Objectives

The objective of this procedure is to collect representative soil vapor and indoor air samples to support risk assessment decisions.

2.14.3. Soil Vapor Intrusion Study Planning

On March 8, 2013, Stone personnel performed a pre-sampling Site walkover of the buildings to be included within the SVI Investigation. These buildings include Buildings 201, 202, 203, 204, 403, and 404. Each building was inspected in accordance with the following documents: 2002 USEPA *Draft Guidance for Evaluating the Vapor Intrusion to Indoor Air Pathway from Groundwater and Soils (Subsurface Vapor Intrusion Guidance);* 2009 Tri-Service Environmental Risk Assessment Workgroup *Department of Defense Vapor Intrusion Handbook;* and 2006 *New York Department of Health (NYDOH) Guidance for Evaluating Soil Vapor Intrusion in the State of New York,* for the following:

- Occupant information, including:
  - Contact information
  - Number of occupants
  - Age of occupants
- Owner or Landlord contact information
- Building Characteristics, including:
  - Use type (e.g., residential or industrial)
  - Number of floors, age, tightness
- Airflow
- Bottom floor construction and entry points for soil vapor
- Heating, venting and air conditioning
- Occupancy
- Factors that may influence air quality
- Site Utilities
- Presence of VOC containing materials.

The results of these inspections were recorded on Indoor Air Quality Questionnaire and Building Inventory forms and will be provided within the SVI Report. Locations for sampling were developed in collaboration with CENAE and are biased to provide spatial coverage, assess areas over the dissolved phase TCE and Carbon Tetrachloride groundwater plumes, and to assess areas where occupants are in an office setting

removed from potential process-related VOC sources. Figure 4 presents the locations of proposed indoor air, soil vapor, and ambient outdoor air sampling locations as well as the locations of observed VOC sources.

2.14.4. Sample Location and Frequency

Based on USEPA, DoD, and NYDOH SVI guidance, Stone will collect sub-slab and indoor air samples to evaluate the possibility of SVI. Up to twenty-eight (14) soil vapor samples, ten (10) indoor air, and six (2) background ambient outdoor air samples will be collected under conservative conditions (e.g., windows and doors closed, heating, ventilating, and/or air conditioning in operation).

Buildings 201, 202, 203, and 204 (Figure 3) are slab-on-grade industrial buildings. Buildings 403 and 404 were constructed such that the slab along east and west sides of both buildings and the north side of 404 and south side of 403 are at the same elevation as a truck deck. The remaining exterior facades (north façade of Building 403 and south side of Building 404) are at grade.

The following assumptions are associated with this task:

- Up to twenty thirty-one (31) samples will be collected from the six buildings and submitted for analysis:
  - A total of ten (10) indoor air samples will be collected from Buildings 201 through 204 from each building on the ground floor:
    - Three (3) samples will be collected from Building 201,
    - Two (2) samples will be collected from Building 202,
    - Three (3) samples will be collected from Building 203, and
    - Two (2) samples will be collected from Building 204.
    - No indoor air samples are proposed for Buildings 403 and 404 due to their lack of tightness with respect to outside air.
  - One (1) duplicate indoor air sample.
  - Two (2) outdoor ambient air samples (will serve as background for all four 200-block buildings and both 400-block buildings) ;
  - A total of ten (11) sub-slab samples will be collected from Buildings 201 through 204, specifically:
    - Three (3) samples will be collected from Building 201,
    - Two (2) samples will be collected from Building 202,
    - Three (3) samples will be collected from Building 203, and
    - Three (3) samples will be collected from Building 204.
  - A total of four (4) sub-slab samples will be collected from Buildings 403 and 404 (two (2) samples each).
  - One (1) duplicate sub-slab soil vapor sample and one (1) trip blank sample;
- Sub-slab soil vapor and indoor air samples will be collected from the lowest occupied floor of each building;

- Sub-slab soil vapor and indoor air samples will be collected from the 200-block buildings will be collected concurrently with an 8-hour duration;
- The SVI samples will be analyzed via EPA Toxic Organic (TO) Method 15. Selected ion monitoring (SIM) analysis shall be performed for carbon tetrachloride and TCE, and their breakdown products;
    - Alpha Analytical Laboratory meets Method T0-15 laboratory certification requirements specified by NYSDoH and is accredited for Method T0-15 according to the DoD Environmental Laboratory Accreditation Program (ELAP);
    - Sample reporting limits will be sufficient to meet NYDDoH and American Conference of Governmental Industrial Hygienists Threshold Limit Values for carbon tetrachloride and TCE, and their breakdown products; and,

2.14.5. Sampling Equipment and Procedures

Stone's SOP for the collection of soil vapor samples for VOC analyses is provided in Attachment B.

2.14.5.1. Sampling Equipment

- Proper protective clothing as specified in the Site Health and Safety Plan including eye protection and hearing protection
- Field Screening Equipment, including:
    - PID equipped with an 11.7 eV lamp to determine risk of exposure to sampling personnel and screen soil vapor sample locations.
    - Carbon dioxide (CO_2)/ Carbon monoxide (CO)/ Oxygen (O_2) meter
- Sample Probe Installation
    - Electric Rotary Hammer Drill with ⅜-inch outside diameter (OD) masonry drill bit
    - ¼-inch OD fluorinated ethylene propylene (FEP) tubing (laboratory grade) of sufficient length to reach between the target depth and surface equipment.
    - Well sand (#1 Silica)
    - Modeling clay
- Leak Testing Equipment
    - Compressed Helium cylinder (small Q size)
    - Regulator
    - 5-gallon pail equipped with 2 bulk head connectors (one to connect to helium, one to connect to sample line.
    - Helium leak detector
    - Tygon tubing (¼-inch inner diameter) to plumb between both the regulator and pail and sample port bulk head fitting and He leak detector
- Summa Method (TO-15)
    - Certified pre-cleaned, 6-Liter Summa Canisters

- For Soil Vapor: Flow controllers calibrated for a 30-minute collection period.
- For Indoor Air: Flow controllers calibrated for a 24-hour collection period.

- Field notebook, sample collection form, or other acceptable medium for recording field data. Ball point pens. Sample forms are contained within SOPs SEI-6.36.1 and -6-49-0, in Attachment B.
- Decontamination equipment
  - Alconox, scrub brushes and containment pails
  - Spring Water
  - Propane torch
- Bentonite chips for backfilling
- Surface patch (hydraulic cement)
- Packing materials
- Chain of Custody

2.14.5.2. Soil Vapor Sampling Procedure

Stone will inspect each proposed sample location for nearby cracks or other conduits that would provide a means for interior air to reach the subsurface. If present, these cracks will be sealed using floor leveling compound, caulk or other patch.

A ⅜-inch diameter hole will be installed in the slab using the rotary hammer drill to a depth of 12 inches or at least 6 inches below the underside of the slab.

Once at depth, a section of ¼-inch OD FEP tubing of sufficient length to reach sampling apparatus on the surface will be inserted into the borehole. The annular space between the tubing and borehole is then filled using silica sand to approximately 2 inches below the surface of the borehole. The final annular space is sealed using modeling clay.

Following installation, the sample train is connected to the handheld PID. The PID serves two purposes: 1) screen the soil vapor sample port to assist the fixed laboratory, and 2) purge entrained atmospheric air from the sample train. One to three sample train volumes should be purged prior to sample collection. Peak PID readings are recorded on field forms. Carbon dioxide, carbon monoxide, and oxygen concentrations are then measured using the multigas meter. Sample purge rate will not exceed 0.2 L/min.

After installation of the sample port and field screening, a leak testing procedure will be performed at each of the sampling locations to demonstrate that the sampling system does not allow for short circuiting to occur between the ambient air and the sampling train.

The leak test procedure includes:

1. Turn on helium detector to allow the unit to warm up.
2. Purge Sample Train by connecting PID to sample side of the leak testing pail using Tygon tubing.
3. Allow unit to draw sample train air for 4 minutes.
4. Record peak value on field form.
5. Measure CO_2/CO/O_2 concentrations with CO_2/CO/O_2 meter and record value on field form.
6. Connect helium detector to the sample side of leak testing bucker
7. Connect the sample train tubing to the sample side of the leak test pail

8. Connect helium regulator to open port of leak test pail
9. Allow helium detector to purge sample train of ambient air (5 minutes)
10. Turn on helium until gas is heard escaping from the bottom of the pail
11. Check for leaks in sample train, as indicated by positive readings on the helium detector.

If no helium is present it can be concluded that no short circuiting has occurred. If helium is found in the sample train, the sample train should be checked for loose fittings and the modeling clay seal inspected for leaks. The procedure should be repeated until it is demonstrated that no short circuiting has occurred. If needed, the soil vapor borehole can be re-drilled; however, to avoid short circuiting of ambient air, the prior borehole should be filled with hydrated bentonite.

Following successful leak testing, plug sample line with a knotted piece of silicon tubing to prevent loss of soil vapor contaminant mass.

2.14.5.2.1. Sample Collection

Following successful leak testing, connect FEP tubing to Summa canister. Open flow control regulator and record start time and initial pressure on sampling form.

At the end of the sampling period (8 hours in the 200-block buildings, 30 minutes in the 400-block buildings), record the time and final pressure, shut regulator valve. The vacuum gauge should still contain vacuum at the end of the sample collection period. If no vacuum remains at the end of the sampling period, either the flow controller or the Summa canister is faulty and the sample should be re-collected. The original summa and flow controller should be marked and returned to the laboratory without requesting analysis. If, following the sample collection period, over 20 inches of Hg remains, sufficient sample volume has not entered the Summa canister, either due to restriction of air flow within the subsurface or a faulty flow controller. Allow more time for sample collection, but record observations on field forms.

Disconnect tubing and repackage Summa for transport to contract laboratory. Complete chain of custody for the sample and complete sample tags for laboratory tracking.

Remove tubing from the borehole and seal borehole to surface with quick setting concrete. Clean residuals and concrete cuttings using a wet-dry vacuum.

2.14.5.3. Indoor Air Sampling Procedure

Indoor air sampling will be performed to assess potential exposure of the building occupants to Site contaminants of concern (COCs). For offsite structures, property owners will be given a list of instructions prior to sampling (see SOP SEI-6.49.0 in Attachment B). Stone will conduct an interview with residents/property owners prior to beginning the sampling. This interview will consist of an information gathering survey that includes an inspection of the home and a question and answer period with the homeowner, to identify sources of indoor air contamination as presented in SOP SEI-6.49.0 in Attachment B of this document.

If potential sources of contamination (e.g., dry cleaning, automotive parts cleaning products) are observed within the structure in the area to be sampled, Stone will relocate these to an area removed from the sampling area and document their occurrence on field forms. Indoor air samples will be collected from the lowest inhabited floor level of each structure.

Two ambient air samples will be collected in the area of Buildings 201, 202, 203, and 204 to serve as the on Site background samples for all buildings.

Sample collection will consist of positioning a sub-atmospheric canister (Summa® canister) within each zone of interest at the approximate height of a child's breathing zone (3 feet off the ground). Each canister will be equipped with a flow controller and allowed to acquire a sample for 8 hours. Final vacuum gauge readings will be recorded at the end of 8 hours and the canister closed.

Sample canisters will travel under chain of custody to Alpha Analytical Laboratory for analysis by low-level modified method TO-15 for TCE, carbon tetrachloride and their degradates.

.

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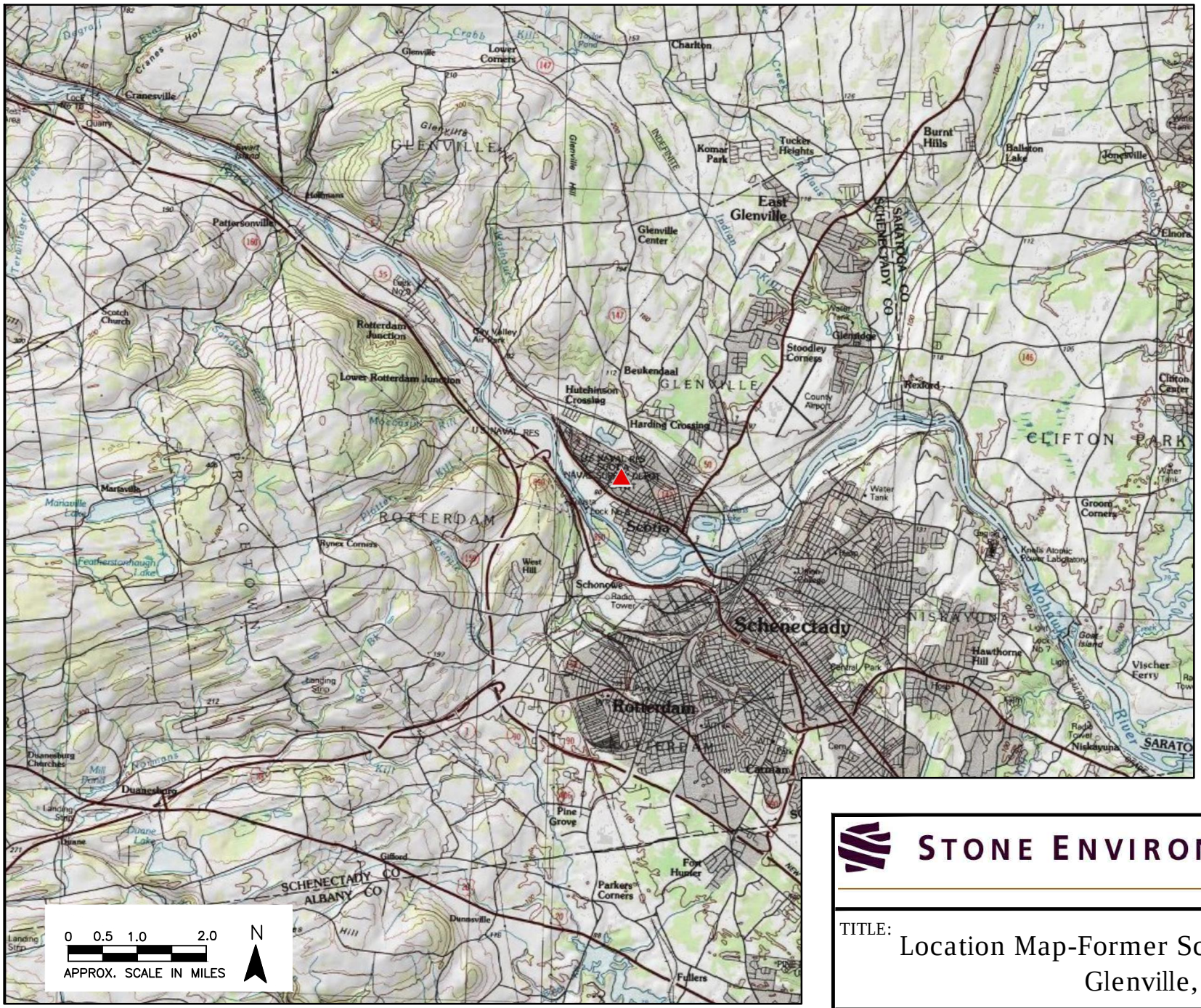
FIGURES

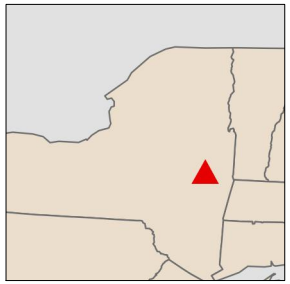
Figure 1: Location Map

Figure 2: Site Vicinity Map

Figure 3: Site Map

Figure 4: Proposed SVI Investigation Sample Locations





DATUMS

Coord. System: N.Y. State Plane
Horizontal Datum: NAD 1983
Vertical Datum: NAVD 1988

Note:
Source- 2011 National Geographic Society, i-cubed

LEGEND

▲ Scotia Naval Depot

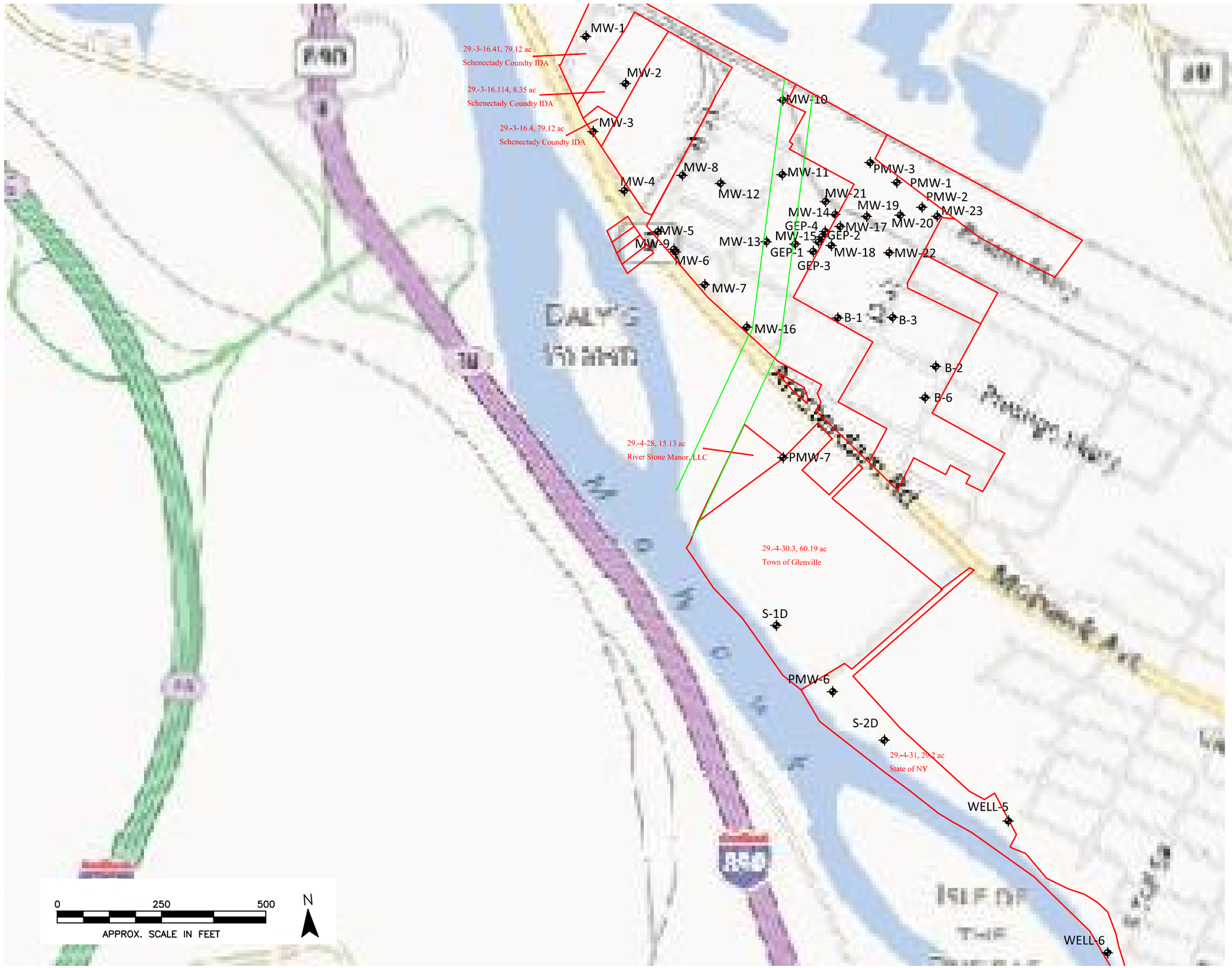
STONE ENVIRONMENTAL INC

TITLE: Location Map-Former Scotia Naval Depot-Glenville, NY

PROJECT: Former Scotia Naval Depot

FIGURE NO.: 1

DATE: 1/4/13 DRAWN BY: DJL CHECKED BY: JS



LEGEND

- Monitoring Well
- Parcel Boundaries with ID#

Note:

Source-Bing Imagery 2013

Monitoring well and plume locations from Figure 3 of the Former Scotia Naval Depot Site- Glenfille, NY -Statement of Work 8/30 /12.

DATUMS

Coord. System: N.Y. State Plane
Horizontal Datum: NAD 1983
Vertical Datum: NAVD 1988

STONE ENVIRONMENTAL INC

TITLE: Site Vicinity Map-Former Scotia Naval Depot- Glenville, NY

PROJECT: Former Scotia Naval Depot

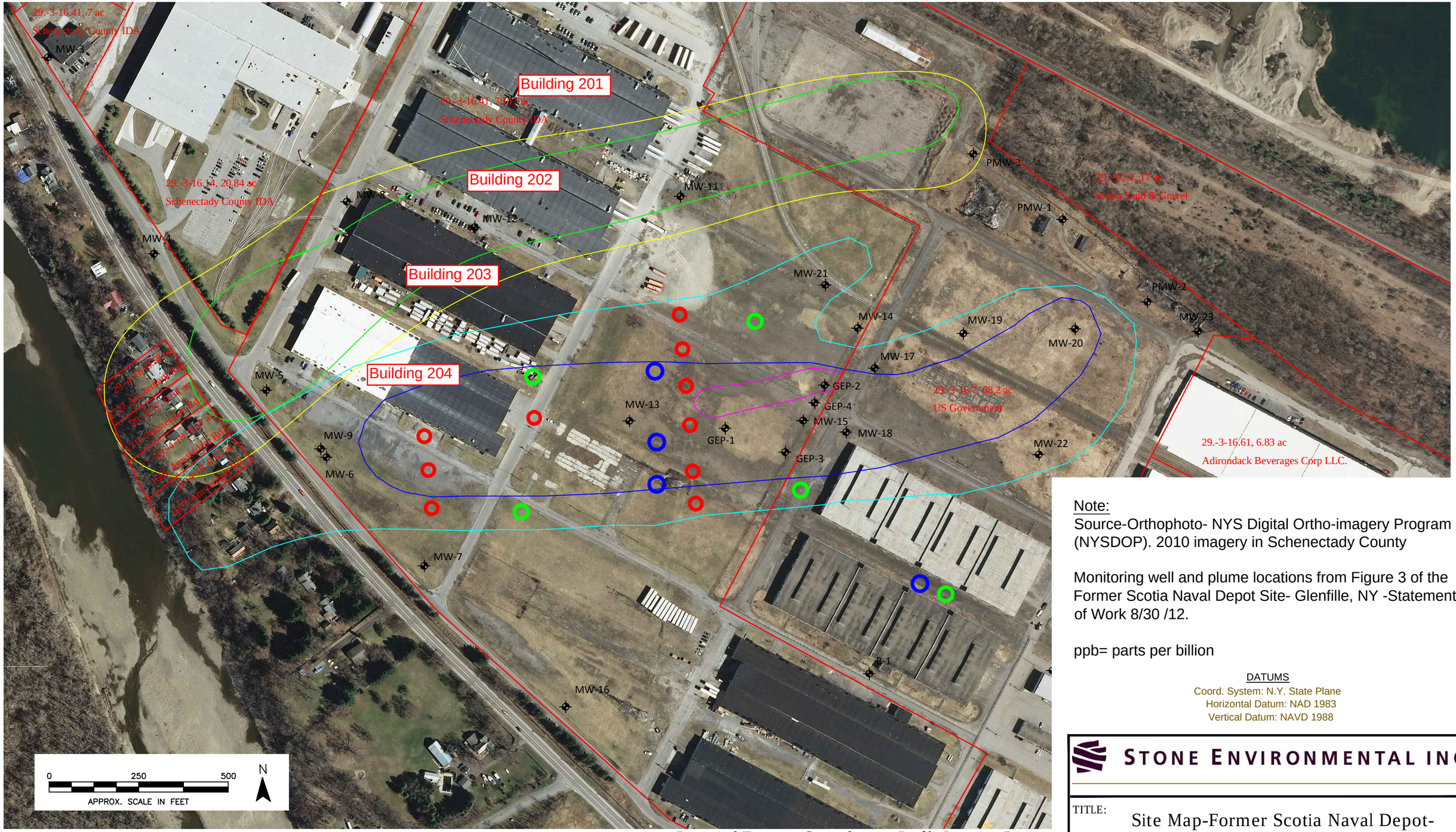
FIGURE NO.: 2

DATE: 1/3/13

DRAWN BY: DJL

CHECKED BY: JS

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LEGEND

- Monitoring Well
- Parcel Boundaries with ID#

June 2005 Carbon Tetrachloride Plume
- 1 ppb
- 5 ppb

June 2005 Trichloroethylene Groundwater Plume
- 10 ppb
- 100 ppb
- 500 ppb

- Proposed Transect Groundwater Profile Location Boring
- Proposed Non-Transect Groundwater Profile Location (subject to change based on the results of the transect groundwater profile results)
- Proposed Monitoring Well Location (subject to change based on the results of the groundwater profile results)

Note:
Source-Orthophoto- NYS Digital Ortho-imagery Program (NYSDOP). 2010 imagery in Schenectady County

Monitoring well and plume locations from Figure 3 of the Former Scotia Naval Depot Site- Glenfille, NY -Statement of Work 8/30 /12.

ppb= parts per billion

DATUMS
Coord. System: N.Y. State Plane
Horizontal Datum: NAD 1983
Vertical Datum: NAVD 1988

STONE ENVIRONMENTAL INC

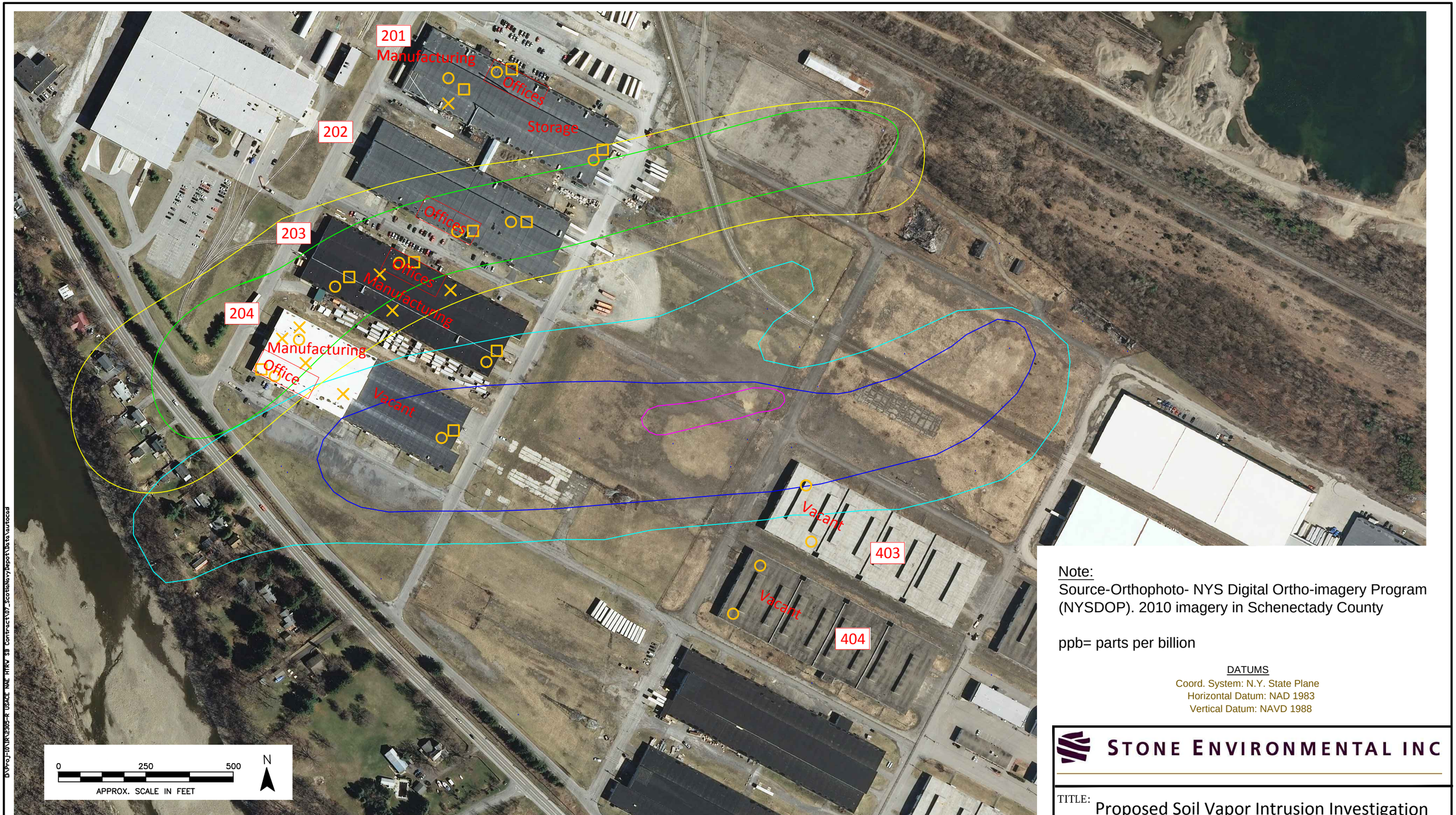
TITLE: Site Map-Former Scotia Naval Depot-Glenville, NY

PROJECT: Former Scotia Naval Depot

FIGURE NO.: 3

DATE: 1/14/13 DRAWN BY: DJL CHECKED BY: JS

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LEGEND

June 2005 Carbon Tetrachloride Plume
- 1 ppb
- 5 ppb

June 2005 Trichloroethylene Groundwater Plume
- 10 ppb
- 100 ppb
- 500 ppb

- Proposed Sub-Slab Soil Vapor Location
- Proposed Indoor Air Sample Location
- Observed VOC Source

Note:
Source-Orthophoto- NYS Digital Ortho-imagery Program (NYSDOP). 2010 imagery in Schenectady County

ppb= parts per billion

DATUMS
Coord. System: N.Y. State Plane
Horizontal Datum: NAD 1983
Vertical Datum: NAVD 1988

STONE ENVIRONMENTAL INC

TITLE: Proposed Soil Vapor Intrusion Investigation Locations

PROJECT: Former Scotia Naval Depot

FIGURE NO.: 4

DATE: 03/15/13 DRAWN BY: JPK CHECKED BY: DV

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ATTACHMENTS

A: QUALITY ASSURANCE PROJECT PLAN

QUALITY ASSURANCE PROJECT PLAN

FINAL

DEFENSE NATIONAL STOCKPILE CENTER
SCOTIA DEPOT SITE
GLENVILLE, NEW YORK

CERCLIS Number: NYN000204534
P2 Project Number: 397651

Contract Number: W912WJ-11-D-0001
Stone Project ID: 102305-R-C
April 2013

Prepared for:

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QAPP WORKSHEET #1: TITLE AND APPROVAL PAGE

☐ Worksheet Not Applicable (State Reason)

Site Name/Project Name: Defense National Stockpile Center Scotia Depot Site

Site Location: Glenville, NY

Document Title: QAPP- Defense National Stockpile Center Scotia Depot Site Pre-Design Investigation

Lead Organization: U.S. Army Corps of Engineers, New England District (NAE)

Preparer's Name and Organizational Affiliation: Lee Rosberg, Stone Environmental, Inc.

Preparer's Address, Telephone Number, and E-mail Address:

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Preparation Date (Day/Month/Year): 28/02/2013

Investigative Organization's Program Manager/Date: _____
Signature

Printed Name/Organization: Seth Pitkin, Stone Environmental, Inc.

Investigative Organization's Project Manager/Date: _____
Signature

Printed Name/Organization: Joseph Schmidl, P.G., Stone Environmental, Inc.

Investigative Organization's Project QA Officer/Date: _____
Signature

Printed Name/Organization: Kim Watson, Stone Environmental, Inc.

Investigative Organization's Project Chemist/Date: _____
Signature

Printed Name/Organization: Michael Rossi, Stone Environmental, Inc.

Lead Organization's Project Manager/Date: _____
Signature

Printed Name/Organization: Gregory Geopfert, USACE New York District (NAN)

Approval Signatures/Date: _____
Signature

Printed Name/Title Dean Brammer, USACE New England District (NAE)

Approval Authority:

Other Approval Signatures/Date: _____
Signature

Printed Name/Title: Jason M. Pelton, New York State Department o Environmental Conservation

Document Control Numbering System: UFPQAPPv2.0.docx

QAPP WORKSHEET #2: QAPP IDENTIFYING INFORMATION

☐ Worksheet Not Applicable (State Reason)

Site Name/Project Name: Defense National Stockpile Center Scotia Depot Site

Title: Quality Assurance Project Plan

Site Location: Glenville, New York

Revision Number: 2

Site Number/Code:

Revision Date:

Operable Unit:

Contractor Name: Stone Environmental, Inc.

Contractor Number: W912WJ-11-D-0001

Contract Title:

Work Assignment Number:

1. Identify regulatory program: New York State Department of Environmental Conservation (NYSDEC) 6 NYCRR Part 375
2. Identify approval entity: Gregory Goepfert, USACE New York District (NAN)
3. The QAPP is (select one): ☐Generic ☒Project Specific
4. List dates of scoping sessions that were held:
5. List dates and titles of QAPP documents written for previous site work, if applicable:

Title	Approval Date

6. List organizational partners (stakeholders) and connection with lead organization:
   Jason M.Pelton, Project Manager, NYSDEC
   Maureen Schuck, Project Manager, NYSDOH
   David Baker, U.S. General Services Administration
   Kenneth Simmons, Construction Control Representative, U.S. General Services Administration
   Lisa Sanders, Depot Supervisor Scotia, Defense Logistics Agency/Defense National Stockpile Center
   David Ahl, Galesi Corporation
   Jeanette Dadusc, US EPA Region 2, Federal Facilities Program
7. List data users:
   Stone Environmental, Inc.

USACE	NYSDEC	NYSDOH
USEPA	GSA	DLA/DNSC

8. If any required QAPP elements and required information are not applicable to the project, then circle the omitted QAPP elements and required information on the attached table. Provide an explanation for their exclusions below:

QAPP WORKSHEET #2: QAPP IDENTIFYING INFORMATION (Continued)

QAPP elements and required information that are not applicable to the project are circled and an explanation is provided in the QAPP.

Required QAPP Element(s) and Corresponding QAPP Section(s)	Required Information	QAPP Worksheet # or Crosswalk to Related Document
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QAPP WORKSHEET #3: DISTRIBUTION LIST

☐ Worksheet Not Applicable (State Reason)

QAPP Recipients	Title	Organization	Telephone Number	Fax Number	E-mail Address	Document Control Number
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Jeanette Dadusc	Federal Facilities Manager	US EPA Region II	212-637-4050		R2fedfac@epamail.epa.gov	
Joseph Schmidl, P.G.	Project Manager	Stone Environmental, Inc.	802-229-5377	802-229-5417	JSchmidl@Stone-Env.com	
Seth Pitkin	Senior Hydrogeologist/Vice President	Stone Environmental, Inc.	802-229-2192	802-229-5417	spitkin@stone-env.com	
David Linari	Field Operations Leader/Site Safety Officer	Stone Environmental, Inc.	802-229-6434	802-229-5417	dlinari@stone-env.com	

QAPP WORKSHEET #3: DISTRIBUTION LIST (Concluded)

QAPP Recipients	Title	Organization	Telephone Number	Fax Number	E-mail Address	Document Control Number
Kim Watson	QA Officer/Health & Safety Officer	Stone Environmental, Inc.	802-229-2196	802-229-5417	kwatson@stone-env.com	
Michael Rossi	Senior Chemist/Laboratory Manager	Stone Environmental, Inc.	802-229-2194	802-229-5417	mrossi@stone-env.com	
Larry Pannell, P.G., P.M.P.	Project Manager	Watermark	978-452-9696	978-453-9988	lawrence.pannell@watermarkenv.com	
Michele Kersey	Project Manager	TestAmerica Laboratories – Savannah	912-354-7858		Betsy.Beauchamp@testamericainc.com	
Phil Dennis	Senior Manager	SiREM Laboratory	519-822-2265 ext. 238	519-822-3151	pdennis@siremlab.com	
Michael Goodfellow.	Owner	New York Leak Detection, Inc.	315-469-4601		mgoodfellow@nyld.com	
Jim Cook	Surveyor	C.T. Male, Inc.	518-786-7605	518-786-7299	j.cook@ctmale.com	
Christopher Anderson	Project Manager	Alpha Analytical	508-898-9220		canderson@alphalab.com	

QAPP WORKSHEET #4: PROJECT PERSONNEL SIGN-OFF SHEET

☐ Worksheet Not Applicable (State Reason)

Organization: U.S. Army Corps of Engineers New England District (NAE)

Project Personnel	Title	Telephone Number	Signature	Date QAPP Read
Gregory Goepfert	Project Manager	917-790-8235		
Dean Brammer, C.H.M.M.	Engineering Technical Lead	978-318-8144		
Ken Heim, Ph.D.	Project Hydrogeologist	978-318-8650		
David Lubianez	Project Chemist	978-318-8311		
Dan Groher, P.E	Innovative Technologies	978-318-8404		
Sheila Harvey	Health & Safety Officer	978-318-8504		
Cynthia Colquitt	Human Health Risk Assessor	978-318-8042		

Organization: Stone Environmental, Inc.

Project Personnel	Title	Telephone Number	Signature	Date QAPP Read
Joseph Schmidl, P.G.	Project Manager	802-229-5377		
Seth Pitkin	Senior Hydrogeologist	802-229-2192		
David Linari	Field Operations Leader/Site Safety Officer	802-229-6434		
Kim Watson	QA Officer/H&S Officer	802-229-2196		
Michael Rossi	Senior Chemist/Laboratory Manager	802-229-2194		

Organization: New York State Department of Environmental Conservation

Project Personnel	Title	Telephone Number	Signature	Date QAPP Read
Jason M. Pelton	Project Manager	518-402-9814		

Organization: New York State Department of Health

Project Personnel	Title	Telephone Number	Signature	Date QAPP Read
Maureen Schuck	Project Manager	518-402-7860		

Organization: Defense Logistics Agency/Defense National Stockpile Center

Project Personnel	Title	Telephone Number	Signature	Date QAPP Read
Lisa Sanders	Project Manager	518-370-3347		

QAPP WORKSHEET #4: PROJECT PERSONNEL SIGN-OFF SHEET (Continued)

Organization: US General Services Agency

Project Personnel	Title	Telephone Number	Signature	Date QAPP Read
David Baker	Property Owner Representative	212-577-7920		
Kenneth Simmons	Construction Control Representative	518-378-8861		

Organization: Galesi Corporation

Project Personnel	Title	Telephone Number	Signature	Date QAPP Read
David Ahl	Project Manager	518-356-4445		

Organization: US EPA Region II

Project Personnel	Title	Telephone Number	Signature	Date QAPP Read
Jeanette Dadusc	Federal Facilities Manager	212-637-4050		

Organization: Watermark

Project Personnel	Title	Telephone Number	Signature	Date QAPP Read
Larry Pannell, P.G., P.M.P.	Project Manager	978-452-9696		

Organization: TestAmerica Laboratories - Savannah

Project Personnel	Title	Telephone Number	Signature	Date QAPP Read
Michele Kersey	Project Manager	912-354-7858		

Organization: Alpha Analytical Laboratory, Inc.

Project Personnel	Title	Telephone Number	Signature	Date QAPP Read
Christopher Anderson	Project Manager	508-898-9220		

Organization: SiREM

Project Personnel	Title	Telephone Number	Signature	Date QAPP Read
Phil Dennis	Senior Manager	519-822-2265 ext. 238		

Organization: New York Leak Detection, Inc.

Project Personnel	Title	Telephone Number	Signature	Date QAPP Read
Michael Goodfellow	Owner	315-469-4601		

Organization: C.T. Male, Inc.

Project Personnel	Title	Telephone Number	Signature	Date QAPP Read
Jim Cook	Surveyor	518-786-7605		

QAPP WORKSHEET #5: PROJECT ORGANIZATIONAL CHART

☐ Worksheet Not Applicable (State Reason)

QAPP WORKSHEET #6: COMMUNICATION PATHWAYS

☐ Worksheet Not Applicable (State Reason)

Communication Drivers	Responsible Entity	Name	Phone Number	Procedure (Timing, Pathways, Etc.)
Point of Contact with CENAE / CENAN	Contractor Project Managers	Seth Pitkin, Joseph Schmidl, P.G	802-229-2192 802-229-5377	All materials and information about the project will be forwarded to Gregory Goepfert
Manage all Project Phases	Contractor Project Managers	Joseph Schmidl, P.G	802-229-5377	Notify Gregory Goepfert of field-related problems by COB the next business day
QAPP changes	QAPP Preparer	Kim Watson	802-229-4600	Will notify about minor changes to the QAPP; prepare QAPP amendments for major changes.
QAPP Amendments	Lead Organization's Project Chemist	David Lubianez	978-318-8021	Any major changes to the QAPP must be approved by David Lubianez before the changes can be implemented.
Daily Field Progress Reports	Sampling Team Leader	Joseph Schmidl, P.G	802-229-5377	Will provide verbal daily field progress reports to Gregory Goepfert
Weekly Field Progress Reports	Field Work Manager	Joseph Schmidl, P.G	802-229-5377	Will provide written reports of weekly field progress to Gregory Goepfert
Recommendations to stop work and initiate corrective action	Field Work Manager	Joseph Schmidl, P.G	802-229-5377	Will Gregory Goepfert where unforeseen circumstances require the stoppage of work and/or immediate remedial actions.
Reporting Lab Data Quality Issues	Laboratory Quality Assurance Officer	Michael Rossi	802-229-2194	All QA/QC issues with project field samples will be reported to the Filed Team Leader by the laboratory QAOs.
Field and Analytical Corrective Actions	Contractor Quality Assurance Officer	Kim Watson	802-229-2196	The need for corrective action for field and analytical issues will be determined.
Release of Analytical Data	Contractor Quality Assurance Officer	Kim Watson	802-229-2196	No analytical data can be released until validation is completed and has approved the release.

QAPP WORKSHEET #7: PERSONNEL RESPONSIBILITIES AND QUALIFICATIONS

☐ Worksheet Not Applicable (State Reason)

Name	Title	Organizational Affiliation	Responsibilities	Education and Experience Qualifications
Gregory Geopfert, P.E.	Lead Organization's Project Manager	CENAN	Oversees project	B.S. Chemical Engineering, M.S. Systems Management, P.E., P.M.P., 22 years experience
Dean Brammer, C.H.M.M	Lead Organization's Technical Manager	CENAE	Supports CENAN PM	B.S. Oceanography, M.S. Forest Hydrology/Water Resources Management, CHMM, 15 years experience
Seth Pitkin	Contractor Project Officer	Stone Environmental, Inc.	Manages project, main point of contact with NAE	B.S. Geology, M.S., Hydrogeology, 25 years experience.
Joseph Schmidl, P.G.	Contractor Project Manager \ Senior Geoscientist	Stone Environmental, Inc.	Manages project, point of contact with NAE, technical hydrogeologic interpretation and review	B.S. Geology, M.S. Environmental Studies, P.G., 22 years experience.
Kim Watson	Contractor QA Officer and H&S Officer	Stone Environmental, Inc.	QA Oversight and Oversees HS for field activities	B.S., Environmental Engineering Technology, RQAP-GLP, 31 years experience.
David Linari	Contractor Field Operations Leader, Site Safety Officer	Stone Environmental, Inc.	Data Collection, Site Health and Safety	B.S. Geo-Environmental Engineering, M.S. Geology, 10 years experience.

QAPP WORKSHEET #8: SPECIAL PERSONNEL TRAINING REQUIREMENTS TABLE

☐ Worksheet Not Applicable (State Reason)

No additional specialized training will be required.

Project Function	Specialized Training- Title or Description of Course	Training Provider	Training Date	Personnel/Groups Receiving Training	Personnel Titles/Organizational Affiliation	Location of Training Records/Certificates
Site Workers	OSHA 1910.120	Worker Specific	Worker-Specific training within the past 14 months	Stone Employees	Seth Pitkin, Project Officer, Stone Joseph Schmidl, Project Manager, Stone Kim Watson, QA/HSO, Stone	Stone Environmental, Inc. 535 Stone Cutters Way, Montpelier, VT 05602

QAPP WORKSHEET #9: PROJECT PLANNING DOCUMENTATION

☐ Worksheet Not Applicable (State Reason)

Project Scoping Session Participants Sheet

Project Name: Pre-Design Investigation **Projected Date(s) of Sampling:** February 2013 **Project Manager:** Joseph Schmidl	**Site Name:** Defense National Stockpile Center Scotia Depot Site **Site Location:** Glenville, NY

Date of Session: 22 August 2012

Scoping Session Purpose: Develop SOW for Pre-Design Investigation

Name	Title	Affiliation	Phone #	E-mail Address	Project Role
Gregory Geopfert	USACE Project Manager	CENAN	917-790-8235	Gregory.J.Goepfert@usace.army.mil	Oversees project
Dean Brammer	USACE Technical Manager	CENAE	978-318-8144	Dean.D.Brammer@usace.army.mil	Supports CENAN PM
Dan Groher	Innovative Technologies	CENAE	978-318-8404	Daniel.M.Groher@usace.army.mil	Supports CENAN PM
Seth Pitkin	Contractor Project Officer	Stone	802-229-2192	spitkin@stone-env.com	Manages Stone's USACE Contract
Joseph Schmidl	Contractor Project Manager \ Geoscientist	Stone	802-229-5377	JSchmidl@Stone-Env.com	Manages project, point of contact with USACE, technical manager
Jason Pelton	Project Manager	NYSDEC	518-402-9814	jmpelton@gw.dec.state.ny.us	Manages project, point of contact with NYSDEC
Joe White	Supervisor	NYSDEC	518-402-9814	ajwhite@gw.dec.state.ny.com	Supervisor, NYSDEC
Michael Mastronardi	Project Manager	DLA / DNSC	518-370-3347	Michael.mastronardi@dla.mil	Property Owner Representative
David Ahl	Project Manager	Galesi Corp.	518-356-4445	dahl@galesi.com	Property Owner Representative
Steve Cox		GSA	518-431-0311	Stephen.cox2@gsa.gov	Property Owner Representative
David Baker	Property Owner Representative	GSA	212-577-7920	David.baker@gsa.gov	Property Owner Representative
Kenneth Simmons	Construction Control Representative	GSA	518-378-8861	Kenneth.simmons@gsa.gov	Construction Control Representative

QAPP WORKSHEET #9: PROJECT PLANNING DOCUMENTATION (Concluded)

Comments/Decisions: Discussed the selected remedy and available data. Identified data gaps, particularly the 3-dimensional extent of the plume and internal concentrations and many geotechnical and geochemical parameters. Decided available data are insufficient to locate and design the selected remedy. Walked Site to assess access and other technical issues. Obtained as-built drawings to support subsurface utility clearance.

Action Items: CENAE to prepare the SOW, to include actions to eliminate as many data gaps as possible under the available budget.

Consensus Decisions: Decided a pre-design investigation to locate and design the selected remedy is feasible.

QAPP WORKSHEET #10: PROBLEM DEFINITION

☐ Worksheet Not Applicable (State Reason)

The problem to be addressed by the project:

1. Perform Remedial Design (RD) pre-design investigations to verify the nature and extent of contamination described in the Expanded Site Investigation Report (NYSDEC/GeoLogic, 2007) and the Feasibility Study (FS) Report (Malcolm Pirnie, 2010), including:
   Additional investigation to verify and further define the horizontal and vertical extent of the TCE plume;
   a. Focused investigations to verify the location and dimensions of the selected remedy; and,
   b. Investigations of the soil vapor intrusion pathway for onsite buildings.
2. Collect the data necessary to support the design of the selected remedy in the Record of Decision (NYSDEC, 2010)..

The environmental questions being asked:

What is the nature and extent of contamination, particularly the 3-dimensional extent of the plume and internal plume concentrations?
What are the geotechnical and geochemical parameters of the plume and aquifer?
What is the optimal location of the selected remedy?
What are the design specifications and estimated costs of the selected remedy?

A synopsis of secondary data or information from Site reports:

In response to the detection of TCE at concentrations below NYSDEC groundwater standards in the Town of Rotterdam and City of Schenectady well fields, NYSDOH performed private water supply sampling in 1991. The private water supply sampling included residences located on NYS Route 5 in the Town of Glenville and hydraulically downgradient of the Site. Volatile organic compounds (VOCs), including TCE, 1,1,1-TCA, and PCE, were detected in groundwater collected in some of the residential wells. Consistent with the groundwater contamination at the Site, TCE was the primary VOC detected in the residential well water samples (at concentrations up to 320 ppb). The homes on NYS Route 5 were subsequently connected to public water provided by the Town of Glenville.

Six subsurface investigations were completed to identify the possible source of TCE in the residential wells and to delineate the extent of the TCE groundwater plume between 1995 and 2007, and focused on the assemblage of properties comprising the former 337-acre Defense National Stockpile Center Scotia Depot. During the investigations, two areas thought to represent possible TCE source areas, including a former burn pit and the Sacandaga Road Landfill were evaluated. Data suggested that although these areas may be contributing to minor amounts of groundwater contamination, they do not represent TCE source areas. Instead, investigation data indicated that TCE disposal occurred in the area near the north corner of the Site.

A site assessment was completed by the Defense National Stockpile Center in advance of transferring unused portions of the site back to the GSA. Based on the site assessment, soil with metals present above background concentrations was excavated from two former metals and ore stockpile areas and two drywells were removed as part of interim remedial measures in 2005.

Additional details are provided in Section 2 of the Work Plan.

The possible classes of contaminants and the affected matrices:

Chlorinated volatile organic compounds (CVOCs) in soils, soil vapor and groundwater.

QAPP WORKSHEET #11: PROJECT QUALITY OBJECTIVES/SYSTEMATIC PLANNING PROCESS STATEMENTS

☐ Worksheet Not Applicable (State Reason)

Who will use the data?
Data will be used by USACE, NYSDEC, NYSDOH, Stone Environmental, Inc., and Stone team subcontractors.

What will the data be used for?
The data collected during the pre-design investigation will be used to document the nature and extent of contamination related to the Site and to determine the optimum location for the selected remedy. Data generated from the treatability study will be used to determine parameters required for the selected remedy to address the contamination, for design and cost estimation purposes.

What types of data are needed? (target analytes, analytical groups, field screening, on-site analytical or off-site laboratory techniques, sampling techniques)
The investigation will include the following data:

- Groundwater Quality: VOCs.
- Groundwater Geochemistry: total organic carbon/total dissolved carbon, total dissolved solids/total suspended solids, oil and grease, pH, hardness, ferrous/ferric iron, manganous/manganic manganese, nitrate/nitrite, sulfate/sulfite, total metals (including chromium, iron, manganese, silicon, and titanium), hexavalent/trivalent chromium, and carbonate. Fixed laboratory analysis will be conducted.
- Subsurface Soil Geotechnical Properties: Grain size, bulk density, effective porosity. Fixed laboratory analysis will be conducted.
- Treatability Testing: Bench-scale treatability testing to evaluate the effectiveness of the selected remedy, and to develop design parameters for the selected remedy. Fixed laboratory analysis will be conducted.
- Soil Vapor : Sub-slab soil vapor and indoor air samples will be collected from 6 onsite buildings and analyzed for CVOCs by a fixed laboratory.
- Nature and Extent of the dissolved-phase groundwater plume: Mobile laboratory analysis will be conducted to screen vertical groundwater profile samples for selected CVOCs; 10% of these samples will be submitted for split analysis by a fixed laboratory.
- Standard operating procedures will be used for sample collection, handling, sample preparation, and analytical methods.
- All samples will be grab samples.
- All groundwater be sampled within as short a time frame as possible in order to provide synchronous results.

How "good" do the data need to be in order to support the environmental decision?
Fixed laboratory data needs to meet NYSDEC, NYSDOH and USACE requirements for data quality. The screening data need to be of sufficient quality to support the design of the selected remedy in the ROD.

QAPP WORKSHEET #11: PROJECT QUALITY OBJECTIVES/SYSTEMATIC PLANNING PROCESS STATEMENTS (Concluded)

☐ Worksheet Not Applicable (State Reason)

How much data are needed? (number of samples for each analytical group, matrix, and concentration)

Groundwater Samples:

- Existing Monitoring Wells: 26 for VOCs, 5 for total organic carbon/total dissolved carbon, total dissolved solids/total suspended solids, oil and grease, pH, hardness, ferrous/ferric iron, manganous/manganic manganese, nitrate/nitrite, sulfate/sulfite, total metals (including chromium, iron, manganese, silicon, and titanium), hexavalent/trivalent chromium, and carbonate
- Vertical Groundwater Profiling 170 for CVOCs (Mobile Laboratory); 17 for VOCs (Fixed Laboartory).

Subsurface Soil: 5 for grain size, bulk density, effective porosity (Fixed Laboartory).

Soil Vapor / Indoor Air: Buildings 403 and 404 will each have 2 soil vapor samples collected; Buildings 201 through 204 will have 2-3 indoor air and 2-3 soil vapor samples collected per building. Two ambient outdoor air samples will be collected to serve as background.

IDW: To be determined number for soil and water VOC analyses (Fixed Laboratory).

Where, when, and how should the data be collected/generated?

Collection techniques include (numbers cross-reference to Worksheet #12):

1. Soil Sampling
2. Groundwater Sampling
3. Soil Vapor / Indoor Air Sampling

Who will collect and generate the data?

Stone Environmental, Inc. and subcontracted laboratories.

How will the data be reported?

The soil and groundwater data will be included in the Pre-Design Investigation Report. The soil vapor data will be presented in a separate report. The laboratories will submit analytical results in SEDD format.

How will the data be archived?

All original data will be electronically filed by Stone and retained for a period of ten years after the project has been closed. Final reports will be stored at a project repository.

QAPP WORKSHEET #12: MEASUREMENT PERFORMANCE CRITERIA

☐ Worksheet Not Applicable (State Reason)

Matrix	Aqueous and Soil Samples
Analytical Group	VOCs (Fixed Lab)
Concentration Level	Low to High

Sampling Procedure[1]	**Analytical Method/SOP[2]**	**Data Indicators (DQIs)**	**Measurement Performance Criteria**	**QC Sample and/or Activity Used to Assess Measurement Performance**	**QC Sample Assesses Error for Sampling (S), Analytical (A) or Both (S&A)**
1, 2	EPA Method 8260B / 13b	Precision	Relative percent difference (RPD) ≤ 30 to 50%	Laboratory Duplicates	A
		Precision	RPD ≤ 30 to 50%[3]	Matrix Spike/Matrix Spike Duplicate (MS/MSD)	A
		Accuracy/Bias	Laboratory control limits[3]	Lab Control Sample (LCS)	A
		Precision-Overall/ Representativeness	RPD ≤ 30% (aqueous), 50% (soils)	Field Duplicates	S&A
		Accuracy/Bias	No reported concentrations above quantitation limits	Trip blank	S&A
		Accuracy/Bias	No reported concentrations above quantitation limits	Equipment blank	S
		Accuracy/Bias	No reported concentrations above ½ quantitation limits	Method blank	A
		Accuracy	Laboratory control limits[3]	Matrix Spike	A
		Accuracy	Laboratory control limits[3]	Surrogate: 4-Bromofluorobenzene Dibromofluoromethane Toluene-d8	A

Note 1: See Worksheet #11 for cross-reference to sampling procedure.
Note 2: See Worksheet #23 for cross-reference to analytical method SOP.
Note 3: LCS and MS/MSD control limits will default to the DoD limits defined in the QSM.

QAPP WORKSHEET #12: MEASUREMENT PERFORMANCE CRITERIA (Continued)

Matrix	Aqueous Samples
Analytical Group	VOCs (SEI-Mobile)
Concentration Level	Low to High

Sampling Procedure[1]	Analytical Method/SOP[2]	Data Indicators (DQIs)	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or Both (S&A)
2	EPA Method 8260C / 14b, 15b	Precision	Relative percent difference (RPD) ≤ 30%	Laboratory Duplicates	A
		Precision	RPD ≤ 30%	Matrix Spike/Matrix Spike Duplicate (MS/MSD)	A
		Accuracy/Bias	Laboratory Control Limits[3]	Lab Control Sample (LCS)	A
		Precision-Overall/ Representativeness	RPD ≤ 30%	Field Duplicates	S&A
		Accuracy/Bias	No reported concentrations above quantitation limits	Trip blank	S&A
		Accuracy/Bias	No reported concentrations above quantitation limits	Equipment blank	S
		Accuracy/Bias	No reported concentrations above ½ quantitation limits	Method blank	A
		Accuracy	50 to 150% recovery[3]	Matrix Spike	A
		Accuracy	75 to 120% recovery	Surrogate: 4-Bromofluorobenzene	A

Note 1: See Worksheet #11 for cross-reference to sampling procedure.
Note 2: See Worksheet #23 for cross-reference to analytical method SOP.
Note 3: LCS and MS/MSD control limits will default to the DoD limits defined in the QSM.

QAPP WORKSHEET #12: MEASUREMENT PERFORMANCE CRITERIA (Continued)

Matrix	Soil Samples
Analytical Group	Geotechnical Parameters (Fixed Lab)
Concentration Level	Not applicable

Sampling Procedure[1]	**Analytical Method/SOP**[2]	**Data Indicators (DQIs)**	**Measurement Performance Criteria**	**QC Sample and/or Activity Used to Assess Measurement Performance**	**QC Sample Assesses Error for Sampling (S), Analytical (A) or Both (S&A)**
1	Grain Size by ASTM Method D422/2b Bulk Density by ASTM Method D2937/3b Effective Porosity by ASTM Method D7263 / 3b	Precision	RPD ≤ 20%	Laboratory Duplicates	A
		Accuracy/Bias	70 to 130% recovery[3]	Reference Sample (if applicable)	A
		Precision-Overall/ Representativeness	RPD ≤ 30%	Field Duplicates	S&A
		Accuracy/Bias	NA	Equipment blank	S
		Accuracy/Bias	No reported concentrations above quantitation limits	Method blank	A

Note 1: See Worksheet #11 for cross-reference to sampling procedure.
Note 2: See Worksheet #23 for cross-reference to analytical method SOP.
Note 3: LCS control limits may be compound-specific; individual control limits are provided in laboratory SOPs.

QAPP WORKSHEET #12: MEASUREMENT PERFORMANCE CRITERIA (Continued)

Matrix	Aqueous Samples
Analytical Group	Total Metals (Fixed Lab)
Concentration Level	Low

Sampling Procedure[1]	**Analytical Method/SOP**[2]	**Data Indicators (DQIs)**	**Measurement Performance Criteria**	**QC Sample and/or Activity Used to Assess Measurement Performance**	**QC Sample Assesses Error for Sampling (S), Analytical (A) or Both (S&A)**
2	Chromium, Iron, Magnesium, Manganese, Silicon, and Titanium by EPA Method 6010B / 4b Hexavalent and Trivalent Chromium by EPA Method 7196A / 4b	Precision	RPD ≤ 20%	Laboratory Duplicates	A
		Accuracy/Bias	80 to 120% recovery[3]	LCS	A
		Precision-Overall/ Representativeness	RPD ≤ 30%	Field Duplicates	S&A
		Accuracy/Bias	No reported concentrations above quantitation limits	Equipment blank	S
		Accuracy/Bias	No reported concentrations above quantitation limits	Method blank	A
		Accuracy/Bias	75 to 125% recovery[3]	Matrix Spike	A

Note 1: See Worksheet #11 for cross-reference to sampling procedure.
Note 2: See Worksheet #23 for cross-reference to analytical method SOP.
Note 3: LCS and MS control limits will default to the DoD limits defined in the QSM.

QAPP WORKSHEET #12: MEASUREMENT PERFORMANCE CRITERIA (Continued)

Matrix	Aqueous Samples
Analytical Group	General Chemistry (Fixed Lab)
Concentration Level	Low

Sampling Procedure[1]	**Analytical Method/SOP**[2]	**Data Indicators (DQIs)**	**Measurement Performance Criteria**	**QC Sample and/or Activity Used to Assess Measurement Performance**	**QC Sample Assesses Error for Sampling (S), Analytical (A) or Both (S&**
2	Total Dissolved Solids by Method SM 2540C / 7b Total Suspended Solids by Method 2540D / 7b pH by EPA Method 9040B / 8b Hardness by SM 2340C / 9b Ferrous and Ferric Iron by ASTM Method D3872-05 (2011) / 4b Carbonate by EPA Method 2320B / 9b	Precision	RPD ≤ 25%	Laboratory Duplicates	A
		Accuracy/Bias	80 to 120% recovery[3]	LCS	A
		Precision-Overall/ Representativeness	RPD ≤ 30%	Field Duplicates	S&A
		Accuracy/Bias	No reported concentrations above ½ quantitation limits	Method blank	A

Note 1: See Worksheet #11 for cross-reference to sampling procedure. Note 2: See Worksheet #23 for cross-reference to analytical method SOP.
Note 3: LCS control limits may be compound-specific; individual control limits are provided in laboratory SOPs.

QAPP WORKSHEET #12: MEASUREMENT PERFORMANCE CRITERIA (Continued)

Matrix	Aqueous Samples
Analytical Group	General Chemistry (Fixed Lab)
Concentration Level	Low

Sampling Procedure[1]	**Analytical Method/SOP**[2]	**Data Indicators (DQIs)**	**Measurement Performance Criteria**	**QC Sample and/or Activity Used to Assess Measurement Performance**	**QC Sample Assesses Error for Sampling (S), Analytical (A) or Both (S&**
2	Manganous and Manganic Manganese by EPA Method 1638, Modified / 16b	Precision	RPD ≤ 20%	Laboratory Duplicates	A
		Accuracy/Bias	75 to 125% recovery	LCS	A
		Precision-Overall/ Representativeness	RPD ≤ 30%	Field Duplicates	S&A
		Accuracy/Bias	No reported concentrations above ½ quantitation limits	Method blank	A
		Accuracy/Bias	75 to 125% recovery	Matrix Spike	A

Note 1: See Worksheet #11 for cross-reference to sampling procedure.
Note 2: See Worksheet #23 for cross-reference to analytical method SOP.

QAPP WORKSHEET #12: MEASUREMENT PERFORMANCE CRITERIA (Continued)

Matrix	Aqueous Samples
Analytical Group	Total Organic Carbon / Total Dissolved Carbon (Fixed Lab)
Concentration Level	Low

Sampling Procedure[1]	**Analytical Method/SOP**[2]	**Data Indicators (DQIs)**	**Measurement Performance Criteria**	**QC Sample and/or Activity Used to Assess Measurement Performance**	**QC Sample Assesses Error for Sampling (S), Analytical (A) or Both (S&A)**
2	EPA Method 9060A / 10b	Precision	RPD ≤ 25%	Laboratory Duplicates	A
		Accuracy/Bias	80 to 120% recovery	LCS	A
		Precision-Overall/ Representativeness	RPD ≤ 30%	Field Duplicates	S&A
		Accuracy/Bias	No reported concentrations above quantitation limits	Equipment blank	S
		Accuracy/Bias	No reported concentrations above quantitation limits	Method blank	A
		Accuracy/Bias	RPD ≤ 25%	Matrix Spike	A

Note 1: See Worksheet #11 for cross-reference to sampling procedure.
Note 2: See Worksheet #23 for cross-reference to analytical method SOP.

QAPP WORKSHEET #12: MEASUREMENT PERFORMANCE CRITERIA (Continued)

Matrix	Aqueous Samples
Analytical Group	Oil & Grease (Fixed Lab)
Concentration Level	Low

Sampling Procedure[1]	**Analytical Method/SOP**[2]	**Data Indicators (DQIs)**	**Measurement Performance Criteria**	**QC Sample and/or Activity Used to Assess Measurement Performance**	**QC Sample Assesses Error for Sampling (S), Analytical (A) or Both (S&A)**
2	Oil & Grease by EPA Method 1664A	Precision	RPD ≤ 18%	Laboratory Duplicates	A
		Accuracy/Bias	78 to 114% recovery	LCS	A
		Precision-Overall/ Representativeness	RPD ≤ 30%	Field Duplicates	S&A
		Accuracy/Bias	No reported concentrations above quantitation limits	Equipment blank	S
		Accuracy/Bias	No reported concentrations above ½ quantitation limits	Method blank	A
		Accuracy/Bias	78 to 114% recovery	Matrix Spike	A

Note 1: See Worksheet #11 for cross-reference to sampling procedure.
Note 2: See Worksheet #23 for cross-reference to analytical method SOP.

QAPP WORKSHEET #12: MEASUREMENT PERFORMANCE CRITERIA (Continued)

Matrix	Aqueous Samples
Analytical Group	General Chemistry (Fixed Lab)
Concentration Level	Low

Sampling Procedure[1]	Analytical Method/SOP[2]	Data Indicators (DQIs)	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or Both (S&A)
2	Nitrate / Nitrite by EPA Method 300.0 / 5b Sulfate / Sulfite by EPA Method 300.0 /5b	Accuracy/Bias	90 to 110% recovery	LCS	A
		Precision-Overall/ Representativeness	RPD ≤ 30%	Field Duplicates	S&A
		Accuracy/Bias	No reported concentrations above quantitation limits	Equipment blank	S
		Accuracy/Bias	No reported concentrations above ½ quantitation limits	Method blank	A
		Accuracy/Bias	90 to 110% recovery	Matrix Spike	A

Note 1: See Worksheet #11 for cross-reference to sampling procedure.
Note 2: See Worksheet #23 for cross-reference to analytical method SOP.

QAPP WORKSHEET #12: MEASUREMENT PERFORMANCE CRITERIA (Concluded)

☐ Worksheet Not Applicable (State Reason)

Matrix	Air
Analytical Group	VOCs (Fixed Lab)
Concentration Level	Low to High

Sampling Procedure[1]	**Analytical Method/SOP**[2]	**Data Indicators (DQIs)**	**Measurement Performance Criteria**	**QC Sample and/or Activity Used to Assess Measurement Performance**	**QC Sample Assesses Error for Sampling (S), Analytical (A) or Both (S&A)**
3	EPA Method TO-15 / 18b	Precision	Relative percent difference (RPD) ≤ 25%	Laboratory Duplicates	A
		Precision	RPD ≤ 25%[3]	Matrix Spike/Matrix Spike Duplicate (MS/MSD)	A
		Accuracy/Bias	Recovery 70 to 130%[3]	Lab Control Sample (LCS)	A
		Precision-Overall/ Representativeness	RPD ≤ 50%	Field Duplicates	S&A
		Accuracy/Bias	No reported concentrations above quantitation limits	Trip blank	S&A
		Accuracy/Bias	No reported concentrations above quantitation limits	Equipment blank	S
		Accuracy/Bias	No reported concentrations above ½ quantitation limits	Method blank	A
		Accuracy/Bias	Laboratory control limits[3]	Matrix Spike	A
		Accuracy	Laboratory control limits[3]	Surrogates: Bromofluorobenzene, Toluene-d8 1,2-Dichloroethane-d4	A

Note 1: See Worksheet #11 for cross-reference to sampling procedure.
Note 2: See Worksheet #23 for cross-reference to analytical method SOP.
Note 3: LCS and MS/MSD control limits will default to the DoD limits defined in the QSM.

QAPP WORKSHEET #13: SECONDARY DATA CRITERIA AND LIMITATIONS TABLE

☐ Worksheet Not Applicable (State Reason)

Secondary Data	Data Source (Originating Organization, Report Title, and Date)	Data Generator(s) (Originating Org., Data Types, Data Generation/ Collection Dates)	How Data Will Be Used	Limitations on Data Use
Groundwater VOC Screening	Stone, *Pre-Design Investigation Report*, 2013	Stone, 2013	Optimize selected remedy location	Not NYSDEC-certified laboratory
Soil headspace screening	Stone, *Pre-Design Investigation Report*, 2013	Stone, 2013	Facilitate geochemical sample selection	Qualitative
Soil headspace screening	Stone, *Pre-Design Investigation Report*, 2013	Stone, 2013	Determine soil vapor sample representativeness	Qualitative

QAPP WORKSHEET #14: SUMMARY OF PROJECT TASKS

☐ Worksheet Not Applicable (State Reason)

Sampling Tasks:

1) Groundwater:
   a) Sample and collect synoptic groundwater elevation measurements for 26 existing wells for VOCs (Subtasks 4A and 4B, Project Plan).
   b) Sample additional monitoring wells, to be installed (Subtask 5F, Project Plan), for VOCs and groundwater elevation data.
   c) Collect groundwater samples in 15 vertical groundwater profile locations (Subtasks 5A and 5C (Optional), and Task 9 (Optional), Project Plan) for CVOC screening analysis (Mobile lab), and 10% confirmatory analysis (Fixed lab).
   d) Collect 6 groundwater samples from monitoring wells in the vicinity of the proposed remedy for geochemical parameters (total organic carbon/total dissolved carbon, total dissolved solids/total suspended solids, oil and grease, pH, hardness, ferrous/ferric iron, manganous/manganic manganese, nitrate/nitrite, sulfate/sulfite, total metals (including chromium, iron, manganese, silicon, and titanium), hexavalent/trivalent chromium, and carbonate) (Subtask 6A, Project Plan).
2) Subsurface Soil: Sample subsurface soil for geotechnical parameters (grain size, bulk density, and effective porosity) (Subtask 5B (Optional), Project Plan).
3) Soil Vapor and Indoor Air: Sample soil vapor and indoor air for CVOCs (Subtasks 12B and C (Optional), Project Plan).

Analysis Tasks:

Stone Environmental, Inc. will analyze CVOCs in a Mobile laboratory, and perform field analyses of natural attenuation parameters. TestAmerica-Savannah., will perform Fixed laboratory analysis of soil and aqueous samples. Alpha Analytical Laboratory will perform Fixed laboratory analysis of air samples

Quality Control Tasks:

Collect duplicate samples for aqueous and soil samples. Collect matrix spike and matrix-spike samples for aqueous samples. Conduct confirmatory analysis of split groundwater screening (Mobile lab) samples.

Secondary Data:

Secondary data will be used at a screening level only.

Data Management Tasks:

Analytical data will be delivered in SEDD format, and after review using ADR software, will be submitted to the CENAE EDMS database.

Documentation and Records:

All new sampling locations will be marked for survey. Field forms will be used to record field sampling data, screening data and conditions at each sampling location. All chains of custody and shipping documents will be retained for the samples.
Laboratory data packages will include a level B reporting package. All documents will be filed in electronic format on Stone's secure local area network, which is backed up daily. Final documents will be stored at a Site specific repository.

Assessment/Audit Tasks:

A field sample collection audit will be performed during the field work.

Data Review Tasks:

Data and all related field logs/notes/records will be reviewed in ADR to assess total measurement error and determine overall usability of the data for project purposes. Data limitations will be determined and data will be compared to Project Quality Objectives and required method detection limits. Corrective action is initiated by the laboratory, as necessary. Final data are placed in database, with any necessary qualifiers, and tables, charts, and graphs are generated.

QAPP WORKSHEET #15: REFERENCE LIMITS AND EVALUATION TABLE

☐ Worksheet Not Applicable (State Reason)

Matrix	Aqueous
Analytical Group	VOCs via EPA Method 8260B (Fixed)
Concentration Level	Low to High

Analyte	CAS Number	Project Action Limit (µg/L)		Project Quantitation Limit (µg/L)	Achievable Laboratory Detection Limits (µg/L)	
		EPA Region 3 RSL[1]	NYSDEC Class GA[2]		Limits of Quantitation	Detection Limits
Acetone	64-67-1	12,000	--	25	5	25
Benzene	71-43-2	0.39	1	1.0	0.25	1.0
Bromochloromethane	74-97-5	83	5*	1.0	0.25	1.0
Bromoform	75-25-2	7.9	--	1.0	0.5	1.0
Bromomethane	74-83-9	7.0	5*	1.0	0.8	1.0
2-Butanone (Methyl Ethyl Ketone)	78-93-3	4,900	--	10	1.0	10
Carbon Disulfide	75-15-0	720	60	2.0	0.6	2.0
Carbon Tetrachloride	56-23-5	0.39	5	1.0	0.5	1.0
Chlorobenzene	108-90-7	72	5*	1.0	0.25	1.0
Chloroethane	75-00-3	--	5*	1.0	1.0	1.0
Chloroform	67-66-3	0.19	7	1.0	0.14	1.0
Chloromethane	74-87-3	190	--	1.0	0.33	1.0
Cyclohexane	110-82-7	--	--	1.0	0.25	1.0
Dibromochloromethane	124-48-1	0.15	5*	1.0	0.1	1.0
1,2-Dibromo-3-Chloropropane	96-12-8	0.00032	0.04	1.0	0.44	1.0
1,2-Dibromoethane	106-93-4	0.0065	--	1.0	0.25	1.0
1,2-Dichlorobenzene	95-50-1	280	3[4]	1.0	0.21	1.0
1,3-Dichlorobenzene	154-73-1	--	3[4]	1.0	0.25	1.0
1,4-Dichlorobenzene	106-46-7	0.42	3[4]	1.0	0.28	1.0
Dichlorodifluoromethane		190	5*	1.0	0.25	1.0
1,1-Dichloroethane	75-34-3	2.4	5*	1.0	0.25	1.0
1,2-Dichloroethane	107-06-2	0.15	0.6	1.0	0.1	1.0
1,1-Dichloroethene	75-35-4	260	5*	1.0	0.11	1.0
1,2-Dichloropropane	78-87-5	0.38	--	1.0	0.13	1.0
cis-1,2-Dichloroethene	156-59-2	28	5*	1.0	0.15	1.0

Matrix	Aqueous
Analytical Group	VOCs via EPA Method 8260B (Fixed)
Concentration Level	Low to High

Analyte	CAS Number	Project Action Limit (µg/L)		Project Quantitation Limit (µg/L)	Achievable Laboratory Detection Limits (µg/L)	
		EPA Region 3 RSL[1]	NYSDEC Class GA[2]		Limits of Quantitation	Detection Limits
trans-1,2-Dichloroethene	166-60-5	86	5*	1.0	0.1	1.0
cis-1,3-Dichloropropene	10061-01-5	0.41[3]	0.4[3]	1.0	0.11	1.0
trans-1,3-Dichloropropene	10061-02-6	0.41[3]	0.4[3]	1.0	0.21	1.0
Ethylbenzene	100-41-4	1.3	5*	1.0	0.11	1.0
2-Hexanone	591-78-6	34	--	10	1.0	10
Isopropylbenzene (Cumene)	98-82-8	390	5*	1.0	0.1	1.0
Methyl acetate	79-20-9	16,000	--	1.0	0.19	1.0
Methylcyclohexane	108-87-2	--	--	1.0	0.1	1.0
4-Methyl-2-Pentanone	108-10-1	1,000	--	10	1.0	10
Methyl tert-Butyl Ether	1634-04-4	12	--	10	0.2	10
Methylene Chloride	79-09-2	--	5*	5.0	1.0	5.0
Styrene	100-42-5	1,100	5*	1.0	0.11	1.0
1,1,2,2-Tetrachloroethane	79-34-5	0.066	5*	1.0	0.18	1.0
Tetrachloroethene	127-18-4	9.7	5*	1.0	0.15	1.0
Toluene	108-88-3	860	5*	1.0	0.33	1.0
1,2,4-Trichlorobenzene	120-82-1	0.99	5*	1.0	0.25	1.0
1,1,1-Trichloroethane	71-55-6	7,500	5*	1.0	0.5	1.0
1,1,2-Trichloroethane	79-00-5	0.24	1	1.0	0.13	1.0
Trichloroethene	79-01-6	0.44	5*	1.0	0.13	1.0
Trichlorofluoromethane	75-69-4	1,100	5*	1.0	0.25	1.0
1,1,2-Trichloro-1,2,2-trifluoroethane	76-13-1	53,000	5*	1.0	0.5	1.0
Vinyl Chloride	75-01-4	0.015	2	1.0	0.18	1.0
Total Xylenes	1330-20-7	190	5*	2.0	0.2	2.0

Note 1: U.S. Environmental Protection Agency, Region 3 Regional Screening Level, dated May 2008, for tap water.
Note 2: NYDEC Groundwater Quality Standards for Class GA groundwater. Note 3: Value for total 1,3-dichloropropene used.
*: The principal organic contaminant standard for groundwater of 5 µg/L applies to this substance.
The three analytes with reporting limits and method detection limits above their Project Action Level are not project contaminants of concern (1,2-Dibromo-3-Chloropropane, 1,2-Dibromoethane, and Vinyl Chloride).

QAPP WORKSHEET #15: REFERENCE LIMITS AND EVALUATION TABLE (Continued)

Matrix	Soil
Analytical Group	VOCs via EPA Method 8260B (Fixed)
Concentration Level	Low to High

Analyte	CAS Number	Project Action Limit (µg/Kg)		Project Quantitation Limit (µg/Kg)	Achievable Laboratory Detection Limits (µg/Kg)	
		EPA Region 3 RSL[1]	NYSDEC SCO [2, 3]		Limits of Quantitation	Detection Limits
Acetone	64-67-1	6.1×10^7	50	50	11	50
Benzene	71-43-2	1,100	60	5.0	0.73	5.0
Bromochloromethane	74-97-5	160,000	--	5.0	0.97	5.0
Bromoform	75-25-2	62,000	--	5.0	1.5	5.0
Bromomethane	74-83-9	7,300	--	5.0	1.5	5.0
2-Butanone (Methyl Ethyl Ketone)	78-93-3	2.8×10^7	*300*	25	2.4	25
Carbon Disulfide	75-15-0	820,000	*2,700*	5.0	1.1	5.0
Carbon Tetrachloride	56-23-5	610	760	5.0	0.83	5.0
Chlorobenzene	108-90-7	290,000	1,100	5.0	0.96	5.0
Chloroethane	75-00-3	--	--	5.0	2.7	5.0
Chloroform	67-66-3	290	370	5.0	1.1	5.0
Chloromethane	74-87-3	120,000	--	5.0	1.0	5.0
Cyclohexane	110-82-7	7×10^6	--	10	1.3	10
Dibromochloromethane	124-48-1	680	--	5.0	1.7	5.0
1,2-Dibromo-3-Chloropropane	96-12-8	5.4	--	10	4.4	10
1,2-Dibromoethane	106-93-4	34	--	5.0	1.5	5.0
1,2-Dichlorobenzene	95-50-1	1.9×10^6	1,100	5.0	1.3	5.0
1,3-Dichlorobenzene	154-73-1	--	2,400	5.0	1.6	5.0
1,4-Dichlorobenzene	106-46-7	2,400	1,800	5.0	0.74	5.0
Dichlorodifluoromethane	75-71-8	94,000	--	5.0	0.94	5.0
1,1-Dichloroethane	75-34-3	3,300	270	5.0	1.1	5.0
1,2-Dichloroethane	107-06-2	430	20	5.0	1.1	5.0
1,1-Dichloroethene	75-35-4	240,000	330	5.0	1.5	5.0
1,2-Dichloropropane	78-87-5	940	--	5.0	086	5.0
cis-1,2-Dichloroethene	156-59-2	160,000	250	5.0	1.4	5.0
trans-1,2-Dichloroethene	166-60-5	150,000	190	5.0	0.63	5.0

Matrix	Soil
Analytical Group	VOCs via EPA Method 8260B (Fixed)
Concentration Level	Low to High

Analyte	CAS Number	Project Action Limit (µg/Kg)		Project Quantitation Limit (µg/Kg)	Achievable Laboratory Detection Limits (µg/Kg)	
		EPA Region 3 RSL[1]	NYSDEC SCO [2, 3]		Limits of Quantitation	Detection Limits
cis-1,3-Dichloropropene	10061-01-5	1,700[4]	--	5.0	0.83	5.0
trans-1,3-Dichloropropene	10061-02-6	1,700[4]	--	5.0	0.87	5.0
Ethylbenzene	100-41-4	5,400	1,000	5.0	1.3	5.0
2-Hexanone	591-78-6	210,000		25	3.3	25
Isopropylbenzene (Cumene)	98-82-8	210,000	*2,300*	5.0	1.9	5.0
4-Isopropyltoluene	99-87-6	--	--	10	5.0	10
Methyl acetate	79-20-9	7.8×10^7	--	10	0.86	10
Methylcyclohexane	108-87-2	--	--	5.0	0.98	5.0
4-Methyl-2-Pentanone	108-10-1	5.3×10^6	*1,000*	25	4.2	25
Methyl tert-Butyl Ether	1634-04-4	43,000	930	10	1.0	10
Styrene	100-42-5	6.3×10^6	--	5.0	0.93	5.0
1,1,2,2-Tetrachloroethane	79-34-5	560	600	5.0	1.6	5.0
Tetrachloroethene	127-18-4	22,000	1,300	5.0	1.9	5.0
Toluene	108-88-3	5×10^6	700	5.0	0.84	5.0
1,2,4-Trichlorobenzene	120-82-1	22,000	*3,400*	5.0	0.89	5.0
1,1,1-Trichloroethane	71-55-6	8.7×10^6	*680*	5.0	0.59	5.0
1,1,2-Trichloroethane	79-00-5	1,100	--	5.0	1.3	5.0
Trichloroethene	79-01-6	910	470	5.0	1.3	5.0
Trichlorofluoromethane	75-69-4	790,000	--	5.0	1.2	5.0
1,1,2-Trichloro-1,2,2-trifluoroethane	76-13-1	4.3×10^7	*6,000*	5.0	1.3	5.0
Vinyl Chloride	75-01-4	6	20	5.0	1.5	5.0
Total Xylenes	1330-20-7	630,000	260	10	1.1	10

Note 1: U.S. Environmental Protection Agency, Region 3 Regional Screening Level, dated May 2008, for residential soil.
Note 2: NYDEC Soil Cleanup Objective.
Note 3: [in *Italics*] NYDEC Supplemental Soil Cleanup Objectives for residential soils or protection of groundwater, whichever is lower.
Note 4: Value for total 1,3-dichloropropene used.
The only analyte with a reporting limit above its Project Action Level has a method detection limit below its Project Action Level (1,2-Dibromo-3-Chloropropane).

QAPP WORKSHEET #15: REFERENCE LIMITS AND EVALUATION TABLE (Continued)

Matrix	Aqueous
Analytical Group	VOCs via EPA Method 8260C modified (SEI-Mobile)
Concentration Level	Low to High

Analyte	CAS Number	Project Action Limit (µg/L)		Project Quantitation Limit (µg/L)	Achievable Laboratory Detection Limits (µg/L)	
		EPA Region 3 RSL[1]	NYSDEC Class GA[2]		Limits of Quantitation	Detection Limits
Benzene	71-43-2	0.39	1.0	2.0	0.20	2.0
Carbon Tetrachloride	56-23-5	0.39	5.0	2.0	0.54	2.0
Chloroform	67-66-3	0.19	7.0	2.0	0.19	2.0
1,1-Dichloroethane	75-34-3	2.4	5*	2.0	0.12	2.0
1,2-Dichloroethane	107-06-2	0.15	0.6	2.0	0.30	2.0
1,1-Dichloroethene	75-35-4	7.0	5*	2.0	0.15	2.0
cis-1,2-Dichloroethene	156-59-2	70	5*	2.0	0.10	2.0
trans-1,2-Dichloroethene	156-60-5	100	5*	2.0	0.32	2.0
Ethylbenzene	100-41-4	700	5*	2.0	0.12	2.0
Naphthalene	91-20-3	--	--	2.0	0.46	2.0
1,1,2,2-Tetrachloroethane	79-34-5	--	5*	2.0	0.37	2.0
Tetrachloroethene	127-18-4	5.0	5*	2.0	0.12	2.0
Toluene	108-88-3	1,000	5*	2.0	0.66	2.0
1,1,2-Trichloroethane	79-00-5	5.0	1.0	2.0	0.20	2.0
Trichloroethene	79-01-6	5.0	5*	2.0	0.19	2.0
1,2,4-Trimethylbenzene	95-63-6	--	5*	2.0	0.52	2.0
1,3,5-Trimethylbenzene	108-67-8	--	5*	2.0	0.43	2.0
Vinyl Chloride	75-01-4	2.0	2.0	2.0	0.59	2.0
m- & p-Xylenes	1330-20-7	10,000[3]	5*	2.0	1.97	2.0
o-Xylene	95-47-6	--	5*	2.0	0.13	2.0

Note 1: U.S. Environmental Protection Agency, Region 3 Regional Screening Level, dated May 2008, for tap water.
Note 2: NYDEC Groundwater Quality Standards for Class GA groundwater.
*: The principal organic contaminant standard for groundwater of 5 µg/L applies to this substance.
Two of the four analytes with reporting limits above their Project Action Level have method detection limits below their Project Action Level (Carbon Tetrachloride and Chloroform), while Benzene and 1,2-Dichloroethane do not.

QAPP WORKSHEET #15: REFERENCE LIMITS AND EVALUATION TABLE (Continued)

Matrix	Soil
Analytical Group	Geotechnical Parameters (Fixed Lab)
Concentration Level	Low

Analyte	CAS Number	Project Action Limit (mg/kg)		Project Quantitation Limit	Achievable Laboratory Limits	
		EPA Region 3 RSL[1]	NYSDEC SCO [2]		Limits of Quantitation	Detection Limits
Grain Size	NA	--	--	6.60	1.40	6.60
Bulk Density	NA	--	--	NA	NA	NA
Effective Porosity	NA	--	--	4.80	0.90	4.80

Note 1: U.S. Environmental Protection Agency, Region 3 Regional Screening Level, dated May 2008, for residential soil.
Note 2: NYDEC Soil Cleanup Objective.
NA - Not applicable.

Matrix	Aqueous
Analytical Group	Total Metals via EPA Method 6010B (Fixed Lab)
Concentration Level	Low

Analyte	CAS Number	Project Action Limit (µg/L)		Project Quantitation Limit (µg/L)	Achievable Laboratory Limits (µg/L)	
		EPA Region 3 RSL[1]	NYSDEC Class GA[2]		Limits of Detection	Detection Limits
Chromium	7440-47-3	--	50	10	1.2	10
Iron	7439-89-6	11,000	300	100	50	100
Magnesium	7439-95-4	--	--	500	9.9	500
Manganese	7439-96-5	--	300	10	2.0	10
Silicon	90337-93-2	--	--	230	23	230
Titanium	7440-32-6	--	--	10	1.0	10

Note 1: U.S. Environmental Protection Agency, Region 3 Regional Screening Level, dated May 2008, for tap water.
Note 2: NYDEC Groundwater Quality Standards for Class GA groundwater.

QAPP WORKSHEET #15: REFERENCE LIMITS AND EVALUATION TABLE (Continued)

Matrix	Aqueous
Analytical Group	Hexavalent and trivalent chromium by EPA Method 7196A (Fixed Lab)
Concentration Level	Low

Analyte	CAS Number	Project Action Limit (mg/L)		Project Quantitation Limit (mg/L)	Achievable Laboratory Limits (mg/L)	
		EPA Region 3 RSL[1]	NYSDEC Class GA[2]		Limits of Detecttion	Detection Limits
Hexavalent Chromium	18540-29-9	0.031	50	0.031	0.003	0.01
Trivalent Chromium	16065-83-1	16,000	--	16,000	0.01	0.01

Note 1: U.S. Environmental Protection Agency, Region 3 Regional Screening Level, dated May 2008, for tap water.

Note 2: NYDEC Groundwater Quality Standards for Class GA groundwater.

Hexavalent chromium has a health-based Project Quantitation Limit below its reporting limit, but above its method detection limit. However, hexavalent chromium is not a Site contaminant of concern, and is being analyzed for geochemical purposes; the reporting limits are sufficient for geochemical purposes.

Matrix	Aqueous
Analytical Group	General Chemistry (Fixed Lab)
Concentration Level	Low

Analyte	CAS Number	Project Action Limit (µg/L)		Project Quantitation Limit (µg/L)	Achievable Laboratory Limits (µg/L)	
		EPA Region 3 RSL[1]	NYSDEC Class GA[2]		Limits of Detection	Detection Limits
Total Dissolved Solids	10052	--	--	5,000	5,000	5,000
Total Suspended Solids	10053	--	--	5,000	5,000	5,000
Hardness	20001	--	--	100	100	1,000
Ferric Iron	STL00113	11,000	300	100	100	100
Ferrous Iron	15438-31-0	11,000	300	100	100	100
Manganous Manganese	NA	--	300	50	10	50
Manganic Manganese	NA	--	300	10	10	50
Carbonate	3812-32-6	--	--	5	5	5

Note 1: U.S. Environmental Protection Agency, Region 3 Regional Screening Level, dated May 2008, for tap water.

Note 2: NYDEC Groundwater Quality Standards for Class GA groundwater.

NA = Not applicable.

QAPP WORKSHEET #15: REFERENCE LIMITS AND EVALUATION TABLE (Continued)

Matrix	Aqueous
Analytical Group	Total Organic Carbon / Total Dissolved Carbon via EPA Method 9060A (Fixed Lab)
Concentration Level	Low

Analyte	CAS Number	Project Action Limit (mg/L)		Project Quantitation Limit (mg/L)	Achievable Laboratory Limits (mg/L)	
		EPA Region 3 RSL[1]	NYSDEC Class GA[2]		Limits of Quantitation	Detection Limits
Total Organic Carbon	7440-44-0	--	--	1.00	0.500	1.00
Dissolved Organic Carbon	7440-44-0	--	--	1.00	0.500	1.00

Note 1: U.S. Environmental Protection Agency, Region 3 Regional Screening Level, dated May 2008, for tap water.

Note 2: NYDEC Groundwater Quality Standards for Class GA groundwater.

Matrix	Aqueous
Analytical Group	Oil & Grease via EPA Method 1664A (Fixed Lab)
Concentration Level	Low

Analyte	CAS Number	Project Action Limit (mg/L)		Project Quantitation Limit (mg/L)	Achievable Laboratory Limits (mg/L)	
		EPA Region 3 RSL[1]	NYSDEC Class GA[2]		Limits of Quantitation	Detection Limits
Oil & Grease	NA	--	--	5	1.4	5

Note 1: U.S. Environmental Protection Agency, Region 3 Regional Screening Level, dated May 2008, for tap water.

Note 2: NYDEC Groundwater Quality Standards for Class GA groundwater.

NA = Not applicable.

QAPP WORKSHEET #15: REFERENCE LIMITS AND EVALUATION TABLE (Continued)

Matrix	Aqueous
Analytical Group	General Chemistry (Fixed Lab)
Concentration Level	Low

Analyte	CAS Number	Project Action Limit (mg/L)		Project Quantitation Limit (mg/L)	Achievable Laboratory Limits (mg/L)	
		EPA Region 3 RSL[1]	NYSDEC Class GA[2]		Limits of Quantitation	Detection Limits
Nitrate	14797-55-8	25,000	10,000	0.25	0.075	0.25
Nitrite	7632-00-0	1,600	10,000	0.25	0.075	0.25
Sulfate	14808-79-8	--	250,000	5.0	2.6	5.0
Sulfite	14265-45-3	--	200	5.0	2.6	5.0

Note 1: U.S. Environmental Protection Agency, Region 3 Regional Screening Level, dated May 2008, for tap water.
Note 2: NYDEC Groundwater Quality Standards for Class GA groundwater.
NA = Not applicable.

QAPP WORKSHEET #15: REFERENCE LIMITS AND EVALUATION TABLE (Concluded)

☐ Worksheet Not Applicable (State Reason)

Matrix	Air
Analytical Group	VOCs via EPA Method TO-15 (Fixed)
Concentration Level	Low to High

Analyte	CAS Number	Project Action Limit (ppbV)			Project Quantitation Limit (ppbV)	Achievable Laboratory Detection Limits (ppbV)	
		EPA Target Indoor Air Concentration[1]	EPA Target Shallow Soil Gas Concentration[2]	NYSDoH Indoor Air Guideline Value[3]		Limits of Quantitation	Limits of Detection
Acetone	67-64-1	150	1,500	--	2.0	2.0	2.96
Acrylonitrile	107-13-1	0.017	0.17	--	0.04	0.04	0.061
Benzene	71-43-2	0.098	0.98	--	0.02	0.02	0.084
Bromodichloromethane	75-27-4	0.021	0.21	--	0.02	0.02	0.032
Bromoform	75-25-2	0.21	2.1	--	0.02	0.02	0.058
Bromomethane	74-83-9	--	--	--	0.50	0.50	0.032
1,3-Butadiene	106-99-0	0.0039	0.039	--	0.02	0.02	0.025
n-Butylbenzene	104-51-8	26	260	--	0.02	0.02	0.18
sec-Butylbenzene	135-98-8	--	--	--	0.10	0.10	0.19
Carbon tetrachloride	56-23-5	0.026	0.26	0.80	0.50	0.50	0.031
Chlorobenzene	108-90-7	13	130	--	0.02	0.02	0.034
Chloroethane	75-00-3	3,800	38,000	--	0.02	0.02	0.028
Chloroform	67-66-3	0.022	0.22	--	0.02	0.02	0.022
Chloromethane	74-87-3	--	--	--	0.02	0.02	0.19
Dibromochloromethane	124-48-1	--	--	--	0.02	0.02	0.030
1,2-Dibromoethane	106-93-4	0.0041	0.041	--	0.02	0.02	0.032
1,2-Dichlorobenzene	95-50-1	33	330	--	0.02	0.02	0.027
1,3-Dichlorobenzene	541-73-1	17	170	--	0.50	0.50	0.028
1,4-Dichlorobenzene	106-46-7	130	1,300	--	0.02	0.02	0.031
Dichlorodifluoromethane	75-71-8	40	400	--	0.02	0.02	0.023
1,1-Dichloroethane	75-34-3	120	1,200	--	0.02	0.02	0.028
1,2-Dichloroethane	107-06-2	0.023	0.23	--	0.02	0.02	0.034
1,1-Dichloroethene	75-35-4	50	500	--	0.05	0.05	0.056
cis-1,2-Dichloroethene	156-59-2	8.8	88	--	0.02	0.02	0.026

Matrix	Air
Analytical Group	VOCs via EPA Method TO-15 (Fixed)
Concentration Level	Low to High

Analyte	CAS Number	Project Action Limit (ppbV)			Project Quantitation Limit (ppbV)	Achievable Laboratory Detection Limits (ppbV)	
		EPA Target Indoor Air Concentration[1]	EPA Target Shallow Soil Gas Concentration[2]	NYSDoH Indoor Air Guideline Value[3]		Limits of Quantitation	Limits of Detection
trans-1,2-Dichloroethene	156-60-5	18	180	--	0.04	0.04	0.023
1,2-Dichloropropane	78-87-5	0.87	8.7	--	0.02	0.02	0.024
cis-1,3-Dichloropropene	10061-01-5	--	--	--	0.02	0.02	0.034
trans-1,3-Dichloropropene	10061-02-6	0.13**	1.3**	--	0.02	0.02	0.032
1,2-Dichlorotetrafluoroethane (Freon 114)	76-14-2	--	--	--	0.05	0.05	0.020
Ethylbenzene	100-41-4	0.51	5.1	--	0.02	0.02	0.029
4-Ethyltoluene	622-96-8	--	--	--	0.02	0.02	0.039
Hexachlorobutadiene	87-68-3	0.010*	0.10*	--	0.02	0.02	0.045
Isopropylbenzene (Cumene)	98-82-8	--	--	--	0.50	0.50	0.19
4-Isopropyltoluene	99-87-6	--	--	--	0.50	0.50	0.19
Methylene Chloride	75-09-2	1.5	15	17.3	1.0	1.0	3.0
Methyl Ethyl Ketone (2-Butanone)	78-93-3	340	3,400	--	0.05	0.05	0.099
Methyl Isobutyl Ketone (2-methyl-4-pentanone)	108-10-1	20	200	--	0.05	0.05	0.17
Methyl tert-butyl ether	1634-04-4	830	8,300	--	0.02	0.02	0.018
Naphthalene	91-20-3	0.57	5.7	--	0.50	0.50	0.048
Styrene	100-42-5	230	2,300	--	0.02	0.02	0.031
1,1,1,2-Tetrachlorethane	79-34-5	0.048	0.048	--	0.02	0.02	0.016
1,1,2,2-Tetrachloroethane	79-34-5	0.0061	0.061	--	0.02	0.02	0.029
Tetrachloroethene	127-18-4	0.12	1.2	14.75	0.02	0.02	0.032
Toluene	108-88-3	110	1,100	--	0.02	0.02	0.039
1,2,3-Trichlorobenzene	86-61-6	--	--	--	0.02	0.05	0.074
1,2,4-Trichlorobenzene	120-82-1	27	270	--	0.02	0.02	0.041
1,1,1-Trichloroethane	71-55-6	400	4,000	18.4	0.50	0.50	0.028
1,1,2-Trichloroethane	79-00-5	0.028	0.28	--	0.05	0.05	0.036
Trichloroethene	79-01-6	0.0041	0.041	0.94	0.02	0.02	0.029

Matrix	Air
Analytical Group	VOCs via EPA Method TO-15 (Fixed)
Concentration Level	Low to High

Analyte	CAS Number	Project Action Limit (ppbV)			Project Quantitation Limit (ppbV)	Achievable Laboratory Detection Limits (ppbV)	
		EPA Target Indoor Air Concentration[1]	EPA Target Shallow Soil Gas Concentration[2]	NYSDoH Indoor Air Guideline Value[3]		Limits of Quantitation	Limits of Detection
Trichlorotrifluoroethane (Freon 113)	76-13-1	--	--	--	0.02	0.02	0.02
Trichlorofluoromethane	75-69-4	120	1,200	--	0.02	0.02	0.024
1,2,4-Trimethylbenzene	95-63-6	1.2	12	--	0.02	0.02	0.028
1,3,5-Trimethylbenzene	108-67-8	1.2	12	--	0.02	0.02	0.019
Vinyl chloride	75-01-4	0.11	1.1	--	0.02	0.02	0.027
m,p-Xylenes	179601-23-1	1,600	16,000	--	0.50	0.50	0.037
Xylene, o-	95-47-6	1,600	16,000	--	0.05	0.05	0.031

Note 1: U.S. Environmental Protection Agency, *OSWER Draft Guidance for Evaluating the Vapor Intrusion to Indoor Air Pathway from Groundwater and Soils*, Table 2c, Target Indoor Air Concentration to Satisfy Both the Prescribed Risk Level and the Target Hazard Index, dated November 2002.

Note 2: U.S. Environmental Protection Agency, *OSWER Draft Guidance for Evaluating the Vapor Intrusion to Indoor Air Pathway from Groundwater and Soils*, Table 2c, Target Shallow Soil Gas Concentration to Satisfy Both the Prescribed Risk Level and the Target Hazard Index, dated November 2002.

Note 3: NYDEC *Guidance for Evaluating Soil Vapor Intrusion in the State of New York*, New York State Department of Health (NYSDoH) values included in Tables 3.1 and 3.3, and Matrices 1 and 2, dated October 2006.

*: The lowest value of the isomers of this compound was used.

**: The value of the total compound was used for this isomer.

ppbV: parts per billion, by volume.

A few analytes have reporting limits and method detection limits above their Project Action Level which are project contaminants of concern (Carbon tetrachloride, 1,1,2,2-Tetrachloroethane, 1,1,2-Trichloroethane, and Trichloroethene). The analytical method is the most sensitive available, and most of the limits of quantitation are below the Project Action Level.

QAPP WORKSHEET #16: PROJECT SCHEDULE TIMELINE TABLE

☐ Worksheet Not Applicable (State Reason)

		Dates (MM/DD/YY)			
Activities	**Organization**	**Anticipated Date(s) of Initiation**	**Anticipated Date(s) of Completion**	**Deliverable**	**Deliverable Due Date**
Soil Sampling	Stone	02/04/13	03/15/2013	*Pre-Design Investigation Report*	05/21/2013
Groundwater Sampling	Stone	02/04/13	02/07/2013	*Pre-Design Investigation Report*	05/21/2013
Soil Vapor Sampling	Stone	03/04/13	05/07/2013	*Soil Vapor Intrusion Investigation Report*	09/03/2013

QAPP WORKSHEET #17: SAMPLING DESIGN AND RATIONALE

☐ Worksheet Not Applicable (State Reason)

Refer to the Section 2 of the *Field Sampling Plan*, for details.

Groundwater Monitoring Activities: Collect groundwater samples from up to 30 groundwater monitoring wells and up to 15 vertical groundwater profiling locations for one or more of the following reasons:

- Obtain current CVOC data from 26 existing and 4 optional, proposed monitoring wells for CVOC analysis. Proposed monitoring wells would be installed to provide additional long-term groundwater contaminant and elevation data.
- Obtain current geochemical data to support the treatability study, which is designed to verify and further define the location and dimensions of the prescribed remedy. Six groundwater samples will be collected from groundwater monitoring well locations in the vicinity of the proposed remedy for total organic carbon/total dissolved carbon, total dissolved solids/total suspended solids, oil and grease, pH, hardness, ferrous/ferric iron, manganous/manganic manganese, nitrate/nitrite, sulfate/sulfite, total metals (including chromium, iron, manganese, silicon, and titanium), hexavalent/trivalent chromium, and carbonate. Obtain additional samples for bench-scale treatability testing.
- Obtain additional CVOC data to verify and further define the nature and extent of the TCE plume, verify the location and dimensions of the selected remedy, and collect data to investigate vapor intrusion pathways for onsite buildings. Up to fifteen (15) vertical groundwater profiles will be completed to obtain this data. Two transects of vertical groundwater profiles will be completed perpendicular to the groundwater flow path across the longitudinal axis of the estimated TCE plume. The first transect will be advanced at the approximate location of the remedy and will consist of up to six (6) locations. The second transect of three (3) profiles is proposed to be located further downgradient in the vicinity of where the estimated TCE plume crosses the property line. A tenth groundwater profile is proposed to be located along the approximate axis of the TCE plume between MW-13 and MW-6/MW-9, with final location to be determined based on the results of the nine previous profiles. Groundwater samples will be collected at five-foot intervals within each vertical profile. Stone estimates 204 groundwater samples will be collected from vertical groundwater profiling locations, these samples will be analyzed for CVOCs and will be used to support the treatability study, which is designed to verify the location and dimensions of the prescribed remedy. Based on near real-time results of the focused investigation up to five (5) additional vertical groundwater profiles will be advanced within the estimated TCE plume.

Geotechnical Assessment: Geotechnical sampling will provide design criteria that will be used to determine the specifications and the cost estimate for the implementation of the proposed remedy. Five (5) soil samples from five (5) ft intervals from depths within the anticipated vertical span of the proposed remedy will be collected and will be submitted for grain size, bulk density, and effective porosity analyses.

Soil Vapor Intrusion Investigation: Collect sub-slab soil vapor samples as follow:

Up to twenty-four (24) samples will be collected from the four onsite buildings and submitted for analysis:

- Two (2) to three (3) indoor air samples from Buildings 201 through 204 on the ground or sub-grade floor (total of ten (10) samples);
- Two (2) to three (3) sub-slab samples from each building (including Buildings 201, 202, 203, 204, 403, and 404), located near the estimated plume areas(total of eight (14) samples);
- Two (2) outdoor ambient air samples (will serve for all four 200-block buildings) (total of two (2)); and;

- two (2) duplicate samples and one (1) trip blank sample;

Sub-slab soil vapor and indoor air samples will be collected from the basement or the lowest occupied floor of each building. The SVI samples will be analyzed via EPA Toxic Organic (TO) Method 15. Selected ion monitoring (SIM) analysis shall be performed for carbon tetrachloride and TCE, and their breakdown products. The laboratory selected to perform the analysis will meet Method T0-15 laboratory certification requirements specified by the state of New York and be accredited for Method T0-15 according to the DoD Environmental Laboratory Accreditation Program (ELAP). Sample reporting limits will be sufficient to meet NYDDOH and American Conference of Governmental Industrial Hygienists Threshold Limit Values for carbon tetrachloride and TCE, and their breakdown products. Stone will perform two identical sampling efforts (winter and summer) to account for seasonal variability, each requiring a separate one-half day mobilization to and demobilization from the Former Scotia Naval Depot.

QAPP WORKSHEET #18: SAMPLING LOCATIONS AND METHODS/SOP REQUIREMENTS TABLE

☐ Worksheet Not Applicable (State Reason)

Sampling Location/ID Number	Matrix	Depth (units)	Analytical Group	Concentration Level	Number of Samples (Identify Field Duplicates)	Sampling SOP Reference[1]	Rationale for Sampling Location
B-1 B-2 B-3 GEP-1 GEP-2 GEP-3 GEP-4 MW-5 MW-6 MW-7 MW-8 MW-9 MW-11 MW-12 MW-13 MW-14 MW-15 MW-16 MW-17 MW-18 MW-19 MW-20 MW-22 MW-23 PMW-3 MW-24 MW-25 MW-26 MW-27	Groundwater	Varies	VOCs	Low	Fixed Lab: 30 samples, 3 Duplicate, 2 MS/MSD	4a, 5a, 6a, 7a, 14a, 15a	To obtain current VOC data in Site groundwater monitoring wells to support the pre-design investigation

Sampling Location/ID Number	Matrix	Depth (units)	Analytical Group	Concentration Level	Number of Samples (Identify Field Duplicates)	Sampling SOP Reference[1]	Rationale for Sampling Location
MW-24 MW-25 MW-26 MW-27	Soil	TBD	Grain Size, Bulk Density, Effective Porosity	NA	Fixed Lab: 5 samples, 1 Duplicate	11a, 16a, 17a, 20a	Provide design criteria used to determine the specifications and cost estimate for the proposed remedy
WP-1 through WP-15	Groundwater	5-ft intervals from water table to ~170 ft bgs at 3 locations and to ~110 ft bgs at 12 locations	CVOCs	Low to High	Mobile Lab: 170 samples, 17 Duplicate, 12 MS/MSD Fixed Lab: 17 samples, 2 Duplicate, 2 MS/MSD	15a, 19a	Obtain CVOC data to verify and further define the nature and extent of the TCE plume, verify the location and dimensions of the selected remedy.

Sampling Location/ID Number	Matrix	Depth (units)	Analytical Group	Concentration Level	Number of Samples (Identify Field Duplicates)	Sampling SOP Reference[1]	Rationale for Sampling Location
MW-24 MW-25 MW-26 MW-27	Groundwater	Various, 65 to 170 ft bgs	Total Organic Carbon/Total Dissolved Carbon Total Dissolved Solids/Total Suspended Solids Oil & Grease ph, Hardness, Ferrous/Ferric Iron Manganous/Manganic Manganese, Nitrate/Nitrite, Sulfate/Sulfite, Total / Dissolved Metals (Chromium, Iron, Magnesium, Manganese, Silicon, and Titanium), Hexavalent/Trivalent Chromium, Carbonate	Low	Fixed Lab: 5 samples, 1 Duplicate	4a, 5a, 6a, 7a, 14a, 15a	Provide design criteria used to determine the specifications and cost estimate for the proposed remedy
		TBD	Treatability Testing	Low to Medium	Fixed Lab: 1 sample, split		
IDW	Aqueous/Soil	NA	VOCs	Low to High	TBD	3a	Determine appropriate disposal/treatment of IDW

Sampling Location/ID Number	Matrix	Depth (units)	Analytical Group	Concentration Level	Number of Samples (Identify Field Duplicates)	Sampling SOP Reference[1]	Rationale for Sampling Location
SV01 to SV14 (soil vapor onsite); AR01 to AR10 (indoor air, onsite); AR11 to AR12 (outdoor ambient air)	Air	Various Indoor Air and 0.5 ft bgs	VOCs	Low to High	Fixed Lab: 45 samples, 5 Duplicates; 3 Trip Blanks	24a, 25a, 26a	Location above groundwater dissolved VOC plume

Note 1: See Worksheet #21 for cross-reference to Sampling SOP.

QAPP WORKSHEET #19: ANALYTICAL SOP REQUIREMENTS TABLE

☐ Worksheet Not Applicable (State Reason)

Matrix	Analytical Group	Concentration Level	Analytical and Preparation Method/SOP Reference	Sample Volume	Containers (number, size and type)	Preservation Requirements (chemical, temperature, light protected)	Maximum Holding Time (preparation/ analysis)
Groundwater	VOCs	Low to High	8260B / 13b	120 mL	3 × 40-mL VOA vials	HCl, 4°C ±2°C	14 days
	CVOCs	Low to High	8260C / 14b, 15b	120 mL	3 × 40-mL VOA vials	HCl, 4°C ±2°C	14 days
	Total Metals	Low	6010B / 1b, 12b	250 mL	1 × 250-mL polyethylene bottle	HNO_3, 4°C ±2°C	180 days
	Total Organic Carbon	Low	9060 / 10b	80 mL	125-mL bottle	H_2SO_4, 4°C ±2°C	28 days
	Total Dissolved Carbon				125-mL bottle (filtered)		
	Total Dissolved Solids	Low	SM 2540C / 7b	1 L	1 × 1 L polyethylene bottle	4°C ±2°C	7 days
	Total Suspended Solids		SM 2540D / 7b				
	Oil & Grease	Low	1664A / 6b	500 mL	1 × 500 mL amber glass bottle	H_2SO_4, 4°C ±2°C	28 days
	pH	Low	9040B / 8b	125 mL	1 × 125 mL polyethylene bottle	4°C ±2°C	24 hours
	Hardness	Low	SM 2340C / 9b	500 mL	1 × 500 mL polyethylene bottle	HNO_3, 4°C ±2°C	6 months
	Carbonate	Low	SM 2320B / 9b	100 mL	1 × 250 mL polyethylene	4°C ±2°C	14 days
	Nitrate / Nitrite, Sulfate / Sulfide	Low	300.0 / 5b	125 mL	1 × 125 mL polyethylene	4°C ±2°C	48 hours
	Ferrous / Ferric Iron	Low	SM 3500-Fe-D /4b	125 mL	1 × 125 mL polyethylene	4°C ±2°C, protect from light	24 hours
	Manganous / Manganic Manganese	Low	1638 modified / 16b	250 mL	2 × 125 mL polyethylene, one filtered	HNO_3, 4°C ±2°C	24 hours

Matrix	Analytical Group	Concentration Level	Analytical and Preparation Method/SOP Reference	Sample Volume	Containers (number, size and type)	Preservation Requirements (chemical, temperature, light protected)	Maximum Holding Time (preparation/ analysis)
	Hexavalent/Trivalent Chromium	Low	7196A / 4b	25 mL	1 × 125mL polyethylene bottle	4°C ±2°C	24 hours
	Treatability Testing	Low	17b	12 gallons	12 × 1 gallon polyethylene bottles and 3 × 40-mL VOA vials	4°C ±2°C	14 days
Soil	Grain Size	NA	ASTM D422-63 / 2b	500 grams	1 × 8 oz. glass jar	4°C ±2°C	None
	Bulk Density	NA	ASTM D2937 / 3b	100 grams	1 × 125-mL glass jar	4°C ±2°C	14 days
	Effective Porosity	NA	ASTM D 7263 / 3b	100 grams	1 × 125-mL glass jar	4°C ±2°C	14 days
IDW	VOCs	Low to High	8260B / 13b	120 mL (Aqueous)	3 × 40-mL VOA vials	HCl, 4°C ±2°C	14 days
				12 grams (Soil)	2 × 40-mL VOA vials, 1 × 40-mL VOA vial	MeOH, 4°C ±2°C, 4°C ±2°C	14 days
Air	VOCs	Low to High	TO-15 / 18b	6 Liters	1 × 6-L Summa canister	None	30 days

QAPP WORKSHEET #20: FIELD QUALITY CONTROL SAMPLE SUMMARY TABLE

☐ Worksheet Not Applicable (State Reason)

Matrix	Analytical Group	Concentration Level	Analytical and Preparation SOP Reference	No. of Sampling Locations	No. of Field Duplicate	No. of MS/MSD	No. of Trip Blanks	No. of Equipment Blanks/ Field Blank	No. of PT Samples	Total No. of Samples to Lab
Groundwater	VOCs (Fixed Lab)	Low to High	8260B /13b	26	3	2	2	3	1	37
	VOCs (Fixed Lab)	Low to High	8260B /13b	21	2	2	5	0	0	30
	CVOCs (Mobile Lab)	Low to High	8260C /14b, 15b	170	17	12	17	17	0	233
	Total Metals	Low	6010B/ 11b, 12b	5	1	0	0	0	0	6
	TOC / TDC	Low	9060/10b	5	1	0	0	0	0	6
	TDS / TSS	Low	SM 2540C / SM 2540D /7b	5	1	0	0	0	0	6
	Oil & Grease	Low	1664A/6b	5	1	0	0	0	0	6
	pH	Low	9040B / 8b	5	1	0	0	0	0	6
	Hardness / Carbonate	Low	SM 2340C / 2320B / 9b	5	1	0	0	0	0	6
	Nitrate / Nitrite Sulfate / Sulfide	Low	300.0 / 5b	5	1	0	0	0	0	6
	Ferrous / Ferric Iron	Low	ASTM 7196A / 4b	5	1	0	0	0	0	6
	Manganous / Manganic Manganese	Low	1638 modified / 16b	5	1	0	0	0	0	6
	Hexavalent / Trivalent Chromium	Low	7196A / 4b	5	1	0	0	0	0	6

Matrix	Analytical Group	Concentration Level	Analytical and Preparation SOP Reference	No. of Sampling Locations	No. of Field Duplicate	No. of MS/MSD	No. of Trip Blanks	No. of Equipment Blanks/ Field Blank	No. of PT Samples	Total No. of Samples to Lab
	Treatability Testing	Low to Medium	17b	2	0	0	1	0	0	3
Soil	Grain Size	NA	ASTM D422 / 2b	5	1	0	0	0	0	6
	Bulk Density	NA	ASTM D2937 / 3b	5	1	0	0	0	0	6
	Effective Porosity	NA	ASTM D 7263 / 3b	5	1	0	0	0	0	6
IDW	VOCs	Low to High	8260C / 13b	TBD	0	0	0	0	0	TBD
Air	VOCs	Low to High	TO-15 / 18b	26	2	0	1	0	0	31

QAPP WORKSHEET #21: PROJECT SAMPLING SOP REFERENCES TABLE

☐ Worksheet Not Applicable (State Reason)

Reference Number	Title, Revision Date and/or Number	Originating Organization	Equipment Type	Modified for Project Work?	Comments
1a	SOP SEI-4.2.7 Chain of Custody Procedures, 05/10/12.	Stone	N/A	No	
2a	SOP SEI-4.5.11 Data Handling, Storage, Retrieval and Error Coding, 05/10/12.	Stone	N/A	No	
3a	SOP SEI-5.1.5 Maintenance and Decontamination of Field Equipment, 02/20/04.	Stone	N/A	No	
4a	SOP SEI-5.26.0 Use, Maintenance and Calibration of the LaMotte Model 2020*e* Turbidity Meter, 06/23/05.	Stone	Turbidity Meter	No	
5a	SOP SEI-5.27.1 Use, Maintenance and Calibration of the Hydrolab MS5 Water Quality Multiprobes, 07/12/12.	Stone	Water Quality Sonde	No	
6a	SOP SEI-5.28.0 Use, Maintenance and Calibration of the Hach L Portable Dissolved Oxygen Meters (Hach Models HQ10 and HQ30d), 02/02/11.	Stone	Dissolved Oxygen Probe	No	
7a	SOP SEI-6.2.6 Water Level Measurement Use, Maintenance and Calibration of Electronic Water Level Indicators, 02/20/04.	Stone	Water Level Indicators	No	
8a	SOP SEI-6.4.5 Installation, Development and Decommissioning of Monitoring Wells and Observation Wells, 08/01/07.	Stone	Drill Rig	No	
9a	SOP SEI-6.16.4 Handling, Collection and Transportation of Samples, 04/16/04.	Stone	N/A	No	
10a	reserved				
11a	SOP SEI-6.19.2 Use, Maintenance and Calibration of the Ionscience Phocheck 1000+ Photionization Detector (PID), 02/04/11, revision number: 2	Stone	Photoionization Detector	No	
12a	SOP SEI-6.11.9 Slug Tests, 01/14/13 (draft).	Stone	Slug	No	
13a	SOP SEI-6.23.1 Observation and Monitoring	Stone	Auto Level or Total Station	No	

Reference Number	Title, Revision Date and/or Number	Originating Organization	Equipment Type	Modified for Project Work?	Comments
	Well Surveying for Elevations, 11/29/05.				
14a	SOP SEI-6.27.3 Groundwater Sampling of Monitoring Wells, 11/29/05.	Stone	Various Sampling Pumps	No	
15a	SOP SEI-6.34.0 Procedure for Sampling Groundwater Monitoring Wells Using Low Stress (Low Flow) Technique, 01/21/05.	Stone	Various Sampling Pumps	No	
16a	SOP SEI-6.45.0 Collection, Handling, and Preservation of Discrete Soil Samples, (draft)	Stone	Soil Augers, etc.	No	
17a	SOP SEI-6.46.0 Geologic Description of Unconsolidated Deposits Using the Unified Soil Classification System, (draft)	Stone	N/A	No	
18a	SOP SEI-6.36.2 Procedure for Collection of Soil Gas Samples for VOC analyses, 03/01/13.	Stone	Coring tools, Summa canisters	Yes	Accommodates DoD, EPA and NYSDEC guidance
19a	SOP SEI-6.43.3 Groundwater Profiling and KPRO Testing, 8/22/12.	Stone	Waterloo$^{APS\ TM}$	No	
20a	SOP SEI-6.1.6 Collection of Soil Samples for Preliminary Site Characterization, 11/18/05.	Stone	Soil Sampler	No	
21a	SOP SEI-5.29.0 Use, Maintenance and Calibration of the MultiRAE IR Multi-Gas Monitor, PGM-54, 2/4/2011.	Stone	Photoionization Detector	No	
22a	SOP SEI-5.30.0 Use, Calibration, and Maintenance of the YSI Model 600XL Multi--Parameter Water Quality Monitoring System (Temperature, Specific Conductance, , pH, Oxidation-Reduction Potential)	Stone	YSI Water Quality Meter	No	
23a	SOP SEI-5.22.2 Maintenance and Calibration of the Troll SP4000 and MiniTroll SSP-100 Datalogger, 2/2/04.	Stone	Troll Data Logger	No	
24a	SOP SEI-6.49.0 Procedure for Collection of Indoor Air Samples for TO-15 Analysis via Summa Canister. 3/22/13.	Stone	Summa canister	Yes	Accommodates NYDES/DOH guidance
25a	SOP SEI-6.36.1 Procedure for Collection of Soil Gas Samples for VOC Analysis. 6/22/07.	Stone	Summa canister	Yes	Accommodates NYDES/DOH guidance

Reference Number	Title, Revision Date and/or Number	Originating Organization	Equipment Type	Modified for Project Work?	Comments
26a	SOP SEI 5.29.0 Use, Maintenance, and Calibration of the MultiRae IR Multi-Gas Monitor, PGM-54, 02/04/11	Stone	Multigas Monitor	No	

QAPP WORKSHEET #22: FIELD EQUIPMENT CALIBRATION, MAINTENANCE, TESTING AND INSPECTION TABLE

☐ Worksheet Not Applicable (State Reason)

Field Equipment	Calibration Activity	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person	SOP Reference[1]
Ionscience Phocheck 1000+ Photionization Detector	Calibrate according to manufacturer's specification	Maintain according to manufacturer's specifications	Check using calibration standards at mid-day	Inspect unit for damage	Daily	± 3%	Manufacturer service or replace unit	Field staff	11a, 18a
LaMotte Model 2020e Turbidimeter	Calibrate according to manufacturer's specification	Maintain according to manufacturer's specifications	Check using calibration standards at mid-day	Inspect unit for damage	Daily	± 3%	Manufacturer service or replace unit	Field staff	4a
Hydrolab MS5 Water Quality Multiprobes	Calibrate according to manufacturer's specification	Maintain according to manufacturer's specifications	Check using calibration standards at mid-day	Inspect unit for damage	Daily	± 3%	Manufacturer service or replace unit	Field staff	5a
Hach L Portable Dissolved Oxygen Meter, Models HQ10 and HQ30d	Calibrate according to manufacturer's specification	Maintain according to manufacturer's specifications	Check using calibration standards at mid-day	Inspect unit for damage	Daily	± 3%	Manufacturer service or replace unit	Field staff	6a
Water level Indicators	Calibrate according to manufacturer's specification	Maintain according to manufacturer's specifications	Check using calibrated bucket at mid-day	Inspect unit for damage	Daily	± 3%	Manufacturer service or replace unit	Field staff	7a
$CO/CO_2/O_2$ Multigas Monitor	Calibrate according to manufacturer's specification	Maintain according to manufacturer's specifications	Check with calibration gas daily	Inspect unit for damage	Daily	± 3%	Manufacturer service or replace unit	Field staff	25a

[1]From the Project Sampling SOP Reference Table (Worksheet #21)

QAPP WORKSHEET #23: ANALYTICAL SOP REFERENCES TABLE

☐ Worksheet Not Applicable (State Reason)

Reference Number	Title, Revision Date, and/or Number	Definitive or Screening Data	Analytical Group(s)	Instrument	Organization Performing Analysis	Modified for Project Work?
1b	reserved					
2b	SOP NO. BR-GT-006 Particle Size Analysis ASTM D 2217 and D422-63, 03/17/10, revision number: 6	Definitive	Grain Size	NA	TestAmerica-Savannah	No
3b	SOP NO. BR-GT-018 Density in Soils by Drive Cylinder Method (ASTM D2937-04), 06/21/10, revision number: 5	Definitive	Bulk Density	NA	TestAmerica-Savannah	No
4b	SOP NO. GE01 Measurement of Analytes Using the Konelab Analyzer, 04/24/2012, revision number: 1G	Definitive	Ferrous / Ferric Iron, Hexavalent / Trivalent Chromium	Konelab Model 20 Analyzer	TestAmerica-Savannah	No
5b	SOP NO. SA-GE-115 Anions by Ion Chromatography, 04/13/2012, revision number: 13	Definitive	Nitrate, Nitrite, Sulfate, Sulfite	Ion Chromatograph (ICP)	TestAmerica-Savannah	No
6b	SOP NO. SA-GE-157 Oil &Grease and Total Petroleum Hydrocarbons (HEM &SGT-HEM)	Definitive	Oil & Grease	NA	TestAmerica-Savannah	No
7b	SOP NO. SA-GE-190 Solid/Residue Determinations, 11/27/2012, revision number: 4	Definitive	TDS/TSS	NA	TestAmerica-Savannah	No
8b	SOP NO. SA-GE-193 Measurement of Analytes using the PC Titrate Auto-Analyzer, 09/17/12, revision number: 3	Definitive	pH	NA	TestAmerica-Savannah	No
9b	SOP NO. SA-GE-201 Total Hardness as $CaCO_3$ by Titrimetric EDTA, 10/15/2011, revision number: 3A	Definitive	Hardness, Carbonate	NA	TestAmerica-Savannah	No

Reference Number	Title, Revision Date, and/or Number	Definitive or Screening Data	Analytical Group(s)	Instrument	Organization Performing Analysis	Modified for Project Work?
10b	SOP NO. SA-GE-204 Carbon Content in Water: Total Carbon (TC), Total Organic Carbon (TOC), and Total Inorganic Carbon (TIC), 04/13/2012, revision number: 2	Definitive	TOC, TDC	Shimadzu TO-VCPN Carbon Analyzer	TestAmerica-Savannah	No
11b	SOP NO. SA-ME-050 Liquid Preparation Procedures for ICP AND ICP/MS, 08/20/2012, revision number: 14	Definitive	Metals	ICP and ICP/MS	TestAmerica-Savannah	No
12b	SOP NO. SA-ME-070 Elements by ICP, 11/23/2012, revision number: 14	Definitive	Metals	ICP	TestAmerica-Savannah	No
13b	SOP NO. SA-VO-004 Volatile Compounds by GC/MS (Methods: EPA 8260B, EPA 624, and SM6200B), 06/04/2012, revision number 0A	Definitive	VOCs	GC/MS	TestAmerica-Savannah	No
14b	SEI-10.15.9 "The Determination of Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry" (GC/MS) (SW846 USEPA Method 8260C), 11/7/12.	Definitive	CVOCs	GC/MS	Stone	Yes
15b	SEI-10.1.6 "Determination of Aromatic and Chlorinated Volatile Organics and Lightweight Petroleum Hydrocarbons (typical range C_1-C_{16}) Compounds Using Solid Phase Microextraction (SPME) And a Gas Chromatograph in Soil and Water Samples (Modified SW846 Methods 8021/8015 & ASTM D6520)", 05/26/09.	Definitive	CVOCs	GC/MS	Stone	Yes
16b	SOP #BR-0060, "Determination of Trace Elements by Inductively Coupled Plasma – Mass Spectrometry using a Perkin-Elmer ELAN DRC II", 05/24/2010.	Definitive	Manganic / Manganous Manganese	ICP/MS	TestAmerica-Savannah	No
17b	SiREM Refs: S1-1725-110212. *Proposal and Cost Estimate for Laboratory ZVI Column Study*. 11/02/12.	Definitive	Treatability Testing	GC/MS	SiREM	Yes

Reference Number	Title, Revision Date, and/or Number	Definitive or Screening Data	Analytical Group(s)	Instrument	Organization Performing Analysis	Modified for Project Work?
18b	SOP No. 2186, Rev. 4, "Determination of Volatile Organic Compounds in Ambient Air Using Specially-Prepared Canisters and Analyzed by GC/MS". 9/25/12.	Definitive	VOCs	GC/MS	Alpha Analytical Laboratory	Yes

QAPP WORKSHEET #24: ANALYTICAL INSTRUMENT CALIBRATION TABLE

☐ Worksheet Not Applicable (State Reason)

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA	SOP Reference[1]
GC/MS (Fixed Lab)	Initial calibration	Prior to analysis of samples and when CCV is not acceptable	5-point Minimum CCC: %RSD < 30% SPCC: RRF_{avg} > 0.050 If %RSD >15%, use curve fit with r^2 > 0.990. GME: Avg %RSD <15%; No single analyte %RSD > 60%.	Establish new calibration curve or service instrument	Michele Kersey	15b
	Continuing calibration verification	Every 12 hours	CCC: %D < 20%. SPCC: RRF > 0.050. Average %D < 20%. No analyte %D > 60%. Poor performers per SOP.	Verify solution integrity and check instrument performance. Perform necessary maintenance and recalibrate instrument. Reanalyze all affected samples		
GC/MS (Fixed Lab)	Initial calibration	Prior to analysis of samples and when CCV is not acceptable	8-point Minimum CCC: %RSD ≤ 30% SPCC: RRF_{avg} > 0.050 If %RSD >15%, use curve fit with r^2 > 0.990. GME: Avg %RSD <15%; No single analyte %RSD > 60%.	Establish new calibration curve or service instrument	Chris Anderson	18b

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA	SOP Reference[1]
	Continuing calibration verification	Every 12 hours	CCC: %D < 20%. SPCC: RRF > 0.050. %D ≤ 30%. No analyte %D > 60%. Poor performers per SOP.	Verify solution integrity and check instrument performance. Perform necessary maintenance and recalibrate instrument. Reanalyze all affected samples		
GC/MS (Mobile Lab)	Initial calibration	Daily or at start of run	5-point correlation coefficient must be 0.995 or higher using linear fit.	Establish new calibration curve or service instrument	Michael Rossi	13b, 14b
	Continuing calibration verification	As needed based on check standards	Minimum RRF and Maximum % RSD on Table 1 or a correlation factor (r) of 0.99 for linear regression.	Verify solution integrity and check instrument performance. Perform necessary maintenance and recalibrate instrument. Reanalyze all affected samples		
ICP	Initial calibration	Daily or at start of run	Single Point: 1 standard and 1 blank, or 3-point correlation coefficient must be 0.998 or higher using a linear fit; additional calibration verifications per control limits provided in SOP.	Establish new calibration curve or service instrument	Michele Kersey	11b, 12b
	Continuing calibration verification	Continuing calibration verification	Continuing calibration verification	Continuing calibration verification		

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA	SOP Reference[1]
	End-of-run calibration verification	End-of-run calibration verification	End-of-run calibration verification	End-of-run calibration verification		
ICP/MS	Initial calibration	Daily or at start of run	3-point correlation coefficient must be 0.998 or higher using a linear fit; additional calibration verifications per control limits provided in SOP.	Establish new calibration curve or service instrument	Michele Kersey	11b, 16b
	Continuing calibration verification	Continuing calibration verification	Continuing calibration verification	Continuing calibration verification		
	End-of-run calibration verification	End-of-run calibration verification	End-of-run calibration verification	End-of-run calibration verification		
Konelab Model 20 Analyzer	Initial Calibration (ICAL)	Every 6 months	The correlation coefficient of the regression curve must be greater than 0.995; ICV within ± 10% of value	Per SOP No. GE01-1G, Appendix I.	Michele Kersey	4b
	Initial Calibration Verification (ICV)	After initial calibration	ICV must be within ± 10% of true value	Per SOP No. GE01-1G, Appendix I.		
	Initial Calibration Blank (ICB/Continuing Calibration Blank (CCB)	Following ICV, and CCB	<½ Reporting Limit	Per SOP No. GE01-1G, Appendix I.		
	Continuing Calibration Verification (CCV)	Every 10 analyses	± 10% of the true value	Per SOP No. GE01-1G, Appendix I.		
Shimadzu TO-VCPN Carbon Analyzer	ICAL	Every 3 months	The correlation coefficient of the regression curve must be greater than 0.995	Per SOP No. SA-GE-204, Sect. 7.3.3.	Michele Kersey	10b
	ICV	Every 3 months	ICV must be within ± 10% of true value	Per SOP No. SA-GE-204, Sect. 9.2.2.		

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA	SOP Reference[1]
	ICB/ CCB	Following ICAL, immediately following end of each batch	<½ Reporting Limit	Per SOP No. SA-GE-204, Sect. 9.2.3.		
	CCV	Every 10 analyses	± 10% of the true value	Per SOP No. SA-GE-204, Sect. 9.2.4.		
	Carbonate/Bicarbonate TIC Check Solution	Once Daily	TIC check standard is less than the RL.	Service Instrument		

From the Analytical SOP References table (Worksheet #23)

QAPP WORKSHEET #25: ANALYTICAL INSTRUMENT AND EQUIPMENT MAINTENANCE, TESTING AND INSPECTION TABLE

☐ Worksheet Not Applicable (State Reason)

Instrument/ Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person	SOP Reference[1]
GC/MS	According to manufacturer's instructions						Michael Rossi/ Michele Kersey/ Chris Anderson	13b, 14b, 15b, 18b
ICP	According to manufacturer's instructions						Michele Kersey	11b, 12b
ICP/MS	According to manufacturer's instructions						Michele Kersey	11b, 16b
Shimadzu TO-VCPN Carbon Analyzer	According to manufacturer's instructions						Michele Kersey	10b

From the Analytical SOP References table (Worksheet #23)

QAPP WORKSHEET #26: SAMPLE HANDLING SYSTEM

☐ Worksheet Not Applicable (State Reason)

Task	
SAMPLE COLLECTION, PACKAGING, AND SHIPMENT	
Sample Collection:	Stone field staff
Sample Packaging:	Stone field staff
Coordination of Shipment:	Stone field staff
Type of Shipment/Carrier:	Coolers, UPS or FedEx overnight
Sample Receipt, Custody and Storage, Preparation, Determinative Analysis	Stone, TestAmerica, Alpha Analytical, SiREM
SAMPLE ARCHIVING	
Field Sample Storage	Stone
Sample Extract/Digestate Storage	See Worksheet #19 for holding times
Biological Sample Storage	N/A
Personnel/Organization	N/A
SAMPLE DISPOSAL	
Personnel/Organization	Stone, TestAmerica, Alpha Analytical

QAPP WORKSHEET #27: SAMPLE CUSTODY REQUIREMENTS

☐ Worksheet Not Applicable (State Reason)

Field Sample Custody Procedures (sample collection, packaging, shipment and delivery): All samples will be placed in appropriate containers according to Worksheet #19. Sample labels will include: site name, date, time, preservative, and analytical method. Preprinted labels provided by Stone Environmental, Inc. will be used as much as possible. All samples will be documented on a Chain of Custody, which will accompany the samples to the laboratory. Except geotechnical soil samples, which will be collected in buckets, all samples will be placed in coolers with wet ice and maintained at 4°C ± 2°C during storage and shipping. Coolers will be packed with sufficient packing material (not Styrofoam) to minimize movement during transit. The coolers will be sealed with Chain of Custody tape and shipped via UPS or FedEx (overnight) to the appropriate laboratory. The laboratory will be responsible for notifying Stone Environmental, Inc. as soon as possible of any inconsistencies or breakage upon receipt. Field blanks will be prepared in the laboratory, where appropriate and shipped with the empty bottles to the Site. Samples of the Chain of Custody, custody tape, and labels are provided on the following page.
Laboratory Sample Custody Procedures (receipt of samples, archiving, disposal): The laboratory will check all samples against the Chain of Custody upon receipt, and will electronically archive all samples before analysis. Samples will be disposed of by the laboratory in accordance with applicable RCRA Hazardous Waste regulations.
Sample Identification Procedures: The first set of characters will be the alphanumeric location ID which will identify the sample location. Samples from existing wells will use the most recent sample name for that well. All other locations will be assigned an individual consecutive number preceded by a two-letter code for the type of sample Each location will be assigned an individual consecutive number preceded by a two letter code for the type of sample. The location ID will be followed by a dash. The second set of characters will identify the depth interval rounded to the nearest foot below ground surface. The third set of characters will identify the type of sample taken ("GW" Groundwater sample, "S" Soil sample, "A" Air sample, "D" Field duplicate, "EB" Equipment blank, "TB" Trip Blank, "MS" Matrix spike sample, "MSD" Matrix spike duplicate sample, "IDW" Investigation Derived Waste sample. Full details are located in Subsection 2.8 of the SAP.

QAPP WORKSHEET #28: QC SAMPLES TABLE

☐ Worksheet Not Applicable (State Reason)

Matrix	Aqueous	**Sampler's Name**	Field Staff		
Analytical Group	CVOCs	**Field Sampling Organization**	Stone		
Sampling SOP	19a	**Analytical Organization**	Stone Mobile Lab		
Analytical Method/ SOP Reference	EPA Method 8260C / 14b, 15b	**No. of Sample Locations**	170		

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Duplicate sample	1 per 20 samples	RPD ≤ 30%	Reanalyze samples or review similarity of samples.	Michael Rossi	Accuracy/Bias-contamination	RPD ≤ 30%
Aqueous VOA Trip Blank	1 per cooler for VOC samples	No reported concentrations ≥ quantitation limits	Review storage and handling procedures; alter as necessary.	Michael Rossi	Contamination	No reported concentrations ≥ quantitation limits
Equipment/Field Blank	1 per event	No reported concentrations ≥ quantitation limits	Review equipment decontamination procedures; alter as necessary.	Michael Rossi	Contamination	No reported concentrations ≥ quantitation limits
Method Blank	1 per 20 samples	No reported concentrations ≥ ½ quantitation limits	Redigest samples with new method blank and reanalyze for analyte.	Michael Rossi	Accuracy	No reported concentrations ≥ quantitation limits
Lab Control Sample	1 per 20 samples	Laboratory Control Limits[1]	Analyte may not be reported without a narrative; rerun batch.	Michael Rossi	Accuracy	±30% recovery
Matrix Spike/Matrix Spike Duplicate	1 per 20 samples or one per matrix type	50 to 150% recovery; RPD ≤ 30%	Note deviation in report.	Michael Rossi	Sensitivity	±25% recovery; RPD ≤ 20%

Note 1: LCS and MS/MSD control limits will default to the DoD limits defined in the QSM.

QAPP WORKSHEET #28: QC SAMPLES TABLE (Continued)

☐ Worksheet Not Applicable (State Reason)

Matrix		Aqueous/Soil		**Sampler's Name**		Field Staff
Analytical Group		VOCs		**Field Sampling Organization**		Stone
Sampling SOP		3a, 4a, 5a, 6a, 7a, 15a		**Analytical Organization**		TestAmerica-Savannah
Analytical Method/ SOP Reference		EPA Method 8260B / 13b		**No. of Sample Locations**		30
QC Sample	**Frequency/Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Action**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria**
Duplicate sample	1 per 20 samples	RPD ≤ 30% (aqueous), 50% (soil)	Reanalyze samples or review similarity of samples.	Michele Kersey	Accuracy/Bias-contamination	RPD ≤ 30% (aqueous), 50% (soil)
Aqueous VOA Trip Blank	1 per cooler for VOC samples	No reported concentrations ≥ quantitation limits	Review storage and handling procedures; alter as necessary.	Michele Kersey	Contamination	No reported concentrations ≥ quantitation limits
Equipment/Field Blank	1 per event	No reported concentrations ≥ quantitation limits	Review equipment decontamination procedures; alter as necessary.	Michele Kersey	Contamination	No reported concentrations ≥ quantitation limits
Method Blank	1 per 20 samples	No reported concentrations ≥ ½ quantitation limits	Redigest samples with new method blank and reanalyze for analyte.	Michele Kersey	Accuracy	No reported concentrations ≥ quantitation limits
Lab Control Sample	1 per 20 samples	Laboratory control limits[1]	Analyte may not be reported without a narrative; rerun batch.	Michele Kersey	Accuracy	±30% recovery
Matrix Spike/Matrix Spike Duplicate	1 per 20 samples or one per matrix type	Laboratory control limits[1]; RPD ≤ 30% (aqueous), 50% (soil)	Note deviation in report.	Michele Kersey	Sensitivity	Laboratory control limits[1]; RPD ≤ 30% (aqueous), 50% (soil)

Note 1: LCS and MS/MSD control limits will default to the DoD limits defined in the QSM.

QAPP WORKSHEET #28: QC SAMPLES TABLE (Continued)

☐ Worksheet Not Applicable (State Reason)

Matrix		Soil		**Sampler's Name**		Field Staff
Analytical Group		Geotechnical Parameters		**Field Sampling Organization**		Stone
Sampling SOP		11a, 16a, 17a, 20a		**Analytical Organization**		Stone MobiLab
Analytical Method/ SOP Reference		ASTM D422, ASTM D2937, ASTM D7263/ 2b, 3b		**No. of Sample Locations**		5
QC Sample	**Frequency/Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Action**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria**
Duplicate sample	1 per 20 samples	RPD ≤ 30%	Reanalyze samples or review similarity of samples.	Michael Rossi	Accuracy/Bias-contamination	RPD ≤ 30%
Method Blank	1 per 20 samples	No reported concentrations ≥ quantitation limits	Redigest samples with new method blank and reanalyze for analyte.	Michael Rossi	Accuracy	No reported concentrations ≥ quantitation limits
Lab Control Sample	1 per 20 samples	70 to 130% recovery	Analyte may not be reported without a narrative; rerun batch.	Michael Rossi	Accuracy	70 to 130% recovery

QAPP WORKSHEET #28: QC SAMPLES TABLE (Continued)

☐ Worksheet Not Applicable (State Reason)

Matrix	Aqueous	**Sampler's Name**	Field Staff
Analytical Group	Total Metals, Hexavalent Chromium, Trivalent Chromium	**Field Sampling Organization**	Stone
Sampling SOP	14a, 15a	**Analytical Organization**	TestAmerica-Savannah
Analytical Method/ SOP Reference	EPA Methods 6010B and 7194A / 4b, 11b, 12b	**No. of Sample Locations**	5

QC Sample	**Frequency/Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Action**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria**
Duplicate sample	1 per 20 samples	RPD ≤ 20%	Reanalyze samples or review similarity of samples.	Michele Kersey	Accuracy/Bias-contamination	RPD ≤ 20%
Equipment/Field Blank	1 per event	No reported concentrations ≥ quantitation limits	Review equipment decontamination procedures; alter as necessary.	Michele Kersey	Contamination	No reported concentrations ≥ quantitation limits
Method Blank	1 per 20 samples	No reported concentrations ≥ ½ quantitation limits	Redigest samples with new method blank and reanalyze for analyte.	Michele Kersey	Accuracy	No reported concentrations ≥ quantitation limits
Lab Control Sample	1 per 20 samples	80 to 120% recovery	Analyte may not be reported without a narrative; rerun batch.	Michele Kersey	Accuracy	80 to 120% recovery
Matrix Spike/Matrix Spike Duplicate	1 per 20 samples or one per matrix type	80 to 120% recovery; RPD ≤ 20%	Note deviation in report.	Michele Kersey	Sensitivity	80 to 120% recovery; RPD ≤ 20%

QAPP WORKSHEET #28: QC SAMPLES TABLE (Continued)

☐ Worksheet Not Applicable (State Reason)

Matrix	Aqueous		**Sampler's Name**		Field Staff
Analytical Group	General Chemistry		**Field Sampling Organization**		Stone
Sampling SOP	14a, 15a		**Analytical Organization**		TestAmerica-Savannah
Analytical Method/ SOP Reference	SM 2340C, 2540C and 2540D; EPA Methods 2320B and 9040D; and ASTM Method D3872-05 (2011) /8b, 9b		**No. of Sample Locations**		5

QC Sample	**Frequency/Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Action**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria**
Duplicate sample	1 per 20 samples	RPD ≤ 25%	Reanalyze samples or review similarity of samples.	Michele Kersey	Accuracy/Bias-contamination	RPD ≤ 25%
Equipment/Field Blank	1 per event	No reported concentrations ≥ quantitation limits	Review equipment decontamination procedures; alter as necessary.	Michele Kersey	Contamination	No reported concentrations ≥ quantitation limits
Method Blank	1 per 20 samples	No reported concentrations ≥ ½ quantitation limits	Redigest samples with new method blank and reanalyze for analyte.	Michele Kersey	Accuracy	No reported concentrations ≥ quantitation limits
Lab Control Sample	1 per 20 samples	80 to 120% recovery	Analyte may not be reported without a narrative; rerun batch.	Michele Kersey	Accuracy	80 to 120% recovery

QAPP WORKSHEET #28: QC SAMPLES TABLE (Continued)

☐ Worksheet Not Applicable (State Reason)

Matrix		Aqueous		**Sampler's Name**		Field Staff
Analytical Group		General Chemistry		**Field Sampling Organization**		Stone
Sampling SOP		14a, 15a		**Analytical Organization**		TestAmerica-Savannah
Analytical Method/ SOP Reference		EPA Method 1638 (Modified) / 16b		**No. of Sample Locations**		5
QC Sample	**Frequency/Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Action**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria**
Duplicate sample	1 per 20 samples	RPD ≤ 20%	Reanalyze samples or review similarity of samples.	Michele Kersey	Accuracy/Bias-contamination	RPD ≤ 20%
Equipment/Field Blank	1 per event	No reported concentrations ≥ quantitation limits	Review equipment decontamination procedures; alter as necessary.	Michele Kersey	Contamination	No reported concentrations ≥ quantitation limits
Method Blank	1 per 20 samples	No reported concentrations ≥ ½ quantitation limits	Redigest samples with new method blank and reanalyze for analyte.	Michele Kersey	Accuracy	No reported concentrations ≥ quantitation limits
Lab Control Sample	1 per 20 samples	75 to 125% recovery	Analyte may not be reported without a narrative; rerun batch.	Michele Kersey	Accuracy	75 to 125% recovery
Matrix Spike/Matrix Spike Duplicate	1 per 10 samples or one per matrix type	75 to 125% recovery; RPD ≤ 20%	Note deviation in report.	Michele Kersey	Sensitivity	75 to 125% recovery; RPD ≤ 20%

QAPP WORKSHEET #28: QC SAMPLES TABLE (Continued)

☐ Worksheet Not Applicable (State Reason)

Matrix		Aqueous		**Sampler's Name**		Field Staff
Analytical Group		Total Organic Carbon / Total Dissolved Carbon		**Field Sampling Organization**		Stone
Sampling SOP		14a, 15a		**Analytical Organization**		TestAmerica-Savannah
Analytical Method/ SOP Reference		EPA Method 9060 / 10b		**No. of Sample Locations**		5
QC Sample	**Frequency/Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Action**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria**
Duplicate sample	1 per 20 samples	RPD ≤ 25%	Reanalyze samples or review similarity of samples.	Michele Kersey	Accuracy/Bias-contamination	RPD ≤ 25%
Equipment/Field Blank	1 per event	No reported concentrations ≥ quantitation limits	Review equipment decontamination procedures; alter as necessary.	Michele Kersey	Contamination	No reported concentrations ≥ quantitation limits
Method Blank	1 per 20 samples	No reported concentrations ≥ ½ quantitation limits	Redigest samples with new method blank and reanalyze for analyte.	Michele Kersey	Accuracy	No reported concentrations ≥ quantitation limits
Lab Control Sample	1 per 20 samples	80 to 120% recovery	Analyte may not be reported without a narrative; rerun batch.	Michele Kersey	Accuracy	80 to 120% recovery

QAPP WORKSHEET #28: QC SAMPLES TABLE (Continued)

☐ Worksheet Not Applicable (State Reason)

Matrix		Aqueous		**Sampler's Name**		Field Staff
Analytical Group		Oil & Grease		**Field Sampling Organization**		Stone
Sampling SOP		14a, 15a		**Analytical Organization**		TestAmerica-Savannah
Analytical Method/ SOP Reference		EPA Method 1664A / 6b		**No. of Sample Locations**		5
QC Sample	**Frequency/Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Action**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria**
Duplicate sample	1 per 20 samples	RPD ≤ 18%	Reanalyze samples or review similarity of samples.	Michele Kersey	Accuracy/Bias-contamination	RPD ≤ 30%
Equipment/Field Blank	1 per event	No reported concentrations ≥ quantitation limits	Review equipment decontamination procedures; alter as necessary.	Michele Kersey	Contamination	No reported concentrations ≥ quantitation limits
Method Blank	1 per 20 samples	No reported concentrations ≥ ½ quantitation limits	Redigest samples with new method blank and reanalyze for analyte.	Michele Kersey	Accuracy	No reported concentrations ≥ quantitation limits
Lab Control Sample	1 per 20 samples	78 to 114% recovery	Analyte may not be reported without a narrative; rerun batch.	Michele Kersey	Accuracy	Laboratory control limits[1]
Matrix Spike/Matrix Spike Duplicate	1 per 20 samples or one per matrix type	Laboratory control limits[1]	Note deviation in report.	Michele Kersey	Sensitivity	Laboratory control limits[1]

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QAPP WORKSHEET #28: QC SAMPLES TABLE (Continued)

☐ Worksheet Not Applicable (State Reason)

Matrix		Aqueous		**Sampler's Name**		Field Staff
Analytical Group		Nitrate / Nitrite, Sulfate / Sulfite		**Field Sampling Organization**		Stone
Sampling SOP		14a, 15a		**Analytical Organization**		TestAmerica-Savannah
Analytical Method/ SOP Reference		EPA Method 300.0 / 5b		**No. of Sample Locations**		5
QC Sample	**Frequency/Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Action**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria**
Duplicate sample	1 per 20 samples	RPD ≤ 30%	Reanalyze samples or review similarity of samples.	Michele Kersey	Accuracy/Bias-contamination	RPD ≤ 30%
Equipment/Field Blank	1 per event	No reported concentrations ≥ quantitation limits	Review equipment decontamination procedures; alter as necessary.	Michele Kersey	Contamination	No reported concentrations ≥ quantitation limits
Method Blank	1 per 20 samples	No reported concentrations ≥ ½ quantitation limits	Redigest samples with new method blank and reanalyze for analyte.	Michele Kersey	Accuracy	No reported concentrations ≥ quantitation limits
Lab Control Sample	1 per 20 samples	90 to 110% recovery	Analyte may not be reported without a narrative; rerun batch.	Michele Kersey	Accuracy	90 to 110% recovery
Matrix Spike/Matrix Spike Duplicate	1 per 20 samples or one per matrix type	90 to 110% recovery; RPD ≤ 30%	Note deviation in report.	Michele Kersey	Sensitivity	90 to 110% recovery; RPD ≤ 30%

QAPP WORKSHEET #28: QC SAMPLES TABLE (Continued)

☐ Worksheet Not Applicable (State Reason)

Matrix	Aqueous	**Sampler's Name**	Field Staff
Analytical Group	Treatability Testing	**Field Sampling Organization**	Stone
Sampling SOP	3a, 4a, 5a, 6a, 7a, 15a	**Analytical Organization**	SiREM
Analytical Method/ SOP Reference	17b	**No. of Sample Locations**	1

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Duplicate sample	1 per 20 samples	RPD ≤ 30%	Reanalyze samples or review similarity of samples.	Phil Dennis	Accuracy/Bias-contamination	RPD ≤ 30%
Aqueous VOA Trip Blank	1 per cooler for VOC samples	No reported concentrations ≥ quantitation limits	Review storage and handling procedures; alter as necessary.	Phil Dennis	Contamination	No reported concentrations ≥ quantitation limits
Equipment/Field Blank	1 per event	No reported concentrations ≥ quantitation limits	Review equipment decontamination procedures; alter as necessary.	Phil Dennis	Contamination	No reported concentrations ≥ quantitation limits
Method Blank	1 per 20 samples	No reported concentrations ≥ ½ quantitation limits	Redigest samples with new method blank and reanalyze for analyte.	Phil Dennis	Accuracy	No reported concentrations ≥ quantitation limits
Lab Control Sample	1 per 20 samples	45 to 150% recovery	Analyte may not be reported without a narrative; rerun batch.	Phil Dennis	Accuracy	±30% recovery
Matrix Spike/Matrix Spike Duplicate	1 per 20 samples or one per matrix type	45 to 150% recovery; RPD ≤ 30%	Note deviation in report.	Phil Dennis	Sensitivity	±25% recovery; RPD ≤ 20%

The above references the VOC analysis that is part of the Treatability Testing.

QAPP WORKSHEET #28: QC SAMPLES TABLE (Concluded)

☐ Worksheet Not Applicable (State Reason)

Matrix		Air		**Sampler's Name**		Field Staff
Analytical Group		VOCs		**Field Sampling Organization**		Stone
Sampling SOP		24a, 25a		**Analytical Organization**		Alpha Analytical Laboratory
Analytical Method/ SOP Reference		EPA Method TO-15 / 18b		**No. of Sample Locations**		14
QC Sample	**Frequency/Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Action**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria**
Duplicate sample	1 per 20 samples	RPD ≤ 50%	Reanalyze samples or review similarity of samples.	Chris Anderson	Accuracy/Bias-contamination	RPD ≤ 50%
Aqueous VOA Trip Blank	1 per cooler for VOC samples	No reported concentrations ≥ quantitation limits	Review storage and handling procedures; alter as necessary.	Chris Anderson	Contamination	No reported concentrations ≥ quantitation limits
Equipment/Field Blank	1 per event	No reported concentrations ≥ quantitation limits	Review equipment decontamination procedures; alter as necessary.	Chris Anderson	Contamination	No reported concentrations ≥ quantitation limits
Method Blank	1 per 20 samples	No reported concentrations ≥ ½ quantitation limits	Redigest samples with new method blank and reanalyze for analyte.	Chris Anderson	Accuracy	No reported concentrations ≥ quantitation limits
Lab Control Sample	1 per 20 samples	Laboratory control limits[1]	Analyte may not be reported without a narrative; rerun batch.	Chris Anderson	Accuracy	±25% recovery
Matrix Spike/Matrix Spike Duplicate	1 per 20 samples or one per matrix type	Laboratory control limits[1]; RPD ≤ 25%; recovery 70 to 130%	Note deviation in report.	Chris Anderson	Sensitivity	Laboratory control limits[1]; RPD ≤ 25%, recovery 70 to 130%

Note 1: LCS and MS/MSD control limits will default to the DoD limits defined in the QSM.

QAPP WORKSHEET #29: PROJECT DOCUMENTS AND RECORDS TABLE

☐ Worksheet Not Applicable (State Reason)

Sample Collection Documents and Records	On-Site Analysis Documents and Records	Off-Site Analysis Documents and Records	Data Assessment Documents and Records	Other
Field Notes	Sample receipt, custody, and tracking records	Sample receipt, custody, and tracking records	Field sampling audit checklist	
Chain of Custody Records	Equipment calibration logs	Equipment calibration logs	Field analysis audit checklist	
Air Bills	Sample collection logs	Samples collection logs	Data validation reports	
Custody Seals	Equipment maintenance, testing and inspection logs	Equipment maintenance, testing and inspection logs	Corrective Action forms	
Telephone logs	Corrective action forms	Corrective action forms	Telephone logs	
Corrective action forms	Field screening result logs	Field screening result logs		
	Reported sample results	Reported sample results		
	Telephone logs	Reported results for standards, QC checks, and QC samples		
		Raw data for samples, standards, QC checks, and QC samples		
		Data package completeness checklist		
		Sample disposal records		
		Telephone logs		
		Extraction/cleanup records		
		Raw data (stored digitally)		

QAPP WORKSHEET #30: ANALYTICAL SERVICES TABLE

☐ Worksheet Not Applicable (State Reason)

Matrix	Analytical Group	Concentration Level	Analytical SOP	Data Package Turnaround Time	Laboratory/ Organization	Backup Laboratory/ Organization
Soil	Grain Size	NA	2b	28 days	TestAmerica-Savannah Michele Kersey 5102 LaRoche Avenue Savannah, GA 31404 912-354-7858	
	Bulk Density	NA	2b	28 days		
	Effective Porosity	NA	3b	28 days		
Aqueous	CVOCs	Low to High	14b, 15	24 hours	Stone Environmental, Inc. Michael Rossi 535 Stone Cutters Way Montpelier, VT 0602 802-229-2194	TestAmerica-Savannah Michele Kersey 5102 LaRoche Avenue Savannah, GA 31404 912-354-7858
	VOCs	Low to High	13b	14 days	TestAmerica-Savannah Michele Kersey 5102 LaRoche Avenue Savannah, GA 31404 912-354-7858	
	Total Metals	Low	11b, 12b	28 days	TestAmerica-Savannah Michele Kersey 5102 LaRoche Avenue Savannah, GA 31404 912-354-7858	
	Total Organic Carbon / Total Dissolved Carbon	Low	10b	28 days		
	Total Dissolved Solids / Total Suspended Solids	Low	7b	28 days		
	Oil & Grease	Low	6b	28 days		
	pH	Low	8b	10 days		
	Nitrate / Nitrite Sulfate / Sulfide	Low	5b	28 days		
	Ferrous / Ferric Iron	Low	4b	28 days		
	Manganous /,Manganic Manganese	Low	16b	28 days		
	Hexavalent/Trivalent Chromium	Low	4b	28 days		

	Hardness / Carbonate	Low	9b	28 days		
Air	VOCs	Low	18B	30 days	Alpha Analytical Laboratory Chris Anderson 8 Walkup Drive Westborough, MA 01581 508-898-9220	

QAPP WORKSHEET #31: PLANNED PROJECT ASSESSMENT TABLE

☐ Worksheet Not Applicable (State Reason)

Assessment Type	Frequency	Internal or External	Organization Performing Assessment	Person(s) Responsible for Performing Assessment (Title and Organizational Affiliation)	Person(s) Responsible for Responding To Assessment Findings (Title and Organizational Affiliation)	Person(s) Responsible for Identifying and Implementing Corrective Actions (CA) (Title and Organizational Affiliation)	Person(s) Responsible for Monitoring Effectiveness of CA (Title and Organizational Affiliation)
Field Sampling Technical Audit	1/At sampling startup	Internal	Stone Environmental, Inc.	Kim Watson, QAO, Stone	Joseph Schmidl, Project Manager, Stone Environmental, Inc.	Joseph Schmidl, Project Manager, Stone Environmental, Inc.	Kim Watson, QAO, Stone Environmental, Inc.
Laboratory Data Audit	1/At receipt of sampling results	External	Stone Environmental, Inc.	Kim Watson, QAO, Stone	Michele Kersey TestAmerica Laboratory or Chris Anderson Alpha Analytical Laboratory OR Kim Watson, Stone Environmental, Inc.	Michele Kersey TestAmerica or Chris Anderson Alpha Analytical Laboratory OR Kim Watson, Stone Environmental, Inc.	Kim Watson, QAO, Stone Environmental, Inc.
Treatability Testing Data Audit	1/At receipt of treatability testing results	External	Stone Environmental, Inc. (team subcontractor Watermark Environmental, Inc.)	Evan Barman, Environmental Engineer, Watermark Environmental, Inc.	Phil Dennis, SiREM, and Evan Barman, Environmental Engineer, Watermark Environmental, Inc.	Phil Dennis, SiREM, and Evan Barman, Environmental Engineer, Watermark Environmental, Inc.	Kim Watson, QAO, Stone Environmental, Inc.

QAPP WORKSHEET #32: ASSESSMENT FINDINGS AND CORRECTIVE ACTION RESPONSES

☐ Worksheet Not Applicable (State Reason)

Assessment Type	Nature of Deficiencies Documentation	Individual(s) Notified of Findings (Name, Title, Organization)	Timeframe of Notification	Nature of Corrective Action Response Documentation	Individual(s) Receiving Corrective Action Response (Name, Title, Org.)	Timeframe for Response
Field Sampling Audit	Written Audit Report	Field Operations Leader (FOL) and Joseph Schmidl, Project Manager, Stone Environmental, Inc.	24 hours after audit	Letter or memorandum	Joseph Schmidl, Project Manager, Stone Environmental, Inc.	24 hours after notification
Laboratory Data Audit / Confirmation Sample Comparisons	Verbal report to laboratory, written report for internal documentation	Michele Kersey or Sara Goff, TestAmerica OR FOL and Joseph Schmidl, Project Manager, and Kim Watson, Stone Environmental, Inc.	24 hours after audit	Verbal report to laboratory, written report for internal documentation	Kim Watson, QAO, Stone Environmental, Inc.	24 hours after notification
Treatability Testing Data Audit	Verbal report to laboratory, written report for internal documentation	Phil Dennis, SiREM, and Evan Barman, Environmental Engineer, Watermark Environmental, Inc.	24 hours after audit	Verbal report to laboratory, written report for internal documentation	Kim Watson, QAO, Stone Environmental, Inc.	24 hours after notification

QAPP WORKSHEET #33: QA MANAGEMENT REPORTS TABLE

☐ Worksheet Not Applicable (State Reason)

Type of Report	Frequency	Projected Delivery Date(s)	Person(s) Responsible for Report Preparation (Title and Org.)	Report Recipient(s) (Title and Org.)
Field Sampling Audit	1/At startup of sampling	02/25/13	Kim Watson, QAO, Stone Environmental, Inc.	FOL and Joseph Schmidl, Project Manager, Stone Environmental, Inc.
Data Usability Assessment Report	1/After all data are generated and checked	04/15/13	Kim Watson, QAO, Stone Environmental, Inc.	Joseph Schmidl, Project Manager, Stone Environmental, Inc.
Final Project Report	1/After QA Management Reports and risk assessment completed	05/21/13	Joseph Schmidl, Project Manager, Stone Environmental, Inc.	Gregory Goepfert, Project Manager, NAN; Joseph Schmidl, Project Manager, Stone Environmental, Inc.

QAPP WORKSHEET #34: VERIFICATION (STEP I) PROCESS TABLE

☐ Worksheet Not Applicable (State Reason)

Verification Input	Description	Internal/External	Responsible for Verification (Name, Organization)
Chain of custody and shipping forms	Chain of custody forms and shipping documentation will be reviewed internally upon their completion and verified against the packed sample coolers they represent. A copy of the chain of custody will be retained at the file site, and the original and remaining copies placed in a Ziploc bag inside the cooler for shipment. See chain of custody SOP for further details.	Internal	Joseph Schmidl, Project Manager, Stone Environmental, Inc.
Audit reports	Upon report completion, a copy of all audit reports will be saved in the site file. If corrective actions are required, a copy of the documented corrective action taken will be attached to the appropriate audit report in the site file. At the beginning of additional sampling and at the completion of site work, site file audit reports will be reviewed internally to ensure that all appropriate corrective actions have been taken and that corrective action reports are attached. If corrective actions have not been taken, the sampling team leader will be notified to ensure corrective action is taken.	Internal	Kim Watson, QAO, Stone Environmental, Inc.
Field Notes	Field notes will be reviewed internally and placed in the site file. A copy of the field notes will be attached to the final report.	Internal	Joseph Schmidl, Project Manager, Stone Environmental, Inc.
Laboratory Data	All laboratory data packages will be verified internally by the laboratory performing the work for completeness and technical accuracy prior to submittal. All received data packages will be verified internally according to the data validation procedures specified in Worksheet #34.	Internal Internal	Kim Watson, QAO, Stone Environmental, Inc. Kim Watson, QAO, Stone Environmental, Inc.

QAPP WORKSHEET #35: VALIDATION (STEPS IIA AND IIB) PROCESS TABLE

☐ Worksheet Not Applicable (State Reason)

Step IIa/IIb	Validation Input	Description	Responsible for Validation (Name, Organization)
IIa	Onsite analytical work	All onsite analytical data will be reviewed against QAPP requirements for completeness and accuracy based on the field calibration records.	Kim Watson, QAO, Stone Environmental, Inc.
IIa	SOPs	Ensure that all sampling and analytical SOPs were followed.	Kim Watson, QAO, Stone Environmental, Inc.
IIa	Documentation of Method QC Results	Establish that all method required QC samples were run and met required limits.	Kim Watson, QAO, Stone Environmental, Inc.
IIa	Documentation of QAPP QC sample results.	Establish that all QAPP required QC samples were run and met required limits.	Kim Watson, QAO, Stone Environmental, Inc.
IIa	Project Quantitation Limits	All sample results met the project quantitation limits specified in the QAPP.	Kim Watson, QAO, Stone Environmental, Inc.

QAPP WORKSHEET #36: VALIDATION (STEPS IIA AND IIB) SUMMARY TABLE

☐ Worksheet Not Applicable (State Reason)

Step IIa/IIb	Matrix	Analytical Group	Concentration Level	Validation Criteria	Data Validator (Title and Org.)
IIa	Aqueous	CVOCs, VOCs, TOC, TDC, TDS, TSS, Oil & Grease, pH, Hardness, Ferrous/Ferric Iron, Manganous/Manganic Manganese, Nitrate/Nitrate, Sulfate/Sulfite, Total Metals, Hexavalent/Trivalent Chromium, Carbonate	Low to High	QAPP Worksheets #12,15 and 28	Kim Watson, QAO, Stone Environmental, Inc.
IIa	Soil	Grain Size, Bulk Density, Effective Porosity	Not Applicable	QAPP Worksheets #12,15 and 28	Kim Watson, QAO, Stone Environmental, Inc.
IIa	Air	VOCs	Low to High	QAPP Worksheets #12,15 and 28	Kim Watson, QAO, Stone Environmental, Inc.

QAPP WORKSHEET #37: USABILITY ASSESSMENT

☐ Worksheet Not Applicable (State Reason)

The Data Usability Assessment will be headed by Kim Watson, QAO, of Stone Environmental, Inc. The FOL will be responsible for assigning task work to the individual task members who will be supporting the Data Usability Assessment. After the Data Usability Assessment has been performed, data deemed appropriate for use will then be used to support the Pre-Design Investigation. The results of the Data Usability Assessment will be presented in the Final Pre-Design Investigation Report. The accuracy, precision and completeness of data will be assessed and conclusions drawn based on their results. Accuracy and precision of analytical results will be evaluated based on criteria specified in the analytical methods used. Such criteria will include, but not be limited to, blank results, matrix spike recoveries, surrogate percent recovery values, correlation coefficients, and detection limits.

Accuracy: The amount of agreement between the true value of a parameter and the measured value. Accuracy is a statistical measurement of correctness and includes components of random error (variability due to imprecision) and systemic error. It therefore reflects the total error associated with a measurement. A measurement is accurate when the value reported does not differ from the true value or known concentration of the spike or standard. Analytical accuracy is measured by comparing the percent recovery of analytes spiked into an LCS to a control limit. For volatile and semi-volatile organic compounds, surrogate compound recoveries are used to assess accuracy and method performance for each sample analyzed. The percent recovery will be calculated. The control limits for percent recovery of spike compounds and surrogates are listed in the laboratory SOP corresponding to the specific analytical method used for analysis.

Precision: The measurement of the agreement between samples from the same population. It is strictly defined as the degree of mutual agreement among independent measurements as the result of repeated application of the same process under similar conditions. Precision can be expressed as the standard deviation between independent samples or as the relative percent difference between duplicate samples.

Completeness: The measure of the amount of validated data obtained compared to that which was expected to be obtained. The number of valid results divided by the number of possible individual analyte results, expressed as a percentage, determines the completeness of a data set. For completeness requirements, valid results are all results not qualified with an "R" flag. An "R" flag is place on the data by the data validator and means that the data are unusable due to deficiencies in the ability to analyze the sample and meet QC criteria.

ATTACHMENT A

eQAPP Data

- Analyte Report
- Precision & Accuracy Report

Project Target Analyte Reporting Limit, Blank Contamination, and Lab & Field Duplicate RPD Criteria

eQapp Name: ScotiaNavyDepot1
Description: 102305-07

Target Analyte Name	Analyte Label (CAS)	Quantitation Limit	Detection Limit	Units	Blank Contamination Rule	LabDup RPD	FieldDup RPD
Method: 1664A Matrix: AQ							
OIL & GREASE	OILGREASE	5	1.4	mg/L	5.00		30.00
Method: 2340B Matrix: AQ							
HARDNESS (AS CACO3)	HARD	10	10	ug/L	5.00		30.00
Method: 300.0 Matrix: AQ							
NITRATE	14797-55-8	0.25	0.075	mg/L	5.00	20.00	30.00
NITRITE	14797-65-0	0.25	0.075	mg/L	5.00	20.00	30.00
SULFATE AS SO4	14808-79-8	5	2.6	mg/L	5.00	20.00	30.00
Method: 3500_F+2_D_Calc Matrix: AQ							
Ferrous Iron	15438-31-0	0.1	0.025	mg/L	5.00	20.00	30.00
Method: 3500_FE_D Matrix: AQ							
Ferric Iron	STL00113	0.1			5.00	20.00	30.00
Ferrous Iron	15438-31-0	0.1			5.00	20.00	30.00
IRON	7439-89-6	0.05			5.00	20.00	30.00
Method: 6010C Matrix: Water							
CALCIUM	7440-70-2	500	96	ug/L	5.00	20.00	30.00
CHROMIUM	7440-47-3	10	1.2	ug/L	5.00	20.00	30.00
IRON	7439-89-6	100	50	ug/L	5.00	20.00	30.00
MAGNESIUM	7439-95-4	500	9.9	ug/L	5.00	20.00	30.00
MANGANESE	7439-96-5	10	2	ug/L	5.00	20.00	30.00
TITANIUM	7440-32-6	10	1	ug/L	5.00	20.00	30.00
Method: 7196A Matrix: AQ							
HEXAVALENT CHROMIUM	18540-29-9	0.01	0.003	ug/L	5.00	20.00	30.00
Method: 7196A_CR3 Matrix: AQ							
CHROMIUM	7440-47-3	0.01	0.01	mg/L	5.00	20.00	30.00
Cr (III)	16065-83-1	0.01	0.01	mg/L	5.00	20.00	30.00

Target Analyte Name	Analyte Label (CAS)	Quantitation Limit	Detection Limit	Units	Blank Contamination Rule	LabDup RPD	FieldDup RPD
Method: 8260B **Matrix: Soil**							
1,1,1,2-TETRACHLOROETHANE	630-20-6			ug/Kg	5.00		50.00
1,1,1-TRICHLOROETHANE	71-55-6			ug/Kg	5.00		50.00
1,1,2,2-TETRACHLOROETHANE	79-34-5			ug/Kg	5.00		50.00
1,1,2-TRICHLOROETHANE	79-00-5			ug/Kg	5.00		50.00
1,1-DICHLOROETHANE	75-34-3			ug/Kg	5.00		50.00
1,1-DICHLOROETHENE	75-35-4			ug/Kg	5.00		50.00
1,1-DICHLOROPROPENE	563-58-6			ug/Kg	5.00		50.00
1,2,3-TRICHLOROBENZENE	87-61-6			ug/Kg	5.00		50.00
1,2,3-TRICHLOROPROPANE	96-18-4			ug/Kg	5.00		50.00
1,2,4-TRICHLOROBENZENE	120-82-1			ug/Kg	5.00		50.00
1,2,4-TRIMETHYLBENZENE	95-63-6			ug/Kg	5.00		50.00
1,2-DIBROMO-3-CHLOROPROPANE	96-12-8			ug/Kg	5.00		50.00
1,2-DIBROMOETHANE	106-93-4			ug/Kg	5.00		50.00
1,2-DICHLOROBENZENE	95-50-1			ug/Kg	5.00		50.00
1,2-DICHLOROETHANE	107-06-2			ug/Kg	5.00		50.00
1,2-DICHLOROPROPANE	78-87-5			ug/Kg	5.00		50.00
1,3,5-TRIMETHYLBENZENE	108-67-8			ug/Kg	5.00		50.00
1,3-DICHLOROBENZENE	541-73-1			ug/Kg	5.00		50.00
1,3-DICHLOROPROPANE	142-28-9			ug/Kg	5.00		50.00
1,4-DICHLOROBENZENE	106-46-7			ug/Kg	5.00		50.00
2,2-DICHLOROPROPANE	594-20-7			ug/Kg	5.00		50.00
2-BUTANONE	78-93-3			ug/Kg	10.00		50.00
2-CHLOROTOLUENE	95-49-8			ug/Kg	5.00		50.00
2-HEXANONE	591-78-6			ug/Kg	5.00		50.00
4-CHLOROTOLUENE	106-43-4			ug/Kg	5.00		50.00
4-METHYL-2-PENTANONE	108-10-1			ug/Kg	5.00		50.00
ACETONE	67-64-1			ug/Kg	10.00		50.00
BENZENE	71-43-2			ug/Kg	5.00		50.00
BROMOBENZENE	108-86-1			ug/Kg	5.00		50.00
BROMOCHLOROMETHANE	74-97-5			ug/Kg	5.00		50.00
BROMODICHLOROMETHANE	75-27-4			ug/Kg	5.00		50.00
BROMOFORM	75-25-2			ug/Kg	5.00		50.00
BROMOMETHANE	74-83-9			ug/Kg	5.00		50.00

Target Analyte Name	Analyte Label (CAS)	Quantitation Limit	Detection Limit	Units	Blank Contamination Rule	LabDup RPD	FieldDup RPD
Method: 8260B **Matrix: Soil**							
CARBON DISULFIDE	75-15-0			ug/Kg	10.00		50.00
CARBON TETRACHLORIDE	56-23-5			ug/Kg	5.00		50.00
CHLOROBENZENE	108-90-7			ug/Kg	5.00		50.00
CHLOROETHANE	75-00-3			ug/Kg	5.00		50.00
CHLOROFORM	67-66-3			ug/Kg	5.00		50.00
CHLOROMETHANE	74-87-3			ug/Kg	5.00		50.00
CIS-1,2-DICHLOROETHENE	156-59-2			ug/Kg	5.00		50.00
CIS-1,3-DICHLOROPROPENE	10061-01-5			ug/Kg	5.00		50.00
DIBROMOCHLOROMETHANE	124-48-1			ug/Kg	5.00		50.00
DIBROMOMETHANE	74-95-3			ug/Kg	5.00		50.00
DICHLORODIFLUOROMETHANE	75-71-8			ug/Kg	5.00		50.00
ETHYLBENZENE	100-41-4			ug/Kg	5.00		50.00
HEXACHLOROBUTADIENE	87-68-3			ug/Kg	5.00		50.00
ISOPROPYLBENZENE	98-82-8			ug/Kg	5.00		50.00
m,p-Xylene	179601-23-1			ug/Kg	5.00		50.00
METHYL TERT-BUTYL ETHER	1634-04-4			ug/Kg	5.00		50.00
METHYLENE CHLORIDE	75-09-2			ug/Kg	10.00		50.00
NAPHTHALENE	91-20-3			ug/Kg	5.00		50.00
N-BUTYLBENZENE	104-51-8			ug/Kg	5.00		50.00
N-PROPYLBENZENE	103-65-1			ug/Kg	5.00		50.00
O-XYLENE	95-47-6			ug/Kg	5.00		50.00
P-ISOPROPYLTOLUENE	99-87-6			ug/Kg	5.00		50.00
SEC-BUTYLBENZENE	135-98-8			ug/Kg	5.00		50.00
STYRENE	100-42-5			ug/Kg	5.00		50.00
TERT-BUTYLBENZENE	98-06-6			ug/Kg	5.00		50.00
TETRACHLOROETHENE	127-18-4			ug/Kg	5.00		50.00
TOLUENE	108-88-3			ug/Kg	5.00		50.00
TRANS-1,2-DICHLOROETHENE	156-60-5			ug/Kg	5.00		50.00
TRANS-1,3-DICHLOROPROPENE	10061-02-6			ug/Kg	5.00		50.00
TRICHLOROETHENE	79-01-6			ug/Kg	5.00		50.00
TRICHLOROFLUOROMETHANE	75-69-4			ug/Kg	5.00		50.00
VINYL CHLORIDE	75-01-4			ug/Kg	5.00		50.00
Xylene (Total)	1330-20-7			ug/Kg	5.00		50.00

Target Analyte Name	Analyte Label (CAS)	Quantitation Limit	Detection Limit	Units	Blank Contamination Rule	LabDup RPD	FieldDup RPD
Method: 8260B **Matrix: Water**							
1,1,1-TRICHLOROETHANE	71-55-6	1	0.5	ug/L	5.00		30.00
1,1,2,2-TETRACHLOROETHANE	79-34-5	1	0.18	ug/L	5.00		30.00
1,1,2-TRICHLORO-1,2,2-TRIFLUOROETHANE	76-13-1	1	0.5	ug/L	5.00		30.00
1,1,2-TRICHLOROETHANE	79-00-5	1	0.13	ug/L	5.00		30.00
1,1-DICHLOROETHANE	75-34-3	1	0.25	ug/L	5.00		30.00
1,1-DICHLOROETHENE	75-35-4	1	0.11	ug/L	5.00		30.00
1,2,4-TRICHLOROBENZENE	120-82-1	1	0.25	ug/L	5.00		30.00
1,2-DIBROMO-3-CHLOROPROPANE	96-12-8	1	0.44	ug/L	5.00		30.00
1,2-DIBROMOETHANE	106-93-4	1	0.25	ug/L	5.00		30.00
1,2-DICHLOROBENZENE	95-50-1	1	0.21	ug/L	5.00		30.00
1,2-DICHLOROETHANE	107-06-2	1	0.1	ug/L	5.00		30.00
1,2-DICHLOROPROPANE	78-87-5	1	0.13	ug/L	5.00		30.00
1,3-DICHLOROBENZENE	541-73-1	1	0.25	ug/L	5.00		30.00
1,4-DICHLOROBENZENE	106-46-7	1	0.28	ug/L	5.00		30.00
2-BUTANONE	78-93-3	10	1	ug/L	10.00		30.00
2-HEXANONE	591-78-6	10	1	ug/L	5.00		30.00
4-METHYL-2-PENTANONE	108-10-1	10	1	ug/L	5.00		30.00
ACETONE	67-64-1	25	5	ug/L	10.00		30.00
BENZENE	71-43-2	1	0.25	ug/L	5.00		30.00
BROMODICHLOROMETHANE	75-27-4	1	0.25	ug/L	5.00		30.00
BROMOFORM	75-25-2	1	0.5	ug/L	5.00		30.00
BROMOMETHANE	74-83-9	1	0.8	ug/L	5.00		30.00
CARBON DISULFIDE	75-15-0	2	0.6	ug/L	10.00		30.00
CARBON TETRACHLORIDE	56-23-5	1	0.5	ug/L	5.00		30.00
CHLOROBENZENE	108-90-7	1	0.25	ug/L	5.00		30.00
CHLOROETHANE	75-00-3	1	1	ug/L	5.00		30.00
CHLOROFORM	67-66-3	1	0.14	ug/L	5.00		30.00
CHLOROMETHANE	74-87-3	1	0.33	ug/L	5.00		30.00
CIS-1,2-DICHLOROETHENE	156-59-2	1	0.15	ug/L	5.00		30.00
CIS-1,3-DICHLOROPROPENE	10061-01-5	1	0.11	ug/L	5.00		30.00
CYCLOHEXANE	110-82-7	1	0.25	mg/L	5.00		30.00
DIBROMOCHLOROMETHANE	124-48-1	1	0.1	ug/L	5.00		30.00
DICHLORODIFLUOROMETHANE	75-71-8	1	0.25	ug/L	5.00		30.00

Target Analyte Name	Analyte Label (CAS)	Quantitation Limit	Detection Limit	Units	Blank Contamination Rule	LabDup RPD	FieldDup RPD
Method: 8260B **Matrix: Water**							
ETHYLBENZENE	100-41-4	1	0.11	ug/L	5.00		30.00
ISOPROPYLBENZENE	98-82-8	1	0.1	ug/L	5.00		30.00
METHYL ACETATE	79-20-9	1	0.19	ug/L	5.00		30.00
METHYL CYCLOHEXANE	108-87-2	1	0.1	ug/L	5.00		30.00
METHYL TERT-BUTYL ETHER	1634-04-4	10	0.2	ug/L	5.00		30.00
METHYLENE CHLORIDE	75-09-2	5	1	ug/L	10.00		30.00
STYRENE	100-42-5	1	0.11	ug/L	5.00		30.00
TETRACHLOROETHENE	127-18-4	1	0.15	ug/L	5.00		30.00
TOLUENE	108-88-3	1	0.33	ug/L	5.00		30.00
TRANS-1,2-DICHLOROETHENE	156-60-5	1	0.2	ug/L	5.00		30.00
TRANS-1,3-DICHLOROPROPENE	10061-02-6	1	0.21	ug/L	5.00		30.00
TRICHLOROETHENE	79-01-6	1	0.13	ug/L	5.00		30.00
TRICHLOROFLUOROMETHANE	75-69-4	1	0.25	ug/L	5.00		30.00
VINYL CHLORIDE	75-01-4	1	0.18	ug/L	5.00		30.00
Xylene (Total)	1330-20-7	2	0.2	ug/L	5.00		30.00
Method: 8260C **Matrix: Water**							
1,1,2,2-TETRACHLOROETHANE	79-34-5	2.0	0.37	ug/L	5.00		30.00
1,1,2-TRICHLOROETHANE	79-00-5	2.0	0.20	ug/L	5.00		30.00
1,1-DICHLOROETHANE	75-34-3	2.0	0.12	ug/L	5.00		30.00
1,1-DICHLOROETHENE	75-35-4	2.0	0.15	ug/L	5.00		30.00
1,2,4-TRIMETHYLBENZENE	95-63-6	2.0	0.52	ug/L	5.00		30.00
1,2-DICHLOROETHANE	107-06-2	2.0	0.30	ug/L	5.00		30.00
1,3,5-TRIMETHYLBENZENE	108-67-8	2.0	0.43	ug/L	5.00		30.00
BENZENE	71-43-2	2.0	0.20	ug/L	5.00		30.00
CARBON TETRACHLORIDE	56-23-5	2.0	0.54	ug/L	5.00		30.00
CHLOROFORM	67-66-3	2.0	0.19	ug/L	5.00		30.00
CIS-1,2-DICHLOROETHENE	156-59-2	2.0	0.10	ug/L	5.00		30.00
ETHYLBENZENE	100-41-4	2.0	0.12	ug/L	5.00		30.00
m,p-Xylene	179601-23-1	2.0	1.97	ug/L	5.00		30.00
NAPHTHALENE	91-20-3	2.0	0.46	ug/L	5.00		30.00
O-XYLENE	95-47-6	2.0	0.13	ug/L	5.00		30.00
TETRACHLOROETHENE	127-18-4	2.0	0.12	ug/L	5.00		30.00

Target Analyte Name	Analyte Label (CAS)	Quantitation Limit	Detection Limit	Units	Blank Contamination Rule	LabDup RPD	FieldDup RPD
Method: 8260C **Matrix: Water**							
TOLUENE	108-88-3	2.0	0.66	ug/L	5.00		30.00
TRANS-1,2-DICHLOROETHENE	156-60-5	2.0	0.32	ug/L	5.00		30.00
TRICHLOROETHENE	79-01-6	2.0	0.19	ug/L	5.00		30.00
VINYL CHLORIDE	75-01-4	2.0	0.59	ug/L	5.00		30.00
Method: 9040B **Matrix: AQ**							
PH	PH			pH		40.00	30.00
Method: 9060A **Matrix: AQ**							
Total Dissolved Carbon	TDC	1	0.5	mg/L	5.00	25.00	30.00
TOTAL ORGANIC CARBON (TOC)	TOC	1	0.5	mg/L	5.00	25.00	30.00
Method: SM 2320B **Matrix: Water**							
Carbonate Alkalinity as CaCO3	STL00154	5	5	mg/L		30.00	30.00
Method: SM2540C **Matrix: AQ**							
TOTAL DISSOLVED SOLIDS	TDS	5	5	mg/L		25.00	30.00
Method: SM2540D **Matrix: AQ**							
Total Suspended Solids	TSS	5	5	mg/L		25.00	30.00

Project Accuracy and Precision Report

eQapp Name: ScotiaNavyDepot1
Description: 102305-07

Analyte Name	Analyte Label (CAS)	Lower Rejection (%)	Lower Recovery (%)	Upper Recovery (%)	Upper Rejection (%)	RPD (%)
Method: 1664A **Matrix: AQ**						
QC Type: Laboratory_Control_Sample						
OIL & GREASE	OILGREASE	10.00	78.00	114.00		18.00
QC Type: Matrix_Spike						
OIL & GREASE	OILGREASE	0.00	78.00	114.00		18.00
Method: 2340B **Matrix: AQ**						
QC Type: Laboratory_Control_Sample						
HARDNESS (AS CACO3)	HARD	10.00	75.00	125.00		30.00
Method: 300.0 **Matrix: AQ**						
QC Type: Laboratory_Control_Sample						
NITRATE	14797-55-8	10.00	90.00	110.00		30.00
NITRITE	14797-65-0	10.00	90.00	110.00		30.00
SULFATE AS SO4	14808-79-8	10.00	90.00	110.00		30.00
QC Type: Matrix_Spike						
NITRATE	14797-55-8		90.00	110.00		30.00
NITRITE	14797-65-0		90.00	110.00		30.00
SULFATE AS SO4	14808-79-8		90.00	110.00		30.00
Method: 3500_F+2_D_Calc **Matrix: AQ**						
QC Type: Laboratory_Control_Sample						
Ferrous Iron	15438-31-0		80.00	120.00		

Analyte Name	Analyte Label (CAS)	Lower Rejection (%)	Lower Recovery (%)	Upper Recovery (%)	Upper Rejection (%)	RPD (%)
Method: 3500_F+2_D_Calc	**Matrix: AQ**					
QC Type: Matrix_Spike						
Ferrous Iron	15438-31-0		80.00	120.00		20.00
Method: 3500_FE_D	**Matrix: AQ**					
QC Type: Laboratory_Control_Sample						
Ferric Iron	STL00113	10.00	80.00	120.00		
Ferrous Iron	15438-31-0	10.00	80.00	120.00		
IRON	7439-89-6	10.00	80.00	120.00		
QC Type: Matrix_Spike						
Ferric Iron	STL00113		80.00	120.00		20.00
Ferrous Iron	15438-31-0		80.00	120.00		20.00
IRON	7439-89-6		80.00	120.00		20.00
Method: 6010C	**Matrix: Water**					
QC Type: Laboratory_Control_Sample						
CALCIUM	7440-70-2	50.00	80.00	120.00		20.00
CHROMIUM	7440-47-3	50.00	80.00	120.00		20.00
IRON	7439-89-6	50.00	80.00	120.00		20.00
MAGNESIUM	7439-95-4	50.00	80.00	120.00		20.00
MANGANESE	7439-96-5	50.00	80.00	120.00		20.00
TITANIUM	7440-32-6	50.00	80.00	120.00		20.00
QC Type: Matrix_Spike						
CALCIUM	7440-70-2		80.00	120.00		20.00
CHROMIUM	7440-47-3		80.00	120.00		20.00
IRON	7439-89-6		80.00	120.00		20.00
MAGNESIUM	7439-95-4		80.00	120.00		20.00
MANGANESE	7439-96-5		80.00	120.00		20.00
TITANIUM	7440-32-6		80.00	120.00		20.00

Analyte Name	Analyte Label (CAS)	Lower Rejection (%)	Lower Recovery (%)	Upper Recovery (%)	Upper Rejection (%)	RPD (%)
Method: 7196A **Matrix: AQ**						
QC Type: Laboratory_Control_Sample						
HEXAVALENT CHROMIUM	18540-29-9	10.00	85.00	115.00		20.00
QC Type: Matrix_Spike						
HEXAVALENT CHROMIUM	18540-29-9		85.00	115.00		20.00
Method: 7196A_CR3 **Matrix: AQ**						
QC Type: Laboratory_Control_Sample						
CHROMIUM	7440-47-3	10.00	85.00	115.00		20.00
Cr (III)	16065-83-1	10.00	85.00	115.00		20.00
QC Type: Matrix_Spike						
CHROMIUM	7440-47-3		85.00	115.00		20.00
Cr (III)	16065-83-1		85.00	115.00		20.00
Method: 8260B **Matrix: Soil**						
QC Type: Laboratory_Control_Sample						
1,1,1,2-TETRACHLOROETHANE	630-20-6	10.00	75.00	125.00		30.00
1,1,1-TRICHLOROETHANE	71-55-6	10.00	70.00	135.00		30.00
1,1,2,2-TETRACHLOROETHANE	79-34-5	10.00	55.00	130.00		30.00
1,1,2-TRICHLOROETHANE	79-00-5	10.00	60.00	125.00		30.00
1,1-DICHLOROETHANE	75-34-3	10.00	75.00	125.00		30.00
1,1-DICHLOROETHENE	75-35-4	10.00	65.00	135.00		30.00
1,1-DICHLOROPROPENE	563-58-6	10.00	70.00	135.00		30.00
1,2,3-TRICHLOROBENZENE	87-61-6	10.00	60.00	135.00		30.00
1,2,3-TRICHLOROPROPANE	96-18-4	10.00	65.00	130.00		30.00
1,2,4-TRICHLOROBENZENE	120-82-1	10.00	65.00	130.00		30.00
1,2,4-TRIMETHYLBENZENE	95-63-6	10.00	65.00	135.00		30.00
1,2-DIBROMO-3-CHLOROPROPANE	96-12-8	10.00	40.00	135.00		30.00
1,2-DIBROMOETHANE	106-93-4	10.00	70.00	125.00		30.00
1,2-DICHLOROBENZENE	95-50-1	10.00	75.00	120.00		30.00
1,2-DICHLOROETHANE	107-06-2	10.00	70.00	135.00		30.00

Analyte Name	Analyte Label (CAS)	Lower Rejection (%)	Lower Recovery (%)	Upper Recovery (%)	Upper Rejection (%)	RPD (%)
Method: 8260B	**Matrix: Soil**					
QC Type: Laboratory_Control_Sample						
1,2-DICHLOROPROPANE	78-87-5	10.00	70.00	120.00		30.00
1,3,5-TRIMETHYLBENZENE	108-67-8	10.00	65.00	135.00		30.00
1,3-DICHLOROBENZENE	541-73-1	10.00	70.00	125.00		30.00
1,3-DICHLOROPROPANE	142-28-9	10.00	75.00	125.00		30.00
1,4-DICHLOROBENZENE	106-46-7	10.00	70.00	125.00		30.00
2,2-DICHLOROPROPANE	594-20-7	10.00	65.00	135.00		30.00
2-BUTANONE	78-93-3	10.00	30.00	160.00		30.00
2-CHLOROTOLUENE	95-49-8	10.00	70.00	130.00		30.00
2-HEXANONE	591-78-6	10.00	45.00	145.00		30.00
4-CHLOROTOLUENE	106-43-4	10.00	75.00	125.00		30.00
4-METHYL-2-PENTANONE	108-10-1	10.00	45.00	145.00		30.00
ACETONE	67-64-1	10.00	20.00	160.00		30.00
BENZENE	71-43-2	10.00	75.00	125.00		30.00
BROMOBENZENE	108-86-1	10.00	65.00	120.00		30.00
BROMOCHLOROMETHANE	74-97-5	10.00	70.00	125.00		30.00
BROMODICHLOROMETHANE	75-27-4	10.00	70.00	130.00		30.00
BROMOFORM	75-25-2	10.00	55.00	135.00		30.00
BROMOMETHANE	74-83-9	10.00	30.00	160.00		30.00
CARBON DISULFIDE	75-15-0	10.00	45.00	160.00		30.00
CARBON TETRACHLORIDE	56-23-5	10.00	65.00	135.00		30.00
CHLOROBENZENE	108-90-7	10.00	75.00	125.00		30.00
CHLOROETHANE	75-00-3	10.00	40.00	155.00		30.00
CHLOROFORM	67-66-3	10.00	70.00	125.00		30.00
CHLOROMETHANE	74-87-3	10.00	50.00	130.00		30.00
CIS-1,2-DICHLOROETHENE	156-59-2	10.00	65.00	125.00		30.00
CIS-1,3-DICHLOROPROPENE	10061-01-5	10.00	70.00	125.00		30.00
DIBROMOCHLOROMETHANE	124-48-1	10.00	65.00	130.00		30.00
DIBROMOMETHANE	74-95-3	10.00	75.00	130.00		30.00
DICHLORODIFLUOROMETHANE	75-71-8	10.00	35.00	135.00		30.00
ETHYLBENZENE	100-41-4	10.00	75.00	125.00		30.00
HEXACHLOROBUTADIENE	87-68-3	10.00	55.00	140.00		30.00
ISOPROPYLBENZENE	98-82-8	10.00	75.00	130.00		30.00

Analyte Name	Analyte Label (CAS)	Lower Rejection (%)	Lower Recovery (%)	Upper Recovery (%)	Upper Rejection (%)	RPD (%)
Method: 8260B	**Matrix:** Soil					
QC Type: Laboratory_Control_Sample						
m,p-Xylene	179601-23-1	10.00	80.00	125.00		30.00
METHYLENE CHLORIDE	75-09-2	10.00	55.00	140.00		30.00
NAPHTHALENE	91-20-3	10.00	40.00	125.00		30.00
N-BUTYLBENZENE	104-51-8	10.00	65.00	140.00		30.00
N-PROPYLBENZENE	103-65-1	10.00	65.00	135.00		30.00
O-XYLENE	95-47-6	10.00	75.00	125.00		30.00
P-ISOPROPYLTOLUENE	99-87-6	10.00	75.00	135.00		30.00
SEC-BUTYLBENZENE	135-98-8	10.00	65.00	130.00		30.00
STYRENE	100-42-5	10.00	75.00	125.00		30.00
TERT-BUTYLBENZENE	98-06-6	10.00	65.00	130.00		30.00
TETRACHLOROETHENE	127-18-4	10.00	65.00	140.00		30.00
TOLUENE	108-88-3	10.00	70.00	125.00		30.00
TRANS-1,2-DICHLOROETHENE	156-60-5	10.00	65.00	135.00		30.00
TRANS-1,3-DICHLOROPROPENE	10061-02-6	10.00	65.00	125.00		30.00
TRICHLOROETHENE	79-01-6	10.00	75.00	125.00		30.00
TRICHLOROFLUOROMETHANE	75-69-4	10.00	25.00	185.00		30.00
VINYL CHLORIDE	75-01-4	10.00	60.00	125.00		30.00
QC Type: Matrix_Spike						
1,1,1,2-TETRACHLOROETHANE	630-20-6	0.00	75.00	125.00		30.00
1,1,1-TRICHLOROETHANE	71-55-6	0.00	70.00	135.00		30.00
1,1,2,2-TETRACHLOROETHANE	79-34-5	0.00	55.00	130.00		30.00
1,1,2-TRICHLOROETHANE	79-00-5	0.00	60.00	125.00		30.00
1,1-DICHLOROETHANE	75-34-3	0.00	75.00	125.00		30.00
1,1-DICHLOROETHENE	75-35-4	0.00	65.00	135.00		30.00
1,1-DICHLOROPROPENE	563-58-6	0.00	70.00	135.00		30.00
1,2,3-TRICHLOROBENZENE	87-61-6	0.00	60.00	135.00		30.00
1,2,3-TRICHLOROPROPANE	96-18-4	0.00	65.00	130.00		30.00
1,2,4-TRICHLOROBENZENE	120-82-1	0.00	65.00	130.00		30.00
1,2,4-TRIMETHYLBENZENE	95-63-6	0.00	65.00	135.00		30.00
1,2-DIBROMO-3-CHLOROPROPANE	96-12-8	0.00	40.00	135.00		30.00
1,2-DIBROMOETHANE	106-93-4	0.00	70.00	125.00		30.00

Analyte Name	Analyte Label (CAS)	Lower Rejection (%)	Lower Recovery (%)	Upper Recovery (%)	Upper Rejection (%)	RPD (%)
Method: 8260B	**Matrix:** Soil					
QC Type: Matrix_Spike						
1,2-DICHLOROBENZENE	95-50-1	0.00	75.00	120.00		30.00
1,2-DICHLOROETHANE	107-06-2	0.00	70.00	135.00		30.00
1,2-DICHLOROPROPANE	78-87-5	0.00	70.00	120.00		30.00
1,3,5-TRIMETHYLBENZENE	108-67-8	0.00	65.00	135.00		30.00
1,3-DICHLOROBENZENE	541-73-1	0.00	70.00	125.00		30.00
1,3-DICHLOROPROPANE	142-28-9	0.00	75.00	125.00		30.00
1,4-DICHLOROBENZENE	106-46-7	0.00	70.00	125.00		30.00
2,2-DICHLOROPROPANE	594-20-7	0.00	65.00	135.00		30.00
2-BUTANONE	78-93-3	0.00	30.00	160.00		30.00
2-CHLOROTOLUENE	95-49-8	0.00	70.00	130.00		30.00
2-HEXANONE	591-78-6	0.00	45.00	145.00		30.00
4-CHLOROTOLUENE	106-43-4	0.00	75.00	125.00		30.00
4-METHYL-2-PENTANONE	108-10-1	0.00	45.00	145.00		30.00
ACETONE	67-64-1	0.00	20.00	160.00		30.00
BENZENE	71-43-2	0.00	75.00	125.00		30.00
BROMOBENZENE	108-86-1	0.00	65.00	120.00		30.00
BROMOCHLOROMETHANE	74-97-5	0.00	70.00	125.00		30.00
BROMODICHLOROMETHANE	75-27-4	0.00	70.00	130.00		30.00
BROMOFORM	75-25-2	0.00	55.00	135.00		30.00
BROMOMETHANE	74-83-9	0.00	30.00	160.00		30.00
CARBON DISULFIDE	75-15-0	0.00	45.00	160.00		30.00
CARBON TETRACHLORIDE	56-23-5	0.00	65.00	135.00		30.00
CHLOROBENZENE	108-90-7	0.00	75.00	125.00		30.00
CHLOROETHANE	75-00-3	0.00	40.00	155.00		30.00
CHLOROFORM	67-66-3	0.00	70.00	125.00		30.00
CHLOROMETHANE	74-87-3	0.00	50.00	130.00		30.00
CIS-1,2-DICHLOROETHENE	156-59-2	0.00	65.00	125.00		30.00
CIS-1,3-DICHLOROPROPENE	10061-01-5	0.00	70.00	125.00		30.00
DIBROMOCHLOROMETHANE	124-48-1	0.00	65.00	130.00		30.00
DIBROMOMETHANE	74-95-3	0.00	75.00	130.00		30.00
DICHLORODIFLUOROMETHANE	75-71-8	0.00	35.00	135.00		30.00
ETHYLBENZENE	100-41-4	0.00	75.00	125.00		30.00

Analyte Name	Analyte Label (CAS)	Lower Rejection (%)	Lower Recovery (%)	Upper Recovery (%)	Upper Rejection (%)	RPD (%)
Method: 8260B	**Matrix: Soil**					
QC Type: Matrix_Spike						
HEXACHLOROBUTADIENE	87-68-3	0.00	55.00	140.00		30.00
ISOPROPYLBENZENE	98-82-8	0.00	75.00	130.00		30.00
m,p-Xylene	179601-23-1	0.00	80.00	125.00		30.00
METHYLENE CHLORIDE	75-09-2	0.00	55.00	140.00		30.00
NAPHTHALENE	91-20-3	0.00	40.00	125.00		30.00
N-BUTYLBENZENE	104-51-8	0.00	65.00	140.00		30.00
N-PROPYLBENZENE	103-65-1	0.00	65.00	135.00		30.00
O-XYLENE	95-47-6	0.00	75.00	125.00		30.00
P-ISOPROPYLTOLUENE	99-87-6	0.00	75.00	135.00		30.00
SEC-BUTYLBENZENE	135-98-8	0.00	65.00	130.00		30.00
STYRENE	100-42-5	0.00	75.00	125.00		30.00
TERT-BUTYLBENZENE	98-06-6	0.00	65.00	130.00		30.00
TETRACHLOROETHENE	127-18-4	0.00	65.00	140.00		30.00
TOLUENE	108-88-3	0.00	70.00	125.00		30.00
TRANS-1,2-DICHLOROETHENE	156-60-5	0.00	65.00	135.00		30.00
TRANS-1,3-DICHLOROPROPENE	10061-02-6	0.00	65.00	125.00		30.00
TRICHLOROETHENE	79-01-6	0.00	75.00	125.00		30.00
TRICHLOROFLUOROMETHANE	75-69-4	0.00	25.00	185.00		30.00
VINYL CHLORIDE	75-01-4	0.00	50.00	125.00		30.00
QC Type: Surrogate						
4-Bromofluorobenzene	460-00-4	0.00	85	120		
TOLUENE-D8	2037-26-5	0.00	85	115		
Method: 8260B	**Matrix: Water**					
QC Type: Laboratory_Control_Sample						
1,1,1-TRICHLOROETHANE	71-55-6	10.00	76.00	126.00		30.00
1,1,2,2-TETRACHLOROETHANE	79-34-5	10.00	71.00	127.00		30.00
1,1,2-TRICHLORO-1,2,2-TRIFLUOROETHANE	76-13-1	10.00	72.00	139.00		30.00
1,1,2-TRICHLOROETHANE	79-00-5	10.00	69.00	127.00		30.00
1,1-DICHLOROETHANE	75-34-3	10.00	69.00	132.00		30.00
1,1-DICHLOROETHENE	75-35-4	10.00	73.00	134.00		30.00

Analyte Name	Analyte Label (CAS)	Lower Rejection (%)	Lower Recovery (%)	Upper Recovery (%)	Upper Rejection (%)	RPD (%)
Method: 8260B	**Matrix:** Water					
QC Type: Laboratory_Control_Sample						
1,2,4-TRICHLOROBENZENE	120-82-1	10.00	67.00	134.00		30.00
1,2-DIBROMO-3-CHLOROPROPANE	96-12-8	10.00	57.00	126.00		50.00
1,2-DIBROMOETHANE	106-93-4	10.00	75.00	127.00		30.00
1,2-DICHLOROBENZENE	95-50-1	10.00	77.00	124.00		30.00
1,2-DICHLOROETHANE	107-06-2	10.00	75.00	120.00		30.00
1,2-DICHLOROPROPANE	78-87-5	10.00	71.00	126.00		30.00
1,3-DICHLOROBENZENE	541-73-1	10.00	79.00	123.00		30.00
1,4-DICHLOROBENZENE	106-46-7	10.00	76.00	124.00		30.00
2-BUTANONE	78-93-3	10.00	55.00	142.00		30.00
2-HEXANONE	591-78-6	10.00	52.00	149.00		30.00
4-METHYL-2-PENTANONE	108-10-1	10.00	51.00	143.00		30.00
ACETONE	67-64-1	10.00	39.00	162.00		50.00
BENZENE	71-43-2	10.00	74.00	123.00		30.00
BROMODICHLOROMETHANE	75-27-4	10.00	72.00	129.00		30.00
BROMOFORM	75-25-2	10.00	60.00	134.00		30.00
BROMOMETHANE	74-83-9	10.00	10.00	171.00		50.00
CARBON DISULFIDE	75-15-0	10.00	63.00	142.00		30.00
CARBON TETRACHLORIDE	56-23-5	10.00	70.00	131.00		30.00
CHLOROBENZENE	108-90-7	10.00	79.00	120.00		30.00
CHLOROETHANE	75-00-3	10.00	47.00	148.00		40.00
CHLOROFORM	67-66-3	10.00	76.00	128.00		30.00
CHLOROMETHANE	74-87-3	10.00	47.00	151.00		30.00
CIS-1,2-DICHLOROETHENE	156-59-2	10.00	78.00	127.00		30.00
CIS-1,3-DICHLOROPROPENE	10061-01-5	10.00	73.00	128.00		30.00
CYCLOHEXANE	110-82-7	10.00	68.00	137.00		30.00
DIBROMOCHLOROMETHANE	124-48-1	10.00	63.00	134.00		50.00
DICHLORODIFLUOROMETHANE	75-71-8	10.00	41.00	165.00		50.00
ETHYLBENZENE	100-41-4	10.00	78.00	125.00		30.00
ISOPROPYLBENZENE	98-82-8	10.00	72.00	129.00		30.00
METHYL ACETATE	79-20-9	10.00	26.00	182.00		30.00
METHYL CYCLOHEXANE	108-87-2	10.00	72.00	133.00		30.00
METHYL TERT-BUTYL ETHER	1634-04-4	10.00	76.00	126.00		30.00

Analyte Name	Analyte Label (CAS)	Lower Rejection (%)	Lower Recovery (%)	Upper Recovery (%)	Upper Rejection (%)	RPD (%)
Method: 8260B	**Matrix: Water**					
QC Type: Laboratory_Control_Sample						
METHYLENE CHLORIDE	75-09-2	10.00	79.00	124.00		30.00
STYRENE	100-42-5	10.00	75.00	129.00		30.00
TETRACHLOROETHENE	127-18-4	10.00	77.00	128.00		30.00
TOLUENE	108-88-3	10.00	77.00	125.00		30.00
TRANS-1,2-DICHLOROETHENE	156-60-5	10.00	78.00	130.00		30.00
TRANS-1,3-DICHLOROPROPENE	10061-02-6	10.00	72.00	127.00		50.00
TRICHLOROETHENE	79-01-6	10.00	80.00	120.00		30.00
TRICHLOROFLUOROMETHANE	75-69-4	10.00	68.00	144.00		30.00
VINYL CHLORIDE	75-01-4	10.00	58.00	141.00		30.00
Xylene (Total)	1330-20-7	10.00	80.00	124.00		30.00
QC Type: Matrix_Spike						
1,1,1-TRICHLOROETHANE	71-55-6	10.00	76.00	126.00		30.00
1,1,2,2-TETRACHLOROETHANE	79-34-5	10.00	71.00	127.00		30.00
1,1,2-TRICHLORO-1,2,2-TRIFLUOROETHANE	76-13-1	10.00	72.00	139.00		30.00
1,1,2-TRICHLOROETHANE	79-00-5	10.00	69.00	127.00		30.00
1,1-DICHLOROETHANE	75-34-3	10.00	69.00	132.00		30.00
1,1-DICHLOROETHENE	75-35-4	10.00	73.00	134.00		30.00
1,2,4-TRICHLOROBENZENE	120-82-1	10.00	67.00	134.00		30.00
1,2-DIBROMO-3-CHLOROPROPANE	96-12-8	10.00	57.00	126.00		50.00
1,2-DIBROMOETHANE	106-93-4	10.00	75.00	127.00		30.00
1,2-DICHLOROBENZENE	95-50-1	10.00	77.00	124.00		30.00
1,2-DICHLOROETHANE	107-06-2	10.00	75.00	120.00		30.00
1,2-DICHLOROPROPANE	78-87-5	10.00	71.00	126.00		30.00
1,3-DICHLOROBENZENE	541-73-1	10.00	79.00	123.00		30.00
1,4-DICHLOROBENZENE	106-46-7	10.00	76.00	124.00		30.00
2-BUTANONE	78-93-3	10.00	55.00	142.00		30.00
2-HEXANONE	591-78-6	10.00	52.00	149.00		30.00
4-METHYL-2-PENTANONE	108-10-1	10.00	51.00	143.00		30.00
ACETONE	67-64-1	10.00	39.00	162.00		50.00
BENZENE	71-43-2	10.00	74.00	123.00		30.00
BROMODICHLOROMETHANE	75-27-4	10.00	72.00	129.00		30.00

Analyte Name	Analyte Label (CAS)	Lower Rejection (%)	Lower Recovery (%)	Upper Recovery (%)	Upper Rejection (%)	RPD (%)
Method: 8260B	**Matrix:** Water					
QC Type: Matrix_Spike						
BROMOFORM	75-25-2	10.00	60.00	134.00		30.00
BROMOMETHANE	74-83-9	10.00	10.00	171.00		50.00
CARBON DISULFIDE	75-15-0	10.00	63.00	142.00		30.00
CARBON TETRACHLORIDE	56-23-5	10.00	70.00	131.00		30.00
CHLOROBENZENE	108-90-7	10.00	79.00	120.00		30.00
CHLOROETHANE	75-00-3	10.00	47.00	148.00		40.00
CHLOROFORM	67-66-3	10.00	76.00	128.00		30.00
CHLOROMETHANE	74-87-3	10.00	47.00	151.00		30.00
CIS-1,2-DICHLOROETHENE	156-59-2	10.00	78.00	127.00		30.00
CIS-1,3-DICHLOROPROPENE	10061-01-5	10.00	73.00	128.00		30.00
CYCLOHEXANE	110-82-7	10.00	68.00	137.00		30.00
DIBROMOCHLOROMETHANE	124-48-1	10.00	63.00	134.00		50.00
DICHLORODIFLUOROMETHANE	75-71-8	10.00	41.00	165.00		50.00
ETHYLBENZENE	100-41-4	10.00	78.00	125.00		30.00
ISOPROPYLBENZENE	98-82-8	10.00	72.00	129.00		30.00
METHYL ACETATE	79-20-9	10.00	26.00	182.00		30.00
METHYL CYCLOHEXANE	108-87-2	10.00	72.00	133.00		30.00
METHYL TERT-BUTYL ETHER	1634-04-4	10.00	76.00	126.00		30.00
METHYLENE CHLORIDE	75-09-2	10.00	79.00	124.00		30.00
STYRENE	100-42-5	10.00	75.00	129.00		30.00
TETRACHLOROETHENE	127-18-4	10.00	77.00	128.00		30.00
TOLUENE	108-88-3	10.00	77.00	125.00		30.00
TRANS-1,2-DICHLOROETHENE	156-60-5	10.00	78.00	130.00		30.00
TRANS-1,3-DICHLOROPROPENE	10061-02-6	10.00	72.00	127.00		50.00
TRICHLOROETHENE	79-01-6	10.00	80.00	120.00		30.00
TRICHLOROFLUOROMETHANE	75-69-4	10.00	68.00	144.00		30.00
VINYL CHLORIDE	75-01-4	10.00	58.00	141.00		30.00
Xylene (Total)	1330-20-7	10.00	80.00	124.00		30.00
QC Type: Surrogate						
1,2-DICHLOROETHANE-D4	17060-07-0	0.00	70	120		
4-Bromofluorobenzene	460-00-4	0.00	75	120		

Analyte Name	Analyte Label (CAS)	Lower Rejection (%)	Lower Recovery (%)	Upper Recovery (%)	Upper Rejection (%)	RPD (%)
Method: 8260B	**Matrix: Water**					
QC Type: Surrogate						
DIBROMOFLUOROMETHANE	1868-53-7	0.00	85	115		
TOLUENE-D8	2037-26-5	0.00	85	120		
Method: 8260C	**Matrix: Water**					
QC Type: Laboratory_Control_Sample						
1,1,2,2-TETRACHLOROETHANE	79-34-5	10.00	65.00	130.00		30.00
1,1,2-TRICHLOROETHANE	79-00-5	10.00	75.00	125.00		30.00
1,1-DICHLOROETHANE	75-34-3	10.00	70.00	135.00		30.00
1,1-DICHLOROETHENE	75-35-4	10.00	70.00	130.00		30.00
1,2,4-TRIMETHYLBENZENE	95-63-6	10.00	75.00	130.00		30.00
1,2-DICHLOROETHANE	107-06-2	10.00	70.00	130.00		30.00
1,3,5-TRIMETHYLBENZENE	108-67-8	10.00	75.00	130.00		30.00
BENZENE	71-43-2	10.00	80.00	120.00		30.00
CARBON TETRACHLORIDE	56-23-5	10.00	65.00	140.00		30.00
CHLOROFORM	67-66-3	10.00	65.00	135.00		30.00
CIS-1,2-DICHLOROETHENE	156-59-2	10.00	70.00	125.00		30.00
ETHYLBENZENE	100-41-4	10.00	75.00	125.00		30.00
m,p-Xylene	179601-23-1	10.00	75.00	130.00		30.00
NAPHTHALENE	91-20-3	10.00	55.00	140.00		30.00
O-XYLENE	95-47-6	10.00	80.00	120.00		30.00
TETRACHLOROETHENE	127-18-4	10.00	45.00	150.00		30.00
TOLUENE	108-88-3	10.00	75.00	120.00		30.00
TRANS-1,2-DICHLOROETHENE	156-60-5	10.00	60.00	140.00		30.00
TRICHLOROETHENE	79-01-6	10.00	70.00	125.00		30.00
VINYL CHLORIDE	75-01-4	10.00	50.00	145.00		30.00
QC Type: Matrix_Spike						
1,1,2,2-TETRACHLOROETHANE	79-34-5	0.00	65.00	130.00		30.00
1,1,2-TRICHLOROETHANE	79-00-5	0.00	75.00	125.00		30.00
1,1-DICHLOROETHANE	75-34-3	0.00	70.00	135.00		30.00
1,1-DICHLOROETHENE	75-35-4	0.00	70.00	130.00		30.00
1,2,4-TRIMETHYLBENZENE	95-63-6	0.00	75.00	130.00		30.00

Analyte Name	Analyte Label (CAS)	Lower Rejection (%)	Lower Recovery (%)	Upper Recovery (%)	Upper Rejection (%)	RPD (%)
Method: 8260C **Matrix: Water**						
QC Type: Matrix_Spike						
1,2-DICHLOROETHANE	107-06-2	0.00	70.00	130.00		30.00
1,3,5-TRIMETHYLBENZENE	108-67-8	0.00	75.00	130.00		30.00
BENZENE	71-43-2	0.00	80.00	120.00		30.00
CARBON TETRACHLORIDE	56-23-5	0.00	65.00	140.00		30.00
CHLOROFORM	67-66-3	0.00	65.00	135.00		30.00
CIS-1,2-DICHLOROETHENE	156-59-2	0.00	70.00	125.00		30.00
ETHYLBENZENE	100-41-4	0.00	75.00	125.00		30.00
m,p-Xylene	179601-23-1	0.00	75.00	130.00		30.00
NAPHTHALENE	91-20-3	0.00	55.00	140.00		30.00
O-XYLENE	95-47-6	0.00	80.00	120.00		30.00
TETRACHLOROETHENE	127-18-4	0.00	45.00	150.00		30.00
TOLUENE	108-88-3	0.00	75.00	120.00		30.00
TRANS-1,2-DICHLOROETHENE	156-60-5	0.00	60.00	140.00		30.00
TRICHLOROETHENE	79-01-6	0.00	70.00	125.00		30.00
VINYL CHLORIDE	75-01-4	0.00	50.00	145.00		30.00
QC Type: Surrogate						
4-Bromofluorobenzene	460-00-4	0.00	75	120		
Method: 9040B **Matrix: AQ**						
QC Type: Laboratory_Control_Sample						
PH	PH		63.00	158.00		40.00
Method: 9060A **Matrix: AQ**						
QC Type: Laboratory_Control_Sample						
Total Dissolved Carbon	TDC	10.00	80.00	120.00		25.00
TOTAL ORGANIC CARBON (TOC)	TOC	10.00	80.00	120.00		25.00
QC Type: Matrix_Spike						
Total Dissolved Carbon	TDC		80.00	120.00		25.00
TOTAL ORGANIC CARBON (TOC)	TOC		80.00	120.00		25.00

Analyte Name	Analyte Label (CAS)	Lower Rejection (%)	Lower Recovery (%)	Upper Recovery (%)	Upper Rejection (%)	RPD (%)
Method: SM 2320B **Matrix: Water**						
QC Type: Laboratory_Control_Sample						
Carbonate Alkalinity as CaCO3	STL00154		80.00	120.00		
Method: SM2540C **Matrix: AQ**						
QC Type: Laboratory_Control_Sample						
TOTAL DISSOLVED SOLIDS	TDS	10.00	80.00	120.00		25.00
Method: SM2540D **Matrix: AQ**						
QC Type: Laboratory_Control_Sample						
Total Suspended Solids	TSS	10.00	80.00	120.00		25.00

Project Accuracy and Precision Report

eQapp Name: ScotiaNavyDepot1
Description: 102305-07

Analyte Name	Analyte Label (CAS)	Lower Rejection (%)	Lower Recovery (%)	Upper Recovery (%)	Upper Rejection (%)	RPD (%)
Method: 1664A **Matrix: AQ**						
QC Type: Laboratory_Control_Sample						
OIL & GREASE	OILGREASE	10.00	78.00	114.00		18.00
QC Type: Matrix_Spike						
OIL & GREASE	OILGREASE	0.00	78.00	114.00		18.00
Method: 2340B **Matrix: AQ**						
QC Type: Laboratory_Control_Sample						
HARDNESS (AS CACO3)	HARD	10.00	75.00	125.00		30.00
Method: 300.0 **Matrix: AQ**						
QC Type: Laboratory_Control_Sample						
NITRATE	14797-55-8	10.00	90.00	110.00		30.00
NITRITE	14797-65-0	10.00	90.00	110.00		30.00
SULFATE AS SO4	14808-79-8	10.00	90.00	110.00		30.00
QC Type: Matrix_Spike						
NITRATE	14797-55-8		90.00	110.00		30.00
NITRITE	14797-65-0		90.00	110.00		30.00
SULFATE AS SO4	14808-79-8		90.00	110.00		30.00
Method: 3500_F+2_D_Calc **Matrix: AQ**						
QC Type: Laboratory_Control_Sample						
Ferrous Iron	15438-31-0		80.00	120.00		

Analyte Name	Analyte Label (CAS)	Lower Rejection (%)	Lower Recovery (%)	Upper Recovery (%)	Upper Rejection (%)	RPD (%)
Method: 3500_F+2_D_Calc	**Matrix: AQ**					
QC Type: Matrix_Spike						
Ferrous Iron	15438-31-0		80.00	120.00		20.00
Method: 3500_FE_D	**Matrix: AQ**					
QC Type: Laboratory_Control_Sample						
Ferric Iron	STL00113	10.00	80.00	120.00		
Ferrous Iron	15438-31-0	10.00	80.00	120.00		
IRON	7439-89-6	10.00	80.00	120.00		
QC Type: Matrix_Spike						
Ferric Iron	STL00113		80.00	120.00		20.00
Ferrous Iron	15438-31-0		80.00	120.00		20.00
IRON	7439-89-6		80.00	120.00		20.00
Method: 6010C	**Matrix: Water**					
QC Type: Laboratory_Control_Sample						
CALCIUM	7440-70-2	50.00	80.00	120.00		20.00
CHROMIUM	7440-47-3	50.00	80.00	120.00		20.00
IRON	7439-89-6	50.00	80.00	120.00		20.00
MAGNESIUM	7439-95-4	50.00	80.00	120.00		20.00
MANGANESE	7439-96-5	50.00	80.00	120.00		20.00
TITANIUM	7440-32-6	50.00	80.00	120.00		20.00
QC Type: Matrix_Spike						
CALCIUM	7440-70-2		80.00	120.00		20.00
CHROMIUM	7440-47-3		80.00	120.00		20.00
IRON	7439-89-6		80.00	120.00		20.00
MAGNESIUM	7439-95-4		80.00	120.00		20.00
MANGANESE	7439-96-5		80.00	120.00		20.00
TITANIUM	7440-32-6		80.00	120.00		20.00

Analyte Name	Analyte Label (CAS)	Lower Rejection (%)	Lower Recovery (%)	Upper Recovery (%)	Upper Rejection (%)	RPD (%)
Method: 7196A **Matrix: AQ**						
QC Type: Laboratory_Control_Sample						
HEXAVALENT CHROMIUM	18540-29-9	10.00	85.00	115.00		20.00
QC Type: Matrix_Spike						
HEXAVALENT CHROMIUM	18540-29-9		85.00	115.00		20.00
Method: 7196A_CR3 **Matrix: AQ**						
QC Type: Laboratory_Control_Sample						
CHROMIUM	7440-47-3	10.00	85.00	115.00		20.00
Cr (III)	16065-83-1	10.00	85.00	115.00		20.00
QC Type: Matrix_Spike						
CHROMIUM	7440-47-3		85.00	115.00		20.00
Cr (III)	16065-83-1		85.00	115.00		20.00
Method: 8260B **Matrix: Soil**						
QC Type: Laboratory_Control_Sample						
1,1,1,2-TETRACHLOROETHANE	630-20-6	10.00	75.00	125.00		30.00
1,1,1-TRICHLOROETHANE	71-55-6	10.00	70.00	135.00		30.00
1,1,2,2-TETRACHLOROETHANE	79-34-5	10.00	55.00	130.00		30.00
1,1,2-TRICHLOROETHANE	79-00-5	10.00	60.00	125.00		30.00
1,1-DICHLOROETHANE	75-34-3	10.00	75.00	125.00		30.00
1,1-DICHLOROETHENE	75-35-4	10.00	65.00	135.00		30.00
1,1-DICHLOROPROPENE	563-58-6	10.00	70.00	135.00		30.00
1,2,3-TRICHLOROBENZENE	87-61-6	10.00	60.00	135.00		30.00
1,2,3-TRICHLOROPROPANE	96-18-4	10.00	65.00	130.00		30.00
1,2,4-TRICHLOROBENZENE	120-82-1	10.00	65.00	130.00		30.00
1,2,4-TRIMETHYLBENZENE	95-63-6	10.00	65.00	135.00		30.00
1,2-DIBROMO-3-CHLOROPROPANE	96-12-8	10.00	40.00	135.00		30.00
1,2-DIBROMOETHANE	106-93-4	10.00	70.00	125.00		30.00
1,2-DICHLOROBENZENE	95-50-1	10.00	75.00	120.00		30.00
1,2-DICHLOROETHANE	107-06-2	10.00	70.00	135.00		30.00

Analyte Name	Analyte Label (CAS)	Lower Rejection (%)	Lower Recovery (%)	Upper Recovery (%)	Upper Rejection (%)	RPD (%)
Method: 8260B	**Matrix:** Soil					
QC Type: Laboratory_Control_Sample						
1,2-DICHLOROPROPANE	78-87-5	10.00	70.00	120.00		30.00
1,3,5-TRIMETHYLBENZENE	108-67-8	10.00	65.00	135.00		30.00
1,3-DICHLOROBENZENE	541-73-1	10.00	70.00	125.00		30.00
1,3-DICHLOROPROPANE	142-28-9	10.00	75.00	125.00		30.00
1,4-DICHLOROBENZENE	106-46-7	10.00	70.00	125.00		30.00
2,2-DICHLOROPROPANE	594-20-7	10.00	65.00	135.00		30.00
2-BUTANONE	78-93-3	10.00	30.00	160.00		30.00
2-CHLOROTOLUENE	95-49-8	10.00	70.00	130.00		30.00
2-HEXANONE	591-78-6	10.00	45.00	145.00		30.00
4-CHLOROTOLUENE	106-43-4	10.00	75.00	125.00		30.00
4-METHYL-2-PENTANONE	108-10-1	10.00	45.00	145.00		30.00
ACETONE	67-64-1	10.00	20.00	160.00		30.00
BENZENE	71-43-2	10.00	75.00	125.00		30.00
BROMOBENZENE	108-86-1	10.00	65.00	120.00		30.00
BROMOCHLOROMETHANE	74-97-5	10.00	70.00	125.00		30.00
BROMODICHLOROMETHANE	75-27-4	10.00	70.00	130.00		30.00
BROMOFORM	75-25-2	10.00	55.00	135.00		30.00
BROMOMETHANE	74-83-9	10.00	30.00	160.00		30.00
CARBON DISULFIDE	75-15-0	10.00	45.00	160.00		30.00
CARBON TETRACHLORIDE	56-23-5	10.00	65.00	135.00		30.00
CHLOROBENZENE	108-90-7	10.00	75.00	125.00		30.00
CHLOROETHANE	75-00-3	10.00	40.00	155.00		30.00
CHLOROFORM	67-66-3	10.00	70.00	125.00		30.00
CHLOROMETHANE	74-87-3	10.00	50.00	130.00		30.00
CIS-1,2-DICHLOROETHENE	156-59-2	10.00	65.00	125.00		30.00
CIS-1,3-DICHLOROPROPENE	10061-01-5	10.00	70.00	125.00		30.00
DIBROMOCHLOROMETHANE	124-48-1	10.00	65.00	130.00		30.00
DIBROMOMETHANE	74-95-3	10.00	75.00	130.00		30.00
DICHLORODIFLUOROMETHANE	75-71-8	10.00	35.00	135.00		30.00
ETHYLBENZENE	100-41-4	10.00	75.00	125.00		30.00
HEXACHLOROBUTADIENE	87-68-3	10.00	55.00	140.00		30.00
ISOPROPYLBENZENE	98-82-8	10.00	75.00	130.00		30.00

Analyte Name	Analyte Label (CAS)	Lower Rejection (%)	Lower Recovery (%)	Upper Recovery (%)	Upper Rejection (%)	RPD (%)
Method: 8260B	**Matrix:** Soil					
QC Type: Laboratory_Control_Sample						
m,p-Xylene	179601-23-1	10.00	80.00	125.00		30.00
METHYLENE CHLORIDE	75-09-2	10.00	55.00	140.00		30.00
NAPHTHALENE	91-20-3	10.00	40.00	125.00		30.00
N-BUTYLBENZENE	104-51-8	10.00	65.00	140.00		30.00
N-PROPYLBENZENE	103-65-1	10.00	65.00	135.00		30.00
O-XYLENE	95-47-6	10.00	75.00	125.00		30.00
P-ISOPROPYLTOLUENE	99-87-6	10.00	75.00	135.00		30.00
SEC-BUTYLBENZENE	135-98-8	10.00	65.00	130.00		30.00
STYRENE	100-42-5	10.00	75.00	125.00		30.00
TERT-BUTYLBENZENE	98-06-6	10.00	65.00	130.00		30.00
TETRACHLOROETHENE	127-18-4	10.00	65.00	140.00		30.00
TOLUENE	108-88-3	10.00	70.00	125.00		30.00
TRANS-1,2-DICHLOROETHENE	156-60-5	10.00	65.00	135.00		30.00
TRANS-1,3-DICHLOROPROPENE	10061-02-6	10.00	65.00	125.00		30.00
TRICHLOROETHENE	79-01-6	10.00	75.00	125.00		30.00
TRICHLOROFLUOROMETHANE	75-69-4	10.00	25.00	185.00		30.00
VINYL CHLORIDE	75-01-4	10.00	60.00	125.00		30.00
QC Type: Matrix_Spike						
1,1,1,2-TETRACHLOROETHANE	630-20-6	0.00	75.00	125.00		30.00
1,1,1-TRICHLOROETHANE	71-55-6	0.00	70.00	135.00		30.00
1,1,2,2-TETRACHLOROETHANE	79-34-5	0.00	55.00	130.00		30.00
1,1,2-TRICHLOROETHANE	79-00-5	0.00	60.00	125.00		30.00
1,1-DICHLOROETHANE	75-34-3	0.00	75.00	125.00		30.00
1,1-DICHLOROETHENE	75-35-4	0.00	65.00	135.00		30.00
1,1-DICHLOROPROPENE	563-58-6	0.00	70.00	135.00		30.00
1,2,3-TRICHLOROBENZENE	87-61-6	0.00	60.00	135.00		30.00
1,2,3-TRICHLOROPROPANE	96-18-4	0.00	65.00	130.00		30.00
1,2,4-TRICHLOROBENZENE	120-82-1	0.00	65.00	130.00		30.00
1,2,4-TRIMETHYLBENZENE	95-63-6	0.00	65.00	135.00		30.00
1,2-DIBROMO-3-CHLOROPROPANE	96-12-8	0.00	40.00	135.00		30.00
1,2-DIBROMOETHANE	106-93-4	0.00	70.00	125.00		30.00

Analyte Name	Analyte Label (CAS)	Lower Rejection (%)	Lower Recovery (%)	Upper Recovery (%)	Upper Rejection (%)	RPD (%)
Method: 8260B	**Matrix:** Soil					
QC Type: Matrix_Spike						
1,2-DICHLOROBENZENE	95-50-1	0.00	75.00	120.00		30.00
1,2-DICHLOROETHANE	107-06-2	0.00	70.00	135.00		30.00
1,2-DICHLOROPROPANE	78-87-5	0.00	70.00	120.00		30.00
1,3,5-TRIMETHYLBENZENE	108-67-8	0.00	65.00	135.00		30.00
1,3-DICHLOROBENZENE	541-73-1	0.00	70.00	125.00		30.00
1,3-DICHLOROPROPANE	142-28-9	0.00	75.00	125.00		30.00
1,4-DICHLOROBENZENE	106-46-7	0.00	70.00	125.00		30.00
2,2-DICHLOROPROPANE	594-20-7	0.00	65.00	135.00		30.00
2-BUTANONE	78-93-3	0.00	30.00	160.00		30.00
2-CHLOROTOLUENE	95-49-8	0.00	70.00	130.00		30.00
2-HEXANONE	591-78-6	0.00	45.00	145.00		30.00
4-CHLOROTOLUENE	106-43-4	0.00	75.00	125.00		30.00
4-METHYL-2-PENTANONE	108-10-1	0.00	45.00	145.00		30.00
ACETONE	67-64-1	0.00	20.00	160.00		30.00
BENZENE	71-43-2	0.00	75.00	125.00		30.00
BROMOBENZENE	108-86-1	0.00	65.00	120.00		30.00
BROMOCHLOROMETHANE	74-97-5	0.00	70.00	125.00		30.00
BROMODICHLOROMETHANE	75-27-4	0.00	70.00	130.00		30.00
BROMOFORM	75-25-2	0.00	55.00	135.00		30.00
BROMOMETHANE	74-83-9	0.00	30.00	160.00		30.00
CARBON DISULFIDE	75-15-0	0.00	45.00	160.00		30.00
CARBON TETRACHLORIDE	56-23-5	0.00	65.00	135.00		30.00
CHLOROBENZENE	108-90-7	0.00	75.00	125.00		30.00
CHLOROETHANE	75-00-3	0.00	40.00	155.00		30.00
CHLOROFORM	67-66-3	0.00	70.00	125.00		30.00
CHLOROMETHANE	74-87-3	0.00	50.00	130.00		30.00
CIS-1,2-DICHLOROETHENE	156-59-2	0.00	65.00	125.00		30.00
CIS-1,3-DICHLOROPROPENE	10061-01-5	0.00	70.00	125.00		30.00
DIBROMOCHLOROMETHANE	124-48-1	0.00	65.00	130.00		30.00
DIBROMOMETHANE	74-95-3	0.00	75.00	130.00		30.00
DICHLORODIFLUOROMETHANE	75-71-8	0.00	35.00	135.00		30.00
ETHYLBENZENE	100-41-4	0.00	75.00	125.00		30.00

Analyte Name	Analyte Label (CAS)	Lower Rejection (%)	Lower Recovery (%)	Upper Recovery (%)	Upper Rejection (%)	RPD (%)
Method: 8260B	**Matrix: Soil**					
QC Type: Matrix_Spike						
HEXACHLOROBUTADIENE	87-68-3	0.00	55.00	140.00		30.00
ISOPROPYLBENZENE	98-82-8	0.00	75.00	130.00		30.00
m,p-Xylene	179601-23-1	0.00	80.00	125.00		30.00
METHYLENE CHLORIDE	75-09-2	0.00	55.00	140.00		30.00
NAPHTHALENE	91-20-3	0.00	40.00	125.00		30.00
N-BUTYLBENZENE	104-51-8	0.00	65.00	140.00		30.00
N-PROPYLBENZENE	103-65-1	0.00	65.00	135.00		30.00
O-XYLENE	95-47-6	0.00	75.00	125.00		30.00
P-ISOPROPYLTOLUENE	99-87-6	0.00	75.00	135.00		30.00
SEC-BUTYLBENZENE	135-98-8	0.00	65.00	130.00		30.00
STYRENE	100-42-5	0.00	75.00	125.00		30.00
TERT-BUTYLBENZENE	98-06-6	0.00	65.00	130.00		30.00
TETRACHLOROETHENE	127-18-4	0.00	65.00	140.00		30.00
TOLUENE	108-88-3	0.00	70.00	125.00		30.00
TRANS-1,2-DICHLOROETHENE	156-60-5	0.00	65.00	135.00		30.00
TRANS-1,3-DICHLOROPROPENE	10061-02-6	0.00	65.00	125.00		30.00
TRICHLOROETHENE	79-01-6	0.00	75.00	125.00		30.00
TRICHLOROFLUOROMETHANE	75-69-4	0.00	25.00	185.00		30.00
VINYL CHLORIDE	75-01-4	0.00	50.00	125.00		30.00
QC Type: Surrogate						
4-Bromofluorobenzene	460-00-4	0.00	85	120		
TOLUENE-D8	2037-26-5	0.00	85	115		
Method: 8260B	**Matrix: Water**					
QC Type: Laboratory_Control_Sample						
1,1,1-TRICHLOROETHANE	71-55-6	10.00	76.00	126.00		30.00
1,1,2,2-TETRACHLOROETHANE	79-34-5	10.00	71.00	127.00		30.00
1,1,2-TRICHLORO-1,2,2-TRIFLUOROETHANE	76-13-1	10.00	72.00	139.00		30.00
1,1,2-TRICHLOROETHANE	79-00-5	10.00	69.00	127.00		30.00
1,1-DICHLOROETHANE	75-34-3	10.00	69.00	132.00		30.00
1,1-DICHLOROETHENE	75-35-4	10.00	73.00	134.00		30.00

Analyte Name	Analyte Label (CAS)	Lower Rejection (%)	Lower Recovery (%)	Upper Recovery (%)	Upper Rejection (%)	RPD (%)
Method: 8260B	**Matrix: Water**					
QC Type: Laboratory_Control_Sample						
1,2,4-TRICHLOROBENZENE	120-82-1	10.00	67.00	134.00		30.00
1,2-DIBROMO-3-CHLOROPROPANE	96-12-8	10.00	57.00	126.00		50.00
1,2-DIBROMOETHANE	106-93-4	10.00	75.00	127.00		30.00
1,2-DICHLOROBENZENE	95-50-1	10.00	77.00	124.00		30.00
1,2-DICHLOROETHANE	107-06-2	10.00	75.00	120.00		30.00
1,2-DICHLOROPROPANE	78-87-5	10.00	71.00	126.00		30.00
1,3-DICHLOROBENZENE	541-73-1	10.00	79.00	123.00		30.00
1,4-DICHLOROBENZENE	106-46-7	10.00	76.00	124.00		30.00
2-BUTANONE	78-93-3	10.00	55.00	142.00		30.00
2-HEXANONE	591-78-6	10.00	52.00	149.00		30.00
4-METHYL-2-PENTANONE	108-10-1	10.00	51.00	143.00		30.00
ACETONE	67-64-1	10.00	39.00	162.00		50.00
BENZENE	71-43-2	10.00	74.00	123.00		30.00
BROMODICHLOROMETHANE	75-27-4	10.00	72.00	129.00		30.00
BROMOFORM	75-25-2	10.00	60.00	134.00		30.00
BROMOMETHANE	74-83-9	10.00	10.00	171.00		50.00
CARBON DISULFIDE	75-15-0	10.00	63.00	142.00		30.00
CARBON TETRACHLORIDE	56-23-5	10.00	70.00	131.00		30.00
CHLOROBENZENE	108-90-7	10.00	79.00	120.00		30.00
CHLOROETHANE	75-00-3	10.00	47.00	148.00		40.00
CHLOROFORM	67-66-3	10.00	76.00	128.00		30.00
CHLOROMETHANE	74-87-3	10.00	47.00	151.00		30.00
CIS-1,2-DICHLOROETHENE	156-59-2	10.00	78.00	127.00		30.00
CIS-1,3-DICHLOROPROPENE	10061-01-5	10.00	73.00	128.00		30.00
CYCLOHEXANE	110-82-7	10.00	68.00	137.00		30.00
DIBROMOCHLOROMETHANE	124-48-1	10.00	63.00	134.00		50.00
DICHLORODIFLUOROMETHANE	75-71-8	10.00	41.00	165.00		50.00
ETHYLBENZENE	100-41-4	10.00	78.00	125.00		30.00
ISOPROPYLBENZENE	98-82-8	10.00	72.00	129.00		30.00
METHYL ACETATE	79-20-9	10.00	26.00	182.00		30.00
METHYL CYCLOHEXANE	108-87-2	10.00	72.00	133.00		30.00
METHYL TERT-BUTYL ETHER	1634-04-4	10.00	76.00	126.00		30.00

Analyte Name	Analyte Label (CAS)	Lower Rejection (%)	Lower Recovery (%)	Upper Recovery (%)	Upper Rejection (%)	RPD (%)
Method: 8260B	**Matrix: Water**					
QC Type: Laboratory_Control_Sample						
METHYLENE CHLORIDE	75-09-2	10.00	79.00	124.00		30.00
STYRENE	100-42-5	10.00	75.00	129.00		30.00
TETRACHLOROETHENE	127-18-4	10.00	77.00	128.00		30.00
TOLUENE	108-88-3	10.00	77.00	125.00		30.00
TRANS-1,2-DICHLOROETHENE	156-60-5	10.00	78.00	130.00		30.00
TRANS-1,3-DICHLOROPROPENE	10061-02-6	10.00	72.00	127.00		50.00
TRICHLOROETHENE	79-01-6	10.00	80.00	120.00		30.00
TRICHLOROFLUOROMETHANE	75-69-4	10.00	68.00	144.00		30.00
VINYL CHLORIDE	75-01-4	10.00	58.00	141.00		30.00
Xylene (Total)	1330-20-7	10.00	80.00	124.00		30.00
QC Type: Matrix_Spike						
1,1,1-TRICHLOROETHANE	71-55-6	10.00	76.00	126.00		30.00
1,1,2,2-TETRACHLOROETHANE	79-34-5	10.00	71.00	127.00		30.00
1,1,2-TRICHLORO-1,2,2-TRIFLUOROETHANE	76-13-1	10.00	72.00	139.00		30.00
1,1,2-TRICHLOROETHANE	79-00-5	10.00	69.00	127.00		30.00
1,1-DICHLOROETHANE	75-34-3	10.00	69.00	132.00		30.00
1,1-DICHLOROETHENE	75-35-4	10.00	73.00	134.00		30.00
1,2,4-TRICHLOROBENZENE	120-82-1	10.00	67.00	134.00		30.00
1,2-DIBROMO-3-CHLOROPROPANE	96-12-8	10.00	57.00	126.00		50.00
1,2-DIBROMOETHANE	106-93-4	10.00	75.00	127.00		30.00
1,2-DICHLOROBENZENE	95-50-1	10.00	77.00	124.00		30.00
1,2-DICHLOROETHANE	107-06-2	10.00	75.00	120.00		30.00
1,2-DICHLOROPROPANE	78-87-5	10.00	71.00	126.00		30.00
1,3-DICHLOROBENZENE	541-73-1	10.00	79.00	123.00		30.00
1,4-DICHLOROBENZENE	106-46-7	10.00	76.00	124.00		30.00
2-BUTANONE	78-93-3	10.00	55.00	142.00		30.00
2-HEXANONE	591-78-6	10.00	52.00	149.00		30.00
4-METHYL-2-PENTANONE	108-10-1	10.00	51.00	143.00		30.00
ACETONE	67-64-1	10.00	39.00	162.00		50.00
BENZENE	71-43-2	10.00	74.00	123.00		30.00
BROMODICHLOROMETHANE	75-27-4	10.00	72.00	129.00		30.00

Analyte Name	Analyte Label (CAS)	Lower Rejection (%)	Lower Recovery (%)	Upper Recovery (%)	Upper Rejection (%)	RPD (%)
Method: 8260B	**Matrix:** Water					
QC Type: Matrix_Spike						
BROMOFORM	75-25-2	10.00	60.00	134.00		30.00
BROMOMETHANE	74-83-9	10.00	10.00	171.00		50.00
CARBON DISULFIDE	75-15-0	10.00	63.00	142.00		30.00
CARBON TETRACHLORIDE	56-23-5	10.00	70.00	131.00		30.00
CHLOROBENZENE	108-90-7	10.00	79.00	120.00		30.00
CHLOROETHANE	75-00-3	10.00	47.00	148.00		40.00
CHLOROFORM	67-66-3	10.00	76.00	128.00		30.00
CHLOROMETHANE	74-87-3	10.00	47.00	151.00		30.00
CIS-1,2-DICHLOROETHENE	156-59-2	10.00	78.00	127.00		30.00
CIS-1,3-DICHLOROPROPENE	10061-01-5	10.00	73.00	128.00		30.00
CYCLOHEXANE	110-82-7	10.00	68.00	137.00		30.00
DIBROMOCHLOROMETHANE	124-48-1	10.00	63.00	134.00		50.00
DICHLORODIFLUOROMETHANE	75-71-8	10.00	41.00	165.00		50.00
ETHYLBENZENE	100-41-4	10.00	78.00	125.00		30.00
ISOPROPYLBENZENE	98-82-8	10.00	72.00	129.00		30.00
METHYL ACETATE	79-20-9	10.00	26.00	182.00		30.00
METHYL CYCLOHEXANE	108-87-2	10.00	72.00	133.00		30.00
METHYL TERT-BUTYL ETHER	1634-04-4	10.00	76.00	126.00		30.00
METHYLENE CHLORIDE	75-09-2	10.00	79.00	124.00		30.00
STYRENE	100-42-5	10.00	75.00	129.00		30.00
TETRACHLOROETHENE	127-18-4	10.00	77.00	128.00		30.00
TOLUENE	108-88-3	10.00	77.00	125.00		30.00
TRANS-1,2-DICHLOROETHENE	156-60-5	10.00	78.00	130.00		30.00
TRANS-1,3-DICHLOROPROPENE	10061-02-6	10.00	72.00	127.00		50.00
TRICHLOROETHENE	79-01-6	10.00	80.00	120.00		30.00
TRICHLOROFLUOROMETHANE	75-69-4	10.00	68.00	144.00		30.00
VINYL CHLORIDE	75-01-4	10.00	58.00	141.00		30.00
Xylene (Total)	1330-20-7	10.00	80.00	124.00		30.00
QC Type: Surrogate						
1,2-DICHLOROETHANE-D4	17060-07-0	0.00	70	120		
4-Bromofluorobenzene	460-00-4	0.00	75	120		

Analyte Name	Analyte Label (CAS)	Lower Rejection (%)	Lower Recovery (%)	Upper Recovery (%)	Upper Rejection (%)	RPD (%)
Method: 8260B	**Matrix: Water**					
QC Type: Surrogate						
DIBROMOFLUOROMETHANE	1868-53-7	0.00	85	115		
TOLUENE-D8	2037-26-5	0.00	85	120		
Method: 8260C	**Matrix: Water**					
QC Type: Laboratory_Control_Sample						
1,1,2,2-TETRACHLOROETHANE	79-34-5	10.00	65.00	130.00		30.00
1,1,2-TRICHLOROETHANE	79-00-5	10.00	75.00	125.00		30.00
1,1-DICHLOROETHANE	75-34-3	10.00	70.00	135.00		30.00
1,1-DICHLOROETHENE	75-35-4	10.00	70.00	130.00		30.00
1,2,4-TRIMETHYLBENZENE	95-63-6	10.00	75.00	130.00		30.00
1,2-DICHLOROETHANE	107-06-2	10.00	70.00	130.00		30.00
1,3,5-TRIMETHYLBENZENE	108-67-8	10.00	75.00	130.00		30.00
BENZENE	71-43-2	10.00	80.00	120.00		30.00
CARBON TETRACHLORIDE	56-23-5	10.00	65.00	140.00		30.00
CHLOROFORM	67-66-3	10.00	65.00	135.00		30.00
CIS-1,2-DICHLOROETHENE	156-59-2	10.00	70.00	125.00		30.00
ETHYLBENZENE	100-41-4	10.00	75.00	125.00		30.00
m,p-Xylene	179601-23-1	10.00	75.00	130.00		30.00
NAPHTHALENE	91-20-3	10.00	55.00	140.00		30.00
O-XYLENE	95-47-6	10.00	80.00	120.00		30.00
TETRACHLOROETHENE	127-18-4	10.00	45.00	150.00		30.00
TOLUENE	108-88-3	10.00	75.00	120.00		30.00
TRANS-1,2-DICHLOROETHENE	156-60-5	10.00	60.00	140.00		30.00
TRICHLOROETHENE	79-01-6	10.00	70.00	125.00		30.00
VINYL CHLORIDE	75-01-4	10.00	50.00	145.00		30.00
QC Type: Matrix_Spike						
1,1,2,2-TETRACHLOROETHANE	79-34-5	0.00	65.00	130.00		30.00
1,1,2-TRICHLOROETHANE	79-00-5	0.00	75.00	125.00		30.00
1,1-DICHLOROETHANE	75-34-3	0.00	70.00	135.00		30.00
1,1-DICHLOROETHENE	75-35-4	0.00	70.00	130.00		30.00
1,2,4-TRIMETHYLBENZENE	95-63-6	0.00	75.00	130.00		30.00

Analyte Name	Analyte Label (CAS)	Lower Rejection (%)	Lower Recovery (%)	Upper Recovery (%)	Upper Rejection (%)	RPD (%)
Method: 8260C **Matrix: Water**						
QC Type: Matrix_Spike						
1,2-DICHLOROETHANE	107-06-2	0.00	70.00	130.00		30.00
1,3,5-TRIMETHYLBENZENE	108-67-8	0.00	75.00	130.00		30.00
BENZENE	71-43-2	0.00	80.00	120.00		30.00
CARBON TETRACHLORIDE	56-23-5	0.00	65.00	140.00		30.00
CHLOROFORM	67-66-3	0.00	65.00	135.00		30.00
CIS-1,2-DICHLOROETHENE	156-59-2	0.00	70.00	125.00		30.00
ETHYLBENZENE	100-41-4	0.00	75.00	125.00		30.00
m,p-Xylene	179601-23-1	0.00	75.00	130.00		30.00
NAPHTHALENE	91-20-3	0.00	55.00	140.00		30.00
O-XYLENE	95-47-6	0.00	80.00	120.00		30.00
TETRACHLOROETHENE	127-18-4	0.00	45.00	150.00		30.00
TOLUENE	108-88-3	0.00	75.00	120.00		30.00
TRANS-1,2-DICHLOROETHENE	156-60-5	0.00	60.00	140.00		30.00
TRICHLOROETHENE	79-01-6	0.00	70.00	125.00		30.00
VINYL CHLORIDE	75-01-4	0.00	50.00	145.00		30.00
QC Type: Surrogate						
4-Bromofluorobenzene	460-00-4	0.00	75	120		
Method: 9040B **Matrix: AQ**						
QC Type: Laboratory_Control_Sample						
PH	PH		63.00	158.00		40.00
Method: 9060A **Matrix: AQ**						
QC Type: Laboratory_Control_Sample						
Total Dissolved Carbon	TDC	10.00	80.00	120.00		25.00
TOTAL ORGANIC CARBON (TOC)	TOC	10.00	80.00	120.00		25.00
QC Type: Matrix_Spike						
Total Dissolved Carbon	TDC		80.00	120.00		25.00
TOTAL ORGANIC CARBON (TOC)	TOC		80.00	120.00		25.00

Analyte Name	Analyte Label (CAS)	Lower Rejection (%)	Lower Recovery (%)	Upper Recovery (%)	Upper Rejection (%)	RPD (%)
Method: SM 2320B **Matrix: Water**						
QC Type: Laboratory_Control_Sample						
Carbonate Alkalinity as CaCO3	STL00154		80.00	120.00		
Method: SM2540C **Matrix: AQ**						
QC Type: Laboratory_Control_Sample						
TOTAL DISSOLVED SOLIDS	TDS	10.00	80.00	120.00		25.00
Method: SM2540D **Matrix: AQ**						
QC Type: Laboratory_Control_Sample						
Total suspended solids	TSS	10.00	80.00	120.00		25.00

Approved

Limit of Detection Study

Alpha Analytical, Inc.
Mansfield, MA

Page 1 of 2	
Analyst:	MB
Date:	2/28/2012
Instrument ID:	Airpiano 2
Prep Method:	N/A
Prep Chemist:	N/A

Method: EPA TO-15SIM 2012

GC **Toxic Organic Air Pollutants** **Air**

	Compounds	DL*	Bottom of LOD Range	Top of LOD Range	Concentration Run at	Results Verified	RL	Comments
		ppbV	ppbV	ppbV	ppbV		ppbV	
1	1,1,1,2-tetrachloroethane	0.00405	0.0081	0.0162	0.01	Verified - RB	0.02	
2	MTBE	0.00444	0.0089	0.0178	0.01	Verified - RB	0.02	
3	1,3,5-trimethylbenzene	0.00464	0.0093	0.0185	0.02	Verified - RB	0.02	
4	Freon-114	0.00495	0.0099	0.0198	0.01	Verified - RB	0.05	
5	chloroform	0.00560	0.0112	0.0224	0.01	Verified - RB	0.02	
6	trans-1,2-dichloroethene	0.00570	0.0114	0.0228	0.01	Verified - RB	0.04	
7	dichlorodifluoromethane	0.00582	0.0116	0.0233	0.01	Verified - RB	0.02	
8	Freon 113	0.00598	0.0120	0.0239	0.01	Verified - RB	0.02	
9	1,2-dichloropropane	0.00605	0.0121	0.0242	0.01	Verified - RB	0.02	
10	1,3-butadiene	0.00636	0.0127	0.0254	0.01	Verified - RB	0.02	
11	1,1-dichloroethene	0.00655	0.0131	0.0262	0.01	Verified - RB	0.05	
12	cis-1,2-dichloroethene	0.00658	0.0132	0.0263	0.01	Verified - RB	0.02	
13	vinyl chloride	0.00663	0.0133	0.0265	0.01	Verified - RB	0.02	
14	1,2-dichlorobenzene	0.00675	0.0135	0.0270	0.01	Verified - RB	0.02	
15	1,2,4-trimethylbenzene	0.00699	0.0140	0.0280	0.02	Verified - RB	0.05	
16	1,3-dichlorobenzene	0.00703	0.0141	0.0281	0.01	Verified - RB	0.50	
17	chloroethane	0.00705	0.0141	0.0282	0.01	Verified - RB	0.02	
18	1,1-dichloroethane	0.00706	0.0141	0.0282	0.01	Verified - RB	0.02	
19	1,1,1-trichloroethane	0.00711	0.0142	0.0284	0.01	Verified - RB	0.50	
20	ethylbenzene	0.00719	0.0144	0.0288	0.01	Verified - RB	0.02	
21	trichloroethene	0.00720	0.0144	0.0288	0.01	Verified - RB	0.02	
22	1,1,2,2-tetrachloroethane	0.00734	0.0147	0.0294	0.02	Verified - RB	0.02	
23	dibromochloromethane	0.00758	0.0152	0.0303	0.02	Verified - RB	0.02	
24	o-xylene	0.00764	0.0153	0.0305	0.01	Verified - RB	0.05	
25	styrene	0.00768	0.0154	0.0307	0.02	Verified - RB	0.02	
26	carbon tetrachloride	0.00784	0.0157	0.0314	0.01	Verified - RB	0.50	
27	1,4-dichlorobenzene	0.00785	0.0157	0.0314	0.01	Verified - RB	0.02	
28	trichlorofluoromethane	0.00785	0.0157	0.0314	0.01	Verified - RB	0.02	
29	bromomethane	0.00800	0.0160	0.0320	0.01	Verified - RB	0.50	
30	trans-1,3-dichloropropene	0.00801	0.0160	0.0321	0.02	Verified - RB	0.02	
31	bromodichloromethane	0.00806	0.0161	0.0323	0.01	Verified - RB	0.02	
32	1,2-dibromoethane	0.00809	0.0162	0.0323	0.01	Verified - RB	0.02	
33	tetrachloroethene	0.00812	0.0162	0.0325	0.01	Verified - RB	0.02	
34	chlorobenzene	0.00844	0.0169	0.0338	0.01	Verified - RB	0.02	
35	cis-1,3-dichloropropene	0.00847	0.0169	0.0339	0.02	Verified - RB	0.02	

Comments: * Airlab7 '08, Airlab 8 '07, Airpiano1 '08
Airpiano2 '09 - highest MDL

QA Review:

Limit of Detection Study

Alpha Analytical, Inc.
Mansfield, MA

Page 2 of 2	
Analyst:	MB
Date:	2/28/2012
Instrument ID:	Airpiano 2
Prep Method:	N/A
Prep Chemist:	N/A

Method: EPA TO-15SIM 2012

GC **Toxic Organic Air Pollutants** **Air**

	Compounds	DL*	Bottom of LOD Range	Top of LOD Range	Concentration Run at	Results Verified	RL	Comments
		ppbV	ppbV	ppbV	ppbV		ppbV	
36	1,2-dichloroethane	0.00849	0.01698	0.03396	0.01	Verified - RB	0.02	
37	1,1,2-trichloroethane	0.00902	0.01804	0.03607	0.01	Verified - RB	0.05	
38	m+p-xylene	0.00917	0.01834	0.03668	0.01	Verified - RB	0.50	
39	toluene	0.00964	0.01928	0.03855	0.01	Verified - RB	0.02	
40	4-ethyl toluene	0.00984	0.01969	0.03938	0.02	Verified - RB	0.02	
41	1,2,4-trichlorobenzene	0.01022	0.02044	0.04088	0.02	Verified - RB	0.02	
42	hexachlorobutadiene	0.01132	0.02265	0.04529	0.02	Verified - RB	0.02	
43	naphthalene	0.01202	0.02404	0.04808	0.02	Verified - RB	0.50	
44	bromoform	0.01457	0.02913	0.05826	0.02	Verified - RB	0.02	
45	acrylonitrile	0.01517	0.03033	0.06067	0.02	Verified - RB	0.04	
46	benzene	0.02096	0.04192	0.08384	0.04	Verified - RB	0.02	
47	2-butanone	0.02466	0.04932	0.09865	0.04	Verified - RB	0.05	
48	4-methyl-2-pentanone	0.04150	0.08300	0.16600	0.10	Verified - RB	0.05	
49	n-butylbenzene	0.04537	0.09074	0.18148	0.10	Verified - RB	0.02	
50	isopropylbenzene	0.04636	0.09272	0.18544	0.10	Verified - RB	0.50	
51	sec-butylbenzene	0.04707	0.09413	0.18826	0.10	Verified - RB	0.10	
52	chloromethane	0.04766	0.09532	0.19064	0.10	Verified - RB	0.02	
53	p-isopropyltoluene	0.04791	0.09581	0.19163	0.10	Verified - RB	0.50	
54	methylene chloride	0.07500	0.15000	0.30000	0.20	Verified - RB	1.00	
55	acetone	0.73901	1.47801	2.95603	2.50	Verified - RB	2.00	
	Additional Analytes							
1	Halothane	0.00762	0.01525	0.03049	0.01	Verified - RB	0.02	
2	1,2,3-Trichlorobenzene	0.01857	0.03714	0.07428	0.04	Verified - RB	0.05	

Comments: * Airlab7 '08, Airlab 8 '07, Airpiano1 '08
Airpiano2 '09 - highest MDL

B: STANDARD OPERATING PROCEDURES

STANDARD OPERATING PROCEDURE

SEI-4.2.7

CHAIN OF CUSTODY PROCEDURES

SOP Number: SEI-4.2.7
Revision Number: 7

Date Issued: 04/09/93
Date of Revision: 5/10/12

1.0 OBJECTIVE

Chain of custody (COC) procedures documents the historical possession of sample containers and samples. Chain of custody records allow the possession and handling of samples to be tracked from the time of field collection until the samples are delivered to the laboratory. Every effort shall be made to keep as few people as possible in the chain of sample possession.

2.0 POLICIES

1. To maintain a record of sample collection, transfer between personnel, shipment, and receipt by the laboratory, a chain-of-custody record will be completed for each sample shipment by the field personnel or samplers.
2. Custody, as defined by this document, is referred as:
    - It is in someone's actual possession
    - It is in someone's view, after being in their physical possession, or
    - It was in someone's possession and then locked, sealed, or secured in a manner which prevents unsuspected tampering, or
    - It is placed in a designated and secured area
3. Upon receipt at the laboratory, it is the responsibility of the laboratory to note if tampering has occurred during sample shipment. If the coolers or containers are opened before receipt at the laboratory, the project manager will be contacted.

3.0 SAFETY ISSUES

1. The toxicity and carcinogenicity of each test substance used has not been fully established. Each chemical should be regarded as a potential health hazard and exposure should be as low as reasonably achievable.
2. All labels affixed to sampling containers must be carefully read and followed.

3. If necessary and appropriate, all chemicals are required to be received with a Material Safety Data Sheets (MSDS) or appropriate application label. These labels or MSDS shall be made available to all personnel involved in the sampling and testing.

4.0 PROCEDURES

4.1 Chain-of-Custody Record

The record of possession of the samples is kept on a COC form that accompanies each package of samples that are shipped to the lab. The exact format of the COC form may vary based on the sampling activities, but all forms must include the following information (example forms are included in Attachment 1):

1. Name, address, and if necessary, the telephone number of Stone Environmental, Inc.
2. Name (s) of the sampler (s).
3. Project or study designation.
4. Sample identification; if numerous samples are included in the package, a separate list of samples may be attached.
5. Date of sample collection (or dates, if samples in container were collected over more than one day).
6. Type of sample (water, soil, etc.).
7. Analyses to be performed.
8. The name and address of the analytical laboratory to which the samples are being shipped.
9. Signatures of all persons receiving/relinquishing the samples (does not include couriers).
10. Date and time of receipt and date and time of relinquishment for each person in the chain.

If possible, use a carbon copy, multiple page chain of custody form in order to maintain copies of the form throughout the shipment stages. If a multiple copy form is not used, photocopies should be made by the Stone personnel who originally ship the samples in order to document shipment.

4.2 Chain of Custody Procedures

1. The samples must be kept in the possession of the sampler until they can be securely packaged. The sampler should seal samples in a sturdy shipping box or other suitable container.
2. Fill out the COC form with all of the information listed in Section 4.1 above, except for items 9 and 10.
3. Attach the COC form to the outside of the shipping container such that the form can be filled out and signed each time the samples change possession or just inside the cooler. Alternately,

if the items transferred will only be relinquished once or shipped overnight via a courier service, the COC should be included within the shipping container(s). If applicable, a note may be made on the outside of the container to indicate the COC record is inside the container(s).

4. When the sampler delivers the sample container to the laboratory or to a courier, the sampler must enter on the COC form the date and time at which she/he relinquishes the container, then sign the form to signify that she/he has relinquished the container. The recipient of the container must then sign the form to signify that she/he has received the container, and she/he must also enter the date and time on the form.

5. This procedure must be followed each time the sample container changes hands. The first person must enter the date and time of relinquishment and sign the form, and then the next person must enter the date and time of receipt and sign the form. At each change of hands, the person relinquishing the container may take a copy of the form for his records, but the original form must remain with the container.

6. The exception to this procedure is that if a commercial courier is used (for example United Parcel Service, FedX, Airborne, etc.), the Stone tracking report, the bill of lading and/or tracking number from the courier will serve as valid COC documentation. **The courier need not sign the COC form to signify receipt of the samples.**

4.3 Laboratory Sample Receipt and Storage

When the laboratory receives the samples, the appropriate lab personnel should note the condition of the shipping container as described in the Laboratory Quality Systems Manual, then sign the COC form and enter the date and time the container is received. If applicable, the temperature of the sample container upon receipt will be recorded on the COC record. The completed, original form or a verified copy of the original should then be returned to Stone to be filed as a record of the sample possession history.

4.4 Documentation

If a multiple page carbon copy form is utilized copies should be maintained as follows:

- When the COC is filled out and ready to be placed in the shipping container, the sampler signs the COC and takes the bottom copy as a record that the samples were shipped.
- After receipt of samples by the testing facility or laboratory, the receiver signs the COC and may retain a copy (second page) of the COC for their records.
- The testing facility / laboratory should then forward the original COC (first page) to Stone for retention.
- Instructions for third party actions will be described on the COC form.

5.0 RESPONSIBILITIES

5.1 Quality Assurance Unit

1. If necessary and appropriate COC records may be audited by the quality assurance unit.

5.2 Principals/Group Officers/Group Leaders/Personnel

1. To maintain a record of sample collection, transfer between personnel, shipment, and receipt by the laboratory, a COC record will be completed for each sample shipment by the field sampler or field team.
2. No employee of Stone will collect or generate data, or perform any other study functions until they have been properly trained and such training is documented in a training record file as specified in SOP SEI-1.4.*n*.responsibilities.
3. It is the responsibility of the individual employees to follow SOPs covering activities in his/her work area or to identify a deviation from the written SOPs.

5.3 Archivist

The archivist is responsible for the following areas: maintaining of the original documents file including archival of all original data and archiving all original data as described in the SOP.

6.0 DEFINITIONS

1. *Raw data* any laboratory worksheets, records, memoranda, notes, or exact copies thereof, that are the result of original observations and activities of a study and are necessary for the reconstruction and evaluation of the report of that study. In the event that exact transcripts of raw data have been prepared (e.g., tapes which have been transcribed verbatim, dated, and verified accurate by signature), the exact copy or exact transcript may be substituted for the original source as raw data. Raw data may include photographs, microfilm or microfiche copies, computer printouts, magnetic media, including dictated observations, and recorded data from automated instruments.

7.0 REFERENCES

40 CFR Part 160 Good Laboratory Practice Standards, August, 1989.

8.0 TABLES, DIAGRAMS, FLOWCHARTS, AND VALIDATION DATA

1. Attachment 1: Sample Stone Chain of Custody Form

9.0 AUTHORIZATION

Reviewed by: John Hanzas Date: 7/5/2012

John Hanzas, Study Director/Senior GeoScientist

Approved by: Date: 6-28-12

Christopher T. Stone, President

10.0 REVISION HISTORY

Revision number 2:

1. Reformatted under new numbering scheme.

Revision number 3:

1. Section 3.0 (e) - added statement to clarify carrier's role in sample custody.
2. Added sample COC form.

Revision number 4:

1. Reformatted
2. Minor wording edits
3. Samplers may sign or initial form (section 2.1)

Revision number 5:

1. Reformatted.
2. Minor wording edits.
3. Add sections 2.2 and 3.7 detailing the use of multiple page COC forms.
4. Delete requirement of signature / initials of samplers (require only name(s)).
5. Allow for COC to be inserted inside container in section 3.3.

Revision number 6:

1. Change title to COC Procedures from COC Records.
2. Minor wording edits in Section 1.0, Objective.
3. Updated style to match SEI Style Guide. Reformatted using MS Word.
4. Added standardized section headers: 2.0 Policies, 3.0 Safety, 5.0 Responsibilities, 6.0 Definitions, 7.0 References, 8.0 Tables, Diagrams, Flowcharts and Validation data. Authorization moved to Section 9.0, and section10.0 Revision History.

Revision number 7:

1. Update Section 4.2 to allow for the COC to be inside the shipping container.
2. Change Federal Express to FedX

ATTACHMENT 1

Sample Stone Chain of Custody Form

STONE ENVIRONMENTAL INC

CHAIN OF CUSTODY RECORD

STONE ENVIRONMENTAL INC

SEI Study # | Project Name/Client Study #

Study Director | Sampling Personnel (name/signature)

SAMPLE INFORMATION

Sample Identification	Date Collected	Type *	Cont.**	Number of Containers	Analyses Requested				
									Ship To:

Relinquished By: (Signature)	Date/Time	Received By: (Signature)	Date/Time
Relinquished By: (Signature)	Date/Time	Received By: (Signature)	Date/Time
Relinquished By: (Signature)	Date/Time	Received By: (Signature)	Date/Time

Special instructions:
* Type of sample (1) water (2) soil
** Container (1) bag (2) bottle (3) shelby tube (4) acetate sleeves (5) other: _____

Samples shipped with:
☐ ambient air ☐ dry ice ☐ ice

Temperature of samples when received by lab:
☐ room temperature ☐ cold ☐ frozen

Please remit completed, white copy to:

Stone Environmental, Inc.
535 Stone Cutters Way
Montpelier, Vermont 05602

Page __of __

white copy: original **yellow copy:** laboratory **pink copy:** SEI

I:\forms\custody\chain7.cdr

STONE ENVIRONMENTAL INC

Project Number:	Site Name/Location:	Samplers (Signature(s)):	Results and Invoice to: Stone Environmental, Inc. 535 Stone Cutters Way Montpelier, VT 05602 Telephone #: 802-229-4541 Fax: 802-229-5417 Project Manager: PM Direct Phone #
Client Name and Address:			
Client Contact:		ANALYSES REQUESTED Container type[2]	

Sample Identification	DATE/TIME Sampled		Matrix[1]	No. of Containers: Composite	No. of Containers: Grab									Remarks	Perservative added and final pH, if known[3]	Internal Billing Subproject Phase

Sampling Bottles/ Preservative Requirements

[1]**Matrix:** SL-Soil, SG-Soil Gas, GW-Groundwater, SD-Sediment, SW-Surface Water, WW-wastewater, L-liquid, Wipe, Solid, Other-specify_____(i.e., oil, concrete)

[2]**Container(s):** V-VOA 40mL vials, T-Tedlar Airbag **Metals**, check one: ___8RCRA, ___13 PP, ___23 TAL, ___ 14MCP. Method: ___6010, ___ 6020, ___200.7 Other:_____

A - Amber 1L, G-Glass, 1L **Dissolved Metals Field Filtered?** ___Yes ___No **Samples shipped:** _____ice, _____dry ice, _____ambient

M-250 mL-Glass Wide Mouth P-poly/plastic, O-other, specify_____ [3]Preservative: HCL, MeOH, HNO3, H2SO4, NaOH, O-Other, Specify_____

Relinquished by: (Signature)	Date/Time	Received by: (Signature)	Date/Time	Shipped Via: Number:
Relinquished by: (Signature)	Date/Time	Received by: (Signature)	Date/Time	**Turnaround Time Requirements:** <14days must have Pre-Authorization.
Relinquished by: (Signature)	Date/Time	Received by: (Signature)	Date/Time	

Notes/Comments: **Custody Seal Intact?:** Y N N/A Sample Temp at Lab.

EDD Required: Specify Type?_____, (i.e., Equis EZLoad, Excel spreadsheet, Other)

Report Package level needed: ___Level II (results, SS recoveries), ___ Level III (results, all QC), ____Level IV (FULL, CLP-like)

STONE ENVIRONMENTAL INC
CHAIN-OF-CUSTODY RECORD

Page _____ of _____

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Revised 8/4/2011
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STANDARD OPERATING PROCEDURE

SEI-4.5.11

DATA HANDLING, STORAGE, RETRIEVAL AND ERROR CODING

SOP Number: SEI-4.5.11
Revision Number: 11

Date Issued: 09/02/93
Date of Revision: 05/10/12

1.0 OBJECTIVE

This standard operating procedure (SOP) describes the framework for handling study specific (or project specific) raw data and documentation obtained during the process of a study. Provided in this SOP are the essential elements necessary to accurately record activities, measurements, and observations made in the field or during the course of a study and during the processing of data that ultimately is used in a project report.

Definition of Raw Data: *Raw data* any laboratory worksheets, records, memoranda, notes, or exact copies thereof, that are the result of original observations and activities of a study and are necessary for the reconstruction and evaluation of the report of that study. In the event that exact transcripts of raw data have been prepared (e.g., tapes which have been transcribed verbatim, dated, and verified accurate by signature), the exact copy or exact transcript may be substituted for the original source as raw data. Raw data may include photographs, microfilm or microfiche copies, computer printouts, magnetic media, including dictated observations, and recorded data from automated instruments.

2.0 POLICIES

1. Personnel will legibly record data and observations in the field to enable others to reconstruct project events and provide sufficient evidence of activities conducted.
2. All data generated during the conduct of a study, except those that are generated by automated data collection systems, shall be recorded directly, promptly, and legibly in indelible ink.
3. All data entries shall be dated on the day of entry and signed or initialed by the person entering the data. If multiple personnel enter data upon a single page, each entry should be uniquely attributed to its author.
4. Field personnel will use a field logbook, observation and remark form or other designated field form to record activities, measurements, and observations made in the field.

5. Any change in entries shall be made so as not to obscure the original entry, shall indicate the reason for such change and shall be dated and signed or identified at the time of the change. Procedures to follow for error coding are discussed in the procedural section of this SOP.
6. Personnel will document the procedures performed in accordance with Good Laboratory Practices (GLP) Standards, 40 CFR Part 160.
7. All data will be stored in project specific files. All raw data will be archived in accordance with SOP SEI-7.2.*n*.
8. Any access into the archives must be documented in the "Archive Access Log", located with the archivist.

3.0 SAFETY ISSUES

Not applicable to this SOP

4.0 PROCEDURES

Determine the required data to be collected. This can be ascertained from several sources, including: (1) an approved protocol, (2) an approved work plan or Quality Assurance Project Plan (QAPP), (3) an awarded bid, (4) or other documentation that sets forth study design agreed upon by the client and Stone.

4.1 Use and Assembly of Field Study Notebooks and Logbooks

Prior to initiating fieldwork, to the extent possible, a field study notebook or logbook should be obtained and/or assembled to provide an accurate and permanent record of activities, measurements, and observation made in the field. The field notebook should be a self-contained binder that will assist in protecting the field forms, or other appropriate recording media from damage or loss. Once the type of data to be collected is known, search of the Forms subdirectory on Stone's network for specific task related forms which can be customized for a project-specific study or project. Blank forms should be created to be study-specific and saved within the project directory on the Stone network in such a manner that it is clear where the forms are located so any study personnel can print forms as needed for project work.

As forms are saved to the project directory, type in all data that may include, but is not limited to:

1. Stone study number or project number
2. Sponsor or client identification number (if available)
3. Project Location
4. Study Director or Project Manager
5. Study Title
6. Wherever practical include event number, target date and sample identification (ID)

Print copies of all forms as needed. Retain originals and insert a master copy into the notebook. The master copy is then available for photocopying should you run out of forms in the field.

The binder should be labeled in an appropriate manner to aid in identification of the field study notebook.

An approved protocol, work plan or reference QAPP should be included in the notebook. Other items to be included in the notebook, may consist of the following:

1. Applicable protocol or method modifications and deviations as necessary and appropriate.
2. Approved Site Health and Safety Plan (only if it cannot be stored on site).
3. SOPs for data collection.

All original loose-leaf field notes shall be submitted for inclusion in the study or project records.

4.2 Field Data Collection and Handling

All field notes and records must be accurate, orderly, complete, and legible.

A pen (preferably black) or an indelible marker should be used for note taking. Label each page with the date, the signature of the person taking notes (initials may also be appropriate), and the page number. All notes, signatures, and other observations should be entered in the field at the time the notes are taken.

If multiple field personnel make entries onto any sheet or form, it must be clear who entered what data via initialing, bracketing, or other methods.

Any changes in entries shall be made so as not to obscure the original entry, shall indicate the reason for such change, and shall be dated and signed or initialed at the time of the change.

A legible record of data and observations recorded in the field should be sufficient to enable others to reconstruct field events and provide sufficient evidence of activities conducted.

Forms may be removed during actual data collection, but should then be placed in a suitable protective booklet or folder (e.g. a metal field notepad). Every effort should be made to protect data sheets from physical elements that may cause the data to become illegible or lost. Data sheets should be returned to the notebooks as soon as possible (i.e. by the end of the day).

Data may also be recorded on the pre-printed Stone Observation and Remarks Form (O&R) and then included in the project files or the field study notebook.

4.2.1 Error Coding

When errors to the raw data are made or discovered, the original entry should be crossed through with one line in a manner as to not obscure the original entry and designated with a

footnote number. The footnote number should be circled in order to separate it from any surrounding data. The correction should be initialed, dated, and justified in a footnote at the bottom of the page or at the point of entry. The code justification or reason for the edit should be circled to limit confusing the code with the editor's initials. Each entry correction must be given an individual footnote.

Each entry correction must be given an individual footnote number and justified, initialed, and dated at the bottom of the page or on the appropriate medium (see example 1). The following data correction symbols may be used in justifying corrections:

C - Calculation Error	TE – Transcription Error
E - Entry Error	LE- Late Entry
S - Spelling Error	F - Form Change
R - Rounding Error	W - Write Over

(1)

September 15, ~~1997~~1998

(1)(E) RWP, 9/15/98 (1)

[(1)(2)(3)] (LE)(E) RWP, 9/15/98 (2)

The person who originally entered the data should make error corrections. If the person who entered the original data is unavailable, the edit must clearly be supported with appropriate documentation and justification for corrections as noted above.

Any correction made at a later date than the original entry **must** be followed by an "LE" (late entry) designation. This must be footnoted and explained at the bottom of the original data form, on an observation and remarks form, or another suitable medium to clarify the reason for the change and/or addition to the original entry. Similar Late Entry changes may be combined with one set of initials and date, but must still be issued with individual footnote numbers and all footnotes must be contained within one set of brackets (see example (2) above).

Please note that if data generated during the conduct of a study are not recorded directly, promptly, and legibly in ink and all data entries were not dated on the day of entry and signed or initialed by the person entering the data, then "Late Entry" corrections must be indicated in the GLP compliance statement. For example, "The activities which occurred prior to October 12, 2000 were not documented directly and promptly. This was a violation of 40 CFR Part 160.130(e). It is not anticipated that these violations will have an adverse impact on the study."

If multiple errors **of the same type** are discovered at the same time (e.g. during review of page prior to sign off), these errors and corrections can be documented similar to LE changes as above (see example 2). *Note: every effort should be made to make corrections/changes to data*

entries at the time of their discovery. DO NOT wait until other data is recorded and then go back and make corrections.

It some cases, in the on-site laboratory when corrections are made only the initials and date of the corrections are necessary since these corrections are always typically documented as an "entry error" and this type of correction is acceptable according to the National Environmental Laboratory Standards.

4.3 Automated Field Data Collection

In automated data collection systems, the individual responsible for direct data input shall be identified at the time of data input. Any change in automated data entries shall be made so as not to obscure the original entry, shall indicate the reason for change, shall be dated, and the responsible individual shall be identified.

4.4 Certification of Exact Copies of Data

"Raw data" means any laboratory worksheets, records, memoranda, notes, or exact copies thereof, that are the result of original observations and activities of a nonclinical laboratory study and are necessary for reconstruction and evaluation of the report of that study. **In the event that exact transcripts of raw data have been prepared (e.g., tapes which have been transcribed verbatim, dated, and verified accurate by signature), the exact copy or exact transcript may be substituted for the original source as raw data.** When raw data is copied, the copy will be stamped with the following:

"This is an exact copy of the original document."

By_____ Date_____

In the event that there are multiple copies required (e.g., weather data for multiple projects) then the top page should be stamped as above and (if available) the number of pages noted on the top page. At the end of a study all raw data is copied and shipped to the sponsor in accordance with SOP SEI-7.1.*n*.

Copies of transcripts or documents that are not considered raw data are not required to be stamped as above. However, if in doubt, all copies should be stamped as noted above unless otherwise noted.

4.5 Storage of Data

Data will be stored in specific project files. Original raw data sheets will be stored in accordance with SOP SEI-7.2.*n*. All raw data, documentation, records, protocols, specimens, and final reports generated as a result of a study shall be retained. Correspondence and other documents relating to interpretation and evaluation of data, other than those documents contained in the final report also shall be retained as described in 40 CFR 160 § 160.190.

Materials retained or referred to in the archives shall be scanned electronically to permit expedient retrieval.

All study specific computer documentation should ultimately be stored on the network hard drive. Back-up copies will be made of all hard drives on a set schedule as described in the SOP SEI-1.6.n.

4.6 Retrieval of Data

All project data, including both active and inactive project data, will be kept in Stone's filing room, which is kept locked at all times. Archived project data shall be kept separate from active project data in locked filing cabinets.

Any active project data may be accessed by staff if accompanied by the archivist. Any access into the archives must be documented in the "Archive Access Log" located with the archivist (see SOP SEI-7.2.*n*).

Appropriate original raw data, protocols, and final reports shall be located in the archives. Authorized personnel may only access archived data.

5.0 RESPONSIBILITIES

1. All personnel will legibly record data and observations (including phone conversations) in accordance with this SOP to enable others to reconstruct project events and provide sufficient evidence of activities conducted.

2. All personnel will label each page with the date, the signature of the person taking notes (initials may also be appropriate), and the page number. All notes, signatures, and other observations should be entered in the field at the time the notes are taken.

3. If multiple personnel make entries onto any sheet or form, it must be clear who entered what data via initialing, bracketing, or other methods.

6.0 DEFINITIONS

1. *Archives* Include an area for orderly storage and expedient retrieval of original raw data, documentation, protocols, specimens, and interim and final reports retained as a result of a study. Conditions of storage shall minimize deterioration of the documents or specimens in accordance with the requirements for the time period of their retention and the nature of the documents of specimens. An individual shall be identified as responsible for the archives. Only authorized personnel shall enter the archives. Material retained or referred to in the archives shall be indexed to permit expedient retrieval. A testing facility may contract with commercial archives to provide a repository for all material to be retained. Raw data and specimens may be retained elsewhere provided that the archives have specific references to those other locations.

2. *EPA* the U.S. Environmental Protection Agency.

3. *FIFRA* the Federal Insecticide, Fungicide, and Rodenticide Act as amended.

4. *Observations & Remarks Form (O&R):* A pre-printed form, which contains mostly blank space for general note taking. The form typically prompts the user for the study or project designation, the SEI project number, the client or sponsor name, the total number of pages (page *n* of n) and requires a signature and date. The form is generally used to capture notes of one person when another, more specific forms is not available.

5. *Quality Assurance Unit (QAU)* any person or organizational element except the study director, designated by testing facility management to perform the duties related to quality assurance of the studies.

6. *Raw data* any laboratory worksheets, records, memoranda, notes, or exact copies thereof, that are the result of original observations and activities of a study and are necessary for the reconstruction and evaluation of the report of that study. In the event that exact transcripts of raw data have been prepared (e.g., tapes which have been transcribed verbatim, dated, and verified accurate by signature), the exact copy or exact transcript may be substituted for the original source as raw data. Raw data may include photographs, microfilm or microfiche copies, computer printouts, magnetic media, including dictated observations, and recorded data from automated instruments.

7. *Study* means any experiment at one or more test sites, in which a test substance is studied in a test system under laboratory conditions or in the environment to determine or help predict its effects, metabolism, product performance (pesticide efficacy studies only as required by 40 CFR 158.640) environmental and chemical fate, persistence, or residue, or other characteristics in humans, other living organisms, or media. The term "study" does not include basic exploratory studies carried out to determine whether a test substance or a test method has any potential utility.

7.0 REFERENCES

40 CFR Part 160 Good Laboratory Practice Standards, August, 1989.

8.0 TABLES, DIAGRAMS, FLOWCHARTS, AND VALIDATION DATA

None

9.0 AUTHORIZATION

Reviewed by: _____ Date: 7/5/2012

John Hanzas, Study Director/Senior GeoScientist

Approved by: _____ Date: 6-27-12

Christopher T. Stone, President

10.0 REVISION HISTORY

Revision number 2 (SEI-4.3.2):

1. Reformatted under new numbering scheme.

Revision number 3 (SEI-4.3.3):

1. Reformatted
2. Minor wording edits.

Revision number 4 (SEI-4.3.4):

1. Reformatted
2. Minor wording edits
3. Add section 2.4 detailing documentation of entries by multiple personnel.

Revision number 5 (4.3.5)

1. Minor wording edits.

Retire SOP SEI-4.3.5, Combine with 4.5.9.

Revision number 1 (4.11.1):

1. Reformatted under new numbering scheme.

Revision number 2 (4.11.2):

1. Reformatted.
2. Minor wording edits.

Retire SOP SEI-4.11.2, Combine with 4.5.9.

Revision number 2 (4.5.2):

1. Reformatted under new numbering scheme.

Revision number 3 (4.5.3):

1. Inserted the requirement of using an "indelible" black ink pen in section 2.1 (2).
2. Added clarification that each entry changed must receive it own footnote number and explanation in section 2.1 (4).

Revision number 4 (4.5.4):

1. Deleted the requirement of using a black ink pen in section 2.1 (2).
2. Deleted EC (entry change) from the list of standard codes.
3. Deleted W (wording change) from the list of standard codes.
4. Added W for "write over".

Revision number 5 (4.5.5):

1. Section 2.1 part 4 - added circling of the footnote number **and** the error code when justifying an error/change in the raw data.

Revision number 6 (4.5.6):

1. Reformatted

Revision number 7 (4.5.7):

1. Reformatted

Revision number 8 (4.5.8):

1. Add detail to section 2.1.4 regarding who can make changes to data.

Revision number 9:

1. Title changed to Data Handling, Error Coding, Storage and Retrieval.
2. Incorporate SEI- SOPs; SEI-4.11.2, Assembly of Field Study Notebooks and SEI-4.3.5 Use of Field Logs into Data Handling SOP.
3. Minor wording edits in Section 1.0, Objective.
4. Updated style to match SEI Style Guide – font and text. Reformatted using MS Word.
5. Added standardized section headers: 2.0 Policies, 3.0 Safety, 5.0 Responsibilities, 6.0 Definitions, 7.0 References, 8.0 Tables, Diagrams, Flowcharts and Validation data. Authorization moved to Section 9.0, and section10.0 Revision History.

Revision number 10:

1. Added Section 4.4 on Copies. As a result, subsequent sections were re-numbered.

Revision number 11:

1. Updated wording to reflect Stone's current formatting.
2. Added "Materials retained or referred to in the archives shall be scanned electronically to permit expedient retrieval" so Section 4.5 Storage of Data.
3. Updated Section 4.6 Retrieval of Data to reflect Stone's new policy of having a locked filing room.

STANDARD OPERATING PROCEDURE

SEI-5.1.5

MAINTENANCE AND DECONTAMINATION OF FIELD EQUIPMENT

SOP Number: SEI-5.1.5
Revision Number: 5

Date Issued: 04/09/93
Date of Revision: 02/20/04

1.0 OBJECTIVE

Proper maintenance and decontamination of field equipment is an essential quality control measure and a safety precaution to ensure sampling equipment is in proper working condition and to remove any chemical or material contamination. The procedure prevents cross-contamination between sampling stations and helps maintain a clean working environment for the safety of all field personnel. Equipment decontamination helps prevent sampling bias. Maintenance records of equipment that "generates, measures, or assesses data" is required under the Good Laboratory Practices (GLPs) and is an important element of a compliant and scientifically sound operation. This standard operating procedure (SOP) defines the difference between maintenance of equipment and general decontamination procedures and sets forth the guideline SEI uses in determining if equipment records are required per Subpart D of 40 CFR Part 160. This SOP describes a general procedure for decontaminating a sampling device, more specific maintenance requirements and decontamination procedures may be described in SOPs specific to each type of equipment.

2.0 POLICIES

1. According to 40 CFR Part 160, Subpart D, Section 160.61, Equipment used in the generation, measurement, or assessment of data and equipment used for facility environmental control shall be of appropriate design and adequate capacity to function according to the protocol and shall be suitable located for operation, inspection, cleaning, and maintenance.
2. Personnel will legibly record data and observations in the field to enable others to reconstruct project events and provide sufficient evidence of activities conducted.
3. All data generated during the conduct of a study, except those that are generated by automated data collection systems, shall be recorded directly, promptly, and legibly in indelible ink.
4. All data entries shall be dated on the day of entry and signed or initialed by the person entering the data. If multiple personnel enter data upon a single page, each entry should be uniquely attributed to its author.
5. Personnel will document the procedures performed in accordance with Good Laboratory Practices (GLP) Standards, 40 CFR Part 160.

3.0 SAFETY ISSUES

1. If necessary and appropriate, a site-specific health and safety plan shall be created for each study site. A template for creating a proper health and safety plan is provided on the SEI network.
2. If necessary and appropriate, all chemicals are required to be received with a Material Safety Data Sheets (MSDS) or appropriate application label. These labels or MSDS shall be made available to all personnel involved in the sampling and testing.

4.0 PROCEDURES

4.1 Equipment types And Maintenance and Calibration Requirements

The majority of SEI's field equipment does not directly generate, measure or assess data and thus does not require the full documentation as described in Subpart D of the GLPs regarding written record keeping of all inspections, maintenance, testing, calibration or standardization operations.

Examples of exempt equipment include, but are not limited to:

1. Hand augers
2. Pump controllers/compressors
3. Generators
4. General hand tools (shovels, hammers, etc.)
5. Well pumps (bladder, electric submersible, etc)
6. Sampling Tools (bailers, dipping tools)

Maintenance documentation may be kept for this type of equipment to assist with inventory or the general condition of the equipment, but the records may not include all items required under 40 CFR Part 160, Subpart D.

Examples of equipment which fall under 40 CFR Part 160, Subpart D include, but are not limited to:

1. pH, conductivity meters.
2. Water level indicators.
3. Troll datalogger (for slug tests, etc.)
4. Guelph permeameter.
5. Scales (balances)

6. Flow meters.

7. Gas Chromatographs

For equipment, which falls under 40 CFR Part 160, Subpart D, at a minimum, the following records must be maintained:

1. An SOP must be written which details the methods, materials and schedules for the routine inspection, cleaning, maintenance, testing, and calibration for the equipment. Details will also include remedial action that will be taken in the event of failure or malfunction of the equipment. The SOP will include (by title if appropriate) the person responsible for maintenance of the equipment (both routine and non-routine).

2. Records will be maintained of all inspection, maintenance, testing, and calibrating operations. These records will contain, at a minimum:

    a. Dates of operation.

    b. Whether maintenance was routine or non-routine.

    c. Whether maintenance followed written SOPs.

3. If non-routine repairs are performed, at a minimum, the following will be documented in addition to the items in 2 above:

    a. Nature of the defect.

    b. How and when the defect was discovered.

    c. Any remedial or correction action taken in response to the defect.

Inspection, maintenance, testing and calibration efforts will be documented. In general, a checklist form or a maintenance logbook, specifically developed for each piece of equipment, should be used to document these procedures. This form or logbook should prompt the user for the maintenance, etc. to be performed. If a form or logbook is not available, actions must still be documented on an appropriate medium. Forms for equipment must be taken to the field for use. Once field efforts are complete, the forms should be returned to SEI's main office for filing. All original equipment maintenance forms will be retained in a filing cabinet. Copies may be made for inclusion in data packages at the request of a client. Records shall be maintained on each major item of equipment and all reference materials significant to the test performed. As indicated above these records shall include documentation on all routine and non-routinge maintenance activities and reference material verification. If available, the records shall include:

1. the name of the item of equipment;

2. The manufacturer's name, type identification, and serial number or other unique identification;

3. Date received and date place in service (if available);

4. Current location; where appropriate;

5. Condition when received (e.g., new, used, reconditioned);

6. Copy of the manufacturer's instructions,

7. Dates and results of calibrations and/or verifications and date of the next calibration and/or verification;

8. Details of maintenance carried out to date and planned for the future; and,

9. History of any damage, malfunction, modification or repair

10. Equipment which is malfunctioning and has been taken out of service, it shall be tagged as such.

If a piece of equipment is dedicated to a sampling site/study, the maintenance forms or logbook may be retained with that equipment until complete or the fieldwork is finished. Once either of these criteria has been fulfilled, the form should be filed in the project file or the general equipment file as noted above.

4.2 Support Equipment

The following are the calibration and maintenance requirements for laboratory analytical support equipment. This applies to all devices that may not be 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 (including thermometers and thermistors, thermal/pressure sample preparation devices and volumetric dispensing devices (such as Eppendorf®, or automatic dilutor/dispensing devices) if quantitative results are dependent on their accuracy, as in standard preparation and dispensing or dilution into a specified volume

1. All support equipment shall be maintained in proper working order. The records of all repair and maintenance activities including service calls shall be kept.

2. All support equipment shall be calibrated or verified at least annually, using NIST traceable references when available, over the entire range of use. The results of such calibration shall be within the specifications required of the application for which this equipment is used or:

    a. The equipment shall be removed from service until repaired; or

b. The laboratory shall maintain records of established correction factors to correct all measurements

3. Raw data records shall be retained to document equipment performance.

4. Prior to use on each working day, balances, ovens, refrigerators, freezers, and water baths shall be checked in the expected use range, with NIST traceable references where available. The acceptability for use or continued use shall be according to the needs of the analysis or application for which the equipment is being used.

5. Mechanical volumetric dispensing devices including burettes (except Class A glassware) shall be checked for accuracy on at least a quarterly use bases. Glass micro liter syringes are to be considered in the same manner as Class A glassware, but must come with a certificate attesting to established accuracy or the accuracy must be initially demonstrated and documented by the laboratory

6. For chemical tests, the temperature, cycle time, and pressure of each run auto claves must be documented by the use of appropriate chemical indicators or temperature recorders and pressure gauges.

4.3 Equipment Decontamination and Personnel

Decontamination of equipment does not constitute maintenance, but is a specific step required for the use of equipment between sampling points. Decontamination is a requirement for use of the equipment but does not effect whether the equipment is in working order. Improper decontamination may effect the quality or integrity of a sample, but does not effect the device collecting the sample. Thus, decontamination is not an action, which must be documented per 40 CFR Part 160, Subpart D.

Decontamination of sampling equipment refers to the physical and chemical steps taken to remove any chemical or material contamination. Equipment decontamination helps prevent sampling bias. All equipment that comes in contact with the sampled material should be free of components that could influence (contaminate) the true physical or chemical composition of the material. Besides the equipment used to collect the samples, any containers or equipment used for sample compositing or for field sub sampling should be free of contamination.

Equipment decontamination also prevents cross-contamination of samples when the equipment is used to collect more than one sample. Disposable equipment or the use of dedicated equipment provides the most effective means of avoiding cross-contamination; however, the use of such equipment is not always practical.

You should decontaminate equipment to a level that meets the minimum requirements for your data collection effort. Your decontamination steps (e.g., use of solvents versus use of only soap and water), therefore, should be selected based on the constituents present, their concentration levels in the waste

or material sampled, and their potential to introduce bias in the sample analysis results if not removed from the sampling equipment. You should describe the project specific decontamination procedures in your planning document for the sampling effort. In addition, items used to clean the equipment, such as bottlebrushes, should e free of contamination.

The following procedures is an example of one you could use to decontaminate sampling device to be used for collecting samples for trace organic or inorganic constituent analyses (from USEPA 1996):

1. Clean the device with tap water and soap, using a brush if necessary to remove particulate matter and surface films.

2. Rinse thoroughly with tap water.

3. Rinse thoroughly with analyte- or organic-free water.

4. If necessary and appropriate, rinse thoroughly with solvent. Do not solvent-rinse PVC or plastic items.

5. Rinse thoroughly with organic/analyte free water, or allow equipment to dry completely.

6. Remove the equipment from the decontamination area. Equipment stored overnight should be wrapped in aluminum foil and covered with clean, unused plastic.

The specifications for cleaning materials are as follows (you should justify and document the use of substitutes):

- "Soap" should be phosphate-free laboratory detergent such as Liquinox®. It must be kept in a labeled, clean, plastic, metal, or glass containers until used and poured directly from the container when in use.

- "Solvent" should be pesticide-grade isopropanol. It must be stored in the unopened original containers until used. It may be applied using the low-pressure nitrogen system fitted with Teflon nozzle, or using Teflon® squeeze bottles. For equipment highly contaminated with organics (such as oily wastes), a laboratory-grade hexane may be more suitable alternative to isopropanol or for volatile organic contaminates purge and trap grade methanol may be more appropriate.

- "Tap Water" may be used from any municipal water treatment system. Use of an untreated potable water supply is not an acceptable substitute unless the source has been determined to be free of contaminates of interest. Tap water may be kept in clean tanks, hand pressure sprayers, squeeze bottles, or applied directly form a hose of tap.

- "Analyte free water (deionized water) is tap water treated by passing through a standard deionizing resin column. At a minimum, it must contain no detectable heavy metals or other inorganic compounds as defined a standard ICP (or equivalent scan. It may be obtained by other methods as long as it meets the analytical criteria. Analyte free water must be stored in clean glass, stainless steel, or plastic containers that can be closed prior to use. IT can be applied from plastic squeeze bottles.

- "Organic/analyte free water" is tap water that has been treated with activated carbon and deionizing units. A potable system to produce such water under field conditions is available. At a minimum, the water must meet the criteria of analyte free water and not contain detectable pesticides, herbicides, or extractable organic compounds, and no volatile organic compounds above minimum detectable levels as determined for a given set of analyses. Organic/analyte free water obtained by other methods is acceptable, as long as it meets the analytical criteria. It may be applied using Teflon® squeeze bottles or with the portable system.

Clean the field equipment prior to field use. Designate a decontamination zone at the site and, if necessary, construct a decontamination pad at a location free of surface contamination. If necessary, you should collect wastewater from decontamination (e.g., via a sump or pit) and remove it frequently for appropriate treatment or disposal. The pad or area should not leak contaminated water into the surrounding environment. You also should collect solvent rinses for proper disposal.

You should always handle field-cleaned equipment in a manner that prevents recontamination. For example, after decontamination but prior to use store, the equipment in a location away from the cleaning area and in an area free of contaminants. If it is not immediately reused, you should cover it with plastic or aluminum foil to prevent recontamination.

At certain sites, decontamination will generate a quantity of wastes called investigation derived waste (IDW). You should address the handling and disposal of IDW in your sampling plan. You must handle this material in accordance with whether it is nonhazardous or suspect of, or know to be, hazardous. You should minimize the generation of hazardous IDW and keep it separated from the nonharzardous IDW. For example, you should control the volume of spent solvents during equipment decontamination by applying the minimum amount of liquid necessary and capturing it separately form the nonhazardous wash water. For additional guidance on handling IDW, see Management of Investigation-Derived Wastes (USEPA 1992).

Decontamination of personnel and their protective gear also is often necessary during hazardous waste sampling. This important type of decontamination protects personnel from chemical exposure and prevents cross-contamination when personnel change locations. The level or degree of such decontamination will depend on site-specific considerations, such as the decontamination procedures in your health and safety plan. For additional information regarding decontamination, See ASTM D5088, Standard Practice for Decontamination of Field Equipment Used at Nonradioactive Waste

Sites. Another source of additional information is "Sampling Equipment Decontamination (USEPA 1994) issued by EPA's Environmental Response Team.

If necessary and appropriate equipment specific decontamination procedures will be outlined in equipment specific SOPs. These SOPs will clearly state the type of documentation required for each type of equipment and state whether the equipment falls under 40 CFR part 160, subpart D. Decontamination may be documented by citing an SOP or directly on a sampling form or other task specific medium.

5.0 RESPONSIBILITIES

1. All personnel will legibly record data and observations (including phone conversations) in accordance with this SOP to enable others to reconstruct project events and provide sufficient evidence of activities conducted.

2. Prior to use and after use, all equipment will be appropriately cleaned and decontaminated in accordance to this SOP.

6.0 DEFINITIONS

1. *Archives* - Include an area for orderly storage and expedient retrieval of original raw data, documentation, protocols, specimens, and interim and final reports retained as a result of a study. Conditions of storage shall minimize deterioration of the documents or specimens in accordance with the requirements for the time period of their retention and the nature of the documents of specimens. An individual shall be identified as responsible for the archives. Only authorized personnel shall enter the archives. Material retained or referred to in the archives shall be indexed to permit expedient retrieval. A testing facility may contract with commercial archives to provide a repository for all material to be retained. Raw data and specimens may be retained elsewhere provided that the archives have specific references to those other locations.
2. *Calibration* – To determine, by measurement or comparison with a standard, the correct value of each scale reading on a meter, instrument, or other device. The levels of the applied calibration standard should bracket the range of planned or expected sample measurement (NELAC).
3. *Decontamination* – Procedures followed to ensure cross contamination does not occur between sampling points or that potential contamination of equipment does not pose a hazard to sampling personnel.
4. *EPA* the U.S. Environmental Protection Agency.
5. *Equipment which Generates Data* – Equipment which directly collects a data point (or multiple data points) necessary for study goals and stores or displays the data for viewing.
6. *Equipment which Measures Data* – Equipment which directly collects a data point (or multiple data points) which includes a unit that can be used to compare the data point (or points which includes a unit that can be used to compare the data point (or points) with other data of the same unit.

7. *Equipment which Assesses Data* - Equipment which collects a data point (or multiple points) and manipulates the data in a manner in which inferences or calculations are made which create data other then what was directly collected. Or, equipment which measures the performance or accuracy of the data it collects.

8. *FIFRA* the Federal Insecticide, Fungicide, and Rodenticide Act as amended.

9. *Maintenance* – Actions performed on equipment to standardize and/or correct the accuracy and precision of a piece of equipment to ensure that the equipment is operating within the manufacturer's specifications and standard values.

10. *Observations & Remarks Form (O&R):* A pre-printed form, which contains mostly blank space for general note taking. The form typically prompts the user for the study or project designation, the SEI project number, the client or sponsor name, the total number of pages (page *n* of n) and requires a signature and date. The form is generally used to capture notes of one person when another, more specific forms is not available.

11. *Quality Assurance Unit (QAU)* any person or organizational element except the study director, designated by testing facility management to perform the duties related to quality assurance of the studies.

12. *Raw data* any laboratory worksheets, records, memoranda, notes, or exact copies thereof, that are the result of original observations and activities of a study and are necessary for the reconstruction and evaluation of the report of that study. In the event that exact transcripts of raw data have been prepared (e.g., tapes which have been transcribed verbatim, dated, and verified accurate by signature), the exact copy or exact transcript may be substituted for the original source as raw data. Raw data may include photographs, microfilm or microfiche copies, computer printouts, magnetic media, including dictated observations, and recorded data from automated instruments.

13. *Study* means any experiment at one or more test sites, in which a test substance is studied in a test system under laboratory conditions or in the environment to determine or help predict its effects, metabolism, product performance (pesticide efficacy studies only as required by 40 CFR 158.640) environmental and chemical fate, persistence, or residue, or other characteristics in humans, other living organisms, or media. The term "study" does not include basic exploratory studies carried out to determine whether a test substance or a test method has any potential utility.

7.0 REFERENCES

40 CFR Part 160 Good Laboratory Practice Standards, August, 1989.

USEPA. 1992. Management of Investigation-Derived Wastes. Directive 9345.3-03FS. NTIS PB92-963353. Office of Solid Waste and Emergency Response. Washington, DC.

USEPA . 1994. "Sampling Equipment Decontamination." Environmental Response Team SOP#2006, Revision#0.0. Edison, NJ. http://www.ert.org/

USEPA. 2000. National Environmental Laboratory Accreditation Conference (NELAC), EPA 600/R-00-0084Office of Research and Development, Las Vegas, NV.

USEPA. August 2002. RCRA Waste Sampling Draft Technical Guidance, EPA 530-D02-002, Office of Solid Waste and Emergency Response. Washington, DC. (5305W)

8.0 TABLES, DIAGRAMS, FLOWCHARTS, AND VALIDATION DATA

None

9.0 AUTHORIZATION

Revised by: Kim B. Watson Date: 2-19-04

Kim B. Watson, Quality Assurance Manager

Approved by: Christopher T. Stone Date: 2-19-04

Christopher T. Stone, President

10.0 REVISION HISTORY

Revision number 1

1. Added Section 3.0 number 10, for storage requirements.
2. Reformatted under new numbering scheme.

Revision number 2:

1. Changed wording of introduction to state that decontamination is a quality *control*, not a quality assurance measure.

Revision number 3:

1. Reformatted
2. Minor wording edits
3. Remove air dry requirement (section 3, # 8)

Revision number 4:

1. Change title.
2. Re-formatted.
3. Added definitions.
4. Added detail of the type of documentation required under subpart D of the GLPs.
5. Added section 3.4 and 3.5 regarding the use of checklists and filing of equipment documentation.
6. Added detail of what decontamination constitutes.

Revision number 5:

1. Minor wording edits in Section 1.0, Objective.
2. Updated style to match SEI Style Guide – font and text. Reformatted using MS Word.
3. Added standardized section headers: 2.0 Policies, 3.0 Safety, 5.0 Responsibilities, 6.0 Definitions, 7.0 References, 8.0 Tables, Diagrams, Flowcharts and Validation data. Authorization moved to Section 9.0, andSection10.0 Revision History.
4. Revised Section 4.2 on Decontamination of Equipment and Personnel to include general decontamination procedures as outlined in the RCRA Waste Sampling Draft Technical Guidance Document (USEPA 2002).
5. Added Section on Support Equipment for the on-site laboratory.
6. Included Section under Section 4.1 on records requirements for equipment.
7. Updated References

STANDARD OPERATING PROCEDURE

SEI-5.26.0

USE, MAINTENANCE AND CALIBRATION OF THE LAMOTTE MODEL 2020e TURBIDITY METER

SOP Number: SEI-5.26.0
Revision Number: NA

Date Issued: 6/23/05
Date of Revision: NA

1.0 OBJECTIVE

This Standard Operating Procedure describes the use, maintenance and calibration of the LaMotte Model 2020e, turbidity meter. Turbidity is measured by detecting and quantifying the scattering of light in water. The 2020 is a portable microprocessor controlled, direct reading colorimeter and nephelometer that uses a tungsten filament light source that meets or exceeds EPA specifications and is designed for a uniform light spot image and stable output. The turbidity meter generates and measures data, and thus must meet the requirements of 40 CFR part 160 subpart D.

2.0 POLICIES

1. According to 40 CFR Part 160, Subpart D, Section 160.61, Equipment used in the generation, measurement, or assessment of data and equipment used for facility environmental control shall be of appropriate design and adequate capacity to function according to the protocol and shall be suitable located for operation, inspection, cleaning, and maintenance.
2. Personnel will legibly record data and observations in the field to enable others to reconstruct project events and provide sufficient evidence of activities conducted.

3.0 SAFETY ISSUES

1. If necessary and appropriate, a site-specific health and safety plan shall be created for each study site. A template for creating a proper health and safety plan is provided on the SEI network.
2. If necessary and appropriate, all chemicals are required to be received with a Material Safety Data Sheets (MSDS) or appropriate application label. These labels or MSDS shall be made available to all personnel involved in the sampling and testing.

4.0 PROCEDURES

4.1 Equipment and Materials

1. LaMotte Model 2020e Turbidity Meter and users manual
2. Appropriate turbidity standard solutions
3. Glass sample tubes
4. Positioning rings
5. Equipment calibration Log or other appropriate medium to record calibration data

Prior to use in the field the user should read and understand the manufacturer's operating instruction manual which is included in the carrying case and a copy is also provided in the equipment maintenance file.

4.2 Selecting a Calibration Curve Reference

1. The default units are NTU (nephelometric turbidity units) and the default calibration curve reference is formazin.

2. Turn the meter on and scroll down to **Options**, press* | **OK**.

3. Scroll down and then press * | **OK** to select **Turbidity**.

4. Scroll down and then press * | **OK** to select **Calibration**.

5. Scroll down and then press * | **OK** to select a **Calibration** curve (formazin). Select a calibration option based on the composition of the standards that will be used to calibrate the meter.

6. For the most accurate results, the Attenuation option should be chosen when samples are over 500 NTU. The range for the Attenuation option is 40-4000 NTU.

4.3 Calibration Procedure

Prior to use, a standard will be scanned. The reading will be verified. The meter will be recalibrated if necessary.

1. Turn the meter on.

2. Press * | **OK** to select **Measure.**

3. Rinse a clean tube three times with a blank. Below 1 NTU, the meter should be blanked with a 0 NTU Primary Standard or prepared turbidity free (<0.1NTU) water. For the most accurate results, use the same tube for the blanks and the sample.

4. Fill the tube to the fill line with the blank. Pour the blank down the inside of the tube to avoid creating bubbles. Cap the tube.

5. Dry the tube with a lint free cloth. Put on a dry positioning ring. Cap the tube. Wipe the tube again with a lint free cloth.

6. Open the meter lid. Insert the tube into the chamber. Align the index notch on the positioning ring with the index arrow on the meter. Close the lid.

7. Press * | **OK** to select **Scan Blank.** Remove the tube.

8. Rinse a clean tube, or the same tube three times with the standard. While the tube is inverted, wipe the lip of the tube to remove droplets trapped under the ring.

9. Fill the tube to the line with the standard. Pour the standard down the inside of the tube to avoid creating bubbles. Cap the tube.

10. Wipe the tube thoroughly again with a lint- free cloth.

11. Open the meter lid. Insert the tube into the chamber. Align the index notch on the positioning ring with the index arrow on the meter. Close the lid.

12. Press * | **OK** to select **Scan Sample.**

13. Observe the result.

14. Scroll down and press * | **OK** to select **Calibrate.**

15. Use the up or down arrows to change the highlighted digits on the display to match the concentration of the turbidity standard. Press * | **OK** to accept a digit and move to the next digit.

16. When the value on the display matches the concentration of the turbidity standard, press * | **OK** to select **Set.**

17. Press * | **OK** to proceed to Turbidity analysis.

NOTE: The meter will remember the last scanned blank reading. It is not necessary to scan a blank each time a test is performed. To use the previous blank reading, instead of scanning a new one, scroll to Scan Sample and proceed. For the greatest accuracy during the calibration procedure, be sure that after the meter is blanked, and the blank is scanned as a sample, the reading is 0.00. If not, re-blank the meter and scan the blank again until reads 0.00. When scanning the sample, scan the calibration standard three times, removing the tube from the chamber each time. The readings should be consistent.

4.4 Turbidity Analysis of Field Samples

1. Turn on the meter
2. Press * | **OK** to select **Measure.**
3. Rinse a clean tube three times with a blank. Below 1 NTU, the meter should be blanked with a 0 NTU Primary Standard or prepared turbidity free (<0.1NTU) water. For the most accurate results, use the same tube for the blanks and the sample.
4. Fill the tube to the fill line with the blank. Pour the blank down the inside of the tube to avoid creating bubbles.
5. Dry the tube with a lint free cloth. Put on a dry positioning ring. Cap the tube. Wipe the tube again with a lint free cloth.
6. Open the meter lid. Inset the tube into the chamber. Align the index notch on the positioning ring with the index arrow on the meter. Close the lid.
7. Press * | **OK** to select **Scan Blank.** Remove the tube.
8. Rinse a clean tube, or the same tube three times with the standard. While the tube is inverted, wipe the lip of the tube to remove droplets trapped under the ring.
9. Fill the tube to the line with the standard. Pour the standard down the inside of the tube to avoid creating bubbles. Cap the tube.
10. Wipe the tube thoroughly again with a lint- free cloth.
11. Open the meter lid. Insert the tube into the chamber. Align the index notch on the positioning ring with the index arrow on the meter. Close the lid.
12. Press * | **OK** to select **Scan Sample.**
13. Record the result.

4.5 Sample Dilution Procedures

If a sample is encountered that is more than 4,000 NTU, a careful dilution with 0 NTU or a very low turbidity water will bring the sample into an acceptable range. However, there is no guarantee that halving the concentration will exactly halve the NTU value. See page 21 of the operators manual for more details.

4.6 General and Annual Maintenance

Individual users are responsible for the calibration, cleaning, repair, and maintenance of the instrument.

Routine inspection and maintenance schedules vary for each piece of equipment. Typically there is minor maintenance that each piece of equipment will need to undergo prior to use in the field (such as cleaning or conditioning). Always consult the manufacturer's instructions for general and routine maintenance.

Specific per use maintenance needs for the model 2020e turbidity meter include:

1. Inspect instrument for physical damage
2. Check battery
3. Clean and dry exterior with a damp, lint free cloth.
4. Clean meter chamber and lens over LED's with a lint- free cloth slightly dampened with alcohol.
5. Scan blank
6. Turbidity verified and re-calibrated, if necessary.

DO NOT ALLOW WATER TO ENTER THE METER CHAMBER OR ANY OTHER PARTS OF THE METER.

All maintenance, repairs, and calibrations are to be documented on an equipment maintenance log or other appropriate medium. Follow the checklist provided on the equipments maintenance log for regular use maintenance needs. Any maintenance must include documentation of whether the maintenance was routine and followed the SOP or not.

Equipment logs shall be brought to the field for documenting use and calibration. The logs will be returned to the office after each field use and filed in the equipment records filing cabinet.

In the event of failure due to breakage or loss of parts, an attempt will be made to repair or replace the necessary parts by the field personnel who discover the malfunction. All repairs will be documented in field notes and/or on a non-routine maintenance log. If an instrument is rendered "out of service" or "broken", it should be tagged as such. If further repair is necessary, return the instrument to the manufacturer following proper shipping procedures.

Non-routine repairs must include documentation of the nature of the defect, how and when the defect was discovered, and any remedial action taken in response to the defect and maintained in the instrument maintenance file.

Annual maintenance is outlined in the annual maintenance checklist for each piece of equipment. All annual maintenance shall be conducted in no more than 13 month increments after the initial use of the equipment.

5.0 RESPONSIBILITIES

1. All personnel will legibly record data and observations (including phone conversations) in accordance with this SOP to enable others to reconstruct project events and provide sufficient evidence of activities conducted.

2. Prior to use and after use, all equipment will be appropriately cleaned, decontaminated and calibrated (if necessary) in accordance with the manufacturer's instructions and this SOP.

6.0 DEFINITIONS

1. *Decontamination* – Procedures followed to ensure cross contamination does not occur between sampling points or that potential contamination of equipment does not pose a hazard to sampling personnel.
2. *EPA* the U.S. Environmental Protection Agency.
3. *Maintenance* – Actions performed on equipment to standardize and/or correct the accuracy and precision of a piece of equipment to ensure that the equipment is operating within the manufacturer's specifications and standard values.
4. *Quality Assurance Unit (QAU)* any person or organizational element except the study director, designated by testing facility management to perform the duties related to quality assurance of the studies.

7.0 REFERENCES

40 CFR Part 160 Good Laboratory Practice Standards, August, 1989.

8.0 TABLES, DIAGRAMS, FLOWCHARTS, AND VALIDATION DATA

Attachment I: Manufacturer's Instruction

9.0 AUTHORIZATION

Revised by: S Nuss for Mike Nuss Date: 6-23-05

Michael Nuss, Staff Scientist

Approved by: [signature] Date: 6-23-05

Christopher T. Stone, President

STANDARD OPERATING PROCEDURE

SEI-5.27.1

USE, MAINTENANCE AND CALIBRATION OF THE HYDROLAB MS5 WATER QUALITY MULTIPROBES

SOP Number: SEI-5.27.1
Revision Number: 1

Date Issued: 4/17/08
Date of Revision: 7/12/12

1.0 OBJECTIVE

The Hydrolab MS5 Water Quality Multiprobe is used to monitor the temperature, specific conductance, pH, oxidation-reduction potential (ORP) and dissolved oxygen concentration (DO) of water flowing through the system's flow-through cell. These parameters are measured to gain knowledge of the quality of the water and to ensure that a representative sample is being collected. To ensure the accuracy of the measurements, each of the probes in the system must be calibrated and have periodic and preventative maintenance.

2.0 POLICIES

1. The water quality parameters of the Hydrolab MS5 Water Quality Multiprobe should be verified each day before use. Maintenance, corrective actions, and calibration should be noted in the "Use, Maintenance and Calibration Log".
2. Personnel will legibly record data and observations in the field to enable others to reconstruct project events and provide sufficient evidence of activities conducted.
3. Field personnel will use a field logbook, observation and remark form (O&R) or other designated field form to record activities, measurements, and observations made in the field.
4. Any change in entries shall be made so as not to obscure the original entry. The recorder will initial corrections.
5. Field personnel will read this SOP and the appropriate sections in the Operator's Manual prior to operating the Hydrolab MS5.

3.0 SAFETY ISSUES

1. Whenever visiting a hazardous or potentially hazardous site, it is important to read, follow and sign the site health and safety plan (HASP).
2. By itself, use of the Hydrolab MS5 poses no risk to human health, however, it may be necessary to don personnel protective equipment as described in the site's HASP. In addition, protective gloves (nitrile

or latex) are recommended while handling calibration solutions as these solutions can stain skin or clothing. pH 4.01/ORP powder pillows may cause slight irritation to eyes and skin and nausea, vomiting or cramps if ingested.

3. Field visits may involve accessing remote areas. Health and safety concerns regarding these field visits are minimal however; slip, trip and fall hazards, and poisonous plant and dangerous animal, as well as getting vehicles stuck in remote areas do present considerable health and safety issues. To help ensure field staff's health and safety with remote areas, all field staff is to have daily communication with the project manager or another appropriate Stone Environmental, Inc. employee.
4. If necessary and appropriate, all chemicals are required to be received with Material Safety Data Sheets (MSDS) or appropriate application label. These labels or MSDS shall be made available to all personnel involved in the sampling and testing.

4.0 PROCEDURES

4.1 Introduction

The units owned and operated by Stone Environmental, Inc. are the Hydrolab MS5 Water Quality Multiprobes. The Hydrolab MS5 is a compact, modular system with a range of components to customize the instrument to various applications and operational modes. Components include the instrument body with a host of options, sensors, and a suite of cables for single instrument operation, network boxes and cables for network operation, external power accessories, and consumable items.

4.2 General Considerations

The Hydrolab MS5 is adversely affected by extreme temperatures and pressures. Refer to the operating manual to determine allowable operating temperatures and pressures.

4.2.1 Equipment

- Hydrolab MS5 Water Quality Multiprobe and operator's manual
- Sensors for Dissolved Oxygen, pH, ORP, and Specific Conductance
- Low Flow Cell
- Cables and power supply
- Software (Hydras 3 LT)
- Calibration solutions – one for each sensor to be calibrated, Deionized water
- Field notebook, soil boring/well log/groundwater profiling log and parameter equilibration forms, or other appropriate medium for recording field data

- Sensor Guard
- Maintenance Kit (replacement o-rings for sensors and Sonde body, spare screws with allen wrench and silicone compound)
- pH salt pellet kit (pH reference electrode/saturated KCL and AgCL)

4.2.2 Use

Water quality sensors – pH, ORP, DO, Specific Conductance – are installed in the instrument's sensor ports. Remove the multiprobe from its shipping case and inspect it for any visible damage. Connect the calibration cable (Cat. No. 013470), or detachable cable (015XXX) to the multiprobe. Align the bigger pin on the multiprobe male connector to the indicator dots on the female cable connector. Do not rotate the cable or force or twist the pins into the connectors, this can cause damage to the connector pins. Connect the power receptacle from the calibration cable and external power adapter to an approved power supply. Connect the other end of the calibration cable to the computer serial port. Start the communications program (Hydras 3 LT). The software will automatically scan for Sondes. All detected Sondes are displayed in the 'Connected Sondes' list in the main window displayed below. If a Sonde is not found, reassign the COM port (File: Options) and press **RE-SCAN FOR SONDES**. Retry until the Sonde(s) are found. Synchronize the clock on the probe with the computer clock. Be sure to condition a new DO sensor for at least half an hour, preferably overnight, before calibrating.

4.2.3 Calibration

Sensors are checked for calibration at the factory; however calibration needs to be specific for a site and application. Check the calibration prior to first use. In addition sensors should be calibrated when fouling has occurred or is noticeable, parameter measurements do not match those of a known calibrated standard and components have been added or removed for different applications or when replacing components. When Waterloo APS profiling, sondes should be calibrated before use, and at least once a week while working. If calibration is required more than once per day, a solution may be used more than once, provided it is not returned to its original container. For general calibration preparations of all sensors select a calibration standard whose values are near the field samples. Clean and prepare the sensors. To ensure accuracy of calibration discard used calibration standards appropriately. Do not reuse calibration standards (if multiple calibrations per day, solution can be reused but should be stored separate from unused solutions). It is important to maintain temperature stability during calibration, take care to keep Sonde out of direct sunlight, heat source, or other conditions that may alter the temperature of the calibration standards during calibration. To calibrate each parameter select the Calibration tab from the main window then select individual parameters

1.) pH Calibration

- Select the pH tab from the Calibration window
- Attach the calibration cup and unscrew its cap
- Thoroughly rinse the Sonde with clean water to make sure each sensor is free from contaminants.
- In a similar manner to the above step rinse the sensors twice with a small amount of the desired calibration standard
- Pour the pH standard to within a centimeter of the top of the cup.
- Enter the value of the pH standard in the field provided
- Click **CALIBRATE**. A "Calibrate Successful!" screen will be displayed.

Note: pH is a two or three-point calibration. pH 6.8 to 7.2 is treated as the "zero" all other values are "slope." First calibrate the "zero" then calibrate the "slope."

2.) ORP Calibration

- Select the ORP tab from the Calibration window
- Attach the calibration cup and unscrew its cap
- Thoroughly rinse the Sonde with clean water to make sure each sensor is free from contaminants.In a similar manner to the above step rinse the sensors twice with a small amount of the desired calibration standard
- Pour the ORP standard to within a centimeter of the top of the cup.
- Enter the value of the ORP standard in the field provided
- Click **CALIBRATE**. A "Calibrate Successful!" screen will be displayed.

3.) Specific Conductance (SpCond) Calibration

- Select one of the SpCond tabs from the Calibration window (mS/cm or μS/cm)
- Attach the calibration cup and unscrew its cap
- Thoroughly rinse the Sonde with clean water to make sure each sensor is free from contaminants.In a similar manner to the above step rinse the sensors twice with a small amount of the desired calibration standard
- Pour the SpCond standard to within a centimeter of the top of the cup.
- Enter the value of the SpCond standard in the field provided

- Click **CALIBRATE**. A "Calibrate Successful!" screen will be displayed.

Note: Specific conductance requires a two-point calibration. Calibrate the sensor to zero and then to the slope buffer. Specific Conductance is the conductivity of a substance at 25°C. When the temperature of a sample and its conductivity at that temperature are known, the software can extrapolate the conductivity to 25°C

4.) Hach LDO Sensor Calibration

- There are three methods for calibrating the Hach LDO sensor. Outlined below is the method used when water saturated air is applied to the sensor. This is the most common field method for calibrating the LDO sensor, refer to the Hach LDO Sensor Instruction Sheet for air saturated water and known calibration standards calibration methods.
- Select the LDO (SAT) tab from the Calibration window
- Attach the calibration cup and unscrew its cap
- Thoroughly rinse the Sonde with clean water to make sure each sensor is free from contaminants.Place the calibration cup with one end sealed so that the calibration cup is facing upwards. The Sonde will be inserted downwards into this cup.
- Fill the calibration cup with approximately ½ inches of deionized water or tap water (specific conductance less than 0.5 mS/cm). Water will not touch the top of the sensor cup.
- Carefully remove any water droplets from the sensor cap and temperature probe with the corner of a tissue, q-tip, or clean cotton cloth.
- Gently set the Sonde with sensors down into the calibration cup blocking any air exchange with the outside environment. Do not screw the calibration cup fully onto the Sonde as this will increase the pressure inside the calibration cup to above the barometric pressure and give false 100% saturation readings.
- Allow the dissolved oxygen and temperature readings to stabilize
- Determine the barometric pressure for entry as the calibration standard and enter into the field provided.
- Click **CALIBRATE**. A "Calibrate Successful!" screen will be displayed.
- The black coating on the LDO sensor cap wears off over time, allowing the blue LED to be visible. If more than 30% of the coating has worn off, the sensor cap should be replaced. Refer to instructions in HydroLab Manual.

4.2.4 Maintenance

Individual users are responsible for the calibration, cleaning, repair, and maintenance of the instrument.

There is minor maintenance the Hydrolab MS5 needs to undergo prior to use in the field (such as cleaning or conditioning). Always consult the manufacturer's instructions for general maintenance.

All maintenance, repairs, and calibrations are to be documented on an equipment maintenance log or other appropriate medium. Follow the checklist provided on the equipments maintenance log for regular use maintenance needs. Any maintenance must include documentation of whether the maintenance was routine and followed the SOP or not.

In the event of failure due to breakage or loss of parts, an attempt will be made to repair or replace the necessary parts by the field personnel who discover the malfunction. All repairs will be documented in field notes and/or on a non-routine maintenance log. If the instrument is rendered "out of service" or "broken", it should be tagged as such. If further repair is necessary, return the instrument to the manufacturer following proper shipping procedures.

Non-routine repairs must include documentation of the nature of the defect, how and when the defect was discovered, and any remedial action taken in response to the defect.

Detailed maintenance must be performed by a qualified technician and usually requires the shipping of the instrument to the manufacturer. Call the manufacturer for shipping instructions prior to sending the Hydrolab MS5.

General Maintenance that may be performed includes but is not limited to:

- Cleaning the Multiprobe housing
- Battery replacement
- Lithium battery replacement
- Multiprobe and sensor storage
- Electrical cable care
- Individual sensor maintenance (pH, ORP, SpCond, and Hach LDO)

Note: Consult manufactures user manual (Cat. No. 003078HY) for detailed maintenance directions.

4.2.5 Storage

For short term storage (less than 24 hours) fill the MS5 calibration cup with one inch of clean tap water and screw the cup on the multiprobe. This works for long term storage as well.

DO NOT store in pH4 solution. Prevent sensors from freezing by storing the multiprobe in conditions that freezing will not occur. Do not remove the lithium battery which powers the multiprobe internal clock. Store equipment in its designated carrying case.

5.0 RESPONSIBILITIES

1. SEI personnel must verify the Hydrolab MS5 is calibrated properly prior to use. If the values returned by the instrument are within pre-determined specifications when compared against known quantities, the instruments most recent calibration can be considered valid. Calibrations should be recorded in the Maintenance and Calibration Log.
2. It is the responsibility of SEI field staff to make sure that the Hydrolab MS5 is in proper working order prior to bringing it onto a site.
3. SEI personnel must read this SOP prior to using the Hydrolab MS5. After reading the SOP, personnel should update their personnel training folder.

6.0 DEFINITIONS

1. *EPA* the U.S. Environmental Protection Agency.
2. *Maintenance* – Actions performed on equipment to standardize and/or correct the accuracy and precision of a piece of equipment to ensure that the equipment is operating within the manufacturer's specifications and standard values.
3. *Observations & Remarks Form (O&R):* A pre-printed form, which contains mostly blank space for general note taking. The form typically prompts the user for the study or project designation, the SEI project number, the client or sponsor name, the total number of pages (page *n* of n) and requires a signature and date. The form is generally used to capture notes of one person when another, more specific form is not available.
4. *PPM*: Parts per million.
5. *Quality Assurance Unit (QAU)* any person or organizational element except the study director, designated by testing facility management to perform the duties related to quality assurance of the studies.
6. *Raw data* any laboratory worksheets, records, memoranda, notes, or exact copies thereof, that are the result of original observations and activities of a study and are necessary for the reconstruction and evaluation of the report of that study. In the event that exact transcripts of raw data have been prepared (e.g., tapes which have been transcribed verbatim, dated, and verified accurate by signature), the exact copy or exact transcript may be substituted for the original source as raw data. Raw data may include photographs, microfilm or microfiche copies, computer printouts, magnetic media, including dictated observations, and recorded data from automated instruments.

7. *Oxidation-Reduction Potential* (ORP): The ability for a compound to gain electrons (Reduce) or lose electrons (Oxidize).
8. *pH:* Negative log rhythm of Hydrogen Ion (H^+) activity in solution. Ranges from 0 (acidic) to 7 (neutral) to 14 (basic).
9. *Conductivity:* The ability of water to conduct electricity as a function of dissolve ionic species, suspended solids, and organic content. Usually measured in Siemens (S) or microsiemens (μS).

7.0 REFERENCES

1. Hydrolab DS5X, DS5 and MS5 Water Quality Multiprobes User Manual, February 2006, Edition 3

8.0 TABLES, DIAGRAMS, FLOWCHARTS, AND VALIDATION DATA

None

9.0 AUTHORIZATION

Authored by: Lee Rosberg Date: 7/13/12

Lee Rosberg, Staff Scientist

Approved by: _____ Date: 7/18/12

Seth Pitkin, Vice President

10.0 REVISION HISTORY

Revision Number 1:

1. Minor grammatical corrections.
2. Updated calibration instructions.

STANDARD OPERATING PROCEDURE

SEI-5.28.0

USE, MAINTENANCE AND CALIBRATION OF THE HACH LDO PORTABLE DISSOLVED OXYGEN METERS (HACH MODELS HQ10 AND HQ30d)

SOP Number: SEI-5.28.0
Revision Number: NA

Date Issued: 2/2/11
Date of Revision: NA

1.0 OBJECTIVE

This Standard Operating Procedure describes the use, maintenance and calibration of the Hach HQ10 and HQ30d portable luminescent dissolved oxygen (LDO) meters. The principle of operation is as follows (as per the Hach website):

The HACH LDO sensor is coated with a luminescent material. Blue light from an LED is transmitted to the sensor surface. The blue light excites the luminescent material. As the material relaxes, it emits red light. The time from when the blue light was sent and the red light is emitted is measured. The more oxygen that is present the shorter the time it takes for the red light to be emitted. This time is measured and correlated to the oxygen concentration. Between the flashes of blue light a red LED is flashed on the sensor and used as an internal reference.

2.0 POLICIES

1. The HACH LDO Portable DO Meter should be verified or calibrated as neccessary before use. Maintenance, corrective actions, and calibration should be noted in the "Maintenance and Calibration Log".
2. Personnel will legibly record data and observations in the field to enable others to reconstruct project events and provide sufficient evidence of activities conducted.
3. Field personnel will use a field logbook, observation and remark form (O&R) or other designated field form to record activities, measurements, and observations made in the field.
4. Any change in entries shall be made so as not to obscure the original entry. The recorder will initial corrections.
5. Field personnel will read this SOP prior to operating the Meter.

3.0 SAFETY ISSUES

1. If necessary and appropriate, a site-specific health and safety plan shall be created for each study site. A template for creating a proper health and safety plan is provided on the SEI network.

2. All chemicals are required to be received with Material Safety Data Sheets (MSDS) or appropriate application label. These labels or MSDS shall be made available to all personnel involved in the sampling and testing.

4.0 PROCEDURES

4.1 Equipment and Materials

1. Hach HQ10 or Hach HQ30d LDO Meter and User's Manual
2. BOD bottle or similar bottle with cap, used to create an environment of water-saturated air (LDO sensor must fit snugly into neck of bottle).
3. Replacement batteries – the LDO meter takes four AA alkaline batteries
4. Dedicated Equipment Maintenance and Calibration Log or other appropriate medium to record calibration data

Prior to use in the field the user should read and understand the manufacturer's operating instruction manual which is included in the carrying case and a copy is also provided in the equipment maintenance file.

4.2 Calibration Procedure

The LDO probe does not require calibration by the user for normal operation. However, the calibration of the LDO sensor should be checked and verified prior to use. The following steps should be followed to first verify the current calibration and, if necessary, perform recalibration in water-saturated air. (Note that these instructions are written for the HQ10 model, but when the instructions are different for the HQ30d model, those different instructions will be included in italicized font in an extra note below the applicable steps.)

1. Add ½ inch of water to the bottom of a BOD bottle or other container (Note: The container must have a cap and fit snugly around the probe. It also must allow the sensor cap to be above the water level).

2. Cap or stopper the BOD bottle and vigorously shake the bottle for 1 minute. This will saturate the entrapped air with water.

3. Press **SETUP** (same as #5 key on the HQ10) on the LDO meter.

4. Use the **UP** and **DOWN** keys to highlight "O_2 Calibration". Press **READ/OK**.

5. From the "O_2 Calibration" menu, use the **UP** and **DOWN** keys to highlight "New 100%". Press **READ/OK**.

*Note: Follow steps 3-6 if calibrating the HQ10, but if calibrating the HQ30d, replace steps 3-6 with the following: Push the **CALIBRATE** key then the **METHODS** key and then select "User Cal- 100%". Push **OK.***

6. Remove the shroud if it is installed (In Stone's case, it usually is not installed).

7. Carefully blot any liquid from the sensor surface and temperature sensor.

8. Remove the stopper from the BOD bottle and insert the probe into the 100% water-saturated air environment. Make sure that the sensor cap is above the surface of the liquid and does not get wet.

9. Press **READ/OK.**

10. While the temperature stabilizes, the meter will display "Stabilizing..." (Note: For faster stabilization, make sure the temperature of the probe is within a few degrees of the water and container temperature.) Press **POWER/CANCEL** at any time during stabilization to return to the O_2 Calibration menu.

11. After the temperature stabilizes, the percent saturation is measured and displayed using the existing calibration. If the displayed percent saturation value is acceptable (95 - 105%), press **POWER/CANCEL** to keep the existing calibration and return to the "O_2 Calibration" menu.

12. To adjust the calibration (i.e., keep the new calibration settings), press **READ/OK.** New calibration data will be displayed.

13. Press **READ/OK** to store the results of the New 100% saturation measurement.

*Note: Follow steps 9-13 if calibrating the HQ10, but if calibrating the HQ30d, replace steps 9-13 with the following: Select READ. The display will show "Stabilizing..." and a progress bar as the probe stabilizes. The display will highlight the standard value. If the value is acceptable (95 – 105%), exit the calibration menu to keep current calibration. Otherwise, select **DONE** to view the calibration summary. Select **STORE** to accept the calibration and return to the measurement mode.*

14. If necessary, the user can always revert back to the default sensor calibration for the HQ10 model, removing all user-entered adjustments made through "New 100%". To do this, follow the following steps:

    i. From "O_2 Calibration", use the **UP** and **DOWN** keys to highlight "Default". Press **READ/OK.**

    ii. Press **READ/OK** to continue. Or press the **POWER/CANCEL** key to return to the "O_2 Calibration" setup menu without changing the default calibration

iii. Again, press **READ/OK** to confirm restoration of default calibration settings and return to dissolved oxygen measurement mode. OR press the **POWER/CANCEL** key to return to the "O_2 Calibration" setup menu without changing the user-entered calibration.

iv. Verify the calibration using steps 1-11 above before use.

4.3 Analysis of Field Samples

1. Insert the LDO probe into the Stone-made custom flow through cell until it fits snugly. Teflon tape may be necessary to ensure a good leak-free seal.

2. Then use tubing to plumb the flow through cell so that it receives purge water from the profiling box or peristaltic pump outflow and so that the water then passes through the LDO flow through cell (water passing by the LDO Sensor) and into the flow cell of the water quality sonde (if a water quality sonde is also being used concurrently for measuring other physicochemical parameters).

3. Turn on the LDO meter

4. Introduce sample water flow into the flow through cell so that the LDO sensor is completely submerged and sample water can flow past the sensor cap.

5. When you are ready to take a reading, press the **READ/OK** button.

6. The meter will display "Stabilizing..." and a progress bar. As the probe stabilizes, the progress bar shrinks, then disappears when stabilization is complete.

7. Record the stabilized reading on field sampling sheet.

4.4 Handling, Storage and Maintenance

Individual users are responsible for the calibration, cleaning, repair, and maintenance of the instrument. Routine inspection and maintenance schedules vary for each piece of equipment. Typically there is minor maintenance that each piece of equipment will need to undergo prior to use in the field (such as cleaning or conditioning). Always consult the User's Manual for general and routine maintenance.

4.4.1 Handling

Take care not to touch the sensor cap with fingers, hand or any surface that may scratch or cut the cap. Also do not touch the metallic button on the side of the probe when holding it. Keep the sensor out of direct sunlight and avoid pointing directly at the sun.

If a clean water rinse alone does not remove dirt or grime from the probe, gently clean the sensor with a mild detergent, water and a soft cloth or cotton swab. Make sure not to touch the sensor or metallic button with hands or fingers and make sure that any debris present does not scratch or mar the surface of the sensor cap during cleaning. Also, do not remove the black colored substrate from the sensor cap.

If water is ever present between the sensor cap and lens, remove the cap and blot dry the sensor cap and lens with a soft dry cloth. Reinstall the sensor cap.

4.4.2 Storage

When not in use, the LDO probes should be stored ***clean and dry***. If possible, the HQ10 and HQ30d models should be stored with fresh desiccant packs in its storage case. The HQ10 LDO probe also comes with a Dry Storage Chamber. A silica gel desiccant cartridge is housed in the chamber to maintain a dry atmosphere for the sensor cap between uses. The desiccant contains a moisture indicator that changes color when it gets wet. To replace the desiccant cartridge, remove a new cartridge from its sealed bag. Open the cap on the bottom of the storage chamber and dry the inside of the chamber if moisture is present. Replace the cartridge and close the cap.

When field use is completed, the sensor should be gently rinsed with clean water and then carefully dried with a soft, lint-free cloth or kimwipe. Then insert the probe gently into the Dry Storage Chamber until it reaches the stop. If there is no dry storage chamber, the probe should be stored with fresh desiccant packs in its storage case.

The desiccant cartridge should be inspected frequently. When the cartridge color has changed, it must be either replaced or regenerated. To regenerate a saturated desiccant cartridge, first remove the saturated cartridge and replace it with a new cartridge. Place the saturated cartridge in a 105°C (221°F) drying oven overnight, or until it changes to the original color. Store regenerated cartridges in a sealed container.

4.4.3 Annual Maintenance

Sensor cap replacement is required every 365 days, or more frequently if the cap becomes damaged or fouled. The meter will keep track of the 365 days and will display a reminder message when there are 30 days of sensor service remaining on the sensor cap. Each sensor cap comes with a sensor-specific update module that will need to be replaced at the same time the sensor cap is replaced. The Hach part number for the replacement sensor cap, with update module and desiccant cartridges is 51812-00 (for the HQ10 model) and 5811200 (for the HQ30d model). Instructions for replacement will be included with the replacement sensor cap for each model.

To replace the sensor cap and update module on the HQ10 model, follow these steps:

1. Remove the old sensor cap and seal and then position the new seal on the probe body. Make sure the narrow shoulder of the seal is facing the probe tip. Slide the seal until it rests against the probe body threads at the bottom end of the threads (i.e., the end closest to the tip).
2. Finger-tighten the new sensor cap onto the probe, just until the seal is compressed. ***Do not overtighten.*** The cap will push the seal over the threads and into position. The narrow shoulder of the seal will now be inside the sensor cap.
3. Visually inspect the probe seal to make sure the narrow shoulder of the seal is inside the cap.
4. Press **SETUP.**
5. Use the **UP** and **DOWN** keys to highlight "New Sensor" in the "O_2 Setup" Menu.
6. Press **READ/OK.**
7. Now, replace the sensor-specific update module, which is provided with each replacement sensor cap. To do so, hold the update module to the back of the meter. First, slide the rounded end of the module until it stops at the probe holder on the back of the meter. The pins on the update module will align with the metal contact pads on the meter. Sensor data are loaded automatically when the meter senses the update module. The update takes less than two seconds. After the sensor data are loaded, the meter will beep twice and display the sensor lot code. Make sure the lot code on the meter display matches the lot code printed on the sensor cap.
8. Press **READ/OK** to store the new sensor setup.

5.0 RESPONSIBILITIES

1. All personnel will legibly record data and observations (including phone conversations) in accordance with this SOP to enable others to reconstruct project events and provide sufficient evidence of activities conducted.
2. Personnel will read, understand the SOP, and document that they have been properly trained prior to the use of Stone's equipment.
3. Prior to use and after use, all equipment will be appropriately cleaned, decontaminated and calibrated (if necessary) in accordance with the manufacturer's instructions and this SOP.

6.0 DEFINITIONS

1. *Decontamination* – Procedures followed to ensure cross contamination does not occur between sampling points or that potential contamination of equipment does not pose a hazard to sampling personnel.

2. *EPA* the U.S. Environmental Protection Agency.

3. *Maintenance* – Actions performed on equipment to standardize and/or correct the accuracy and precision of a piece of equipment to ensure that the equipment is operating within the manufacturer's specifications and standard values.

7.0 REFERENCES

40 CFR Part 160 Good Laboratory Practice Standards, August, 1989.

8.0 TABLES, DIAGRAMS, FLOWCHARTS, AND VALIDATION DATA

None

9.0 AUTHORIZATION

Revised by: _____ Date: 2/4/11

Morgan Greenwald, Staff Scientist

Approved by: _____ Date: 2-4-11

Christopher T. Stone, President

STANDARD OPERATING PROCEDURE

SEI-6.2.6

WATER LEVEL MEASUREMENT USE, MAINTENANCE AND CALIBRATION OF ELECTRONIC WATER LEVEL INDICATORS

SOP Number: SEI-6.2.6
Revision Number: 6

Date Issued: 04/09/93
Date of Revision: 02/20/04

1.0 OBJECTIVE

The objective of water level measurement is to determine the depth or elevation of the water table in a well. This standard operating procedure shall set forth in sufficient detail the methods, materials, and schedules to be used in the routine inspection, cleaning, maintenance, testing, and calibration, for electronic water level indicators used for water level measurement. Electronic water level meters generate and measure data, and thus must meet the requirements of 40 CFR Part 160 Subpart D.

2.0 POLICIES

1. According to 40 CFR Part 160, Subpart D, Section 160.61, Equipment used in the generation, measurement, or assessment of data and equipment used for facility environmental control shall be of appropriate design and adequate capacity to function according to the protocol and shall be suitable located for operation, inspection, cleaning, and maintenance.
2. Personnel will legibly record data and observations in the field to enable others to reconstruct project events and provide sufficient evidence of activities conducted.

3.0 SAFETY ISSUES

1. If necessary and appropriate, a site-specific health and safety plan shall be created for each study site. A template for creating a proper health and safety plan is provided on the SEI network.
2. If necessary and appropriate, all chemicals are required to be received with a Material Safety Data Sheets (MSDS) or appropriate application label. These labels or MSDS shall be made available to all personnel involved in the sampling and testing.

4.0 PROCEDURES

4.1 Decontamination

A water level indicator shall be approximately triple rinsed with distilled or de-ionized water between wells if necessary to prevent cross contamination, and at the end of use on site, unless other decontamination procedures have been outlined. If other procedures are outlined in an approved protocol or Quality Assurance Project Plan (QAPP), or special situations call for different decontamination or cleaning procedures, these procedures shall be followed.

Every effort should be made to keep the water level indicator from contact with the ground. If the water level indicator does come in contact with the ground, it shall be decontaminated prior to use.

4.2 Calibration

To calibrate a water level indicator, the indicator will be unraveled to a distance of at least 50 feet. Measurement increments will then be checked along a tape measure to ensure accuracy and that there are no missing, frayed or damaged links.

This calibration procedure shall be done after any major repair due to malfunction or breakage that may have effected the total length of the measurement cable.

Unless specified, calibration shall be done in a clean environment by a staff scientist or staff technician and documented on the appropriate water level calibration form.

4.3 Field Water Level Measurement

4.3.1 Field Equipment Needed:

1. Map of location(s) of well(s) to be measured.
2. Keys to locked wells.
3. Proper protective clothing (if necessary or as designated in the protocol or QAPP).
4. Field notebook or other acceptable recording medium.
5. Water level measuring device (electronic water indicator, interface probe, or other device).
6. Sampling gloves.
7. Decontamination materials, as necessary.
8. Clean (new) poly/plastic sheeting or other material on which well cap and other equipment may be placed.

4.3.2 Field Procedure

1. Locate wells on a well defined site map.
2. Unlock and remove the well guard if one is present, then remove the well cap. Be careful to prevent the inside of the well cap from touching the ground in order to prevent possible contamination. Clean, poly/plastic sheeting (or other appropriate material) may be spread on the ground around the well to set the cap on.
3. Determine the established measuring point, which should be marked on the well casing with indelible ink. If no mark exists, establish a measuring point at the highest point on the rim of the well casing and mark it with indelible ink. The measuring point must be on the well casing itself and not on a separate well guard, since the well guard may shift due to ground surface settling or frost heaves. If a pump head assembly is dedicated to the well, a mark should be established on the head assembly (near the access port).
4. Prior to inserting anything down the well, be sure to don clean sampling gloves and to decontaminate the measuring device.
5. Whenever possible, use an electric water level indicator to measure the water level. The water indicator consists of a graduated electric cable with a weighted probe at the end. The probe consists of an open electric connection; when the probe come in contact with the water, the connection is closed and a current travels through the wire, causing a signal to be activated. The signal may be an audible alarm or a light that is turned on. Lower the probe into the well slowly until the signal is noted. Put the graduated wire against the measurement point, then raise and lower the probe with decreasing increments to determine precisely where the water surface is.
    a. Record in the field notebook or appropriate form the water level based on the graduation of the tape; typically this is to the nearest $1/100^{th}$ of a foot
6. If a non-aqueous phase liquid (NAPL) is known or expected to be present, the measurements shall be made with an interface probe. The probe is lowered slowly into the well until a liquid is encountered. The probe signifies a low conductance liquid such as NAPL with a continuous tone. The probe is then slowly lowered deeper into the well to determine the position of the NAPL/water interface. The presence of a high conductance fluid such as water is indicated by an intermittent tone.
7. Other measuring devices, such as a measuring tape or a string, may be used to make approximate water level measurements, but they should be used only if an electronic water indicator is not available.
8. Note the water level, and the NAPL level if applicable, in a field notebook or other acceptable medium as a measurement below the top of the well casing (BTOC). Also note the time of the water level measurement.

9. Decontaminate the measuring instrument after each measurement in accordance with SOP-SEI-5.1.*n*.

10. The sequence of water level measurements shall be from least likely contaminated to most likely contaminated sampling points, or if contamination levels are unknown, from upgradient to downgradient locations, in order to reduce the potential for cross contamination.

11. Once the water level is measured, and the measurement recorded, the well cap is then set back on the well casing, and the locking well guard re-locked (if applicable). If the lock or guard needs lubrication, be sure to take appropriate steps to prevent any possible contamination of the site, well, or well casing.

4.4 General and Annual Maintenance

Individual users are responsible for the calibration, cleaning, repair, and maintenance of the water level indicator equipment.

Routine inspection and maintenance schedules vary from each piece of equipment. Typically there are minor maintenance needs each piece of equipment will need to undergo prior to use in the field (such as cleaning or conditioning). Always consult the manufacturer's instructions for general maintenance.

Specific per use maintenance needs for the water level indicator include, but is not limited to:

- Check battery/light buzzer.
- Clean probe.
- Clean tape.
- Clean outside of housing reel.
- Alconox/Liquinox rinse (refer to study requirements).
- Distilled or Deionized water rinse.
- Inspect tape for damage
- SOP followed.
- Non-routine maintenance.

The water level indicator shall be stored in a clean dry place. Care should be given to keep the instrument from dust and contamination.

All maintenance, repairs, and calibrations are to be documented on an equipment maintenance log or other appropriate medium. Follow the checklist provided on the equipments maintenance log for regular use maintenance needs. Any maintenance must include documentation of whether the maintenance was routine and followed the SOP or not.

Equipment logs shall be brought to the field for documenting use and calibration. The logs will be returned to the office after each field use and filed in the equipment records filing cabinet or within the project files.

In the event of failure due to breakage or loss of parts, an attempt will be made to repair or replace the necessary parts by the field personnel who discover the malfunction. A backup is often kept on site and may be substituted. This substitution will be documented in field notes. All repairs will be documented in field notes and/or on a non-routine maintenance log. If further repair is necessary, return the instrument to the manufacturer following proper shipping procedures. A "out of service" or "broken" tag should be placed on the piece of equipment identifying the need for maintenance and that it should not be used until further notice.

Non-routine repairs must include documentation of the nature of the defect, how and when the defect was discovered, and any remedial action taken in response to the defect.

Annual maintenance is outlined in the annual maintenance checklist for each piece of equipment. A staff technician shall perform this maintenance or other staff designated by the sampling team leader. If appropriate and necessary, all annual maintenance shall be conducted in no more than 13-month increments after the initial use of the equipment.

5.0 RESPONSIBILITIES

1. All personnel will legibly record data and observations (including phone conversations) in accordance with this SOP to enable others to reconstruct project events and provide sufficient evidence of activities conducted.

2. Prior to use and after use, all equipment will be appropriately cleaned, decontaminated and if necessary calibrated in accordance with the manufacturer's instructions.

6.0 DEFINITIONS

1. *Decontamination* – Procedures followed to ensure cross contamination does not occur between sampling points or that potential contamination of equipment does not pose a hazard to sampling personnel.
2. *EPA* the U.S. Environmental Protection Agency.
3. *FIFRA* the Federal Insecticide, Fungicide, and Rodenticide Act as amended.

4. *Maintenance* – Actions performed on equipment to standardize and/or correct the accuracy and precision of a piece of equipment to ensure that the equipment is operating within the manufacturer's specifications and standard values.

5. *Quality Assurance Unit (QAU)* any person or organizational element except the study director, designated by testing facility management to perform the duties related to quality assurance of the studies.

6. *Study* means any experiment at one or more test sites, in which a test substance is studied in a test system under laboratory conditions or in the environment to determine or help predict its effects, metabolism, product performance (pesticide efficacy studies only as required by 40 CFR 158.640) environmental and chemical fate, persistence, or residue, or other characteristics in humans, other living organisms, or media. The term "study" does not include basic exploratory studies carried out to determine whether a test substance or a test method has any potential utility.

7.0 REFERENCES

40 CFR Part 160 Good Laboratory Practice Standards, August, 1989.

8.0 TABLES, DIAGRAMS, FLOWCHARTS, AND VALIDATION DATA

None

9.0 AUTHORIZATION

Revised by: _____ Date: 2-20-04

Glenn Schellinger, Project Geoscientist

Approved by: _____ Date: 2-19-04

Christopher T. Stone, President

10.0 REVISION HISTORY

Revision number 1 (5.7.1):

1. Added Section 5.0 to detail in-house storage of water level marker.
2. Reformatted under new numbering scheme.

Revision number 2 (5.7.2):

1. Delete documentation of "use" (old section 2.1).
2. Delete sections 3.3 and 3.4 detailing annual inspections
3. Alter title to reflect that the SOP is for all of SEI's electronic water level markers and wording changed to water level "indicators".
4. Reformatted

Revision number 3 (5.7.3):

1. Removed reference to use.
2. Edited section 2.0 (General Procedure), renamed (Decontamination).
3. Removed sections 3.0 (Maintenance) and 5.0 (Storage Requirements).
4. Added section 4.0 (General and Annual Maintenance).
5. Reformatted.

Revision number 1 (6.2.1):

1. Reformatted under new numbering scheme.

Revision number 2 (6.2.2):

1. Reformatted

Revision number 3 (6.2.3):

1. Reformatted.
2. Minor wording edits.

Revision number 4 (6.2.4):

1. Added detail of measuring point for wells with dedicated sampling equipment in section 3.2.
2. Added detail on lubrication of well guard/locks in section 3.9

Revision number 5 (6.2.5):

1. Add section 3.3 on wearing sampling gloves and decontaminating measuring devices prior to lowering into a well.

SEI-5.7.3 retired, all information and procedures included in Water Level Measurement SOP SEI-6.2.6

Revision number 6 (6.2.6 Water Level Measurement, SEI-5.7.3 included in Water Level Measurements):

1. Added Use, Maintenance and Calibration of Electronic Water Level Indicators to the title.
2. Included procedures as stated in SOP SEI-5.7.3
3. Minor wording edits in Section 1.0, Objective.
4. Updated style to match SEI Style Guide – font and text. Reformatted using MS Word.
5. Added standardized section headers: 2.0 Policies, 3.0 Safety, 5.0 Responsibilities, 6.0 Definitions, 7.0 References, 8.0 Tables, Diagrams, Flowcharts and Validation data. Authorization moved to Section 9.0, andSection10.0 Revision History.
6. Deleted section on logs being given to the QAU.

STANDARD OPERATING PROCEDURE

SEI-6.4.5

INSTALLATION, DEVELOPMENT AND DECOMMISSIONING OF MONITORING WELLS AND OBSERVATION WELLS

SOP Number: SEI-6.4.5
Revision Number: 5

Date Issued: 04/09/93
Date of Revision: 08/01/07

1.0 OBJECTIVE

In this Standard Operating Procedure, there is little distinction made between a monitoring well and an observation well. Unless otherwise noted, both a monitoring well and an observation well will collectively be referred to as a well.

Well construction, development and decommissioning are outlined in this procedure. A well consists of a rigid well casing with a screened interval to allow groundwater to flow into the well, and a filter pack surrounding the well screen. The well casing material, the grain size of the filter pack, and the backfill materials above the filter pack may vary considerably. In general, the main difference between a monitoring well and an observation well is that a monitoring well is sometimes constructed of inert material, such as stainless steal or Teflon-coated material, and their primary use is to access the saturated zone for groundwater sample collection. An observation well may be installed in a circumstance where groundwater levels are of interest but the collection of groundwater samples is not necessary. An observation well can also differ from a monitoring well in that it does not necessarily have a sand pack around the well screen. A clear work plan for approximate placement of the wells should be laid out and documented in the study or project files.

The purposes for installing a well may include:

1. Monitoring water levels and groundwater geochemistry
2. Mapping the local potentiometeric surfaces
3. Determining hydraulic parameters of the surficial aquifer
4. Saturated hydraulic conductivity testing.
5. Provide stratigraphic information.
6. Collecting groundwater samples.

Need for well development should be defined in the work plan on a case-by-case basis. If necessary, the procedures for well development are described below.

2.0 POLICIES

1. According to 40 CFR Part 160, Subpart E, Section 160.81, a testing facility shall have standard operating procedures in writing setting forth study methods that management is satisfied are adequate to insure the quality and integrity of the data generated in the course of a study.
2. Personnel will legibly record data and observations in the field to enable others to reconstruct project events and provide sufficient evidence of activities conducted.

3.0 SAFETY ISSUES

1. If necessary and appropriate, a site-specific health and safety plan shall be created for each study site. A template for creating a proper health and safety plan is provided on the SEI network.
2. Care must always be taken when approaching a sampling location. Do not, under any circumstances, place yourself in danger to collect a sample.
3. If necessary and appropriate, all chemicals are required to be received with a Material Safety Data Sheets (MSDS) or appropriate application label. These labels or MSDS shall be made available to all personnel involved in the sampling and testing.

4.0 PROCEDURES

4.1 Equipment and Materials

1. Hand auger, backhoe, drill rig or Geoprobe (operated by qualified personnel).
2. Well casing (screen and riser pipe).
3. Filter pack materials.
4. If required, protective well guard.
5. Field notebook, well construction log forms, or other acceptable medium for recording field notes.
6. Ruler or tape for measuring borehole depth and diameter, casing lengths, split spoon recovery, and other parameters.
7. Appropriate site personnel protective equipment as required or outlined in the Site Health and Safety Plan (e.g. hardhats, steel toed boots.)
8. If required, Photo-Ionization Detector for field screening of organic compounds.
9. Note: All drilling **must** be authorized by *DIG SAFE* (1-888-DIG-SAFE) and/or other appropriate agencies.

4.2 Borehole Drilling

Borings may be advanced by a variety of methods. If split spoon sampling of the soils is desired, a hollow –stem auger rig may be used. Solid core augers or other drilling techniques may be also used depending on the goals of a study. A hand auger may be used if the wells will be shallow and the soil is not prohibitively coarse.

Boreholes are most commonly drilled using hollow stem augers or by advancing bits or casing utilizing rotary boring rigs; solid stem augers may also be used. Other boring techniques may be used provided the quality and accuracy of the end product is not compromised, the variations are well documented, and the variations are approved by the on-site project manager/drilling supervisor prior to their implementation.

All augers, rods, split spoons, drilling tools or other tools that will come in contact with soil or groundwater must be cleaned at a minimum with a high temperature, high pressure steam before drilling is started. Steam cleaning should be performed after the rig has arrived on-site, but based on the potential for cross contamination, the project manager may elect to forgo on-site cleaning if it can be determined that the rig was, at a minimum, cleaned after leaving the last site the rig drilled. All actions must be documented.

On sites of known or suspected contamination, all drilling equipment that will come in contact with soil or groundwater must also be steam cleaned between boreholes to prevent cross contamination during the drilling.

The drill rig should be level, and the proper clearance between the drill rig and overhead utilities and obstructions must be maintained. Even though the drilling contractor usually establishes this, it is the responsibility of the on-site project manager/drilling supervisor to assure that the regulation and safety procedures are established.

4.3 Soil Sampling During Well Installation

Soil samples are generally collected using a split spoon sampler or other soil coring device/method. Bedrock may be sampled using a rock coring device or other appropriate means.

If required, split spoon samples are collected by driving the spoon into the soil using a hammer. The length and inside diameter of the split spoons should be recorded, and if blow counts are logged, the weight of the hammer and the dropping distance should also be recorded. The frequency of split spoon sample collection and the length of the split spoon used depend on the objectives of the investigation. The split spoon sampler should be washed with clean water prior to re-use. Detergent or steam cleaning may be required, depending on the objectives of the investigation.

Samples shall be qualitatively **described and logged** as appropriate as to:

1. Depth interval.
2. Blow counts (optional).
3. Length of sample recovered.
4. Sample description, including field texture, color, moisture content (dry, moist, or wet), mottles (if present), and other observable features.
5. Indications of contamination (if applicable).
6. Other, as appropriate.

Item 4 above may be performed at a later date if necessary. The color description should be performed in light similar to natural sunlight (if possible), and every attempt should be made to preserve field moisture conditions. If descriptions are performed at a time other than during field collection, this should be noted on the description sheet.

The collection of each sample will be **recorded** at the time of sample collection in a field log book or on a specified sample collection log form. Information to be recorded will also include the following (items italicized below may also be included to facilitate sample identification):

1. Date of sample collection.
2. Name of personnel collecting and describing the sample.
3. Sample identification.
4. Sample location.
5. Sample depth.

Samples for Laboratory Analysis: If samples are to be collected for laboratory analysis, they should be placed into a plastic sample bag, or other appropriate container, which will then be sealed and **labeled** with at least the following information:

1. Test system (sample matrix).
2. Study identification (name or study number).
3. Date of collection.
4. Sample identification number.
5. Name or initials of the sample collector.

Additional label information may be required per project specific protocol and/or quality assurance project plan.

A clear description of each sample will be noted in a field notebook, sample log form, or other acceptable medium. The description will include the details on the sample label, as well as the color, field texture, and moisture content of the sample.

Samples should be packaged securely for shipping, in accordance with standard operating procedures for sample handling, storage, and shipping.

A completed chain of custody form should accompany the samples to the laboratory, following standard operation procedures for chain of custody. A copy of the chain of custody form or list of samples transferred must be kept.

4.4 Materials

Screen and Casing. PVC is the most common well material used, but studies may require more inert materials such as stainless steel or Teflon. Polyethylene or fiberglass may be used in some situations. All joints should be made with flush-coupled threads; no glues or solvents should be used.

Annular Space. The annular space is the space between the outside of the well casing and the side of the borehole. Clean silica sand is typically used as a filter pack around the well screen. The filter pack typically extends one to two feet above the top of the screen. A bentonite seal approximately two feet thick is installed on top of the sand pack, above which the annulus may be filled either with native fill, bentonite grout (generally Portland cement with a small amount of powdered bentonite added to prevent shrinkage), bentonite slurry (a mixture of bentonite and water), bentonite chips/pellets, sand, or other material, depending on the requirements of the study. All backfill materials and depth intervals should be recorded in the field notes.

Well Guard. If required, a metal, locking well guard may be cemented into the ground surface to protect the well. A locking, PVC well cap may also be used in some cases.

State or local codes requiring different materials for installation supercede the above items. Clearly identify any changes from the above procedure.

4.5 Well Installation

When the borehole has been completed to the desired depth, the drill rods and plug are removed from the borehole and the well screen and riser pipe are lowered through the inside of the augers. The pipe should be capped or plugged at both ends to prevent the introduction of foreign objects. When the well is complete, the riser pipe should stick up above ground surface by 1-2 feet unless this would cause a hazard to vehicles or pedestrians or study needs require a different height.

Clean silica sand should be poured slowly into the annular space between the well pipe and the auger. The sand will form a filter pack around the well screen. The augers should be pulled up slowly as the sand is added to help ensure an even, continuous filter pack (If the filter pack tends to bridge, water made be poured in the annulus with the sand). The sand pack should extend from the bottom of the well up to 1-2 feet above the top of the screen.

A bentonite seal should be placed immediately above the sand pack. The seal may consist of bentonite pellets or chips, granular bentonite, bentonite slurry, or some combination thereof depending on site conditions and local code requirements. Generally, if moisture is present, granular bentonite will stick to the auger walls and cause "bridging" to occur. This problem can often be avoided by using pelletized bentonite. The plug should be at a minimum of two feet in thickness. The remainder of the annular space can be filled with bentonite grout, bentonite slurry, native material, clean silica sand, or other material, depending on the requirements of the study.

A surface plug of cement, a cement/bentonite mixture, or a bentonite/sand/native material mixture should be installed in the upper 1-2 feet of the borehole.

A locking well guard may be installed to protect the well and inhibit tampering.

State or local codes requiring different seal/backfill materials should supercede the above items. Clearly identify any changes from the above procedure.

4.6 Well Development (If Required)

After it is completed, the monitoring well should be developed to ensure that fine sediment is removed from around the well. Suspended sediment in water samples can interfere with chemical analyses. Well development also restores the natural permeability of the formation adjacent to the well and allows groundwater to flow freely into the well.

Pumping and/or surging are used to accomplish well development. Surging can be accomplished using either a surge block or reversing the pump direction to re-inject previously pumped water back down the well. If a surge block is used, ensure that the surge block is of appropriate diameter for the well and is fitted with a handle of adequate length to produce maximum water displacement and flow through the well screen. Following surging, the well would then be evacuated using either a submersible (impeller or bladder) pump, centrifugal pump, peristaltic pump or bottom discharge/filling bailer (for sediment removal). The evacuation method used will depend on well diameter, depth to water table, aquifer permeability and general yield. Depending on well conditions, it may be necessary to alternate between surging and evacuation to satisfactorily complete development.

If possible, the well will be pumped at a rate higher rate than that which would be used to purge or sample the well. By pumping the well at a higher rate, the mobilized particulates may be removed, thereby providing a cleaner well for sampling. During development, water should be removed throughout the entire water column in the well by periodically lowering and raising the pump intake.

During well evacuation, pH, specific conductivity and turbidity will be monitored and recorded every 5 minutes. Flow-through cells and/or other comparable instrumentation will be used to measure the parameters. Wells will be considered developed if the evacuated water is visibly clear and/or the parameters have stabilized to within 10 percent for three successive measurements.

The rate of flow of water should be measured and recorded during evacuation of the well. Measurements may be made using a flow meter or a watch and calibrated container such as a bucket or graduated cylinder. Evacuated water will be handled as indictated by federal or state-specific regulatory guidance depending upon the expected chemistry of the water.

The development technique used, as well as the duration of the development, volume of water removed and parameter measurement data should be recorded in a field notebook, well log form, or other acceptable medium.

4.7 Well Log

A well log should be completed with diagrams of the well construction. The following information should be included:

1. General

    a. Project designation.
    b. Well designation.
    c. Date of construction.
    d. Name of field sampling personnel.
    e. Name of drilling contractor.
    f. Drilling method (e.g. hollow stemmed auger).
    g. Inside diameter of hollow stemmed augers, if applicable.
    h. Borehole diameter or outside diameter of auger (the two should be equal).
    i. Total depth of boring.

2. Well Materials

    a. Casing diameter.
    b. Riser pipe material and length.
    c. Screen material, length, depth interval, and slot size.
    d. Sump interval, if applicable.
    e. Type of well guard.
    f. Backfill details

3. Well Development Data

    a. Date of well development.
    b. Development methods.
    c. Approximate volume evacuated from well.
    d. pH measurements.
    e. Specific Conductivity (SC) measurements.
    f. Turbidity measurements.
    g. Containment and/or disposal methods.

4. Survey and Water Level Information

    a. Top of casing elevation (optional).
    b. Ground surface elevation (optional).
    c. Length of stick-up.
    d. Water level in well (hydraulic head) at or near time of completion (if possible, this should be measured after the water level has had time to equilibrate).

All of this information should be recorded in the field at the time of well construction, except for elevation data that is not available until a survey has been completed. The information should be copied carefully from the survey onto the well log form (identify as transcribed data), or a note should be made on the form indicating that this data can be obtained from the site survey.

4.8 Water Level Measurements

A datum point at the top of the well casing should be marked with an indelible pen. This point should be the highest point on the rim of the well casing, and all water level measurements must be made from this point.

If the water levels in more than one well on a site are to be used to draw groundwater contours, the elevations of the well casings should be surveyed by an appropriate method, using the datum marks on the wells as survey points.

The location of the well should be plotted on a sketch map of the site at the time of installation. The well location may be determined by tape and compass, transit survey, or other means as appropriate.

4.9 Well Decommissioning (If Required)

Well decommissioning procedure must comply with all appropriate local and state regulations. Proper well decommissioning is necessary to:

1. Eliminate aboveground physical hazards.
2. Conserve yield and hydrostatic head of aquifers.
3. Prevent intermingling of subsurface waters.
4. Prevent contamination of the groundwater from the surface.
5. Ensure complete sealing of the borehole and prevent vertical migration of contaminants.

Depending on state and local regulations, and any prior agreements with the landowner, the manner in which a well is decommissioned and the type of equipment used for decommissioning will vary. In short, well decommissioning consists of safely extracting and removing from site, and properly disposing of all well materials, followed by permanently sealing the resulting hole.

Complete and accurate records will be kept of the decommissioning procedures to provide documentation for the study records and, if necessary, regulatory agencies. Documentation will include at least the following:

1. The project designation and/or study number.
2. The date that the well was decommissioned.
3. The personnel involved in the fieldwork.
4. Any subcontracting companies employed (drillers, backhoe operators, etc.)
5. All procedures used to remove the well materials from the borehole.
6. Any well materials left in the hole, and the approximate depths of those materials.
7. All backfill materials used, and the approximate depths of those materials.

5.0 RESPONSIBILITIES

1. All personnel will legibly record data and observations (including phone conversations) in accordance with this Standard Operating Procedure to enable others to reconstruct project events and provide sufficient evidence of activities conducted.

6.0 DEFINITIONS

1. *Study* means any experiment at one or more test sites, in which a test substance is studied in a test system under laboratory conditions or in the environment to determine or help predict its effects, metabolism, product performance (pesticide efficacy studies only as required by 40 CFR 158.640) environmental and chemical fate, persistence, or residue, or other characteristics in humans, other living organisms, or media. The term "study" does not include basic exploratory studies carried out to determine whether a test substance or a test method has any potential utility.

7.0 REFERENCES

40 CFR Part 160 Good Laboratory Practice Standards, August, 1989.

8.0 TABLES, DIAGRAMS, FLOWCHARTS, AND VALIDATION DATA

None

9.0 AUTHORIZATION

Revised by: _____ Date: 8/1/07

Gerald Edwards, PG

Approved by: _____ Date: 8/1/07

Seth E. Pitkin, Vice President

10.0 REVISION HISTORY

Revision number 1 (SEI-6.4.1):

1. Section 1.0 (Introduction): clarify purposes of monitoring wells and the designation of "observation well" in some groundwater studies.
2. Section 2.0: edit equipment list.
3. Section 3.1: make blow count recording optional; amend sample log and sample label requirements.
4. Section 3.3: make bentonite seal "approximately" two feet thick instead of "at least" two feet thick; distinguish between bentonite grout and bentonite slurry.
5. Section 3.4: change "bentonite plug" to "bentonite seal"; allow for bentonite/sand/native material mixture in surface plug.
6. Section 3.6: edit well log information.
7. Reformatted under new numbering scheme.

Revision number 2 (SEI-6.4.2):

1. Reformatted.
2. Remove inspection of rig (section 3.1).
3. Add review of state drilling codes (sections 3.3.4 & 3.4.6).
4. Add pouring water with filter pack if bridging occurs (section3.4.2).

Revision number 3 (SEI-6.4.3):

1. Adjust recommended equipment list in section 2.0.
2. Add details to section 3.1.
3. Add that state and local codes supercede construction/backfill materials per SOP.
4. Change label and logging requirements throughout section 3.2.
5. Add ability to have a stick-up less then 1 foot in section 3.4.1.
6. Delete reference to compressed air in section 3.5.2.
7. Add survey data requirements for elevations in section 3.6.2.
8. Reformatted.
9. Minor wording edits.

Revision number 1 (SEI-6.5.1):

1. Section 1.0: amend the Introduction to clarify the difference between the observation wells described in this SOP and the PVC monitoring wells that are called "observation wells" for some groundwater studies.
2. Add Section 2.0, a list of recommended equipment.
3. Add Section 3.2, split spoon sampling (cut and pasted form monitoring well SOP).
4. Section 3.3: minor edits to installation procedures.
5. Section 3.4: make the survey requirements more general.
6. Reformatted under new numbering scheme.

Revision number 2 (SEI-6.5.2):

1. Reformatted.
2. Minor wording edits.

Revision number 3 (SEI-6.5.3):

1. Reformatted.
2. Minor wording edits.
3. Adjust recommended equipment list in Section 2.0.
4. Delete Section 3.1.3.
5. Add details to Section 3.1.4 (now Section 3.1.3).

Observation Well Installation and Construction SOP included in SOP SEI-6.4.4. SOP SEI-6.5.3 retired.

Revision number 1 (SEI-6.9.1):

1. Reformatted.

Revision number 2 (SEI-6.9.2):

1. Reformatted.
2. Add discussion on sealing the borehole from the water table to the surface in Section 2.5.

Observation and Monitoring Well Decommissioning SOP included in SOP SEI-6.4.4. SOP SEI-6.9.2 retired.

Revision number 4 (SEI-6.4.4):

1. Change Title to read, "Installation, Development and Decommissioning of Monitoring Wells and Observation Wells."
2. Wording edit in Objective Section, to include references to monitoring and observation wells. Under purpose #3 add saturated. Made note about Well Development being required on a case by case basis...."
3. Minor wording edits under Equipment (i.e., hand auger). Add note on Dig Safe.

4. Change Geologic Sampling to read "Soil Sampling During Well Construction".
5. Add definition to annular space.
6. Add Section on Water Level Measurement from SOP SEI-6.5.3.
7. Add Section on Well Decommissioning from SOP SEI-6.9.2.
8. Updated style to match SEI Style Guide – font and text. Reformatted using MS Word.
9. Added standardized section headers: 2.0 Policies, 3.0 Safety, 5.0 Responsibilities, 6.0 Definitions, 7.0 References, 8.0 Tables, Diagrams, Flowcharts and Validation data. Authorization moved to Section 9.0, andSection10.0 Revision History.

Revision number 5 (SEI-6.4.5):

1. Added specificity in regard to well development in Section 4.6 Well Development.
2. Added additional well development-related data requirements to Section 4.7 Well Log, Bullet 3. Well Development Data.

STANDARD OPERATING PROCEDURE

SEI-6.16.4
HANDLING, COLLECTION AND TRANSPORTATION OF SAMPLES

SOP Number: SEI-6.16.4
Revision Number: 4

Date Issued: 11/22/93
Date of Revision: 04/16/04

1.0 OBJECTIVE

Proper handling of samples is an integral component of obtaining representative and accurate data. The handling of a sample from the time it is removed from its medium until it is placed in its designated storage area must be done with care and must be thoroughly documented. This standard operating procedure describes the basic steps to follow in proper collection and transportation of samples

2.0 POLICIES

1. According to 40 CFR Part 160, Subpart E, Section 160.81, a testing facility shall have standard operating procedures in writing setting forth study methods that management is satisfied are adequate to insure the quality and integrity of the data generated in the course of a study.
2. Personnel will legibly record data and observations in the field to enable others to reconstruct project events and provide sufficient evidence of activities conducted.

3.0 SAFETY ISSUES

1. If necessary and appropriate, a site-specific health and safety plan shall be created for each study site. A template for creating a proper health and safety plan is provided on the SEI network.
2. Care must always be taken when approaching a sampling location. Do not, under any circumstances, place yourself in danger to collect a sample.
3. If necessary and appropriate, all chemicals are required to be received with a Material Safety Data Sheets (MSDS) or appropriate application label. These labels or MSDS shall be made available to all personnel involved in the sampling and testing.

4.0 PROCEDURES

4.1 General Procedures for Handling Samples

Handling, of samples will often be outlined in an approved study protocol and/or Quality Assurance Project Plan (QAPP). If this is the case, the instructions in the protocol or QAPP will supersede this SOP.

Samples should be handled in a manner to limit any unanticipated, or unwanted exposure to temperature or pressure extremes, direct sunlight, or cross contamination, etc.

Documentation of the handling of samples should be recorded on an appropriate medium (field log book, observation and remark sheets, etc.). Documentation may include but is not limited to:

1. Sample location.
2. Sampling personnel.
3. Date of sampling.
4. Sample ID.
5. Interim storage procedure.
6. Study number.
7. Sample container description.
8. SOP or Protocol followed.

All records generated during collection and transportation of samples should be included as part of the raw data package.

A site must be adequately prepared, characterized, and instrumented according to the protocol and/or regulatory guidelines prior to application of the test substance.

A detailed site characterization shall be performed to fully identify the surface and subsurface soils, depth to groundwater, groundwater direction flow, hydraulic conductivity in the saturated and unsaturated zones, and all other parameters as outlined in an approved protocol.

All observations shall be recorded in the field notebook or on Observations and Remarks sheets.

4.2 Collection and Transportation of Soil Samples

Collection of soil samples should be documented on appropriate soil boring or sampling logs as provided for specific projects. This information should be documented as outlined in SOP SEI-6.1.*n*, SEI-6.4.*n*, SEI-6.5.*n*, and SEI-6.10.*n* or specified in an approved protocol.

If procedures for sample handling are outlined in an approved protocol, those procedures supersede any information described in this SOP. Any deviations from those procedures must be identified.

Samples should be placed into an appropriate container, and labeled according to the appropriate SOP or approved Protocol.

Samples should be kept from direct sunlight and extreme temperatures.

The samples should be protected from spillage, leakage, and cross-contamination to ensure that sample integrity is not compromised while on-site or during transport.

4.3 Collection and Transportation of Water Samples

The same procedures as described in section 3 apply to water samples. Extra precautions must be taken to ensure a good seal on each sample container to prevent the sample from leaking.

If samples are to be frozen, sample containers should be filled to allow for expansion of the sample while it freezes. Therefore, the sample container should be chosen with the desired sample size in mind.

5.0 RESPONSIBILITIES

1. All data generated during the conduct of a study, except those that are generated by automated data collection systems, shall be recorded directly, promptly, and legible in ink. All data entries shall be dated on the day of entry and signed or initialed by the person entering the data. Any change in entries shall be made so as not to obscure the original entry, shall indicate the reason for such change, and shall be dated and signed or identified at the time of the change. In automated data collection systems, the individual responsible for direct data input shall be identified at the time of data input. Any change in automated data entries shall be made so as not to obscure the original entry, shall indicate the reason for change, shall be dated, and the responsible individual shall be identified.

2. All personnel will legibly record data and observations (including phone conversations) in accordance with this Standard Operating Procedure to enable others to reconstruct project events and provide sufficient evidence of activities conducted.

6.0 DEFINITIONS

1. *Observations & Remarks Form (O&R):* A pre-printed form, which contains mostly blank space for general note taking. The form typically prompts the user for the study or project designation, the SEI project number, the client or sponsor name, the total number of pages (page *n* of n) and requires a signature and date. The form is generally used to capture notes of one person when another, more specific forms is not available.

2. *Raw data* any laboratory worksheets, records, memoranda, notes, or exact copies thereof, that are the result of original observations and activities of a study and are necessary for the reconstruction and evaluation of the report of that study. In the event that exact transcripts of raw data have been prepared (e.g., tapes

which have been transcribed verbatim, dated, and verified accurate by signature), the exact copy or exact transcript may be substituted for the original source as raw data. Raw data may include photographs, microfilm or microfiche copies, computer printouts, magnetic media, including dictated observations, and recorded data from automated instruments.

3. *Study* means any experiment at one or more test sites, in which a test substance is studied in a test system under laboratory conditions or in the environment to determine or help predict its effects, metabolism, product performance (pesticide efficacy studies only as required by 40 CFR 158.640) environmental and chemical fate, persistence, or residue, or other characteristics in humans, other living organisms, or media. The term "study" does not include basic exploratory studies carried out to determine whether a test substance or a test method has a This standard operating procedure shall be used as a guideline in ensuring that all steps have been taken in preparing the test system. ny potential utility.

7.0 REFERENCES

40 CFR Part 160 Good Laboratory Practice Standards, August, 1989.

Guidance for Prospective Ground-Water Monitoring Studies, September 16, 1998

USEPA, Pesticide Assessment Guidelines, Subdivision N, Chemistry, Environmental Fate, 1982

8.0 TABLES, DIAGRAMS, FLOWCHARTS, AND VALIDATION DATA

None

9.0 AUTHORIZATION

Revised by: _____ Date: 4-14-04

Glenn Schellinger, Project Geoscientist

Approved by: _____ Date: 4-16-04

Christopher T. Stone, President

10.0 REVISION HISTORY

Revision number 1 (SEI-6.16.1):

1. Reformatted under new numbering scheme

Revision number 2 (SEI-6.16.2):

1. Reformatted.
2. Minor wording edits.

Revision number 3 (SEI-6.16.3):

1. Reformatted.
2. Minor wording edits.
3. Delete section 3.6 detailing *requirement* of monitoring temperature of soil samples.

Revision number4 (SEI-6.16.4):

1. Titled Sections "General Procedures for Sample Handling, Collection and Transportation of Soil Samples, and Collection and Transportation of Water Samples".
2. Updated style to match SEI Style Guide – font and text. Reformatted using MS Word.
3. Added standardized section headers: 2.0 Policies, 3.0 Safety, 5.0 Responsibilities, 6.0 Definitions, 7.0 References, 8.0 Tables, Diagrams, Flowcharts and Validation data. Authorization moved to Section 9.0, andSection10.0 Revision History.

STANDARD OPERATING PROCEDURE

SEI-6.19.2

USE, MAINTENANCE AND CALIBRATION OF THE IONSCIENCE PHOCHECK 1000+ PHOTOIONIZATION DETECTOR (PID)

SOP Number: SEI-6.19.2
Revision Number: 2

Date Issued: 7/19/99
Date of Revision: 02/04/2011

1.0 OBJECTIVE

A photoionization detector PID is often used to determine the presence or absence of volatile organic compounds (VOCs) in soil and water with ionization potential. Although a PID cannot necessarily differentiate between individual VOCs, it is an indispensable tool for the initial screening of a potentially hazardous site. It is therefore important to understand its operation and to keep the PID in optimal operating conditions.

The goal of soil headspace screening is to acquire preliminary information regarding volatile organic or hydrocarbon contamination in the vadose zone. The process is not meant to identify specific VOCs or hydrocarbon compounds. Rather, it will detect the relative amount of volatile organic hydrocarbons accumulated in the headspace of the sample container within the ionization potential of the lamp. This information should be used with other site-specific information to formulate an assessment of the presence or absence of contamination at the subject site.

2.0 POLICIES

1. The PID should be calibrated each day before use. Maintenance, corrective actions, and calibration should be noted in the "Maintenance and Calibration Log".
2. Personnel will legibly record data and observations in the field to enable others to reconstruct project events and provide sufficient evidence of activities conducted.
3. Field personnel will use a field logbook, observation and remark form (O&R) or other designated field form to record activities, measurements, and observations made in the field.
4. Any change in entries shall be made so as not to obscure the original entry. The recorder will initial corrections.
5. Field personnel will read this SOP prior to operating the PID.

3.0 SAFETY ISSUES

1. Whenever visiting a hazardous or potentially hazardous site, it is important to read, follow and sign the site health and safety protocol (HASP).

2. By itself, use of the PID poses no risk to human health; however, if elevated levels of VOCs are detected by the PID, it may be necessary to don personnel protective equipment as described in the site's HASP.

3. Field visits may involve accessing remote areas. Health and safety concerns regarding these field visits are minimal however; slip, trip and fall hazards, and poisonous plant and dangerous animal, as well as getting vehicles stuck in remote areas do present considerable health and safety issues. To help ensure field staff's health and safety with remote areas, all field staff is to have daily communication with the project manager or another appropriate Stone Environmental, Inc. (SEI) employee.

4.0 PROCEDURES

4.1 Introduction

The unit owned and operated by Stone Environmental, Inc. is the IonScience PhoCheck 1000+ Model PID. Although there are numerous models of PIDs available, all PIDs are based on the following principle:

> A sampling pump continuously draws air sample into the ionization chamber. The ionization chamber for the PhoCheck 1000+ PID unit is constructed as a small cavity in front of the UV lamp. The electrode is made of a wire mesh so that the high energy UV light can shine through the mesh and excite the gas molecules when they pass across the face of the UV window. The ions collected on the electrode are measured by an electrometer.
>
> A single chip microcomputer is used to control the operation of the lamp, alarm buzzer, LED, sampling pump, power supply and electrometer. It measures the electrometer readings and calculates the gas concentrations based on calibration to a known reference gas. An LCD display is used to show the readings. The user interacts with the unit though six keys on the front panel.
>
> A rechargeable lead acid battery powers the unit. A built-in charging unit allows the batteries to be charged directly from an AC power source.

4.2 General Considerations

The PhoCheck 1000+ is adversely affected by both damp and windy conditions, as well as temperature. Ensure that the PhoCheck 1000+ is used with its probe and a clean sample filter at all times. Replace the filter frequently. Refer to the operating manual to determine the lowest allowable operating temperature and humidity.

There is only one operating mode used by the PhoCheck 1000+ Model: Survey Mode. Survey mode is used for discrete VOC measurements where the peak concentration is desired. SEI will utilize the survey mode for its operations.

4.2.1 Equipment

- PhoCheck 1000+ PID and Operations Manual
- Calibration gas and regulator

- Carbon filter canister
- Spare sample filters
- Phillips head screwdriver, 7/16" wrench
- Maintenance and Calibration Log
- Clean, smooth-top jars (125 mL soil jars) and aluminum foil, or; freezer quality *zip-lock* baggies, one quart size
- Stainless steel spoon
- Distilled water
- Field notebook, soil boring/well log forms, or other appropriate medium for recording field data

4.2.2 Use

The PhoCheck 1000+ has only six controls: Power, Enter, Escape, Up Arrow, Down Arrow and Zero. The "Power" control turns the power on and off. The "Enter" control selects options and confirms settings. The "Escape" control is used to return the display to the previous screen and to abort an adjustment. Repeated pressing of this control key will return the display to the DISPLAY/FUNCTION/SELECTION screen. The "Up" and "Down" arrows allow the user to scroll up or down or to change selections. The "Zero" control zeroes out background readings.

The following provides basic instruction on the PhoCheck 1000+ menu and control functions. Once the PhoCheck 1000+ is powered on, press the "Escape" control repeatedly until the DISPLAY/FUNCTION/SELECTION screen is displayed (this screen has three icons: a graph, a number 1, and a wrench). Highlight the function menu (represented by the wrench icon) and press "Enter". Then use the "Up" and "Down" arrows to view the available functions, then press "Enter" to select the function of choice. Once a given function is selected, a cursor (highlighted symbol or number) can be moved from one selection to another by using the "Up" and "Down" keys. Pressing "Enter" will cause the selection to flash. When flashing, use the "Up" and "Down" keys to adjust its value. When you have adjusted the parameter to an acceptable level, press "Enter", which will store the adjusted parameter. In many cases, a checkmark will also appear to confirm the action. You can abort a selection by pressing the "Escape" control. Note: It is best to read the Operations Manual before attempting to operate the PID.

4.2.3 Calibration

Due to the linear output of the IonScience PID, the PhoCheck 1000+ only requires a two-point calibration. The PhoCheck 1000+ scales its linear output across a ZERO level (clean air reference) and the SPAN (a user defined gas concentration). There are two options for calibration: Factory Calibration and Custom Calibration. Factory calibration offers a safe set of calibration data. This should be used if the custom calibration fails and will keep the unit working until a good custom calibration is completed. Stone typically uses the Custom Calibration (see steps below).

The PhoCheck 1000+ is calibrated at zero using the provided carbon filter and at 100 ppm using the isobutylene calibration gas. The date and initials of the individual who performed the calibration will be documented in the maintenance and calibration log. Follow these steps for a custom calibration:

1. Select the Custom Calibration procedure from the Function Menu. A checkmark will appear to confirm the selection. Pressing "Enter" allows the user to enter the Custom Calibration menu.
2. Scroll down and select the flashing "ZERO".
3. Remove the caps from both ends of the carbon canister included in the Cal Kit Sample supplied with your PhoCheck 1000+, remove the probe and filter from the instrument and fit the carbon canister. Alternatively a known clean air supply can be used. Note – the carbon canister simply pushes onto end fitting – do not force; no turning is required.
4. Press "Enter" to start the ZERO process. A counter will count down, showing how long the carbon canister needs to remain connected.
5. At the end of the countdown, a checkmark will appear, indicating that the Zero has been accepted. Press "Escape".
6. Disconnect the carbon canister and replace the cap ends. The useful life of the carbon canister will be shortened if the canister is open to atmosphere for prolonged periods.
7. Scroll down and select the flashing "VOC".
8. The next screen shows the gas and gas concentration you have previously set up. If the information is correct, select "Enter".
9. A screen will show "VOC", a gas bottle icon and a flashing highlighted number.
10. Connect the calibration gas (Isobutylene) to the PhoCheck 1000+ and open the gas valve. Once gas is presented to the PhoCheck, press "Enter". The number will begin to count down, showing how long the instrument needs to be presented with the gas. A checkmark will appear indicating that the calibration has been accepted. "Escape" out of the calibration menu.
11. Now replace the instrument's filter and probe so PhoCheck is ready for use.
12. Carry out a Bump Test to ensure calibration was successful. In other words, expose the PID to a known concentration of gas (e.g., 100 ppm isobutylene calibration gas) and make sure it responds appropriately. Instrument is now ready for use.
13. Record value after calibrating in the Maintenance and Calibration Log.

If you are unsuccessful in calibrating the instrument, check the following points:

- The carbon canister may be contaminated
- The calibration gas may be low or empty
- If using a connector pipe, there may be a kink restricting the flow of gas

- PID lamp may require cleaning or replacement

4.2.4 Maintenance

Some tools are included with the field kit of the PhoCheck 1000+ that allow the user to perform minor maintenance. However, it is imperative that anyone attempting to clean or otherwise maintain the PID be in total comprehension of the inner controls of the PhoCheck 1000+ before opening the main body. **Please refer to the PhoCheck 1000+User Manual when performing maintenance.**

The following maintenance should be performed on a regular basis.

Maintenance	Schedule (Use dependent)
Change sample filter	Weekly
Blow out PID cell with can of computer dustoff	As needed
Replace secondary probe filter	Annually or as needed
Clean UV lamp with aluminum oxide	Annually or as needed

Detailed maintenance must be performed by a qualified technician and usually requires the shipping of the PID to the manufacturer. Stone typically sends the PID to the manufacturer annually for lamp cleaning and general maintenance – or more frequently as needed. Call the manufacturer for shipping instructions prior to sending the PID.

4.2.5 Documentation

A record of calibrations and any maintenance performed on the PID must be made in the Maintenance and Calibration log.

Typically in field use, PID values are recorded onto soil boring logs (see attached). If screening is being conducted of miscellaneous soil samples, such as those from surface samples, values should be recorded on "Observations and Remarks (O&R)" forms.

4.3 Collection of Soil Samples for Headspace Screening

Fill a clean jar or baggie approximately half full using the stainless steel spoon with a "consistent amount" of soil. Take special care to ensure that sample and container are not cross-contaminated from previous sample by rinsing spoon between samples with distilled water. It is also important to ensure that the sample is not frozen prior to analysis.

Seal top of jar with two layers of aluminum foil, or insure that bagged is properly sealed. Secure aluminum foil with either a rubber band or screw lid that fits jar. If using screw lid, do not include solid top with rubber gasket included with new mason jars.

Allow VOC concentrations to develop in the headspace of the sample container for five minutes. During this headspace development period, the sample should be shaken vigorously for a total of one minute.

Immediately pierce the aluminum foil seal (or un-zip an approximate one-inch section of baggie) with the probe of PID and note the highest (peak) reading, and sustained reading. Take care not to submerge PID tip into soil.

Record time of sample collection and peak value as ppm onto appropriate form (Soil Boring Log or O&R).

4.4 Documentation

A record of the soil screening must be made in the field at the time the peak sample headspace concentration is read from the PID. It may also be helpful to record the weather or any site-specific conditions such as excessive heat or cold that may affect the readings from the PID. Be sure that field notes are properly labeled to the project number and date.

5.0 RESPONSIBILITIES

1. Stone personnel must calibrate the PhoCheck 1000+ prior to use. Calibrations should be recorded in the Maintenance and Calibration Log.
2. It is the responsibility of Stone field staff to make sure that the PID is in proper working order prior to bringing it onto a site.
3. Stone personnel must read this SOP prior to using the PhoCheck 1000+. After reading the SOP, personnel should update their personnel training folder.

6.0 DEFINITIONS

1. *EPA* the U.S. Environmental Protection Agency.
2. *Observations & Remarks Form (O&R):* A pre-printed form, which contains mostly blank space for general note taking. The form typically prompts the user for the study or project designation, the SEI project number, the client or sponsor name, the total number of pages (page *n* of n) and requires a signature and date. The form is generally used to capture notes of one person when another, more specific forms is not available.
3. *PID*: Photoionization Detector
4. *PPM*: Parts per million.
5. *Quality Assurance Unit (QAU)* any person or organizational element except the study director, designated by testing facility management to perform the duties related to quality assurance of the studies.
6. *Raw data* any laboratory worksheets, records, memoranda, notes, or exact copies thereof, that are the result of original observations and activities of a study and are necessary for the reconstruction and evaluation of the report of that study. In the event that exact transcripts of raw data have been prepared (e.g., tapes which have been transcribed verbatim, dated, and verified accurate by signature), the exact copy or exact transcript may be substituted for the original source as raw data. Raw data may include photographs, microfilm or microfiche

copies, computer printouts, magnetic media, including dictated observations, and recorded data from automated instruments.

7. *VOC*: Volatile Organic Compound

7.0 REFERENCES

1. Vermont State Agency of Natural Resources, Waste Management Division "Agency Guidelines for Petroleum Contaminated Soils and Debris" August 1996.
2. Ion Science Ltd., "PhoCheck+ Insturment User Manual v2.4", November 2008.

8.0 TABLES, DIAGRAMS, FLOWCHARTS, AND VALIDATION DATA

None

9.0 AUTHORIZATION

Revised by: _____ Date: 2/4/11

Morgan J. Greenwald, Staff Scientist

Approved by: _____ Date: 2-4-11

Christopher T. Stone, President

10.0 REVISION HISTORY

Revision 1

1. Reformatted under new numbering scheme.
2. Combined with SOP draft 5.8.0. Title changed to "Use, Maintenance and Calibration of the Mini-Rae Photo Ionization Detector (PID)".
3. Added documentation and safety issues items from SOP 4.5.9.
4. Minor wording edits in Section 1.0, Objective.
5. Updated style to match SEI Style Guide – font and text. Reformatted using MS Word.
6. Added standardized section headers: 2.0 Policies, 3.0 Safety, 5.0 Responsibilities, 6.0 Definitions, 7.0 References, 8.0 Tables, Diagrams, Flowcharts and Validation data. Authorization moved to Section 9.0, and section10.0 Revision History.

Revision 2

1. Updated SOP to refer to our new PID, the IonScience PhoCheck 1000+ PID, rather than the old PID, the Mini-Rae. Changed title, any references to PID and updated all use and calibration instructions based on the PhoCheck 1000+ User Manual.

SOIL BORING LOG

Project Name: _____ Site Name: _____

SEI Study #: _____

BORING ID:	Date Drilled:	Location:
SOP followed:		
Deviations? (If YES, attach details):		
Drilling Method:		
Drilling contractor:	Inside diameter:	
Sampling method:		
Core Length: Inside diameter:		

Comments:

Depth Interval (feet bgs)	Blow Counts (per 0.5 ft)	PID (ppm)	Recovery (inches or feet)	Sample Description (color, texture, moisture, remarks)

Geoscientist:	Signature:	Date:

Geoscientist:	Signature:	Date:

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Page ____ of ____

SOIL BORING LOG (continued)			Boring ID:	SEI Study #
Depth Interval (feet bgs)	Blow Counts (per 0.5 ft)	PID (ppm)	Recovery (inches or feet)	Sample Description (color, texture, moisture, remarks)

Geoscientist:	Signature:	Date:
Geoscientist:	Signature:	Date:

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STANDARD OPERATING PROCEDURE

SEI-6.11.8

SLUG TESTS

SOP Number: SEI-6.11.8
Revision Number: 8

Date Issued: 04/12/93
Date of Revision: 03/02/06

1.0 OBJECTIVE

Slug tests are used to measure the saturated hydraulic conductivity of the soil surrounding the screened interval of a well. The water level in the well is changed suddenly by adding or removing either a volume of water or a slug bar, and then the water level is monitored as it returns to its equilibrium level. The hydraulic conductivity of the soil is related to the rate at which the water level recovers to its original, equilibrium level.

There are several methods and equations that can be used to calculate hydraulic conductivity from water level recovery data. This SOP details the method of Bouwer and Rice (Bouwer, 1989). The Bouwer and Rice method is the most common method used for shallow, unconfined aquifers, but other methods, such as that of Hvorslev (1951) may be appropriate depending on the objectives of the study. If a method other than Bouwer and Rice (Bouwer, 1989) is used, the method and calculations must be documented in detail.

A slug test indicates an average value of hydraulic conductivity across the entire surface area of the well screen, and does not take into account any heterogeneity or anisotropy in the soils. Also, the test only measures the hydraulic conductivity near the well; extrapolation of the test results to other parts of the aquifer must be done with care.

2.0 POLICIES

1. According to 40 CFR Part 160, Subpart E, Section 160.81, a testing facility shall have standard operating procedures in writing setting forth study methods that management is satisfied are adequate to insure the quality and integrity of the data generated in the course of a study.
2. Personnel will legibly record data and observations in the field to enable others to reconstruct project events and provide sufficient evidence of activities conducted.

3.0 SAFETY ISSUES

1. If necessary and appropriate, a site-specific health and safety plan shall be created for each study site. A template for creating a proper health and safety plan is provided on the SEI network.
2. Care must always be taken when approaching a sampling location. Do not, under any circumstances, place yourself in danger to collect a sample.

3. If necessary and appropriate, all chemicals are required to be received with a Material Safety Data Sheets (MSDS) or appropriate application label. These labels or MSDS shall be made available to all personnel involved in the sampling and testing.

4.0 PROCEDURES

4.1 Equipment and Materials

May include but is not limited to:

1. Water marker.
2. Distilled water or other decontamination equipment, as necessary.
3. Rubbish can and trash bags for storage of dedicated pumps if removal is necessary.
4. Bailer or slug bar (any cylindrical bar dense enough to sink in water) with outside diameter small enough to fit into the well and sufficient cable or line to allow complete submersion below the static level in the well. If a datalogger is used, the bailer or bar must fit in the well with the transducer cable in place. Disposable bailers should be used if slug tests are conducted at multiple sites to prevent cross contamination.
5. Optional: An automated data logging system with a pressure transducer. A list of specific equipment required for use of the Troll SP4000 system is located in SOP 5.22.*n*. A list of specific equipment required for use of the Hermit SE1000C datalogger is located in SOP 5.9.*n*.
6. Clock, watch, or other timepiece.
7. Field notebook with slug test forms or other acceptable recording medium.

4.2 Field Procedure

4.2.1 Slug Tests Using the Hermit SE1000C datalogger.

Measure the water level in the well, following standard operating procedures for water level measurement. This is the equilibrium water level. Note the time of the measurement.

Check water level against documented screened interval for the well. If the equilibrium water level is at or above the top of the well screen, then both a falling head test and a rising head test may be performed on the well. However, if the equilibrium water level is below the top of the screen, then only a rising head test will be valid.

If the well is equipped with a dedicated pump and associated tubing that will interfere with the transducer, cable, and bailer/slugbar, remove the pump assembly and coil it into a container lined with a new trash bag. Avoid letting the pump assembly contact the ground or any other potentially contaminated surfaces.

Decontaminate the pressure transducer and approximately five feet of cable by rinsing with deionized/distilled water and/or Alconox as appropriate.

Insert the transducer into the well. Gently lower it to the bottom of the well, then raise it slightly off the bottom. Note that the transducer has a response range, so that it is only accurate within a range of water depths. If the bottom of the well is outside the response range, raise the transducer until it is within range. Secure the transducer so that it will not move over the course of the test. Note the time at which the transducer is set in place.

Connect the transducer cable to the datalogger.

If a rising head test will be performed, carefully lower the bailer or slug bar into the well, making sure to raise and lower the bailer so that it completely fills with water.

Monitor the water level until it returns to equilibrium. Document the water level and time at which the water level reaches equilibrium.

Set up the datalogger according to the Supplemental Instructions for Using the Hermit SE1000C Datalogger (Attachment 2)

Rising Head Slug Test

In a rising head slug test, the water level in the well is caused to drop suddenly, either by bailing a volume of water from the well or by pulling a slug bar from the well. If a falling head test has just been completed, a rising head test can be done immediately afterward, as long as the water level has returned to equilibrium.

Start the datalogger and quickly remove the bailer or slug bar from the well. Make sure that the transducer is not pulled up with the slug, and, if a bailer is used, pull it completely out of the well to make sure that water does not leak back into the well. Empty water away from well.

Take a water level reading and record the time. Repeat this every couple of minutes until equilibrium is reached. In very permeable soils, the water level can recover in less than a minute, but it is good to collect data for at least three minutes to make sure the test is completed. Stop the test after the water level has returned to equilibrium.

Take a final water level reading and record the time.

Falling Head Slug Test

In a falling head test, the water level in the well is caused to rise quickly, either by inserting a slug bar into the well or by pouring water into the well. If water is used, it should be distilled or deionized so that it will not interfere with any water samples that might be collected from

the well. If a slug bar is used, care must be taken that it does not touch the transducer during the course of the slug test. One way to ensure that this does not happen is to tie off the slug bar line before starting the test, so that the bar cannot go far enough into the well to touch the transducer.

Begin the test after all equipment is in place and the water level has returned to equilibrium. First start the datalogger, then immediately drop the slug bar or pour the water into the well.

Take a water level reading and record the time. Repeat this every couple of minutes until equilibrium is reached. In very permeable soils, the water level can recover in less than a minute, but it is good to collect data for at least three minutes to make sure the test is completed. Stop the test after the water level has returned to equilibrium.

Take a final water level reading and record the time.

Download the test data from according to the Supplemental Instructions for Using the Hermit SE1000C Datalogger (Attachment 2). Print a hard copy of the datalogger data as soon as is practical after the test is completed. Sign or initial, date, and write the study number on each page of the printout, and retain these print-outs as raw data. The total number of pages for each test should be documented on the first page of each test print-out.

4.2.2 Slug Tests Using the Troll SP4000 System

Measure the water level in the well, following standard operating procedures for water level measurement. This is the equilibrium water level. Note the time of the measurement.

Check water level against documented screened interval for the well. If the equilibrium water level is at or above the top of the well screen, then both a falling head test and a rising head test may be performed on the well. However, if the equilibrium water level is below the top of the screen, then only a rising head test will be valid.

If the well is equipped with a dedicated pump and associated tubing that will interfere with the transducer, cable, and bailer/slugbar, remove the pump assembly and coil it into a container lined with a new trash bag. Wear clean gloves whenever handling a pump assembly. Avoid letting the pump assembly contact the ground or any other potentially contaminated surfaces.

Connect the cable to the Troll probe.

Decontaminate the Troll and approximately five feet of cable by rinsing with deionized/distilled water and/or Alconox as appropriate. Remove the probe tip and decontaminate it with a gentle stream of water. Be careful not to damage the transducer's membrane.

The Troll SP4000 can be damaged if it is deployed more than35 feet below the water surface. If the water in the well is deeper than 35 feet, insert the Troll into the well and lower it to 10 - 20 feet below the water surface. If the water in the well is not too deep, lower the Troll to the

bottom of the well, and then raise it slightly off the bottom. Secure the Troll so that it will not move over the course of the test.

Connect the end of the yellow (or green) cable to the grey cable and the grey cable to the computer's serial port (Com. port 1).

If a rising head test will be performed, carefully lower the bailer or slug bar into the well using an inert line, making sure to raise and lower the bailer so that it completely fills with water.

Monitor the water level until it returns to equilibrium. Document the water level and time at which the water level reaches equilibrium.

Set up the datalogger

1. Turn computer on and launch the "Win-Situ" program.
2. Highlight the troll and go to "Network" menu and select "Locate Device".
3. Under "Device" menu, select "Test Facility". A list of tests will open. Select a new test (numbered 1-16) and click on the "Define" button. A window pops up - select each option based on information listed on the sample form. Under "Test Channels" window, highlight temperature and hit spacebar to de-select (only pressure needs to be selected).

Rising Head Slug Test

In a rising head slug test, the water level in the well is caused to drop suddenly, either by bailing a volume of water from the well or by pulling a slug bar from the well. If a falling head test has just been completed, a rising head test can be done immediately afterward, as long as the water level has returned to equilibrium.

Click the start button; you will be prompted to confirm the action. Click "Yes" and then quickly pull the bailer or slugbar out of the well and note the time. Make sure that the Troll is not pulled up with the slug, and, if a bailer is used, pull it completely out of the well to make sure that water does not leak back into the well. Dump water away from well.

Falling Head Slug Test

In a falling head test, the water level in the well is caused to rise quickly, either by inserting a slug bar into the well or by pouring water into the well. If water is used, it should be distilled or deionized so that it will not interfere with any water samples that might be collected from the well. If a slug bar is used, care must be taken that it does not touch the probe during the course of the slug test. One way to ensure that this does not happen is to tie off the slug bar line before starting the test, so that the bar cannot go far enough into the well to touch the probe.

Once the water level has returned to equilibrium, start the test from the computer. Click "Yes" to confirm the action and then quickly drop the slug bar or pour the water into the well.

The remainder of the procedure applies to both rising head and falling head tests. The water level can be displayed as the test proceeds, so the operator can tell if the test is proceeding properly. Approximately 30 seconds into the test, select extract from the Win-Situ menu. The extracted data can then be displayed in graphical form by selecting the graphing button. Tests may be aborted at this point if the instrument has clearly malfunctioned.

Take a water level reading and record the time. Repeat this every couple of minutes. In very permeable soils, the water level can recover in less than a minute, but it is good to collect data for at least three minutes to make sure the test is completed. Tests in slower soils can take much longer. Stop the test from the computer after the water level has returned to equilibrium. If an error message is displayed and the test is not valid, shutdown Win-Situ and restart the computer.

Take a final water level reading and record the time.

On the computer, click the "Extract" button and then select "Save" at the next window that pops up. Save the file with an appropriate name.

Remove the Troll from the well. Reinstall the dedicated pump if necessary.

Print a hard copy of the data file as soon as is practical after the test is completed. Sign or initial, date, and write the study number on each page of the printout, and retain these print-outs as raw data. The total number of pages for each test should be documented on the first page of each test print-out.

4.2.3 Slug Tests Using Manual Water Level Measurements

In soils with low hydraulic conductivities, it may be possible to perform a slug test without a datalogger. Perform the tests essentially as detailed above, ensuring that the water level is at equilibrium before the slug is dropped or pulled.

Pull or drop the slug and document the time.

Measure the water level as soon as possible after the slug is pulled or dropped and document the time of the measurement.

Measure the water level repeatedly throughout the test until the water level has returned to equilibrium. For each water level measurement, document the time of the measurement. The frequency of measurements necessary will depend on the hydraulic conductivity of the soil. For slow soils, minutes may pass without appreciable change in the water level, whereas the water level in fast soils may rise so fast that it is impossible to monitor.

4.3 CALCULATIONS

Overview of the Bouwer and Rice method (Bouwer, 1989).

The method of Bouwer and Rice is based on the following equation:

$$K = \frac{r_c^2 \ln(R_e/r_w)}{2L_e} \frac{1}{t} \ln \frac{y_0}{y_t} \qquad (1a)$$

where:

K = hydraulic conductivity
r_c = radius of the well casing
R_e = effective radial distance over which y is dissipated
r_w = radius of the borehole
L_e = length of well screen (or length of water inlet into well)
t = time elapsed after initial change in water level
y_0 = distance between equilibrium water level and initial water level to which water is lowered or raised
y_t = distance between equilibrium water level and water level at time t

These parameters are shown diagrammatically in Bouwer (1989). The value of R_e is not calculated directly, but the value of [ln (R_e / r_w)] is calculated for each well using one of the following equations from Bouwer (1989):

if $L_w < H$:

$$\ln \frac{R_e}{r_w} = \left[\frac{1.1}{\ln(L_w/r_w)} + \frac{A + B \ln[(H - L_w)/r_w]}{L_e/r_w} \right]^{-1} \qquad (2a)$$

if Lw = H (i.e., if there is an impermeable layer at the bottom of the well screen):

$$\ln \frac{R_e}{r_w} = \left[\frac{1.1}{\ln(L_w/r_w)} + \frac{CA + B \ln[(H - L_w)/r_w]}{L_e/r_w} \right]^{-1} \qquad (3a)$$

where:

R_e = effective radial distance over which y is dissipated
r_w = radius of the borehole
L_w = distance between equilibrium water level and bottom of screen
H = distance between equilibrium water level and the first impermeable layer below well screen

A, B, and C = dimensionless parameters described in Bouwer (1989)

The values of t, y_0, and y_t are determined by plotting the difference between the water level in the well and the equilibrium water level over the course of the slug test.

Data Reduction

The goal of the data reduction process is to create a plot of y versus t, where t is the elapsed time from the beginning of the test and y is the distance between the water level at time t and the equilibrium water level. When this plot is drawn on semi-logarithmic paper, with y on the log axis and t on the normal axis, the data points should form a straight line, and the slope of this line will be used to calculate the hydraulic conductivity of the soil. This generality holds true when using AquiferTest© software for slug test analysis as well.

Examine the data. For each reading, the datalogger records the time of the reading (DT) and the water level (WL) at that time. DT may be in clock time or in elapsed time. WL will be recorded either as an elevation (either relative or absolute) or as a distance from the top of the well casing (TOC). The way that each of these parameters was recorded should be written in the field notes.

For readings made before the slug was pulled (or dropped), the values for WL will be fairly constant; this is the equilibrium water level (EWL). The values change suddenly for readings made at the time the slug was pulled or dropped, then return toward EWL for the remainder of the test. Whether the values increase or decrease depends on whether the test is a rising head or falling head test, and whether the water level readings are recorded as elevations or as distances below TOC, as shown in Table 1 below.

Table 1
Behavior of WL values when slug is pulled or dropped

Type of WL Measurement	Type of Test	
	Rising Head (slug pulled)	Falling Head (slug dropped)
Elevation	decrease	increase
Distance below TOC	increase	decrease

Determine the value for the equilibrium water level (EWL) measured by the transducer (not to be confused with the water level measured from the top of the casing). The datalogger will record slight variations in water level even before the slug is pulled, and some judgement must be used in determining the best value for equilibrium. Document the value used for EWL.

Calculate "y", which is the distance between the equilibrium water level and the water level at each reading. If the values for WL decrease when the slug is pulled, then:

$$y = EWL - WL \qquad (1)$$

If the values for WL increase when the slug is pulled, then:

$$y = WL - EWL \qquad (2)$$

In an ideal test, the calculated value of y will always be positive. However, do not simply use the absolute value of y calculated using equation (1) or (2), because in practice the water level can fluctuate so much when the slug is pulled that y becomes negative for a short time (this is most common in falling head tests). If the absolute value were taken, these anomalous y values would become positive, which could result in erroneous hydraulic conductivity values. For any readings where y is negative, change the y value to the minimum accuracy of the datalogger. For example, if the datalogger is accurate to 0.001 feet, change any negative y values to 0.001 feet. This is necessary because calculating hydraulic conductivity necessitates log transforming the y values, and the log of a negative number does not exist.

Determine DT_0, the value of DT (time logged by the datalogger) at which y reaches its maximum value.

Calculate "t", the elapsed time from the start of the test, using the following equation:

$$t = DT - DT_0 \qquad (3)$$

Note that by using this equation, t equals zero at the start of the test.

Manually Plotting Slug Test Data (This section is unnecessary if using AquiferTest© software)

Plot y versus t on semi-logarithmic paper, with y on the Y- axis with a log scale, and t on the X-axis with a normal scale. Data points should be plotted from t = 0 until the water level has returned or nearly returned to its equilibrium level.

Examine the graph and determine where the data points form a straight line. Draw a best-fit line through these data points and extend the line so it crosses the Y-axis and either the X-axis or the right edge of the graph. Determine the value of y at t = 0 (y_0) and at another value of t (y_t).

The graph must be interpreted with care. A variety of factors, especially the drainage of the sand pack around the well screen, can affect the shape of the graph and make it difficult to determine the best straight line to draw through the data points. In rising head tests, drainage of the sand pack can cause a double straight line effect: an initial, steep line represents drainage from the sand pack, followed by a second straight line with a shallower slope that reflects the conductivity of the aquifer (Bouwer, 1989). In some cases, it may only be possible to determine a range of possible values for the hydraulic

conductivity. If the graph of the data is curvilinear, some justification should be given for the line drawn through the data points.

Calculations Using Manually Plotted Slug Test Data (This section is unnecessary if using AquiferTest© software)

The calculations for each slug test must be documented in detail. They may be done on a pre-printed worksheet, such as the one included in Attachment 1, or on another medium as appropriate. Each page of the documentation must include the well identification; the signature or initials of the personnel doing the calculations, and the date the calculations were completed.

Values for initial variables are defined as follows:

EWL Equilibrium Water Level (units = length)
DTS Depth to Top of Screen (units = length)
DBS Depth to Bottom of Screen (units = length)
DIL Depth to the shallowest impermeable layer below the well screen (if it is not known, this parameter may be estimated) (units = length)
SL Length of the well screen (units = length)
r_c Radius of well casing (units = length)
r_w Radius of well boring (units = length)

EWL, DTS, DBS, and DIL are entered as distances below a measuring point, either below ground surface (bgs) or below the top of the well casing (btoc). All four values must be referenced to the same measuring point. Document the measuring point and the measurement units for each value. Also document the measurement units for r_c and r_w.

Calculate L_e as follows:

if: DTS> or = EWL, then: L_e = SL
if: DTS < EWL, then: L_e = DBS - EWL.

Calculate L_w: Distance between the equilibrium water level and the bottom of the well screen (units = length):

L_w = DBS - EWL

Calculate H: Distance between the equilibrium water level and the first impermeable layer below the well screen (units = length):

H = DIL - EWL

Calculate L_e/r_w: Value used to determine A, B, and C, dimensionless parameters used in the Bouwer and Rice slug test method.

Determine A, B, and C (units = none):

A, B, and C are constants used in the calculation of ln (R_e/r_w). If $L_w < H$, only A and B are needed. If $L_w = H$, only C is needed. A, B, and C are derived by matching the calculated value of L_e/r_w to curves on a graph given in Figure 2 in Bouwer (1989).

Calculate $\ln(R_e/r_w)$ (units = none)

If $L_w < H$, use Equation (2a) in Section 4.3 to calculate $\ln(R_e/r_w)$. This is the case for most tests. If $L_w = H$, use Equation (3a) in Section 4.3 above; this indicates that the bottom of the well screen is lying directly on an impermeable layer.

Determine values for t (units = time); y0 (units = length); and y_t (units = length): these parameters are taken from the plot of y versus t. The t value here is the one corresponding to y_t.

Calculate the hydraulic conductivity, K (units = length/time), by inserting the values of $\ln(R_e/r_w)$, t, y_0, y_t, and r_c into Equation 1a in Section 4.3.

Calculating Input Parameters for AquiferTest© Analysis

When the Bouwer and Rice slug test analysis method is selected in AquiferTest©, the following input parameters are required:

r Inside radius of well casing (units = feet)
R Borehole radius (units = feet)
L Length of screen within the saturated zone under static conditions (feet)
b Height of water column (feet)
D Saturated aquifer thickness (feet)

Calculation of the above parameters depends on the following additional parameters, which should be documented on slug test logs, well construction logs, and/or a site survey:

EWL Equilibrium Water Level (feet)
DTS Depth to Top of Screen (feet)
DBS Depth to Bottom of Screen (feet)
DIL Depth to the shallowest impermeable layer below the well screen (if it is not known, this parameter may be estimated) (feet)
SL Length of the well screen (feet)

EWL, DTS, DBS, and DIL are entered as distances below a measuring point, either below ground surface (bgs) or below the top of the well casing (btoc). All four values must reference the same measuring point.

Calculate b as follows: b = DBS - EWL

Calculate L as follows:

if: DTS> or = EWL, then: L = SL
if: DTS < EWL, then: L = DBS - EWL.

Calculate D (feet) as: D = DIL - EWL

In the calculation of D, the saturated aquifer thickness, we must make an assumption about the depth to the first impervious layer (DIL) when this is unknown. For consistency, DIL is frequently assumed to equal 40 feet bgs. Changing the value for depth to the shallowest impermeable layer has a relatively small effect on the resulting values for hydraulic conductivity (K), so long as D>>L.

Where the change in water level during the slug test occurs within the screened interval, it is necessary to use r effective (r_{eff}) in place of r in hydraulic conductivity calculations to obtain accurate results (Bouwer, 1989; Kruseman and de Ridder, 1994). AquiferTest© will calculate r_{eff} by default when the water level starts within or falls within the screened interval during a slug test. If water level change occurs within the screened interval, verify that r_{eff} is used in slug test calculations by checking that the r_{eff} button is selected in the settings window. The r_{eff}, the effective radial distance over which change in water level is dissipated, is calculated as

$r_{eff} = [r^2(1 - n) + nR^2]^{0.5}$, where n = porosity of the gravel or sand pack r_{eff}

Where r_{eff}, is calculated, a value for porosity of the sand pack must be entered. Because the well packing materials are not routinely tested, a constant must be assumed. Unless study specific information is available for the porosity of the sand pack, a literature value for porosity of 0.35 (35%) should be used (Keller, 1987).

Using AquiferTest© Software to Calculate K from Pre-Processed Slug Test Data

Open AquiferTest© and create a new project, with a new well and slug test. Save the project according to the Stone project name or project number.

Fill in *Preferences* under *File*, including the company information and logo.

Make sure that the units for Wells and Slug Tests are set appropriately, in *Project* | *Units* (feet rather than meters).

Right-click on the *Wells* folder and select "New Well". For the well name, enter the well ID from the slug test form. In the window that appears enter the following:

- Well Name: *well I.D*
- X-coordinate, Y-coordinate, Elevation (amsl), and Benchmark: leave at 0

- Enter the predetermined values for L, r, and R = Borehole radius.
- Uncheck the "Fully penetrating well" box (unless the well is actually fully penetrating).

Right-click on the *Slug Tests* folder and select "New Slug Test". For the slug test name, enter the well ID. Select the appropriate test well from the list in the dialog box. In the window that appears enter the following:

- Performed by, Date, Time: enter from slug test form
- Sat. aquifer thickness: enter predetermined value for D
- Depth to static WL: Enter equilibrium water level before test
- Water level at t=0: enter "0"
- b: Enter height of water column (b) from predetermined values

Right-click on the table in the lower left part of the screen and select *Import...*

Select the Excel file with well ID corresponding to the slug test name for import. Note: Excel files must be saved as Version 5.0/7.0 in order to import properly. Files saved in Excel 2003 format will not import.

In the pre-processed slug test data file, select the adjusted elapsed time and associated water level data from t=0 to the last reading. Click the *Import* button to paste the slug test data into the AquiferTest© data window.

Right-click again on the now-populated data table and select *Create Analysis | Bouwer-Rice.*

Choose Bouwer-Rice under *Method, Select.*

Open the *Settings* window under *Method.* Values displayed for r, R, L and b should reflect your previous entries in the *well options* box. A value for *D* must be entered, and a value for *Gravel Pack Porosity* (e.g., 0.35) must be entered if R_{eff} is used. Select *OK*, and the slug test will run automatically.

AquiferTest© will calculate a best fit line to all the data entered and a K value in ft/min. This line may be adjusted to fit only the meaningful data using the arrow keys. For wells where the water table is above the screened interval, a straight line is typically obvious, barring initial fluctuations. Trailing points will deviate from this straight line. Do not use such points to fit the trend line; place more emphasis on points where y > 0.01 feet. For wells where the water table changes within the screened interval of the well, there is generally a steep, straight line of points at the beginning of the test as the sand pack drains into the well, followed by a second, shallower straight line. Position the trend line through the second data range. The break in slope between the two lines may be difficult to

determine and requires some judgement. AquiferTest© will continually recalculate K as the trend line is moved.

Zooming in to the relevant portions of the data in the graphing mode of AquiferTest© can help in accurately fitting the trend line and improve the appearance of the resulting graph. Zooming will not affect the calculated K value.

Print the final AquiferTest© plot (which includes the calculated K value). Sign and date the print-out and file in the study files.

The rules of significant figures are followed according to standard operating procedures. Significant figures used in calculations should be that as displayed on the calculator, however, do not report more significant figures than necessary. Units must be documented and kept consistent throughout the calculations.

5.0 RESPONSIBILITIES

1. All data generated during the conduct of a study, except those that are generated by automated data collection systems, shall be recorded directly, promptly, and legible in ink. All data entries shall be dated on the day of entry and signed or initialed by the person entering the data. Any change in entries shall be made so as not to obscure the original entry, shall indicate the reason for such change, and shall be dated and signed or identified at the time of the change. In automated data collection systems, the individual responsible for direct data input shall be identified at the time of data input. Any change in automated data entries shall be made so as not to obscure the original entry, shall indicate the reason for change, shall be dated, and the responsible individual shall be identified.

2. All personnel will legibly record data and observations (including phone conversations) to enable others to reconstruct project events and provide sufficient evidence of activities conducted.

6.0 DEFINITIONS

1. *Observations & Remarks Form (O&R):* A pre-printed form, which contains mostly blank space for general note taking. The form typically prompts the user for the study or project designation, the SEI project number, the client or sponsor name, the total number of pages (page *n* of n) and requires a signature and date. The form is generally used to capture notes of one person when another, more specific forms is not available.

2. *Raw data* means any laboratory worksheets, records, memoranda, notes, or exact copies thereof, that are the result of original observations and activities of a study and are necessary for the reconstruction and evaluation of the report of that study. In the event that exact transcripts of raw data have been prepared (e.g., tapes which have been transcribed verbatim, dated, and verified accurate by signature), the exact copy or exact transcript may be substituted for the original source as raw data. Raw data may include

photographs, microfilm or microfiche copies, computer printouts, magnetic media, including dictated observations, and recorded data from automated instruments.

7.0 REFERENCES

40 CFR Part 160 Good Laboratory Practice Standards, August, 1989.

Bouwer, H., 1989. The Bouwer and Rice slug test - an update. Groundwater, volume 27, number 3, pages 304 - 309.

Keller, E.A. 1987. Environmental Geology. 5th edition. Merrill Publishing Company, Columbus, Ohio. p. 38.Kruseman, G.P., and N.A. de Ridder. 1994. Analysis and Evaluation of Pumping Test Data. 2nd edition. International Institute for Land Reclamation and Improvement. Wageningen, Netherlands. p. 246.

8.0 TABLES, DIAGRAMS, FLOWCHARTS, AND VALIDATION DATA

Attachment 1: Slug Test Calculations Worksheet (Example)

Attachment 2: Supplemental Instructions for Using the Hermit SE1000C Datalogger

9.0 AUTHORIZATION

Revised by: _____ Date: 3-2-06

Patrick Larsen, Project Scientist

Approved by: _____ Date: 3-2-06

Chris T. Stone, President

10.0 REVISION HISTORY

Revision number 2:

1. Re-worded Section 1.0
2. Added a clock to the equipment list.
3. Consolidated the field procedures.
4. Added detail to the data reduction and calculation section. The revised sections detail the method of Bouwer (1989), but as discussed in Section 1.0, other methods may be used if applicable.
5. Reformatted under new numbering scheme.

Revision number 3:

1. Re-worded Section 1.0.
2. Added a clock to the equipment list.
3. Consolidated the field procedures.
4. Added detail to the data reduction and calculation section. The revised sections detail the method of Bouwer (1989), but as discussed in Section 1.0, other methods may be used if applicable.
5. Reformatted under new numbering scheme.
6. Revision two (2) was incorrectly released as SEI-6.11.1 on the SOP and correctly named SEI-6.11.2 on the Master Index. This new revision corrects that and is issued as 6.11.3.Revision number 4:

Revision number 4:

1. Reformatted
2. Add documentation of the total number of pages from attachment.

Revision number 5:

1. Reformatted

Revision number 6:

1. Remove Attachment 1, change Attachment 2 to 1 and Attachment 3 to 2.
2. Updated style to match SEI Style Guide – font and text. Reformatted using MS Word.
3. Added standardized section headers: 2.0 Policies, 3.0 Safety, 5.0 Responsibilities, 6.0 Definitions, 7.0 References, 8.0 Tables, Diagrams, Flowcharts and Validation data. Authorization moved to Section 9.0, andSection10.0 Revision History

Revision number 7:

1. Updated Section 4.3 to reflect current version of AquiferTest© software.

Attachment 1
Slug Test Calculations Worksheet
(Example)

SLUG TEST CALCULATIONS WORKSHEET

WELL/TEST IDENTIFICATION: _____

SIGNATURE OR INITIALS: _____

DATE: _____

☐ Rising head test ☐ Falling head test

SEI STUDY NUMBER: _____

EWL =

DTS =

DBS =

DIL =

SL =

r_c = r_w =

if DTS ≥ EWL :
L_e = SL

if DTS < EWL :
L_e = DBS - EWL =

L_w = DBS - EWL =

L_e/r_w =

H = DIL - EWL =

if L_w < H:
A =
B =

if L_w = H:
C =

$$\ln[R_e/r_w] = \left[\frac{1.1}{\ln(L_w/r_w)} + \frac{A+B\ln[(H-L_w)/r_w]}{L_e/r_w}\right]^{-1} \quad \text{OR} \quad \ln[R_e/r_w] = \left[\frac{1.1}{\ln L_w/r_w} + \frac{C}{L_e/r_w}\right]^{-1}$$

t =

y_0 =

y_t =

$\frac{1}{t}\ln[y_0/y_t]$ =

$$K = \frac{r_c^2 \ln[R_e/r_w]}{2L_e}\left(\frac{1}{t}\ln[y_0/y_t]\right)$$

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Attachment 2
Supplemental Instructions for Using the Hermit SE1000C Datalogger

SETTING UP THE DATALOGGER

Setting the Date and Time

a) Turn on the datalogger by pressing any key on the keypad. The display will show a single period (".")

b) Press the "CLOCK" key while holding down the "ENTER" key. A date will show on the screen with the first digit flashing. The first two digits indicate the month and the last two digits indicate the day

c) Change the date by using the arrow keys to increase or decrease the value of a digit and the "STOP/NEXT" key to move from one digit to the next.

d) When the correct date is showing, press "ENTER". If an invalid date is entered, such as "13 12", the display will show the word "Error", then it will display the time again. When a valid time is entered, the display will show a time in hours and minutes (on a 24-hour clock).

e) Follow a similar procedure to enter the correct time, and press "ENTER". The display will return to the "." prompt.

The ENTER DATA Menu

a) If the datalogger is not already on, turn it on by pressing any key on the keypad. The display will show the "." prompt.

b) Access the ENTER DATA menu on the datalogger by pressing the "DATA" key while holding down the "ENTER" key. The display will show "SEL *n*", where *n* is a flashing number that refers to the first available test number. Test numbers lower than *n* have already been used and have data associated with them.

c) Press the up and down arrow keys to change the test number. When the desired test number is reached, press "ENTER". If a number lower than the default number is selected, the data in all tests above this will be erased, so make sure this is OK. (Note that datalogger will not allow a number higher than the default to be selected).

d) When the test number is selected, the a series of dashes will show on the display momentarily, then the word "rAtE" will appear. At this point, the user can select the time step at which readings will be taken. Press "ENTER", then press the arrow keys until the word "LOg" is shown. Press "ENTER" again to select the log rate scale. This way, the time between readings will increase periodically as the test proceeds.

e) Now a number will flash on the display indicating the longest time step between readings (the maximum sample interval). Press the "STOP/NEXT" key to move the cursor between digits and the arrow keys to change the value of each digit until the display reads "0001", indicating that the time between readings will increase until the readings are 1 minute apart, then readings will be taken at 1 minute intervals until the test is stopped. Press "ENTER", and the display will again show "rAtE".

f) Press the "ENTER" key once, and the display will show "InP. n", where n is either 1 or 2. If n is 2, press enter, and it will begin to flash. Press the down arrow key to change it to 1, then press return. If n is already 1, proceed to step g).

g) Press the down arrow key so that the display shows "tyPE", and press "ENTER". Press the arrow keys until the display shows "LEVEL" and press "ENTER"; this will tell the datalogger that the readings from the pressure transducer are to be converted to water levels.

h) The display will again show "tyPE". Press the "STOP/NEXT" key, and a number will show briefly on the display. This is the remaining number of data points that the datalogger's memory has room to store. Note this number as a check to make sure the datalogger is indeed collecting data. The number will show for only 1-2 seconds, then the display will return to the "." prompt.

The ENTER XD Menu

a) Connect the pressure transducer cable to the "INPUT 1" port on the datalogger.

b) With the display at the "." prompt, press the "XD" key while holding down the "ENTER" key. The display will show "rEF.".

c) Reference: Set the reference water level to zero [note that although the rest of the ENTER XD menu can be set up at any time before the test begins, this step must be done with the transducer set in the well and hooked up to the datalogger]. Press "ENTER", and a number will show on the display with the first digit flashing. Using the arrow keys to change the values of the digits and the "STOP/NEXT" key to move the cursor between digits, change the number to 000.00 and press "ENTER". A series of dashes will flash on the screen momentarily as the datalogger takes a reading from the transducer and translates it to zero feet of water, then the display will show "rEF." again. If the pressure transducer is not connected to the datalogger, the word "Error" will flash on the screen, the "rEF.".

d) Scale: Press the arrow key once, and the display will show "Scale". Press "ENTER", and a number will show. Using the arrow and "STOP/NEXT" keys, change the number to match the scale value stamped on the side of the pressure transducer under "QUAD COEF." (14.98). Note that the datalogger will only accept values to the nearest 0.01, so round the transducer's scale factor to the nearest 0.01 and enter that number. Press "ENTER", and the display will again show "Scale". Record the scale value.

e) Offset: Press the arrow key once, and the display will show "OFFS.". Press "ENTER", and a number will show. Using the arrow and "STOP/NEXT" keys, change the number to match the offset value stamped on the side of the pressure transducer under "QUAD COEF." (0.02). Note that the datalogger will only accept values to the nearest 0.01, so round the transducer's offset factor to the nearest 0.01 and enter that number. Press "ENTER", and the display will again show "OFFS.". Record the offset value.

f) Linearity: Press the arrow key once, and the display will show "Lin.". Press "ENTER", and a number will show. Using the arrow and "STOP/NEXT" keys, change the number to match the linearity value

stamped on the side of the pressure transducer under "Quadratic " (0.11). Note that the datalogger will only accept values to the nearest 0.01, so round the transducer's linearity factor to the nearest 0.01 and enter that number. Press "ENTER", and the display will again show "Lin.". Record the linearity value.

g) Delay: Press the arrow key once, and the display will show "dELAy". Press "ENTER", and a number will show. Using the arrow and "STOP/NEXT" keys, change the number to match the minimum warmup delay (in milliseconds) required by the transducer before it can take a reading. This number should be stamped on the side of the pressure transducer, and is usually 50 milliseconds. Press "ENTER", and the display will again show "dELAy".

h) Display: Press the arrow key once, and the display will show "dSP.". Press "ENTER", and some letters will show. Press the arrow keys until the display shows "En:Sur". This will tell the datalogger to record the water levels in English units (feet) as elevations of the water surface relative to the reference datum (as opposed to depths below top of casing). Press "ENTER", and the display will again show "dSP.".

i) I.D.: Press the arrow key three time (scrolling past the alarm settings which are not necessary) until the display shows "i.d.". Press "ENTER", and a number will show. The user may now enter a five digit code number as an identification number for the test. Use the arrow and "STOP/NEXT" keys to enter the desired ID number on the display, and press the "ENTER" key. The display will again show "i.d.". Record the ID value.

j) The ENTER XD menu is complete. Press the "STOP/NEXT" to return to the "." prompt.

CONDUCTING THE TEST

a) Start the test: With the datalogger at the "." prompt, press the "START" key, and the display will show the word "StArt"; the datalogger is ready to begin collecting data. Make sure that the transducer and slug are set in place in the well, the water level has returned to equilibrium, and the transducer is connected to the datalogger. Now press the "ENTER" key and quickly pull the slug out of the water. The datalogger starts to collect data as soon as the "ENTER" key is pressed, so pull the slug as soon as possible to collect the most data. If a solid slug bar is used, it only needs to be pulled up far enough so it is completely out of the water, but a bailer should be pulled completely out of the well to ensure that water does not leak back into the well and disrupt the test.

Note the time when the test is started.

As the test proceeds, the display will show "LOg n", where n is the log cycle indicating the frequency of readings (in Cycle 1 the datalogger makes one reading every 0.2 seconds, in Cycle 2 the datalogger makes one reading every second, etc.). After ten minutes, the display changes to "run." and stays that way until the test is stopped. The data cannot be viewed while "LOg n" is displayed.

b) Manual water level measurements: Measure the water level periodically during the test using the water marker. Record the times of the measurements.

c) Ending the test: When the water levels show that the well has returned to equilibrium, stop the test by pressing the "STOP" key while holding down the "ENTER" key. If this is done while the display shows "LOg n", the datalogger will stop recording and return to the "." prompt. If "ENTER" and "STOP" are pressed after the first ten minutes of the tests (while the display shows "run."), the display will then show "StOP" and the user will need to press "ENTER" again to confirm the stop. Note the time when the test is stopped.

DOWNLOADING THE DATA

Connect the communications cable to the RS232 port on datalogger and to the serial port (Com 1 or Com 2) on a computer.

a) Load the In-Situ Inc. data transfer programs DATATRAN.EXE and DT.BAT onto the computer's hard drive.

b) From the directory where the data transfer programs are stored, run DT.BAT, which starts the data transfer program, and get to the screen entitled "HERMIT Data Transfer Utility".

c) At the prompt for "Header + numeric file name:", type the name of the file in which the test data will be stored. The program downloads one test at a time, so each test will has a separate file name. A full pathname may be entered if desired; otherwise, the file will be downloaded to the directory where DATATRAN.EXE is stored.

d) At the prompt "Create numeric data file:", enter "No".

e) At the bottom of the screen are the communications parameters. Ensure that the parameters on the "HERMIT Data Transfer Utility" screen match those of the serial port that the datalogger is connected to.

f) Press <F1> on the computer keyboard; screen will then show the prompt:

"Wait until: HERMIT 1000 shows "out" or SEnd"; Then press any key to begin transfer..."

Make sure the cable connections are secure. From the "." prompt on the datalogger, press the "DATA" key. The display will show "Out. n", where n is the number of a test. Use the arrow keys to change n to the number of the test to be downloaded, then press "ENTER". [If the display shows "dSP. n" when the "DATA" key is pressed, the computer connections are not right; check all connections and communications parameter settings, then try again.]

g) The display will show "SP:000" (percentage point at which to start download); press "ENTER". The display will then show "EP:000" (percentage point at which to end download); press "ENTER" again. The display will now show "SEnd".

h) Press any key on the computer keyboard to start the downloading process. The computer screen will show the message "Transferring data..." until the download is complete, then it will show the message "End of data" and the cursor will return to the "Header + numeric data file name" prompt.

i) Enter the name of the next file to be downloaded and repeat the download process. When there are no more tests to download, press the <Esc> key on the computer to end the data transfer program.

DATA FILE CONFIGURATION-"Header and Numeric Data File"

a) Line 1: blank

b) Lines 2 and 3: "SE 1000C Environmental Logger" (identifies the Hermit model datalogger)

c) Line 4: date and time of printout

d) Line 5: blank

e) Line 6: "Unit # nnnnn" (the datalogger serial number) and "Test n" (the test number)

f) Line 7: blank

g) Lines 8-12: Identifies the test set-up. "Type" is "level" indicating that the readings are recorded as water levels; "Mode" is "surface", indicating that the water levels are recorded as relative elevations rather than relative depths; "I.D." is the test identification code entered in the ENTER XD menu.

h) Line 13: blank

i) Line 14: Reference. This is the reference value for equilibrium water level entered in the ENTER XD menu.

j) Lines 15-18: pressure transducer parameters entered in the ENTER XD menu.

k) Line 19: blank

l) Line 20: "Step 0" (following the procedure above, the tests are not stepped), then the date and time that the test is started.

m) Line 21: blank

n) Lines 22-23: Column headings for water level data

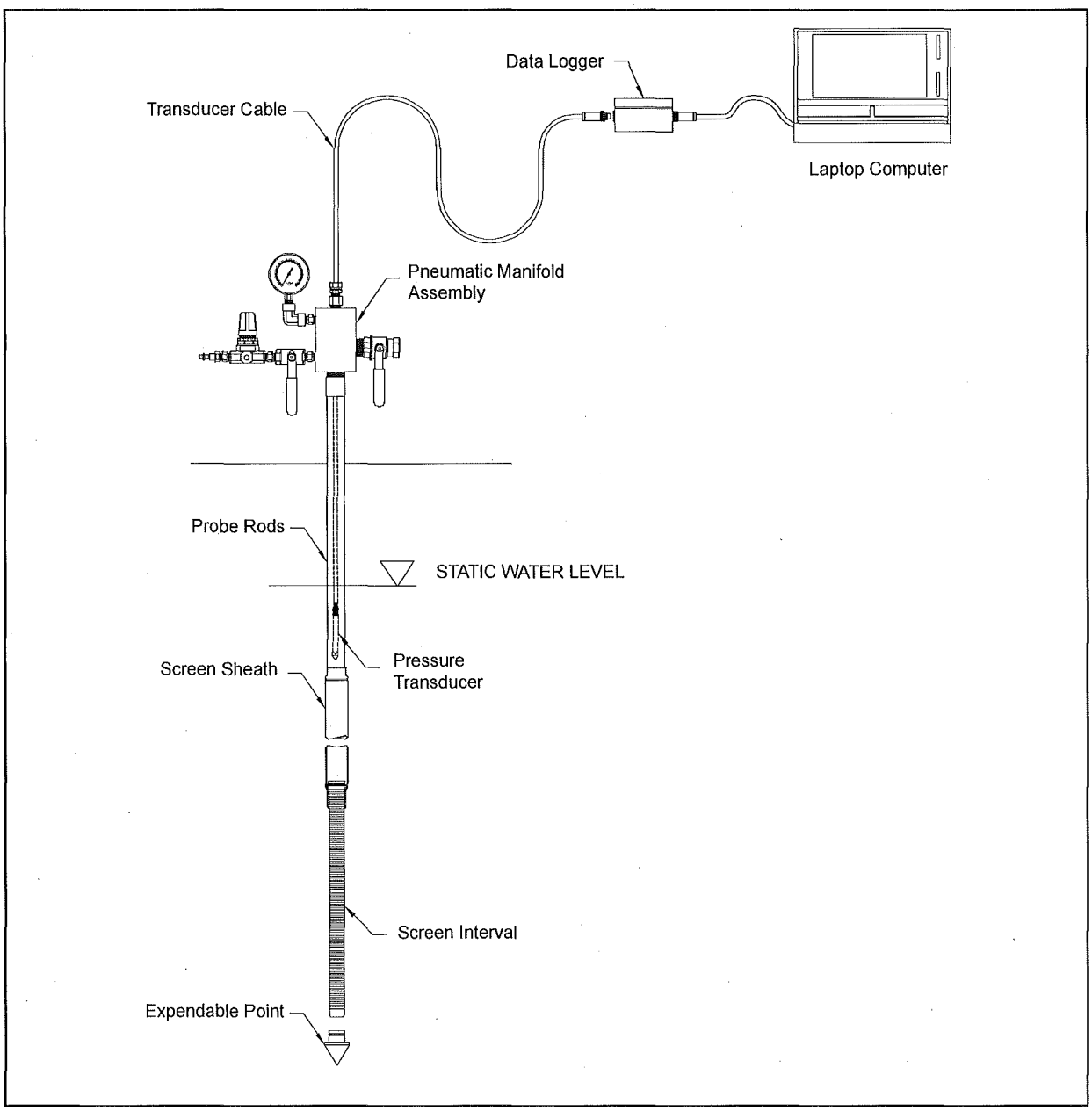
o) Lines 24 and following: The water level data. In the first column is "Elapsed Time", the time of each reading, recorded in minutes from the start of the test. In the second column is "Input 1", the water level reading in feet above the equilibrium water level (the reference level set in the ENTER XD menu). If a second numeric data file is created in the downloading process, that file will contain only these lines, without the column headings.

GEOPROBE® PNEUMATIC SLUG TEST KIT

STANDARD OPERATING PROCEDURE

Technical Bulletin No. 19344

PREPARED: February, 2002
REVISED: January, 2011



PNEUMATIC SLUG TESTING WITH A SP16 GROUNDWATER SAMPLER

Geoprobe® Systems

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1.0 Ojective

The Pneumatic Slug Test Kit is used in conjunction with a groundwater sampler such as the Screen Point 16 or a monitoring well to conduct pneumatic slug tests in saturated formations of unconsolidated soils or sediments. This document identifies the equipment and techniques required to successfully operate the Pneumatic Slug Test Kit (GW1600) and obtain the necessary data to determine the formation hydraulic conductivity.

2.0 Background

2.1 Definitions

Geoprobe®: A brand name of high quality, hydraulically-powered machines that utilize both static force and percussion to advance sampling and logging tools into the subsurface. The Geoprobe® brand name refers to both machines and tools manufactured by Geoprobe Systems®, Salina, Kansas. Geoprobe® tools are used to perform soil core and soil gas sampling, groundwater sampling and testing, soil conductivity and contaminant logging, grouting, and materials injection.
**Geoprobe® is a registered trademark of Kejr, Inc., Salina, Kansas.*

Slug Test: Fetter (1994) defines a slug test as "an aquifer test made by pouring a small instantaneous charge of water into a well or by withdrawing a slug of water from the well" (e.g. with a bailer). The initial change in the water level or head (H_o) is recorded and then the time (seconds) and changing water level (H_t) at each time increment are recorded. The water level changes at each time increment are normalized by the initial change of water level (H_t/H_o) and plotted on semilog graph paper (Fig. 2.1). Information from this graph is then used to calculate the hydraulic conductivity of the formation.

Hydraulic Conductivity (K): Again, Fetter 1994 defines hydraulic conductivity (K) as "a coefficient of proportionality describing the **rate at which water <u>can</u> move through a permeable medium**" (emphasis added). Note that this definition says "can move". To know the actual flow rate at a location, the groundwater gradient must also be measured. There are two primary reasons why we are interested in the "K-value" of a formation, or portion of a formation. First, this helps us know if the formation is a viable aquifer, that is can it yield enough water for a well. Second, the K-value gives us a measure of how fast contaminants could move through a formation and reach a water supply well. This information can be used to determine the potential 'risk' caused by the presence of contaminants in the subsurface for risk based corrective action (RBCA) models (ASTM E 1739) and for evaluating the application of monitored natural attenuation (MNA) as a remedial option (EPA 1998). The formation hydraulic conductivity is also a key piece of information required to properly design many groundwater remediation systems.

2.2 Discussion

Water should not be added to a well located in a contaminated aquifer when performing a slug test. Likewise, as little water as possible should be removed from the well in order to minimize waste handling. Because of this, investigators slug testing for environmental purposes began using "mechanical slugs" to initiate slug tests and pressure transducers to record the data. A pressure transducer is especially needed in fast recovering wells.

A mechanical slug is simply a length of pipe filled with sand, capped on both ends, with a cord on one end to lower and recover the slug from the well. This method (Fig. 2.2) works well in many formations, but usually causes some splashing in the well as the slug is lowered or raised. When the slug test is over in 20 or 30 seconds, as for many sandy aquifers, this splashing 'noise' can interfere with the data collection and the results may be useless or questionable at best. This is why pneumatic slug testing was developed (Prosser 1981, Butler 1997).

In pneumatic slug testing the well head is sealed and air pressure is used to displace/lower the water level. As air pressure in the well is increased, the water level falls (Fig. 2.3) until the water pressure 'up' and the air pressure 'down' are equal. Once the water level is stable, a release valve is quickly opened, instantaneously releasing the air pressure. The water level recovers (rising head test) without splashing and the pressure transducer and data logger/computer record the changes in water level and time.

Pneumatic slug testing can provide very high quality data with essentially no "noise" or interference from splashing as is caused by the mechanical slug method. This becomes more important in very high-K formations which yield an oscillatory response to slug testing.

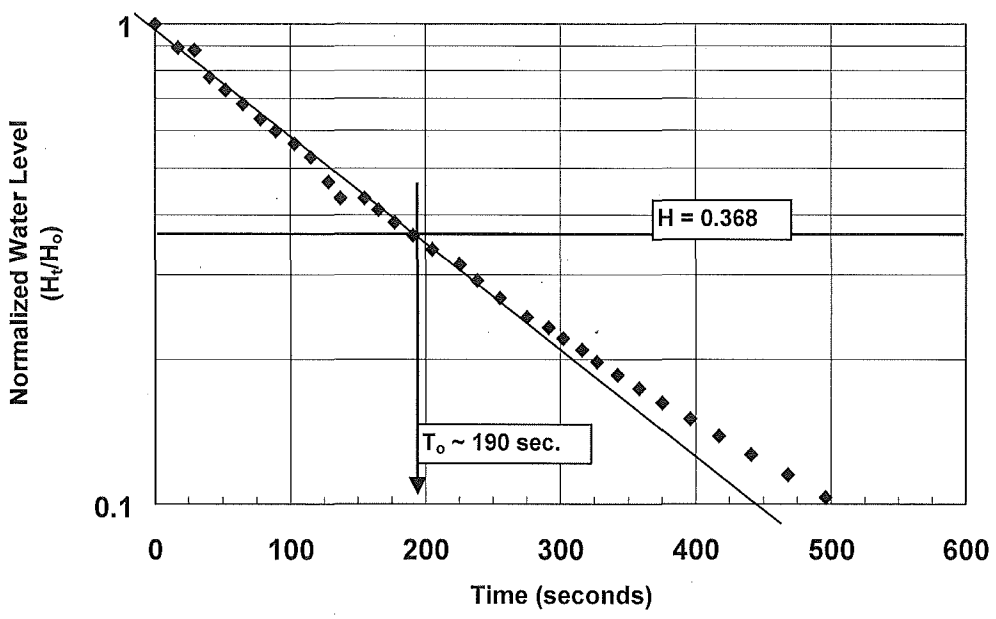


FIGURE 2.1:
Plot of normalized water levels recorded during a pneumatic slug test performed using a SP16 Groundwater Sampler.

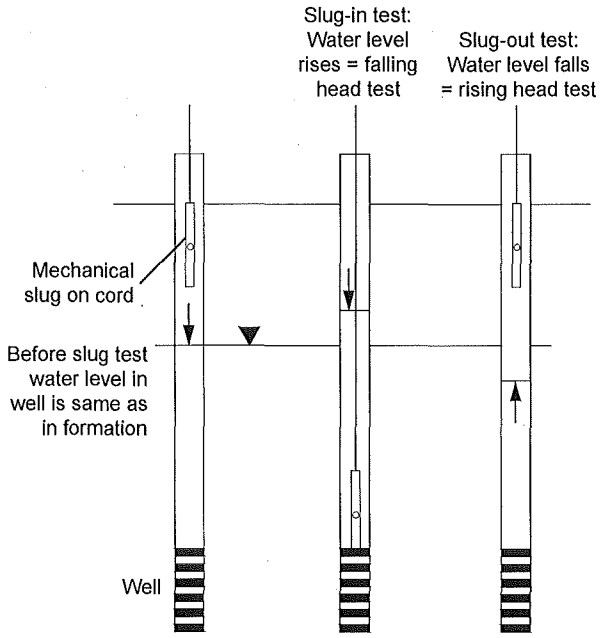


FIGURE 2.2:
Mechanical slug testing.

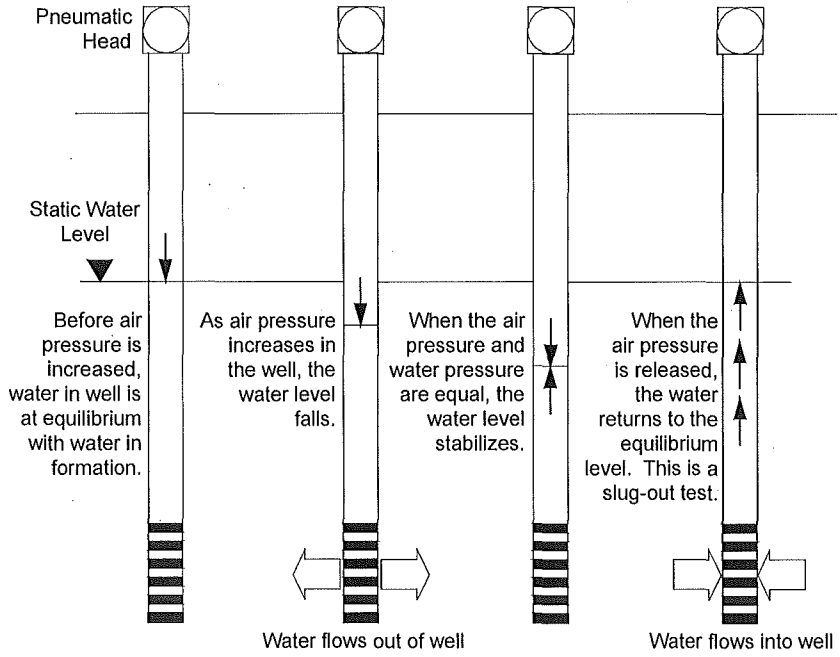


FIGURE 2.3:
Pneumatic slug testing.

3.0 Required Equipment

The following equipment is required in order to perform a pneumatic slug test with the GW1600 Slug Test Kit. Refer to Figure 3.1 for identification of the major parts included in the GW1600 kit.

Pneumatic Slug Test Kit (GW1600) includes:

Description	Quantity	Part Number
Pneumatic Head Assembly	(1)	17631
Low-Pressure Hose Assembly	(1)	17644
Foot Pump Assembly	(1)	17643
Transducer Assembly	(1)	19345
Data Logger	(1)	GW1610
Integral Data/Power Cable	(1)	19174
Power Cord	(1)	16839
Power Inverter (12VDC to 120VAC)	(1)	SC152
12V Socket Adapter (direct push machine to inverter)	(1)	10138
Data Acquisition Software (CD)	(1)	GW1615
Probe Rod Adapters:		
1.25-inch Pin x 1.5-inch Box	(1)	15813
2.125-inch rod adapter with valve	(1)	19165
Leak Test Fluid (8-ounce bottle)	(1)	12356
Teflon® Tape (1/2-inch x 50 feet roll)	(1)	8819
O-rings:		
1.25-inch Probe Rods	(1)	AT1250R
1.5-inch Probe Rods	(1)	15389
2.25-inch Probe Rods	(1)	AT2100R
Carrying Case*	(1)	19329

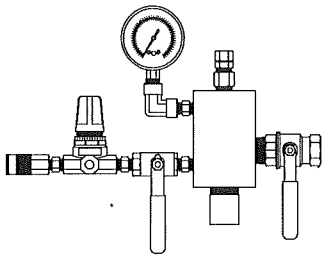
*Total weight of the Pneumatic Slug Test Kit and carrying case is approximately 29.8 lbs. (13.5 Kg).

Other materials or equipment that may be required for slug testing with this kit include:

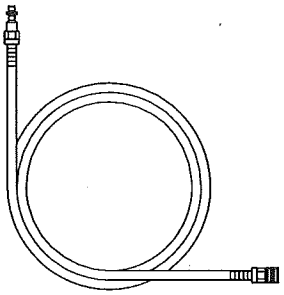
Well or Groundwater Sampler (installed and developed)
Lap Top Computer (for running data acquisition software)
Power Supply (120V line current or 12V vehicle battery connection)
Poly Tubing (TB25L) and Check Valve (GW4210) or other device for well development
Miscellaneous Hand Tools (wrenches, pliers, screw drivers, etc.)

PVC casing adapters will be required for slug testing monitoring wells with PVC casing:

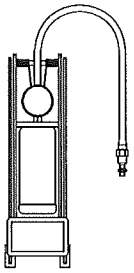
PVC Casing	Adapter Description	Quantity	Part Number
1/2-inch Schedule 80	Bushing, adapts 17558 to 1/2-inch PVC	(1)	17559
3/4-inch Schedule 40	Bushing, adapts 17558 to 3/4-inch PVC	(1)	19424
1-inch Schedule 40	Adapter, connects 17631 to 1-inch PVC	(1)	17558
2-inch Schedule 40	Coupling, adapts 19165 to 2-inch PVC	(1)	2164
Extension Tube, 18-inch, PVC (threads between 17631 and 17558)		(1)	17821



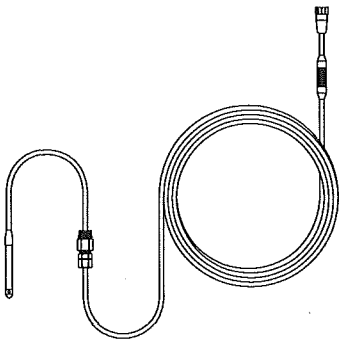
Pneumatic Head Assembly
(17631)



Low-Pressure Hose Assembly
(17644)

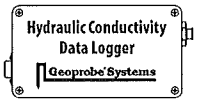


Foot Pump Assembly
(17643)

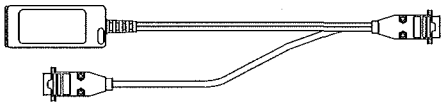


Transducer Assembly
(19345)

Data Logger
(GW160)

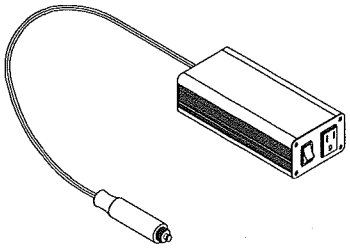


Integral Data/Power Cable
(19174)

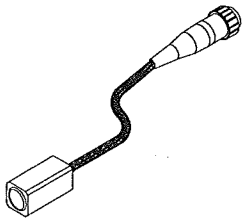


Power Cord
(16839)

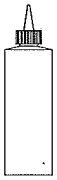




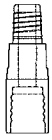
Power Inverter
(SC152)



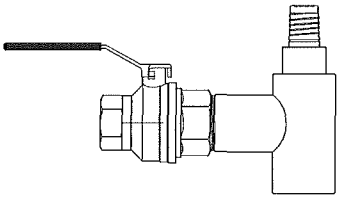
12V Socket Adapter
(10138)



Leak Test Fluid
(12356)



Adapter, 1.25 Pin x 1.5 Box
(15813)



2.125-inch Rod Adapter with Valve
(17532)

FIGURE 3.1:
Major components of the GW1600 Pneumatic Slug Test Kit. (Items not to scale)

4.0 Groundwater Sampler or Monitoring Well Installation

During the installation of the ground water sampler or monitoring well, the appropriate O-rings must be used on each casing or rod joint to assure that an airtight system is installed for pneumatic slug testing. To help preserve the integrity of the O-rings, first drive the rod to near ground surface, remove the drive cap, then install the O-ring and the next rod. When installing the SP16 sampler, be sure to use the appropriate O-rings on each part of the assembly as specified in the SOP. O-ring kits are available for the SP16 groundwater sampler.

Follow the manufacturer's standard operating procedure (SOP) for installation of the sampler or monitoring well. Geoprobe® SOPs are available for the Screen Point 16 Groundwater Sampler (Tech. Bull. No. 95-1500), Dual Tube (DT22) Groundwater Profiler Kit Instructions (Bull. No. 19275) or the Geoprobe® Prepacked Screen Monitoring Well SOPs (Tech. Bull. Nos. 96-2000 or 99-2500). Free copies of these bulletins may be obtained by contacting Geoprobe Systems®.

Standard Guides and Practices are also available from the American Society of Standards and Materials (ASTM D 6001, D 6724, D 6725) to provide further assistance on proper installation practices if needed. These Standards are available by contacting ASTM at 610-832-9585 or via the internet at www.astm.org.

In this Standard Operating Procedure (SOP) the Screen Point 16 groundwater sampler will be used to illustrate how the Pneumatic Slug Test Kit is used to conduct slug testing. The pneumatic slug testing procedures described here will be essentially the same for any Geoprobe® groundwater sampler or monitoring well. Only the adapter for attaching the pneumatic head to the well or sampler will be different.

The SP16 sampler is assembled and advanced to the bottom of the interval to be tested. Geoprobe® extension rods and screen push adapter (GW1535) are used to hold the screen in position as the rods and screen sheath are retracted with the direct push machine (Fig. 4.1). It is important to know the length of screen exposed to the formation so that an accurate K-value can be determined. For this reason, the length of screen to be exposed to the formation is measured and marked off on the extension rods before retraction is started. Approximately 2 inches (50 mm) must be added to the retraction distance to obtain the desired exposed screen length because of the expendable point and solid tip on the screen (Fig. 4.1, inset). Once the screen is in position, the sampler is ready for development.

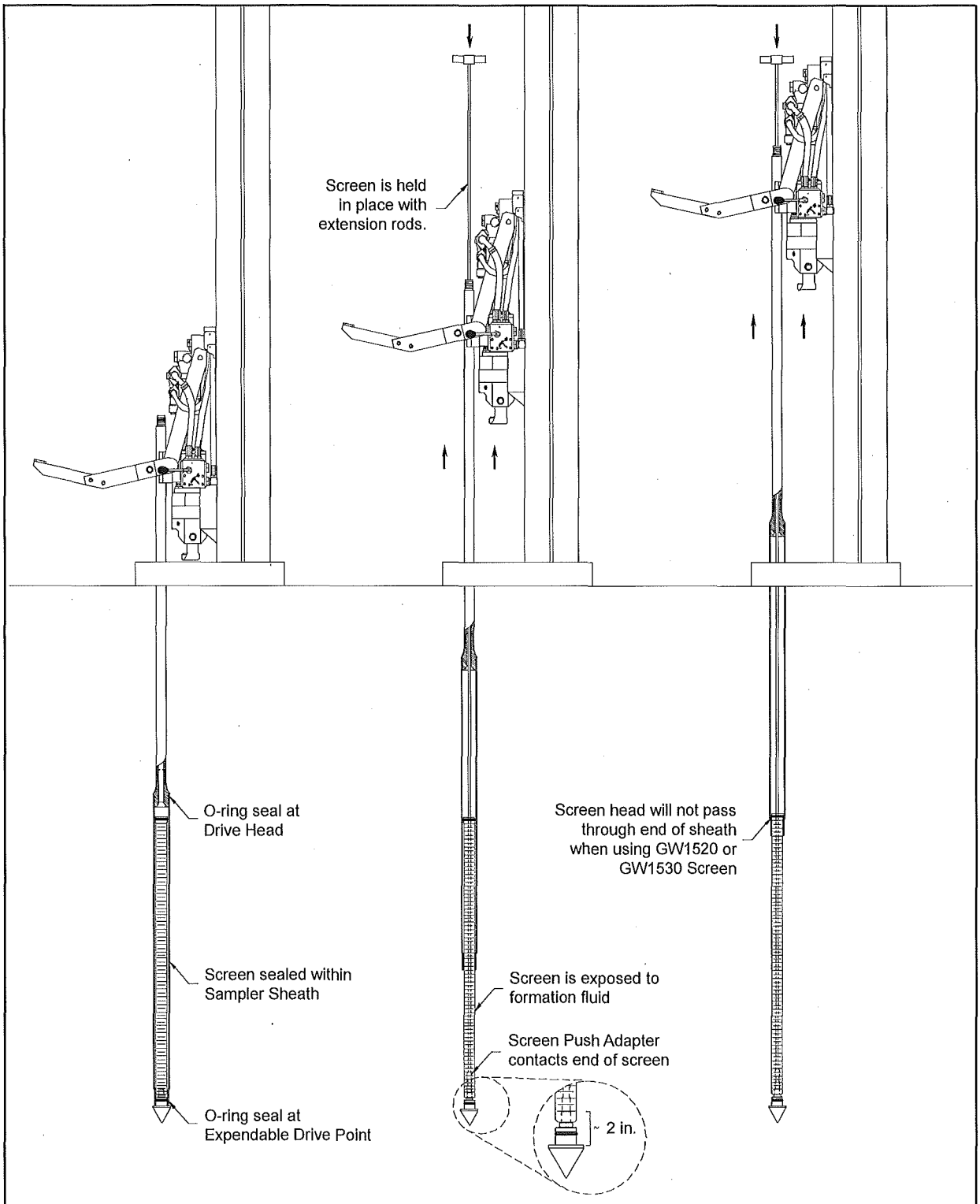


FIGURE 4.1:
Screen deployment for SP16 Sampler

5.0 Development of Screened Interval

Appropriate well development is absolutely essential in order to obtain representative slug test results from a well or sampler. Without adequate development the K-value determined from slug testing may be more than ten times lower than the true value (Butler 1997). In ASTM Guide D 5521 *well development* is defined as:

> - the act of repairing damage to the borehole caused by the drilling process and removing fine grained materials or drilling fluids, or both, from the formation materials so that natural hydraulic conditions are restored ...

While the direct push method of well installation usually does not involve rotary drilling or drilling fluids, formation damage often occurs. This is usually compression of the formation as the tool string is advanced and may include smearing of clays on the borehole wall in formations containing these fine grained materials. In poorly cohesive materials such as saturated sands, reparation of the damage is usually easy and relatively quick. The simple tubing check valve (GW4210) and polyethylene tubing are lowered into the screen interval and vigorously oscillated up and down (Fig. 5.1). The vigorous oscillating action both surges the screen interval and purges dislodged fines from the well simultaneously.

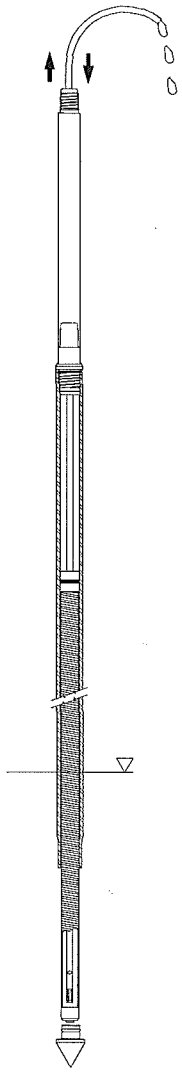


FIGURE 5.1:
Surging with tubing and check valve assembly

Even in what are generally termed "clean sands", turbid, muddy water is purged from the well. Surging and purging should be conducted throughout the length of the exposed screen to properly develop the entire screen exposed to the formation. For each one-foot (30 cm) of screen length, purging from one to three gallons (4 to 12 liters) of water with the tubing check valve method is usually sufficient to obtain representative results. In sandy formations this may require from 5 to 20 minutes depending on the yield of the formation. When development of the well is complete, repeated slug tests will provide consistent results. Therefore, one way of checking for adequate development is to run three or more slug tests and see if the response curves are consistent and symmetrical. If the results change noticeably with each slug test, further development may be required.

In formations containing a significant proportion of clays, care must be taken in the development process so that clogging of the screen and irreversible well damage do not occur. In some of these formations, purging with a peristaltic pump or bladder pump may be the best option for development. In very low hydraulic conductivity formations, it may be necessary to core the formation across the interval to be screened in order to prevent significant damage to the formation which may not be repaired with usual development methods. One possible method for slug testing in very low-K formations is discussed in Section 13.2 (Slug Tests with the Dual Tube Groundwater Profiler).

6.0 Well Parameters Required for Calculation of Hydraulic Conductivity

Accurate documentation of well construction parameters (Fetter 1994, Butler 1997) and site specific geology are necessary so slug test data can be used to calculate the formation hydraulic conductivity. Without this information the slug test data is of little value. Establish a consistent reference mark for measuring all depths (top of casing or other) and use consistent units. Be sure to accurately document the following well construction parameters (Figure 6.1) and site geologic information:

L_e: Effective Screen Length, this will include length of any artificial sand pack extending above the well screen.

L_s: True Screen Length, only the length of screen exposed to the formation.

L_w: Length of the water column in the well (TD – SWL)

R_s: Screen radius

R_B: Radius of the filter pack or borehole over the screened interval

***R_c:** Casing radius (true internal radius of casing where water level changes occur)

r_t: Radius of transducer cable (required for wells one inch or less in diameter)

T_s: Depth the transducer is submerged below the SWL

SWL: Static water level as measured from reference point

TD: Total depth of well/screen as measured from reference point

h: Saturated thickness of the aquifer

H_o: Initial head change at instant slug test started. For the pneumatic slug test system this may be estimated from the stabilized pressure gauge readout in inches.

Aquifer Type: Is the aquifer confined or unconfined? (Review soil cores, well logs, or DP e-logs to make this determination.)

Formation Type: Give a verbal description of the formation being tested (clay, silt, sand, gravel, silty-sand, etc.) Best if this is described from a sample collected from the interval being tested.

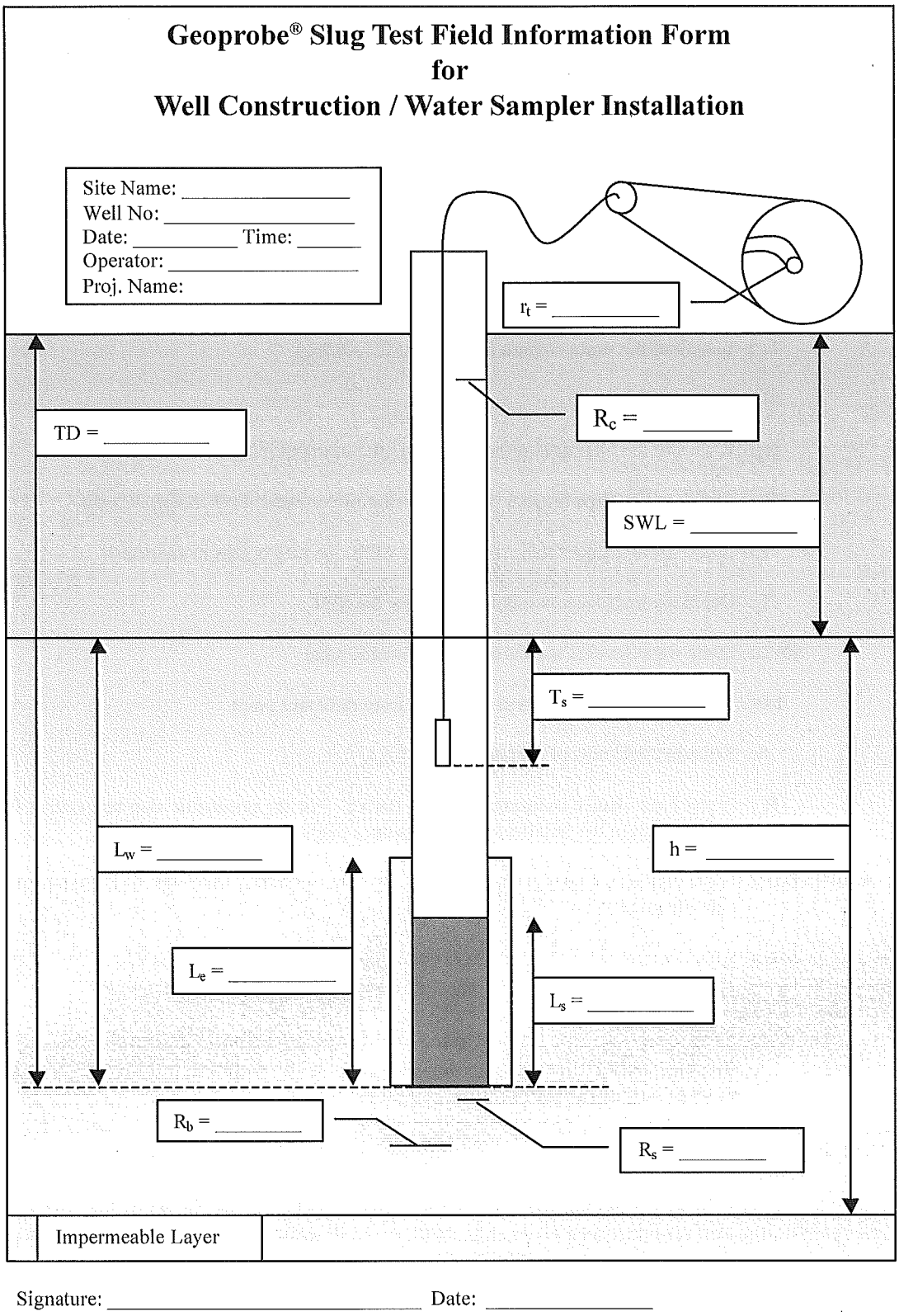
***NOTE:** **Casing radius must stay the same over the interval that the water level changes during the slug test or rate of water level change in the well will not be constant and errors in data will occur and results will be inaccurate.**

6.1 Correction of Casing Radius

For wells or piezometers less than two inches (50 mm) in diameter the radius of the casing should be corrected for the radius of the transducer cable. In these small wells the transducer cable begins to displace a significant proportion of the well bore and as such will cause a measureable error in the calculated K-value if not corrected for in the model equation. The corrected casing radius (Rcc) is calculated as follows:

$$Rcc = (R_c^2 - r_t^2)^{1/2}$$ (after Butler et al., 2002)

Geoprobe® Slug Test Field Information Form
for
Well Construction / Water Sampler Installation



Signature: _____ Date: _____

FIGURE 6.1:
Copy and use this form in the field to record construction and installation parameters for slug testing

7.0 Installation of the Pneumatic Manifold and Set-up for Slug Testing

Once the well or groundwater sampler is installed and developed, you are ready to install the Pneumatic Manifold Assembly (17631) on the casing or probe rods. The pneumatic manifold (Fig. 7.1) includes all connections, valves, and controls required to conduct pneumatic slug tests. The Pneumatic Head Assembly comes with an adapter that allows it to be connected directly to Geoprobe® 1.25-inch probe rods (Fig. 7.2). Other adapters included with the kit allow the pneumatic head to be attached to 1.5-inch and 2.125-inch Geoprobe® probe rods. Adapters for standard size PVC casings (1/2-, 3/4-, 1-, and 2-inch) are also available that allow for slug testing in direct push installed or conventional design monitoring wells or piezometers.

To install the pneumatic head on the SP16 rod string, use an O-ring (AT1250R) or Teflon® tape to seal the head on the probe rods. If probe rods of a diameter other than the 1.25 inches are being used, the appropriate adapter must be installed to attach the pneumatic head to the rod string. If the pneumatic head is being installed on PVC casing, use the correct adapter for the nominal casing size of the well (see Section 3.0).

7.1 Transducer

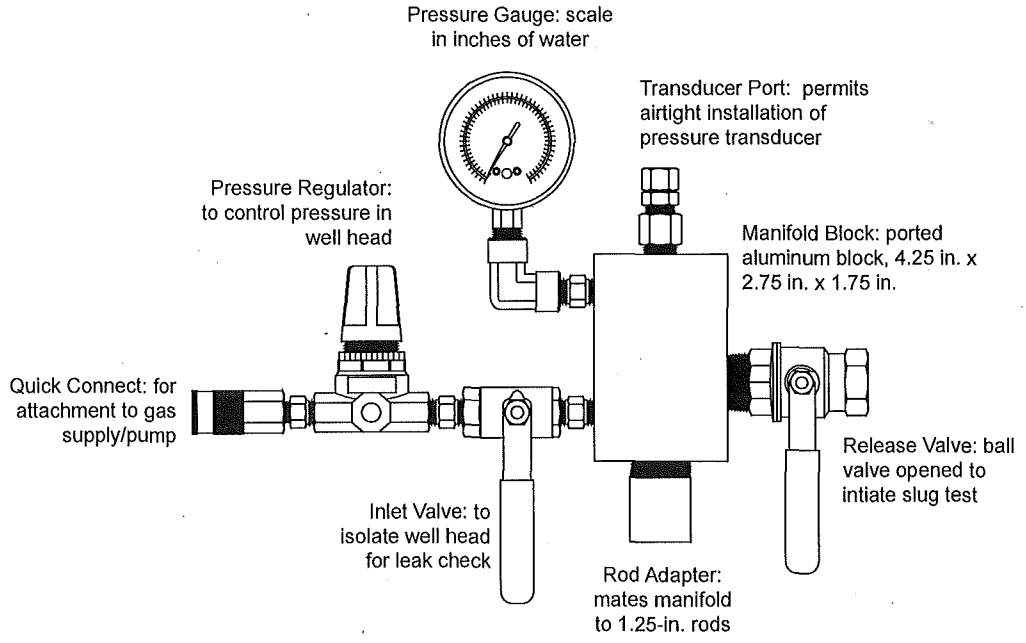


FIGURE 7.1:
Pneumatic Manifold Assembly (17631)

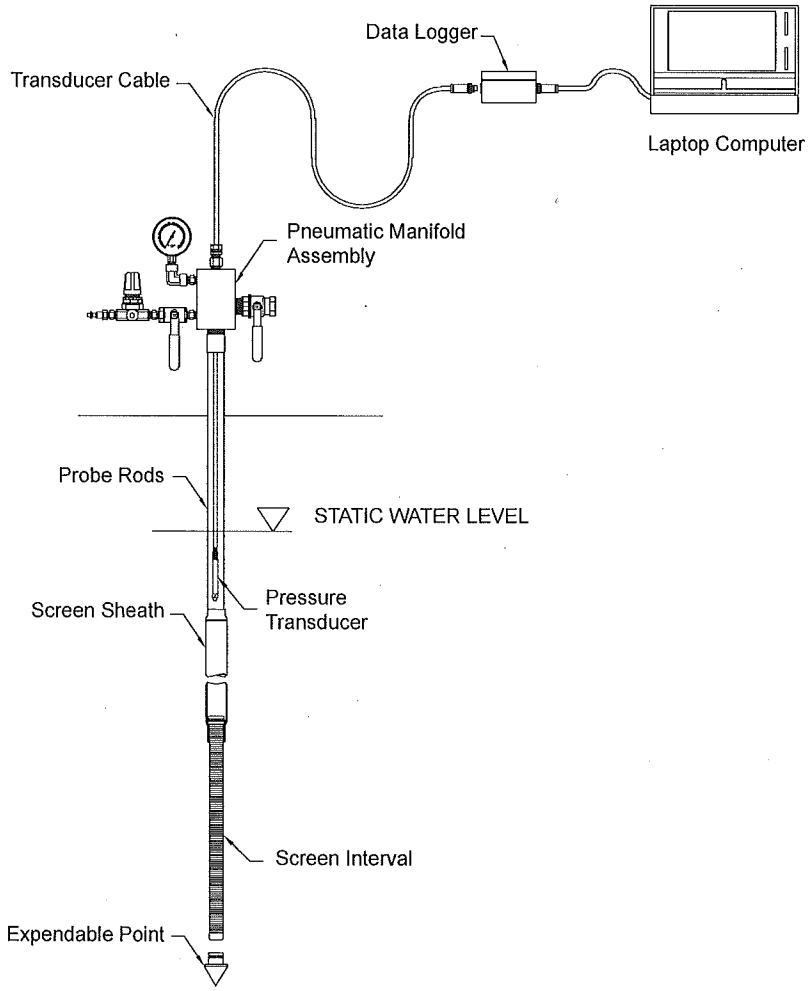


FIGURE 7.2:
Slug test setup for SP16 Groundwater Sampler

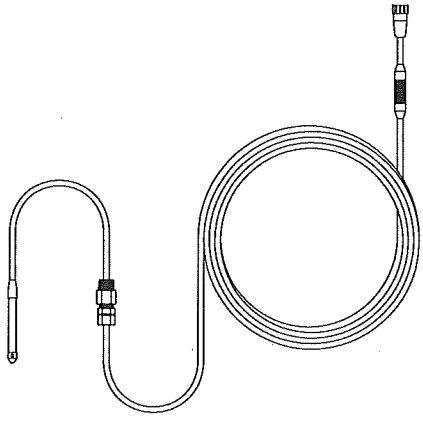


FIGURE 7.3:
Transducer assembly (19345)

The pressure transducer (Fig. 7.3) supplied with the pneumatic slug test kit is vented. A breathable membrane located near the top end of the cable keeps the transducer at ambient atmospheric pressure.

CAUTION: Keep the breathable membrane clean and do not crimp the transducer cable as this will crimp the vent tube and interfere with accurate transducer response.

The specifications of the transducer supplied with the Pneumatic Slug Test Kit are as shown in Table 7.1.

Specification	English Units	Metric Units
Range	10psi g	689.5 millibars or 6.89×10^4 Pascals
Transducer diameter	0.39 inches	9.91 mm
Transducer length	4 inches	101.6 mm
Supply current	10 V	10 V
Sensitivity (approximate)	100 mV	100mV
Non-linearity & Hysteresis	± 0.1 % baseline	± 0.1 % baseline
Temperature Compensated Range	28.4 to 86° F	-2 to 30° C
Temperature error band	± 0.5%	± 0.5%
Cable length	100 ft	34.8 m
Cable diameter	0.225 inches	5.71 mm

TABLE 7.1:
Pressure transducer specifications.

Once the pneumatic head is in place the transducer assembly is installed. The transducer itself (Fig. 7.4) is inserted through the port on the top of the pneumatic head and lowered into the well. For most slug testing the transducer is set about 18 to 24 inches (45 to 60 cm) below the static water level.

NOTE: Be sure to cool the transducer to the ambient groundwater temperature before zeroing, usually three to five minutes required.

After zeroing the transducer (see Section 9.3), the 1/4-inch NPT x 1/4-inch tube nylon fitting is threaded into the transducer port. Teflon® tape is used to improve the seal on the threads. Wrap Teflon® tape around the transducer cable at the fitting (Fig. 7.5), lower the fitting cap into place, and thread the cap onto the fitting. The cap will compress the Teflon® tape around the transducer cable and provide an airtight seal.

CAUTION: Do not over tighten the nylon cap as the transducer cable or vent tube may be damaged. Finger tighten and snug as needed to obtain an air-tight fit. Well head pressures generally do not exceed 1 to 2 psi (about 10 to 20 inches of water, 25 to 50 cm of water, or 25 to 50 millibars).

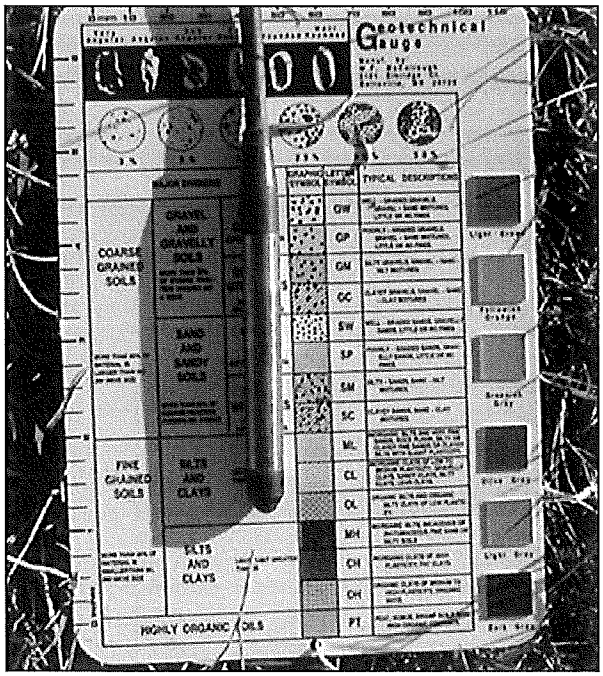


FIGURE 7.4:
The small outside diameter (0.40 inches) of the pressure transducer allows it to be used with Geoprobe® 1.25-inch OD probe rods and 0.5-inch Schedule 80 PVC casing.

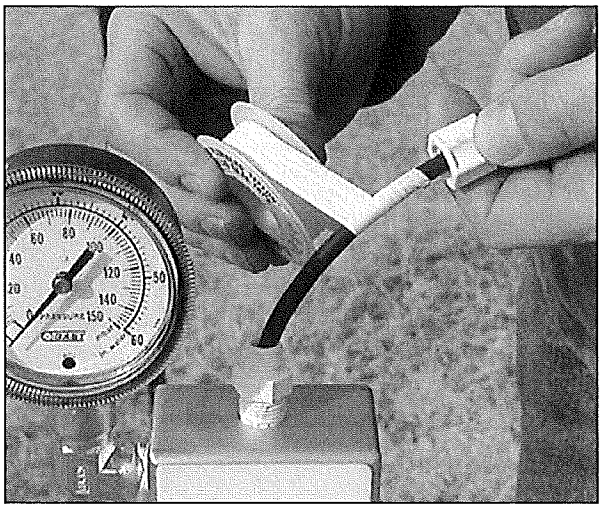


FIGURE 7.5:
Wrap Teflon® tape around the transducer cable to promote an airtight seal at the nylon cap.

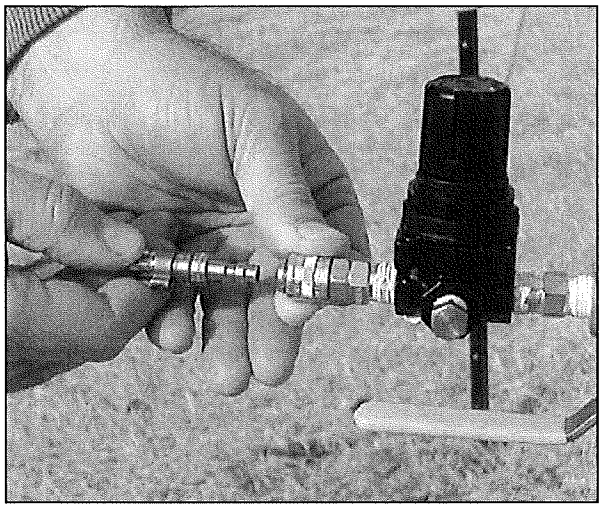


FIGURE 7.6:
Attach the foot pump to the pneumatic head using the low-pressure hose with quick connects.

7.2 Connecting Pump and Air Hose

The foot pump assembly (17643) included with the pneumatic slug test kit is used to supply air to pressurize the well head. Connect the foot pump to the inlet side of the pneumatic head with the low-pressure hose and quick-connect fittings (Figure 7.6). Typically 30 to 40 psi of air pressure is maintained in the supply hose during slug testing.

CAUTION: The pressure rating of the hose is low (45 psi) do not over-inflate and burst the hose.

You are now ready to connect the transducer to the data logger and the data logger to the laptop computer.

8.0 Data Logger

A single channel data logger (GW1610) and all necessary cables are supplied with the pneumatic slug test kit. Specifications of the data logger are shown in Table 8.1.

Specification	English Units	Metric Units
Size (L x W x H)	7.4 x 4.7 x 2.25 inches	188 x 120.2 x 56.8 mm
Weight	1.49 lbs	0.675 kg
Sensor Port	Twist-on, 8 pin connector, male	
Computer Port	Serial, female RS232 (DB-9)	
Supply Voltage	12 V	
Output Voltage (to transducer)	10 V	
Sampling Frequency	120 Hz	
Available Output Frequencies	1, 2, 10 and 38 Hz	

TABLE 8.1:
Data Logger specifications

The female 9-pin connector on the transducer is connected to the male 9-pin connector on the Data Logger. Next the Data/Power Cable (19174, Fig. 8.1) is connected to the serial port on the data logger and then the serial port on the laptop computer. The small in-line power transformer (Fig. 8.1) is connected to standard line current (115VAC). Line current may be obtained from a generator, small inverter (SC152) plugged into vehicle lighter socket, or conventional wall outlet if available. The same power supply will also operate the laptop computer if needed.

The slug test system is set up and you are now ready to initialize the software and begin data collection.

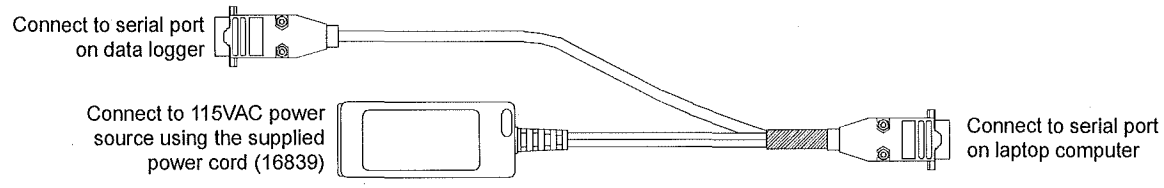


FIGURE 8.1:
Data/Power Cable (19174)

9.0 Slug Test Data Acquisition Software Version 1.00

Geoprobe Systems® has designed a simple, user friendly software system that allows you to acquire and file the transducer data on a lap top computer. The data files are stored in ASCII format for easy export to spreadsheet and data analysis programs. This is solely a data acquisition software system and does not provide data modeling or K-calculation options. The minimum computer requirements for installation and operation of the Data Acquisition Software are:

CPU:	Pentium 90 MHz
RAM:	16 megabytes minimum
Available Hard Drive Space:	12 megabytes minimum
Operating Software:	Windows 95, 98, 2000, NT, ME

9.1 Installation

The Data Acquisition Software comes on a compact disc (CD) with the Pneumatic Slug Test Kit, or can be purchased separately. Simply insert the CD into your computer CD drive, and from the "START" menu in Windows, select the "RUN" option to run the setup program on the CD. Follow the simple on-screen instructions to enter your personal identification information. The software installation should take less than three minutes. The installation will automatically place an icon on your Windows Screen labeled "SLUG TEST". Simply double-click on the icon to start the software. The following information provides a brief summary of the software operation for acquisition of slug test data.

9.2 Selecting Options

After initializing the software by double-clicking on the windows icon, the first window you see will let you select between using either English units or Metric units on the data display (Fig. 9.1). If English units are selected, the water level (head) will be displayed on screen in feet (ft). If Metric units are selected, the head will be displayed in millimeters (mm) of water. Simply click on the desired option.

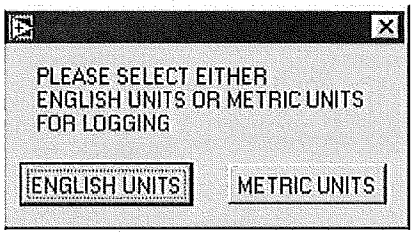
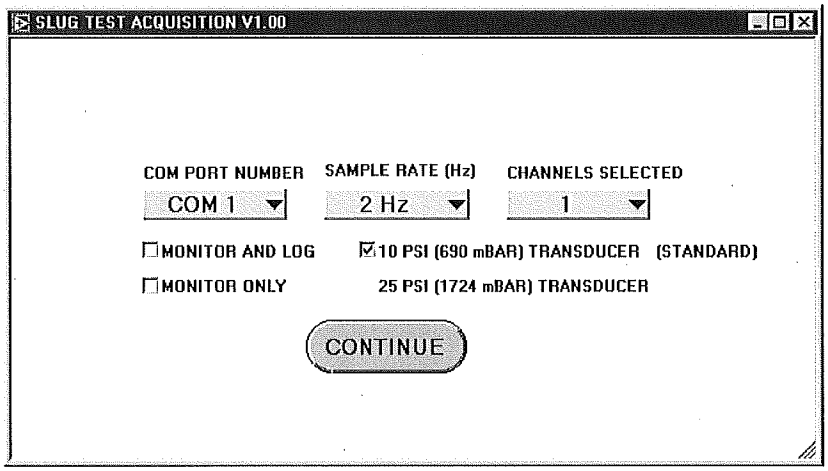
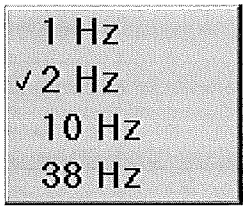


FIGURE 9.1:
Units Selection Window (click on desired option)

The next window (Fig 9.2) allows you to specify several options for system operation and data acquisition. Drop-down menus allow you to choose either Com Port 1 or 2 for your computer connection to the data logger, as well as the sampling rate or frequency in Hertz (Hz).

The sample rate determines the number of sample points that will be plotted on screen and saved in your file each second. The default rate is 2 Hz. You may select either 1, 2, 10, or 38 Hz from the drop-down menu.





Example drop down menu for selection of sampling frequency.

FIGURE 9.2:
Test Options Window (select options using pull down menus and check boxes)

In formations that are very slow recovering from a slug test (30 minutes or more), a sampling frequency of 1Hz is sufficient. If you are slug testing a very high hydraulic conductivity formation which recovers from a slug test in less than 10 to 20 seconds, you may wish to choose 10Hz or even 38Hz. When you select a higher sampling rate, the baseline will appear more noisy because less averaging is conducted to obtain each data point.

A 10 psi transducer comes with the kit and is the default value for this option.

You may choose to MONITOR ONLY or to MONITOR AND LOG from this screen. The MONITOR ONLY option will allow you to monitor the readout from the transducer, but you can not save the data to a file. The MONITOR AND LOG option will allow you to initially monitor the transducer readout and then begin saving the data to a file when desired.

If you select the MONITOR ONLY option, you will go directly to the MONITOR ONLY MODE screen when you click on the CONTINUE button (Fig 9.3). If you select the MONITOR AND LOG option you will proceed to the SITE INFORMATION screen when you click on the CONTINUE button (Fig. 9.4).

The MONITOR ONLY MODE may be used when first inserting the transducer into the well or sampler to verify that the transducer has cooled to ambient temperature (refer to notes on Figure 9.3). Gently oscillating the transducer up-and-down in the water will also dislodge any air bubbles from the transducer opening (Fig. 9.3).

The SITE INFORMATION window (Fig. 9.4) lets you enter a file name (6 characters) and transducer serial number. Tab between data entry boxes and click on CONTINUE to proceed to the next window.

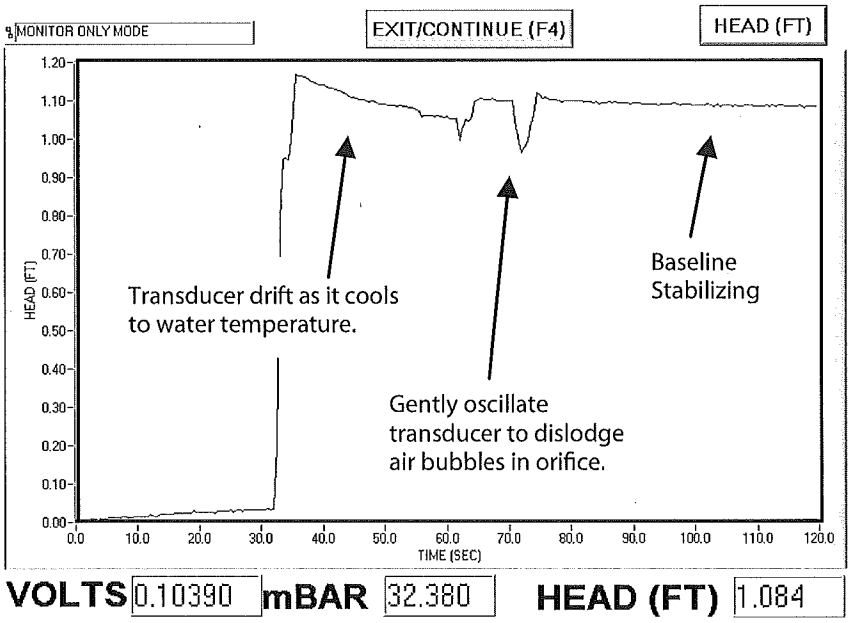


FIGURE 9.3:
Screen output when running in MONITOR ONLY MODE

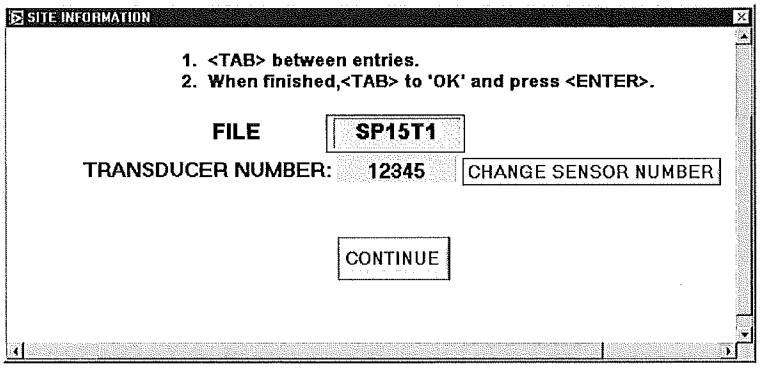


FIGURE 9.4:
Enter site information after selecting MONITOR AND LOG (Fig. 9.2)

9.3 Zero Transducer

Once you click on the CONTINUE button you will proceed to the TRANSDUCER ZERO window (Fig. 9.5). When the software is first initialized, you must zero the transducer before acquiring slug test data. First place the transducer in the groundwater to equilibrate with the ambient temperature. Then the transducer should be removed from the water to zero it at atmospheric pressure. If you cycle back through the software to run additional slug tests in separate files, you may choose to BYPASS the zero option if desired. Simply click on the BYPASS button to do so. It is recommend that the operator zero the transducer initially and then bypass this function if desired as additional files and slug tests are conducted.

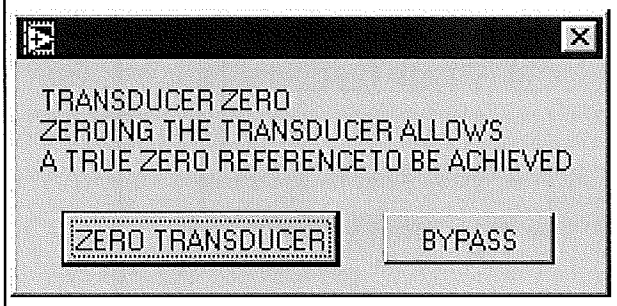


FIGURE 9.5
Select or bypass transducer zero function

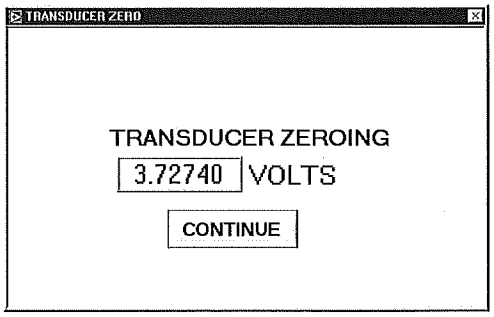


FIGURE 9.6
Transducer Zero Window

When the ZERO TRANSDUCER function is selected, the real-time transducer voltage is displayed in the VOLTS box (Fig. 9.6). Click on the CONTINUE button when the voltage has stabilized and you are ready to proceed with logging the slug test. If you selected a higher sampling rate (e.g. 10 or 38Hz), the voltage readout will be more variable.

9.4 Data Acquisition and Viewing Graph

After clicking on the CONTINUE button in the TRANSDUCER ZERO window, you will enter the Collect-and-Graph window (Figure 9.7). If the transducer is still above the water level, you will observe a very magnified baseline as the data points are plotted and scroll across the screen. Once the transducer is submerged below the water level several inches or more, what at first appeared to be a noisy baseline is seen to be a relatively quiet line at normal operating head/pressure values (Figure 9.8).

Once the transducer is stable and you are ready to start saving data, you may click on the BEGIN SAVING button or press the F1 key on the keyboard. When this is done the word **SAVING** is displayed near the top-center of the window (Figure 9.8). The filename with directory path is displayed in the top-left side of the window.

A mouse click on the HEAD button at the top-right of the window allows you to toggle between a Y-axis in feet of head (or mm of head in metric units) and the transducer voltage readout if desired. Once you have completed your slug test(s), a mouse click on the STOP button or pressing the F5 key will stop data collection and freeze the screen. This will enable you to review the collected data and briefly evaluate your results before continuing with further work.

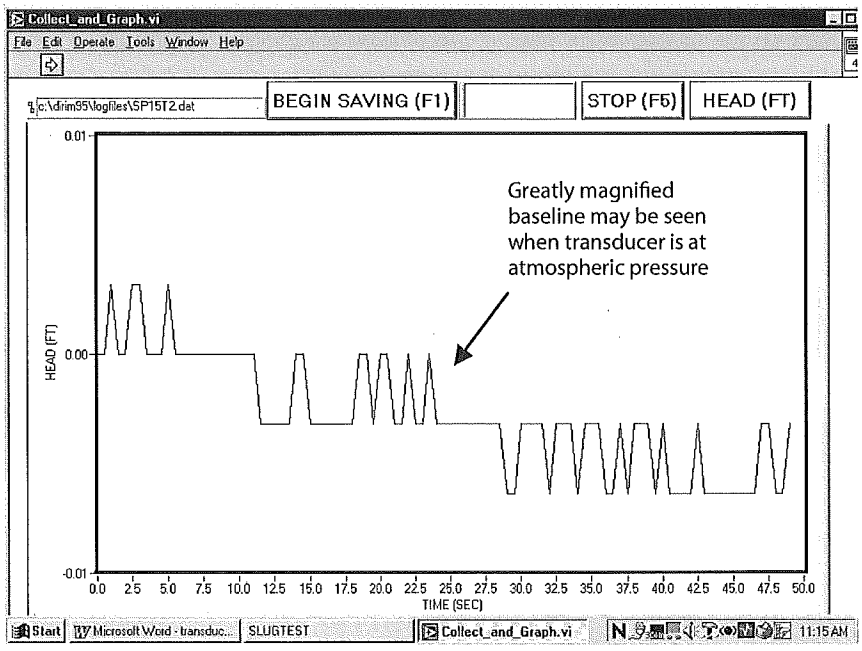


Figure 9.7

Real-time transducer data is plotted on the Collect-and-Graph window. The baseline will be greatly magnified and appear noisy when the transducer is above the water level.

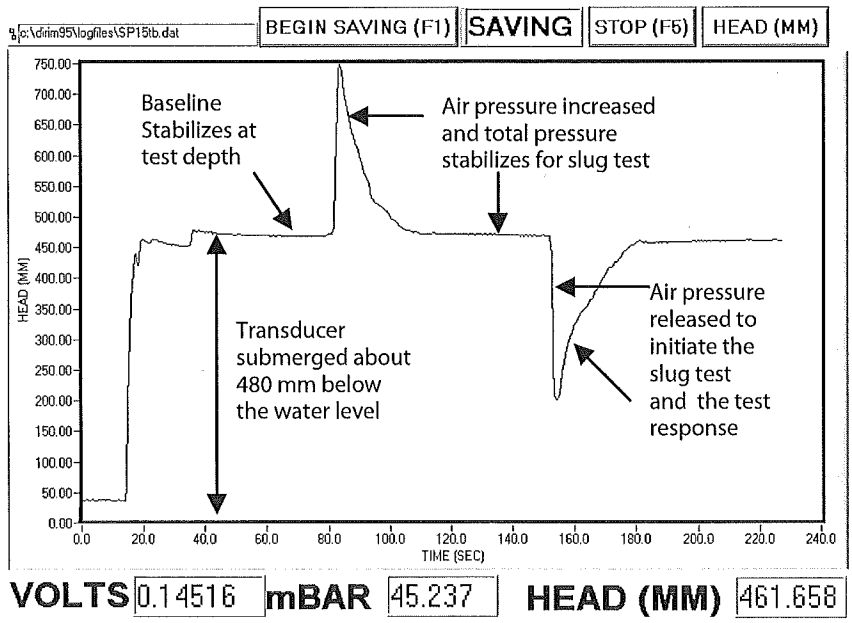


Figure 9.8

Simulated transducer data during a slug test (well head pressurized, stabilization, and release of pressure to initiate the slug test response)

9.5 Exiting Program

When the STOP button (or F5 key) is pressed on the Collect-and-Graph window, the QUIT/CONTINUE button appears in the top-center of the screen. Data collection is halted and the transducer log may now be reviewed without affecting the data file.

Click the QUIT/CONTINUE button when you are finished reviewing the slug test response and are ready to either exit the program or continue with additional slug testing. This brings up the QUIT window shown in Figure 9.9. Click on the YES button if you are ready to quit and exit the program. Click the NO button to go back to the Options window (Figure 9.2) to set up for additional slug test data acquisition.

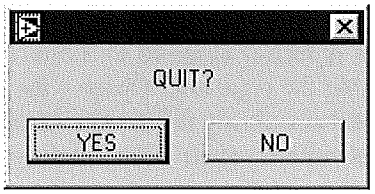


FIGURE 9.9:
Select YES to exit the program or NO to continue data aquisition with a new file

9.6 File Storage and Data Format

Two files are generated each time you enter a file name and click on the BEGIN SAVE button or press the F1 key while in the Collect-and-Graph window (Figure 9.7). The first file is where the time and transducer data are saved and it is denoted as a ".dat" file in the directory. The second file is an information file and it is denoted as a ".inf" file in the directory. Both file types are saved in the C:\dirim95\logfiles*.* directory on the computer hard drive. The data in the *.dat file is saved in columns in the following order:

Time (seconds) Transducer (voltage) Head (feet) Head (millimeters) Pressure (millibars)

The *.inf file saves some basic date and time started/ended information. It also specifies the column format of the data file for future reference if needed. This file may be edited to add information such as site name, location, operator, and client information as needed to meet project specifications. An example *.inf file is shown in Figure 9.10 below.

```
SITE INFORMATION -- SLUG TEST REV. 1.0


START DATE: 11/30/2001
START TIME: 4:03 PM
DATA FILE NAME: C:\DIRIM95\LOGFILES\SP15TB.DAT
DATA FILE FORMAT:
TIME(S)  TRANSDUCER(V)  HEAD(FT)  HEAD(MM)  PRESSURE(MILLIBAR)
UNITS:  METRIC
TRANSDUCER NUMBER:  1388621   10 PSI (690 MILLIBAR)
END DATE:  11/30/2001
END TIME:  4:08 PM
```

FIGURE 9.10:
Format of the *.inf file saved with each data (*.dat) file

10.0 Running a Pneumatic Slug Test

The following summary is a step by step outline of the procedure required to run a pneumatic slug test.

1. Close inlet and release valves and close the pressure regulator on the pneumatic manifold assembly (Fig. 10.1).

   NOTE: Adjust zero setting on the pressure gauge if needed.

2. Operate foot pump to pressurize supply hose to ~ 30 to 40psi

3. Open inlet valve on pneumatic head (Fig. 10.1)

4. Slowly open the pressure regulator (Fig. 10.1). From the fully closed position it takes about five revolutions to begin opening the regulator. Observe the pressure gauge on the pneumatic head. Let the pressure in the well head rise slowly to a few inches above the level desired for testing (e.g. if you want to initiate the slug test with H_o of 10 inches let the gauge rise to about 12 inches).

5. Quickly close the inlet valve and allow the pressure observed from the transducer in the well head (as shown on the computer screen - Fig. 9.8) to return to equilibrium and stabilize. Record the stabilized gauge pressure. Observing the Collect-and-Graph window of the Slug Test software, the readings should return to the levels noted before pressurization was started (Fig. 9.8). For very permeable and porous formations, return to equilibrium (baseline) happens quickly (Fig. 10.2). For moderate to lower-K formations (silty-clayey sands) this may take several minutes (Fig. 10.3). For very low-K formations (silts/clays) it could take hours for the water level to return to equilibrium. In this situation, a mechanical slug test should be conducted to save considerable time.

6. Leak test the fittings on the pneumatic head and connection to the rods with the leak test fluid (12356) or equivalent. Tighten fittings if necessary and retest. If a leak appears to be downhole, tighten the probe rods with a wrench. If the leak persists and is slow relative to well recovery rate, the regulator may be used to stabilize the pressure in the well head. It is preferable to locate and correct the slow leak if possible before continuing with the slug test.

   NOTE: For slow leaks the on-screen transducer readout will appear stable because the water level rises as quickly as the air escapes, keeping the readout constant. You must monitor the pressure gauge on the pneumatic head to determine if a slow air pressure drop is occurring.

7. Once the transducer readout is back to equilibrium and stable, you are ready to initiate the slug test. To initiate the slug test, open the release valve as quickly as possible (Fig. 10.1). You should hear a "swoosh" of air venting from the valve if you listen closely.

8. Watch the Collect-and-Graph screen on the computer. You should observe a very rapid drop in the transducer readout (head) as the air is instantaneously released (Figs. 10.2 and 10.3). Then the rise/ recovery of the water level to the pre-test equilibrium level (baseline) will occur. Once the water level has returned to the pretest equilibrium level and is stable, the slug test is complete.

9. It is strongly recommended that you run at least three slug tests of different initial head values (H_o) to use for verification of appropriate well performance and development. If the well or sampler is responding properly, the normalized data from all three tests should overlay each other in the normalized data plot. If there is significant deviation between the repeat tests, additional development of the well or sampler may be necessary or other actions may be required to obtain valid data.

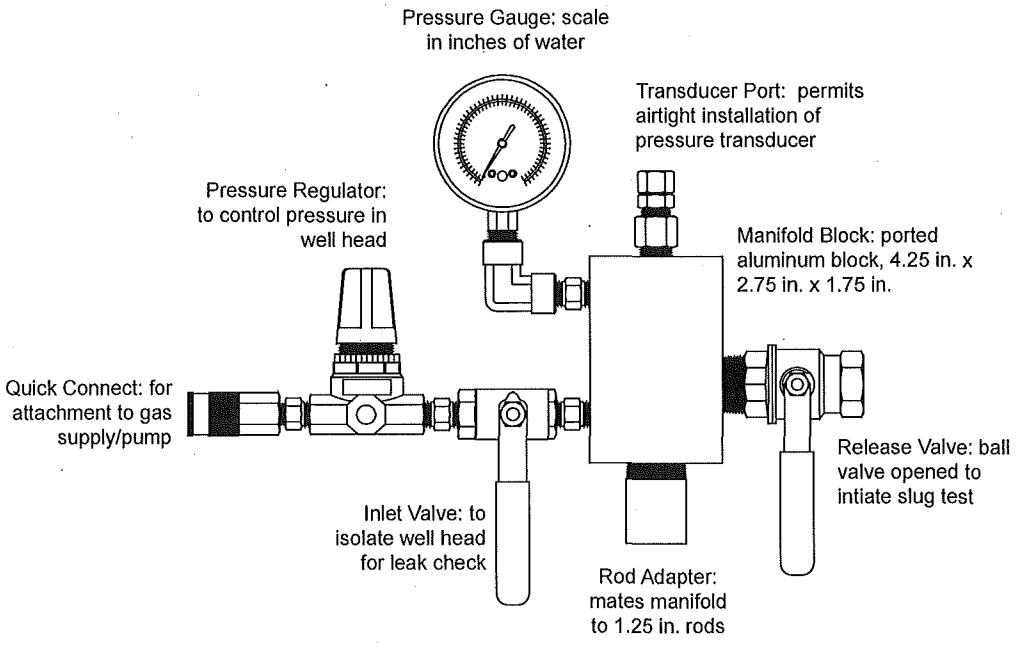


FIGURE 10.1:
Pneumatic Manifold Assembly (17631)

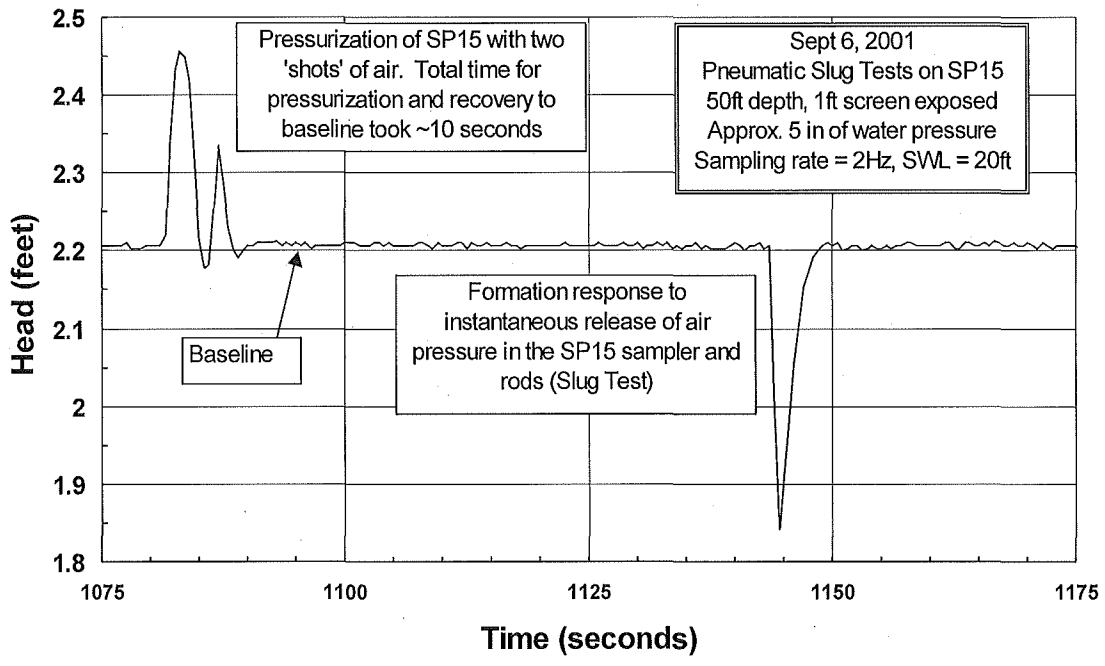


FIGURE 10.2:
SP16 slug test in a high-K formation

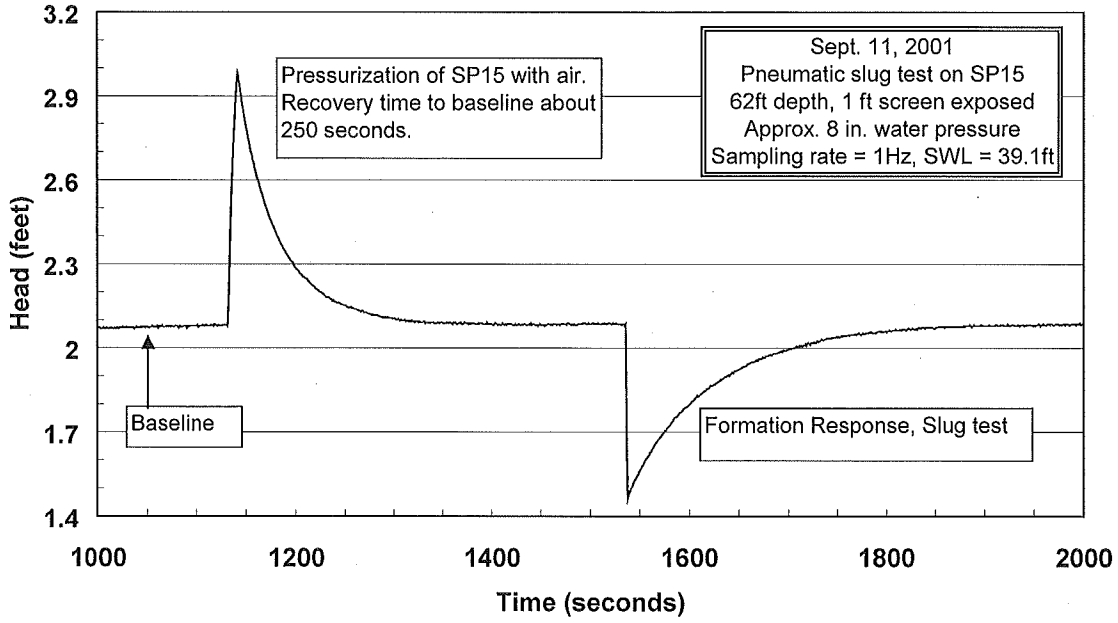


FIGURE 10.3:
SP16 slug test in a moderate-K formation

11.0 What the Data Looks Like

Response to slug tests takes on two general forms. The observed responses are comparable to a spring that has been stretched and released. If a large weight is attached to the end of the spring it slowly slides back to the equilibrium point and stops. If a small weight is attached to the spring it may oscillate back-and-forth around the equilibrium point a few times before coming to rest. This first type of behavior is called "overdamped" response and the second is called "underdamped" or oscillatory response (the water level oscillates up-and-down in the well in response to the slug test) (Butler 1997).

Overdamped response (Figs. 10.2 and 10.3) is what is most commonly observed during slug testing and is characteristic of formations that range from fine-grained materials (silts and clays) to sandy materials. The underdamped response (Figure 11.1) is less commonly observed and is characteristic of high hydraulic conductivity formations such as coarse sands and gravels. There is also a special case of critically damped response that lies between the underdamped and overdamped behaviors (Butler 1997, ASTM D 5581).

The portion of the slug test curve and data used in the modeling process to determine the hydraulic conductivity is the response and recovery that begins immediately after the pressure is released (pneumatic slug test initiated) and ends when the water level has returned to its pretest equilibrium pressure (see Figures 10.3, 10.4, and 11.1).

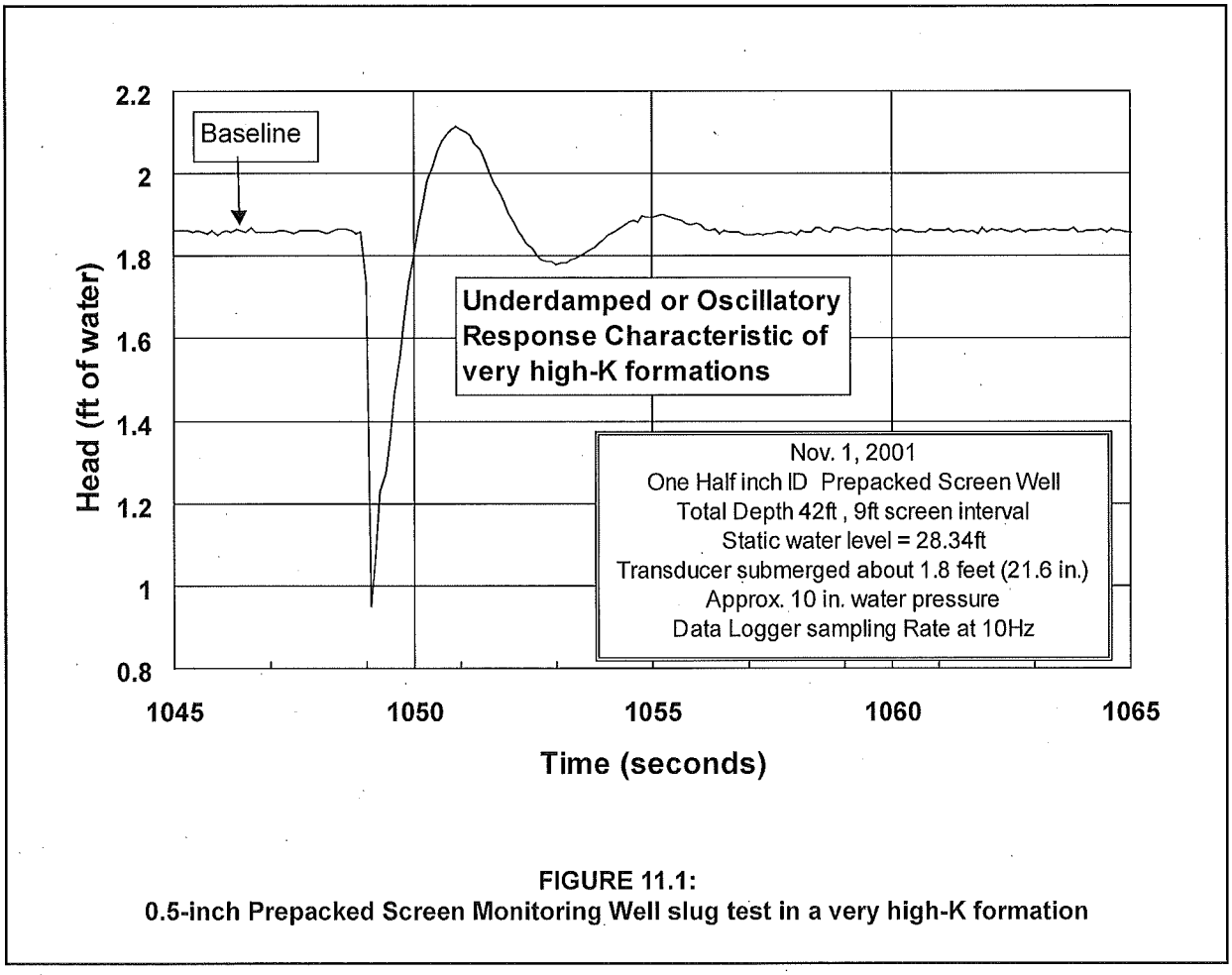


FIGURE 11.1:
0.5-inch Prepacked Screen Monitoring Well slug test in a very high-K formation

12.0 Data Analysis Methods

The same data analysis methods that are applied to larger-diameter wells are used for the smaller-diameter direct push tools and wells. These conventional analysis methods provide accurate results for the small-diameter tools (Butler et al. 2002) under almost all conditions. However, when tools or wells less than one inch inside diameter are used for slug testing in very high hydraulic conductivity formations (K > 250ft/day, >$8x10^{-2}$cm/sec), a simple linear correction is applied to determine the K-value (Butler, 2002). Probably just a fraction of the formations to be tested will have K-values exceeding this range.

Two factors must be considered in the selection of the appropriate data analysis method. First, is the formation being tested an unconfined aquifer or a confined aquifer? Second, is the aquifer response overdamped behavior or underdamped and oscillatory in nature? The following is a very brief selection of possible modeling methods that may be appropriate. An authoritative review of slug test data analysis methods is provided by Butler (1997).

Overdamped:

- Unconfined = Bouwer & Rice (1976) method, see also Bouwer (1989)
  ASTM D 5912
- Confined = Hvorslev (1951) method
  ASTM D 4104

Underdamped:

- Unconfined = Springer and Gelhar (1991) also know as the Hi-K Bouwer & Rice Method (Butler 1997, Butler and Garnett 2001)
- Confined = van der Kamp (1976) method also known as the Hi-K Hvorslev Method (Butler and Garnett 2001)
  ASTM D 5785

Critically Damped:

- Confined = Kipp (1985) method
  ASTM D 5881

Additional information on slug testing field procedures and data analysis methods is available in any of several ASTM Standard Guides and Practices (ASTM D 4043, D 4044, D 4104, D 5785, D 5881, D 5912). The Standard Guide D 4043 includes a decision tree for selection of the appropriate test and data analysis methods while Standard Method D 4044 provides information on field procedures for slug testing.

The selection and application of the appropriate data analysis method(s) is beyond the scope of this SOP for field techiniques. Many literature resources (Butler 1997, Fetter 1994, Freeze and Cherry 1979) and software resources (AQTESOLV, AquiferTest, etc.) are available for guidance in selection and application of the appropriate model to determine the formation hydraulic conductivity, transmissivity, and other formation parameters from the slug test data.

13.0 Pneumatic Slug Testing in Groundwater Profiling Tools and Monitoring Wells

The Pneumatic Slug Test kit can be installed on many of the available Geoprobe® groundwater sampling tools to conduct slug testing. The kit may also be used on PVC casing ranging in size from nominal 1/2-inch to 2-inch. The diagrams below indicate how the pneumatic slug test kit is used with several of the groundwater sampling tools and PVC well casings. Reference the appropriate standard operating procedures or instruction bulletins for details of sampler installation or well construction.

13.1 Slug Tests with the Groundwater Profiler (GW1600)

The Groundwater Profiler (GW1600) may be telescoped through Geoprobe® 2.125-inch rods when fine-grained materials overlay the sandy formation to be sampled and slug tested (Fig. 13.1). The profiler is available with either a 6.0-inch or 12.0-inch (length) stainless steel screen and may be advanced incrementally for sampling and slug testing at multiple depths in one borehole.

The groundwater profiler is advanced with Geoprobe® 1.25-inch rods so the pneumatic head will readily connect to the sampling system for slug testing. Because the 1.25-inch drive rods have an ID of only 0.625 inches, the calculated hydraulic conductivity value must be corrected if the test result indicates a K-value ≥ 200 ft/day ($7x10^{-2}$ cm/sec) (Butler, 2002). Clogging of the exposed screen as it is advanced to multiple depths can be problematic, even in relatively clean sands. Development for slug testing becomes critical.

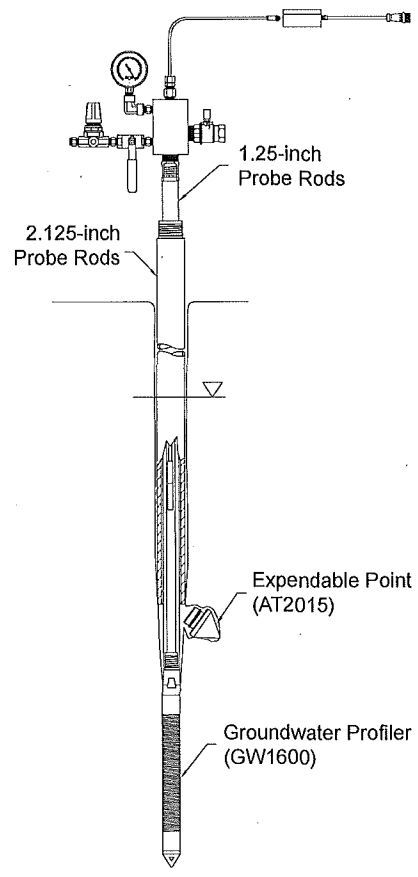


FIGURE 13.1:
Pneumatic slug testing with the Groundwater Profiler (GW1600)

13.2 Slug Tests with the Dual Tube Groundwater Profiler (GW2100)

The Dual Tube (DT22) Groundwater Profiler system (GW2100) may be used to conduct soil sampling, groundwater sampling, and slug testing (Fig. 13.2) at multiple depths in one probe advancement (Instructional Bulletin No. 19275). The 2.125-inch OD by 1.5-inch ID rods are the outer casing for this system and may be used to conduct slug tests in formations of very high hydraulic conductivity where the K-values exceed 500 ft/day ($1.76x10^{-1}$ cm/sec) (McCall et al. 2002). The pneumatic head adapter (PN 19165) for the 2.125-inch rods, which has a larger diameter release valve, is required for slug testing in very high-K formations.

The Dual Tube (DT22) Groundwater Profiler system (GW2100) may also be used to conduct pneumatic or mechanical slug tests in moderate to low hydraulic conductivity formations (Figure 13.3). When slug testing in cohesive materials with significant proportions of clay, a small coring device should be used to core the formation ahead of the outer rods and cutting shoe. This minimizes damage and compression of the formation allowing for accurate determination of K-values in these sensitive materials. After the pre-core is removed, the DT22 Profiler screen is installed in the core hole. An adapter for nominal 1/2-inch Schedule 80 PVC (PN 17561) is assembled onto 1/2-inch PVC casing and installed in the top of the DT22 profiler screen (Figure 13.3). This reduces the casing ID and significantly increases the recovery rate of the slug test.

In moderate-K formations (e.g. < 10^{-3} cm/sec to > 10^{-6} cm/sec), a PVC casing adapter and bushing (PN 17558 and 17559) may be used to attach the pneumatic head to the PVC casing so pneumatic slug tests may be conducted. In low- to very low-K formations (e.g. less than 10^{-5} cm/sec) polyethylene tubing (TB25L) may be placed over the transducer cable to further reduce the effective casing radius and speed up the slug test recovery rate. In this configuration the transducer and poly-shrouded cable become a mechanical slug-transducer. Mechanical slug testing with this configuration eliminates the need to wait for re-equilibration of the water level needed when pneumatic slug testing is conducted. In these low-K formations this re-equilibration of the water level needed for pneumatic slug testing will more than double the length of the slug test, which may be several hours in length. So use of the mechanical slug-transducer configuration can save over 50% of the time required for slug testing in these low-K formations.

13.3 Slug Tests in Monitoring Wells

Small-diameter direct push installed monitoring wells (Fig. 13.4) and conventional 2.0-inch PVC wells may also be used for slug testing with the Pneumatic Slug Test kit. Adapters and bushings to attach the pneumatic head assembly to nominal 1/2-inch up to 2-inch PVC casing are available (see Section 3.0). Follow the manufacturer's standard operating procedure (Geoprobe® Technical Bulletins 96-2000, 99-2500) or appropriate ASTM Standard Guide (D 6724) or Standard Practices (D 6725 or D 5092) for well installation and construction. Remember that appropriate development (ASTM D 5521) is necessary to obtain representative slug test results and K-values in small- or large-diameter wells. Once the well has been installed and developed, the PVC casing adapter (and bushing if required) is used to attach the pneumatic head assembly to the well casing (Fig. 13.4). Slug testing then follows the same procedures as described in the above sections. Remember that the pneumatic method can not be used in wells where the static water level is within the screen interval or will be lowered into the screen interval during the pressurization of the well head. Under these conditions mechanical slug test methods must be applied.

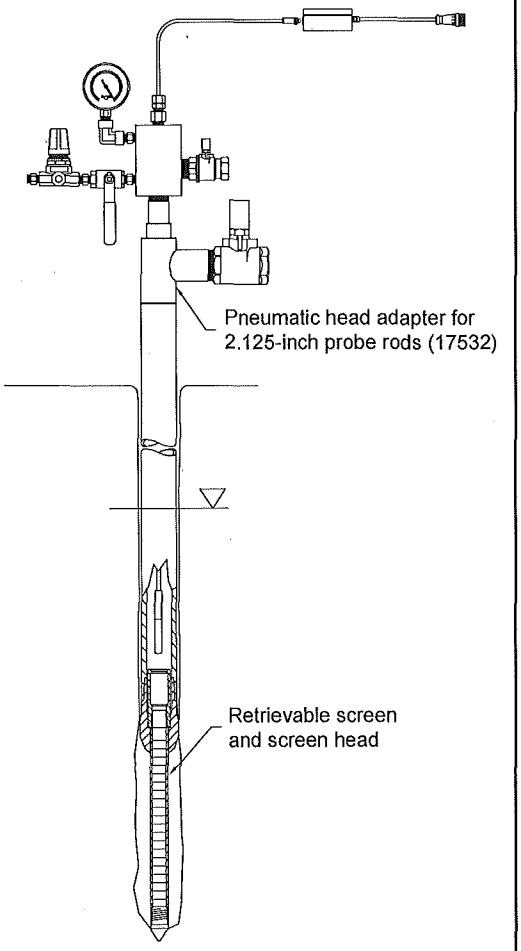


FIGURE 13.2:
Pneumatic slug testing with the Dual Tube Groundwater Profiler (GW2100)

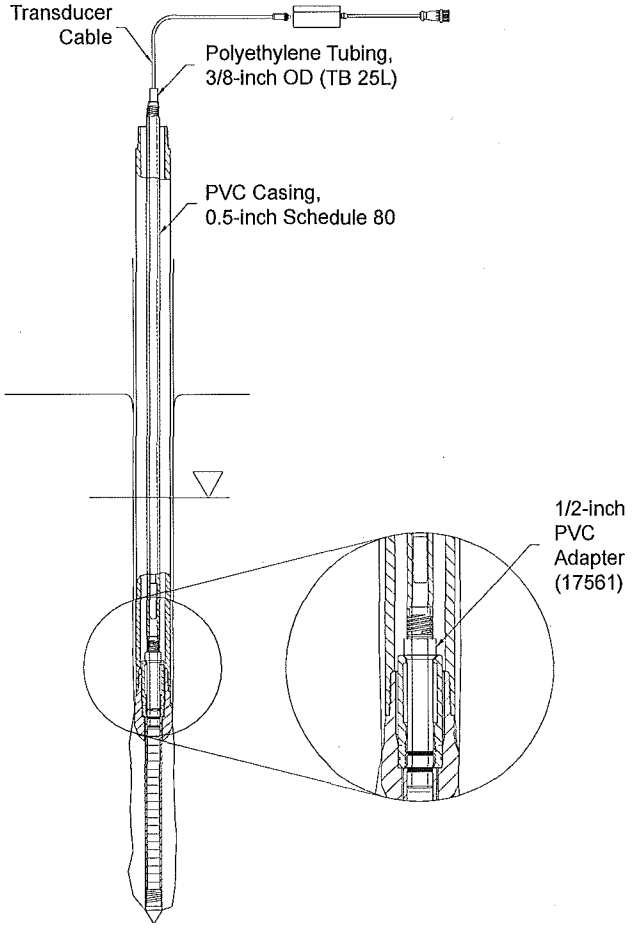


FIGURE 13.3:
Mechanical slug testing with the Dual Tube Groundwater Profiler (GW2100)

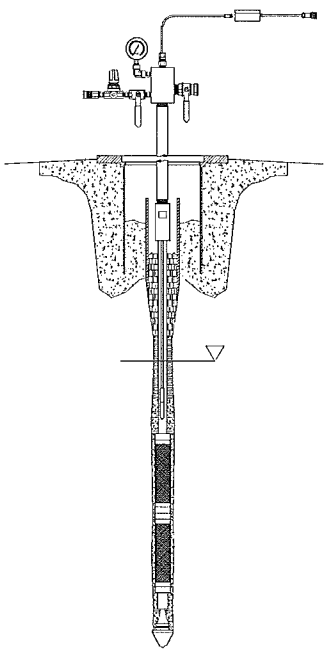


FIGURE 13.4:
Pneumatic slug testing with a prepacked screen monitoring well

14.0 Hints and Tips

Development!! Do it or just don't run the slug test.

Do not over-pressurize the well head as this can result in a damaged transducer and/or air in the formation which will result in inaccurate results.

Run multiple tests with varying initial head (H_o) to verify aquifer response and development meet model requirements (e.g. When normalized data for tests with different H_o are normalized and plotted on semi-log paper the results over lay one another).

Cool transducer to ambient groundwater temperature before zeroing and beginning slug test to prevent baseline drift.

After cooling the transducer in the groundwater, raise it just above the water level to zero at atmospheric pressure.

Watch the air pressure gauge on the pneumatic head to detect slow leaks. The water level will rise in the well as quickly as air escapes from a slow leak. The transducer readout on screen may appear stable when in fact the water level and air pressure in the well are both changing to maintain equilibrium in response to the slow leak.

Use smaller initial head values (H_o) especially in very high-K formations to be sure to get accurate results. In higher-K formations, using a large initial head will result in erroneous well response and incorrect results. Do not use initial head of over 20 inches (~50cm) under most conditions (less is preferable in high-K formations).

For smaller-diameter wells (1-inch or less), be sure to correct the casing radius (r_c) for the transducer cable radius before calculating the hydraulic conductivity. If this correction is not made, errors in the calculated K-value of 10% or greater may occur.

15.0 References

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Appendix I: Field Calibration of the Transducer

The transducer, data logger, and software system are designed with a built-in calibration that will calculate a head value in feet and millimeters, and a pressure value in millibars. This internal calibration factor is not exact, but should provide head and pressure values within 10% of the true values. Instrument drift over time is a normal phenomenon and results in these variations. While the precision of the data will be very good over time, the accuracy may shift from the true value due to this instrument drift.

Since the data is normalized (H_t/H_o) for use in modeling and calculation of the hydraulic conductivity, it is not mandatory that the reported head or pressure values be exactly accurate. Normalizing the data removes the units so that modeling is done with simple ratios. It would be possible to use simply the transducer voltage readout and normalize these by the H_o voltage value to obtain the same ratios as calculated with normalized head values originally stated in feet or inches. However, if for your project it is necessary to have accurate as well as precise head data, then a simple field calibration may be conducted prior to running slug tests to obtain an accurate calibration line.

Take a clear plastic four-foot long Macro-Core® soil sample liner or DT22 soil sample liner, cap the bottom, and tape it sealed. Stand the tube upright and fill it to over-running with clean water. (Refer to Sections 7.0, 8.0, and 9.0 for more information on the following actions.) Connect the transducer to the data logger and the data logger to the power supply and laptop computer. Turn on the computer and initialize the slug test software. Let the transducer equilibrate at ambient working temperature. Begin saving the *.dat file in the software (e.g. filename CAL730). With the transducer stabilized at atmospheric pressure, record data for at least one minute. Next, insert the transducer exactly 6 inches (152 mm) below the water level in the water-filled liner. Again take at least one minute of data at this water level. Lower the transducer to depths of 12, 18, 24, 30, and 36 inches (305, 457, 610, 762, and 914 mm) in the water column and leave it at each depth for a minimum of one minute after it has stabilized. Once the information for each depth interval is obtained exit from the file.

This data file may be imported into a spreadsheet or scientific software system for plotting and calculation of the calibration equation. An example of a portion of the raw data file is shown in Table A-1. Plotting this data (Figure A-1) is recommended for visual review and selection of the time intervals to be used to calculate the average transducer reading at each water depth interval and atmospheric pressure. Table A-2 shows the time interval and transducer voltage readouts for each water depth and atmospheric pressure used to calculate the average voltage for that condition. As an example, for the condition of 12 inches of water the plot (Fig. A-1) indicated that the transducer readout was stable from the 550- to 600-second interval. The average voltage over this interval was calculated by summing the voltage readout at each time increment and then dividing by the number of readings (n) summed:

$$\mathbf{(Sum(V_{550}\ to\ V_{600}))/n} = \text{average voltage for 12 inches of water over the time interval specified.}$$

This same calculation is conducted for each water depth interval monitored for an equal time period so equal weight is given to each depth interval. Then a linear regression analysis is applied to the paired data points (water depth, average voltage) to determine the least squares best fit regression line for calibration of the transducer under ambient conditions (Table A-3). Most spreadsheet and scientific software programs have a function or macro to calculate the regression line parameters and correlation coefficient. Rerunning the field calibration is recommended if the correlation coefficient is less than 0.99. If a correlation coefficient of 0.99 or better can not be obtained, the transducer may need maintenance or possibly replacement if it is considerably of range.

The slope of the regression line and the Y-intercept determined from the least squares fit can now be used to calculate a very accurate head level (see Table A-3) for slug tests run under this calibration. The transducer voltage is multiplied by the slope (m) and the intercept value is added to the result to get the true head reading. If very accurate data is required, calibrations may be conducted daily (or more often) if instrument drift is significant.

Field Calibration of 10psi transducer
Nov. 30, 2001
Wes McCall

Raw Data File:

Time (seconds)	Transducer (volts)	Head (feet)	Head (mm)	Pressure (millibars)
0	0.03545	0.36989	112.7417	11.04739
0.5	0.03545	0.36989	112.7417	11.04739
1	0.03545	0.36989	112.7417	11.04739
1.5	0.0217	0.2264	69.00566	6.76176
2	-0.01039	-0.10842	-33.045	-3.23803
2.5	-0.01192	-0.12436	-37.9045	-3.71421
3	-0.01253	-0.13074	-39.8483	-3.90468
3.5	-0.01222	-0.12755	-38.8764	-3.80944
4	-0.01192	-0.12436	-37.9045	-3.71421
4.5	-0.01131	-0.11798	-35.9607	-3.52373
5	-0.01131	-0.11798	-35.9607	-3.52373
5.5	-0.011	-0.11479	-34.9888	-3.4285
6	-0.0107	-0.1116	-34.0169	-3.33326
6.5	-0.0107	-0.1116	-34.0169	-3.33326
7	-0.01008	-0.10523	-32.0731	-3.14279
7.5	-0.00978	-0.10204	-31.1011	-3.04755
8	-0.00947	-0.09885	-30.1292	-2.95232
8.5	-0.00917	-0.09566	-29.1573	-2.85708
9	-0.00856	-0.08928	-27.2135	-2.66661
9.5	-0.00825	-0.08609	-26.2416	-2.57137
10	-0.00795	-0.08291	-25.2697	-2.47614
10.5	-0.00733	-0.07653	-23.3259	-2.28567
11	-0.00703	-0.07334	-22.354	-2.19043
11.5	-0.00672	-0.07015	-21.382	-2.09519
12	-0.00611	-0.06377	-19.4382	-1.90472
12.5	-0.00611	-0.06377	-19.4382	-1.90472
13	-0.0055	-0.0574	-17.4944	-1.71425
13.5	-0.0055	-0.0574	-17.4944	-1.71425
14	-0.00489	-0.05102	-15.5506	-1.52378
14.5	-0.00489	-0.05102	-15.5506	-1.52378
15	-0.00458	-0.04783	-14.5787	-1.42854
15.5	-0.00428	-0.04464	-13.6068	-1.33331
16	-0.00397	-0.04145	-12.6348	-1.23807
16.5	-0.00367	-0.03826	-11.6629	-1.14283
17	-0.00367	-0.03826	-11.6629	-1.14283
17.5	-0.00306	-0.03189	-9.71911	-0.95236
18	-0.00306	-0.03189	-9.71911	-0.95236
18.5	-0.00275	-0.0287	-8.7472	-0.85712
19	-0.00244	-0.02551	-7.77529	-0.76189
19.5	-0.00214	-0.02232	-6.80338	-0.66665
20	-0.00214	-0.02232	-6.80338	-0.66665
20.5	-0.00183	-0.01913	-5.83146	-0.57142
21	-0.00183	-0.01913	-5.83146	-0.57142
21.5	-0.00122	-0.01275	-3.88764	-0.38094
22	-0.00122	-0.01275	-3.88764	-0.38094
22.5	-0.00092	-0.00957	-2.91573	-0.28571
23	-0.00061	-0.00638	-1.94382	-0.19047
23.5	-0.00061	-0.00638	-1.94382	-0.19047
24	-0.00031	-0.00319	-0.97191	-0.09524
24.5	0	0	0	0
25	0	0	0	0
25.5	0.00031	0.00319	0.97191	0.09524
26	0.00061	0.00638	1.94382	0.19047
26.5	0.00061	0.00638	1.94382	0.19047
27	0.00061	0.00638	1.94382	0.19047
27.5	0.00092	0.00957	2.91573	0.28571
28	0.00122	0.01275	3.88764	0.38094
28.5	0.00183	0.01913	5.83146	0.57142

TABLE A-1:
Example of raw data file from field calibration of transducer

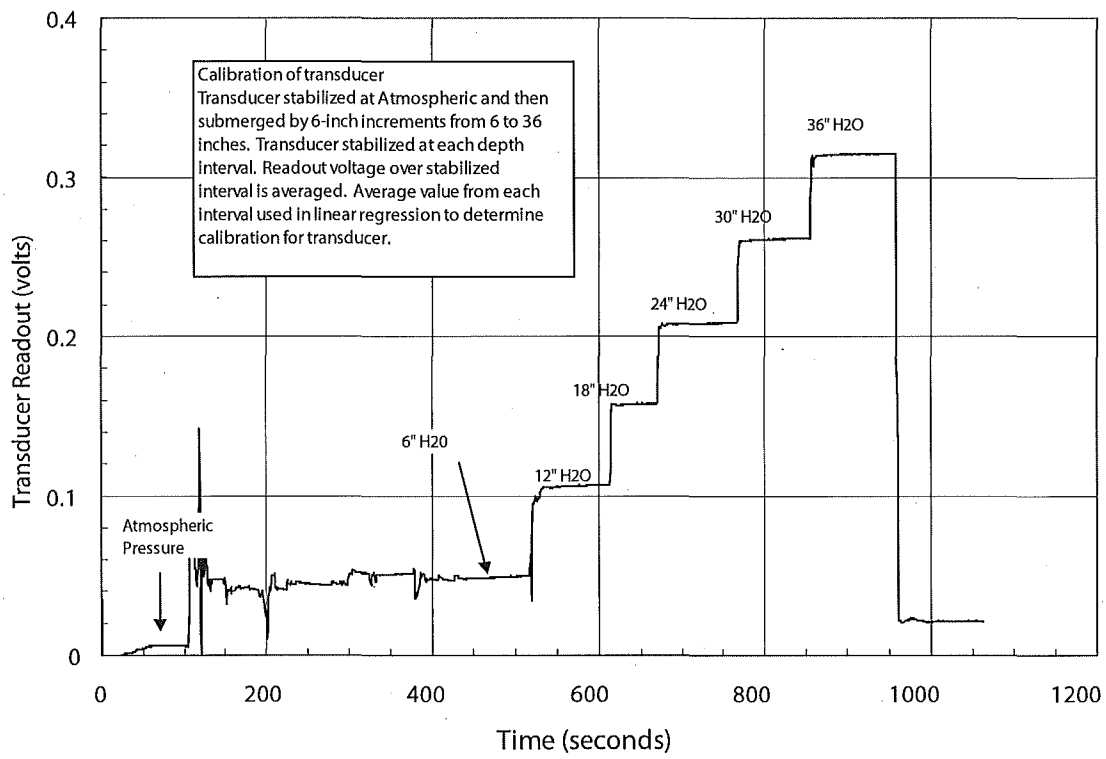


FIGURE A-1:
Plot of transducer voltage (raw data) collected during field calibration

Atmospheric Calibration		6" water Calibration		12" water Calibration		18" water Calibration		24" water Calibration		30" water Calibration		36" water Calibration	
50 to 100 seconds		450 to 500 seconds		550 to 600 seconds		625 to 675 seconds		700 to 750 seconds		800 to 850 seconds		900 to 950 seconds	
time (sec)	Voltage	time (sec)	Voltage	time (sec)	Voltage	time (sec)	Voltage	time (sec)	Voltage	time (sec)	Voltage	time (sec)	Voltage
50	0.00458	450	0.04828	550	0.10574	625	0.15708	700	0.20811	800	0.26068	900	0.31477
50.5	0.00428	450.5	0.04828	550.5	0.10574	625.5	0.15708	700.5	0.20811	800.5	0.26098	900.5	0.31446
51	0.00458	451	0.04828	551	0.10604	626	0.15708	701	0.20811	801	0.26098	901	0.31446
51.5	0.00458	451.5	0.04828	551.5	0.10604	626.5	0.15738	701.5	0.20811	801.5	0.26098	901.5	0.31446
52	0.00458	452	0.04828	552	0.10574	627	0.15708	702	0.20811	802	0.26098	902	0.31477
52.5	0.00458	452.5	0.04828	552.5	0.10574	627.5	0.15738	702.5	0.20811	802.5	0.26098	902.5	0.31446
53	0.00489	453	0.04859	553	0.10574	628	0.15738	703	0.20811	803	0.26098	903	0.31446
53.5	0.00458	453.5	0.04828	553.5	0.10574	628.5	0.15738	703.5	0.20842	803.5	0.26098	903.5	0.31477
54	0.00489	454	0.04828	554	0.10574	629	0.15738	704	0.20811	804	0.26098	904	0.31477
54.5	0.00489	454.5	0.04859	554.5	0.10574	629.5	0.15708	704.5	0.20811	804.5	0.26098	904.5	0.31477
55	0.00489	455	0.04828	555	0.10604	630	0.15738	705	0.20842	805	0.26129	905	0.31477
55.5	0.00489	455.5	0.04859	555.5	0.10604	630.5	0.15738	705.5	0.20811	805.5	0.26098	905.5	0.31477
56	0.0052	456	0.04859	556	0.10604	631	0.15738	706	0.20811	806	0.26098	906	0.31446
56.5	0.0052	456.5	0.04828	556.5	0.10604	631.5	0.15738	706.5	0.20842	806.5	0.26098	906.5	0.31446
57	0.0052	457	0.04828	557	0.10604	632	0.15738	707	0.20811	807	0.26098	907	0.31446
57.5	0.0055	457.5	0.04859	557.5	0.10604	632.5	0.15738	707.5	0.20811	807.5	0.26098	907.5	0.31446
58	0.0052	458	0.04859	558	0.10604	633	0.15738	708	0.20811	808	0.26098	908	0.31477
58.5	0.0055	458.5	0.04859	558.5	0.10604	633.5	0.15738	708.5	0.20811	808.5	0.26098	908.5	0.31477
59	0.0052	459	0.04859	559	0.10604	634	0.15769	709	0.20811	809	0.26098	909	0.31477
59.5	0.0052	459.5	0.04859	559.5	0.10604	634.5	0.15738	709.5	0.20811	809.5	0.26098	909.5	0.31477
60	0.0055	460	0.04859	560	0.10604	635	0.15769	710	0.20811	810	0.26129	910	0.31507
60.5	0.0055	460.5	0.04859	560.5	0.10604	635.5	0.15769	710.5	0.20811	810.5	0.26129	910.5	0.31477
61	0.0055	461	0.04859	561	0.10604	636	0.15738	711	0.20811	811	0.26098	911	0.31477
61.5	0.0055	461.5	0.04859	561.5	0.10604	636.5	0.15738	711.5	0.20811	811.5	0.26129	911.5	0.31477
62	0.0055	462	0.04859	562	0.10604	637	0.15738	712	0.20842	812	0.26098	912	0.31446
62.5	0.0055	462.5	0.04859	562.5	0.10604	637.5	0.15738	712.5	0.20811	812.5	0.26098	912.5	0.31446
63	0.00581	463	0.04859	563	0.10604	638	0.15769	713	0.20811	813	0.26129	913	0.31446
63.5	0.0055	463.5	0.04859	563.5	0.10604	638.5	0.15738	713.5	0.20811	813.5	0.26129	913.5	0.31477
64	0.00581	464	0.04859	564	0.10604	639	0.15769	714	0.20811	814	0.26098	914	0.31446
64.5	0.00581	464.5	0.04859	564.5	0.10604	639.5	0.15769	714.5	0.20811	814.5	0.26098	914.5	0.31477
65	0.00581	465	0.04859	565	0.10604	640	0.15738	715	0.20811	815	0.26098	915	0.31477
65.5	0.00611	465.5	0.04859	565.5	0.10604	640.5	0.15738	715.5	0.20842	815.5	0.26129	915.5	0.31477
66	0.00581	466	0.04859	566	0.10604	641	0.15769	716	0.20842	816	0.26098	916	0.31446
66.5	0.00581	466.5	0.04859	566.5	0.10604	641.5	0.15738	716.5	0.20842	816.5	0.26098	916.5	0.31477
67	0.00581	467	0.04859	567	0.10604	642	0.15769	717	0.20842	817	0.26098	917	0.31477
67.5	0.00581	467.5	0.04859	567.5	0.10604	642.5	0.15769	717.5	0.20842	817.5	0.26129	917.5	0.31477
68	0.00581	468	0.04859	568	0.10604	643	0.15769	718	0.20811	818	0.26098	918	0.31477
68.5	0.00581	468.5	0.04859	568.5	0.10604	643.5	0.15769	718.5	0.20811	818.5	0.26129	918.5	0.31477
69	0.00581	469	0.0489	569	0.10635	644	0.15769	719	0.20811	819	0.26129	919	0.31477
69.5	0.00611	469.5	0.04859	569.5	0.10604	644.5	0.15769	719.5	0.20842	819.5	0.26129	919.5	0.31477
70	0.00581	470	0.0489	570	0.10635	645	0.15769	720	0.20811	820	0.26098	920	0.31477
70.5	0.00611	470.5	0.04859	570.5	0.10635	645.5	0.15769	720.5	0.20811	820.5	0.26098	920.5	0.31477
71	0.00581	471	0.04859	571	0.10635	646	0.15769	721	0.20842	821	0.26129	921	0.31477
71.5	0.00611	471.5	0.0489	571.5	0.10604	646.5	0.158	721.5	0.20842	821.5	0.26098	921.5	0.31477
72	0.00611	472	0.04859	572	0.10635	647	0.15769	722	0.20842	822	0.26129	922	0.31477
72.5	0.00611	472.5	0.04859	572.5	0.10604	647.5	0.15769	722.5	0.20842	822.5	0.26129	922.5	0.31477
73	0.00611	473	0.0489	573	0.10635	648	0.15769	723	0.20842	823	0.26129	923	0.31477
73.5	0.00611	473.5	0.04859	573.5	0.10635	648.5	0.15769	723.5	0.20842	823.5	0.26098	923.5	0.31446
74	0.00611	474	0.04859	574	0.10635	649	0.15769	724	0.20842	824	0.26098	924	0.31477
74.5	0.00611	474.5	0.0489	574.5	0.10635	649.5	0.15769	724.5	0.20842	824.5	0.26129	924.5	0.31477
75	0.00611	475	0.0489	575	0.10635	650	0.15769	725	0.20842	825	0.26129	925	0.31477
75.5	0.00611	475.5	0.0489	575.5	0.10635	650.5	0.15769	725.5	0.20842	825.5	0.26129	925.5	0.31446
76	0.00611	476	0.0489	576	0.10635	651	0.15769	726	0.20842	826	0.26129	926	0.31477
76.5	0.00611	476.5	0.0489	576.5	0.10665	651.5	0.15769	726.5	0.20842	826.5	0.26129	926.5	0.31477
77	0.00611	477	0.0489	577	0.10635	652	0.158	727	0.20842	827	0.26159	927	0.31446
77.5	0.00611	477.5	0.0489	577.5	0.10635	652.5	0.15769	727.5	0.20842	827.5	0.26129	927.5	0.31477
78	0.00611	478	0.0489	578	0.10635	653	0.15769	728	0.20842	828	0.26129	928	0.31446
78.5	0.00611	478.5	0.0489	578.5	0.10635	653.5	0.15769	728.5	0.20842	828.5	0.26129	928.5	0.31477
79	0.00611	479	0.0489	579	0.10635	654	0.15769	729	0.20842	829	0.26129	929	0.31477
79.5	0.00581	479.5	0.0489	579.5	0.10635	654.5	0.15769	729.5	0.20842	829.5	0.26129	929.5	0.31477
80	0.00581	480	0.0489	580	0.10635	655	0.15769	730	0.20842	830	0.26129	930	0.31477
80.5	0.00581	480.5	0.0489	580.5	0.10635	655.5	0.15769	730.5	0.20842	830.5	0.26129	930.5	0.31477
81	0.00611	481	0.0489	581	0.10635	656	0.15769	731	0.20842	831	0.26129	931	0.31477
81.5	0.00581	481.5	0.0489	581.5	0.10635	656.5	0.15769	731.5	0.20872	831.5	0.26129	931.5	0.31477
82	0.00581	482	0.0489	582	0.10635	657	0.158	732	0.20842	832	0.26129	932	0.31446
82.5	0.00581	482.5	0.0489	582.5	0.10635	657.5	0.15769	732.5	0.20842	832.5	0.26129	932.5	0.31477
83	0.00611	483	0.0489	583	0.10635	658	0.15769	733	0.20872	833	0.26129	933	0.31507
83.5	0.00611	483.5	0.0489	583.5	0.10635	658.5	0.15769	733.5	0.20842	833.5	0.26129	933.5	0.31477
84	0.00611	484	0.0489	584	0.10635	659	0.158	734	0.20872	834	0.26129	934	0.31507
84.5	0.00581	484.5	0.0489	584.5	0.10665	659.5	0.158	734.5	0.20842	834.5	0.26129	934.5	0.31477
85	0.00611	485	0.0492	585	0.10635	660	0.15769	735	0.20842	835	0.26129	935	0.31477
85.5	0.00611	485.5	0.0489	585.5	0.10665	660.5	0.15769	735.5	0.20842	835.5	0.26129	935.5	0.31446
86	0.00611	486	0.0492	586	0.10665	661	0.158	736	0.20842	836	0.26159	936	0.31477
86.5	0.00581	486.5	0.0489	586.5	0.10665	661.5	0.15769	736.5	0.20872	836.5	0.26129	936.5	0.31477
87	0.00581	487	0.0489	587	0.10665	662	0.15769	737	0.20842	837	0.26129	937	0.31446
87.5	0.00581	487.5	0.0492	587.5	0.10665	662.5	0.15769	737.5	0.20872	837.5	0.26129	937.5	0.31477
88	0.00581	488	0.0489	588	0.10665	663	0.158	738	0.20842	838	0.26159	938	0.31477
88.5	0.00611	488.5	0.0489	588.5	0.10665	663.5	0.158	738.5	0.20842	838.5	0.26129	938.5	0.31477
89	0.00581	489	0.0489	589	0.10665	664	0.15769	739	0.20842	839	0.26129	939	0.31507
89.5	0.00581	489.5	0.0489	589.5	0.10665	664.5	0.15769	739.5	0.20872	839.5	0.26159	939.5	0.31477
90	0.00581	490	0.0489	590	0.10665	665	0.158	740	0.20842	840	0.26129	940	0.31477
90.5	0.00581	490.5	0.0489	590.5	0.10665	665.5	0.15769	740.5	0.20872	840.5	0.26159	940.5	0.31477
91	0.00581	491	0.0492	591	0.10665	666	0.158	741	0.20872	841	0.26129	941	0.31477
91.5	0.00581	491.5	0.0492	591.5	0.10665	666.5	0.158	741.5	0.20842	841.5	0.26129	941.5	0.31477
92	0.00581	492	0.0492	592	0.10665	667	0.158	742	0.20872	842	0.26129	942	0.31477
92.5	0.00581	492.5	0.0492	592.5	0.10665	667.5	0.15769	742.5	0.20872	842.5	0.26129	942.5	0.31477
93	0.00581	493	0.0492	593	0.10665	668	0.158	743	0.20842	843	0.26159	943	0.31477
93.5	0.0055	493.5	0.0492	593.5	0.10665	668.5	0.158	743.5	0.20842	843.5	0.26129	943.5	0.31477
94	0.00581	494	0.04951	594	0.10665	669	0.15769	744	0.20872	844	0.26129	944	0.31477
94.5	0.00581	494.5	0.0492	594.5	0.10665	669.5	0.158	744.5	0.20872	844.5	0.26159	944.5	0.31477
95	0.00581	495	0.0489	595	0.10696	670	0.16655	745	0.20872	845	0.26129	945	0.31477
95.5	0.00581	495.5	0.0489	595.5	0.10696	670.5	0.19253	745.5	0.20842	845.5	0.26159	945.5	0.31477
96	0.0055	496	0.0492	596	0.10696	671	0.20506	746	0.20872	846	0.26159	946	0.31477
96.5	0.00581	496.5	0.0492	596.5	0.10696	671.5	0.20597	746.5	0.20872	846.5	0.26159	946.5	0.31507
97	0.00581	497	0.0492	597	0.10665	672	0.20567	747	0.20872	847	0.26129	947	0.31477
97.5	0.0055	497.5	0.0492	597.5	0.10696	672.5	0.20567	747.5	0.20872	847.5	0.26129	947.5	0.31507
98	0.00581	498	0.0492	598	0.10696	673	0.20536	748	0.20872	848	0.26129	948	0.31477
98.5	0.00581	498.5	0.0492	598.5	0.10696	673.5	0.20536	748.5	0.20842	848.5	0.26159	948.5	0.31477
99	0.0055	499	0.0492	599	0.10696	674	0.20536	749	0.20872	849	0.26159	949	0.31477
99.5	0.00581	499.5	0.0492	599.5	0.10696	674.5	0.20689	749.5	0.20872	849.5	0.26159	949.5	0.31477
100	0.0055	500	0.0492	600	0.10696	675	0.2075	750	0.20872	850	0.26129	950	0.31477
Average	0.00567	Average	0.04879	Average	0.10632	Average	0.16236	Average	0.208384	Average	0.261221	Average	0.314723
Voltage at atmospheric		Voltage at 6"		Voltage at 12"		Voltage at 18"		Voltage at 24"		Voltage at 30"		Voltage at 36"	

TABLE A-2:
Calculate average transducer voltage for each depth increment

Transducer Calibration Factor

Inches of Water	Averaged Transducer Voltage
0	0.005677
6	0.048791
12	0.106326
18	0.162364
24	0.205384
30	0.261221
36	0.314723

Regression Output:

Constant (b)	-0.264402
Std Err of Y Est	0.410288
R Squared	0.999165
No. of Observations	7
Degrees of Freedom	5
X Coefficient (m)	115.4423
Std Err of Coef.	1.492468

Regression Line Y = mX + b

where:
Y = Inches of water
155.44 = m = slope of regression line
X = Transducer voltage
-0.2644 = b = Y-intercept

Example:
If transducer voltage = 0.1458
Then inches - water = 16.57

TABLE A-3:
Linear regression analysis of average transducer voltage for each water depth (from TABLE A-2) provides calibration data for transducer

Equipment and tool specifications, including weights, dimensions, materials, and operating specifications included in this brochure are subject to change without notice. Where specifications are critical to your application, please consult Geoprobe Systems®.

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STANDARD OPERATING PROCEDURE

SEI-6.23.1

OBSERVATION AND MONITORING WELL SURVEYING FOR ELEVATIONS

SOP Number: SEI-6.23.1
Revision Number: 1

Date Issued: 7/19/99
Date of Revision: 11/29/05

1.0 OBJECTIVE

A field survey control program is conducted by using standard survey techniques to document piezometer and monitoring well elevations relative to a common datum or relative to each other. There are many types of leveling instruments available to conduct a survey program. This Standard Operating Procedure (SOP) describes the procedure for determining the relative elevations (altitudes) of observation and monitoring wells in the field.

2.0 POLICIES

1. Personnel will legibly record data and observations in the field to enable others to reconstruct project events and provide sufficient evidence of activities conducted.
2. Field personnel will use a field logbook, observation and remark form (O&R) or other designated field forms to record activities, measurements, and observations made in the field.

3.0 SAFETY ISSUES

1. Whenever visiting a hazardous or a study site, it is important to read, follow and sign the site health and safety protocol (HASP), if available.
2. Field visits may involve accessing remote areas. Health and safety concerns regarding these field visits are minimal however; slip, trip and fall hazards, and poisonous plant and dangerous animal, as well as getting vehicles stuck in remote areas do present considerable health and safety issues. To help ensure field staff's health and safety with remote areas, all field staff is to have daily communication with the project manager or another appropriate Stone Environmental, Inc. (Stone) employee.

4.0 PROCEDURES

4.1 Equipment and Materials

1. Level.
2. Tripod.
3. Stadia rod.

4. Field forms or other appropriate media.

4.2 Procedure

The following procedure describes a simple method to determine relative field elevations.

The method described here uses a traverse starting from a known elevation working towards unknown elevations. If there are no known elevations, then the starting point is to be given an arbitrary elevation of a temporary benchmark. The traverse will be a closed loop so that the starting point also is the ending point in the loop. This method enables determining the accuracy of the measurements by taking the difference between the initial elevation of the starting point and the final elevation of the starting point.

Surveying requires two persons, one to operate the level and one to hold the stadia rod.

1. Set up a table in the field notebook or on field data sheets with columns labeled as point location, backshot(+), foreshot(-), and elevation.
2. Set the tripod up some distance away from the starting point. (The distance depends on the ocular power of the instrument). Approximately 150 feet is a good distance.
3. Level the instrument according to the manufacturer's instructions.
4. The stadia rod is to be held vertically over the measuring point of a piezometer (monitoring well) or on a point of some known elevation. Note that this measuring point is the same point from which water levels in the piezometers (monitoring wells) are to be measured. If there is no mark, one should be made in black indelible ink. Be very careful to keep dirt or other debris from falling into the well. It may be necessary to cover the well with a plastic baggie for protection.
5. With the instrument leveled, read the measurement on the stadia at the middle cross hair. Record this measurement as the backshot (+).
6. Move the stadia rod to the next position beyond the instrument roughly the same distance as the backshot. This is designated as a turning point (could be the next piezometer or monitoring well).
7. Pivot the level around being careful not to disturb the tripod. Read the stadia rod and record as the foreshot (-).
8. Move the instrument with its tripod and level, roughly the same distance beyond the stadia rod as before (this can be done by pacing).
9. Pivot the stadia rod being careful not to change the elevation.
10. Read the measurement on the stadia rod at the turning point. Record as the backshot.
11. Repeat steps 6 through 10 working around the field to close the loop until the last foreshot is made at the original starting point.

Example of Leveling and Recording Measurements

The following example depicts the above method of leveling and recording measurements. P-1 is designated at an arbitrary elevation. P-2 is measured relative to P-1. TP is the turning point, b = backshot, f = foreshot, $I_{1,2}$ = instrument location.

1. P-1 is the starting point and is designated at an arbitrary elevation of 100.00 feet.
2. The stadia rod is at P-1. The instrument is at I_1. Read the stadia rod backshot, b = 2.00 feet. The instrument height = 100.00 + 2.00 = 102.00 feet.
3. The instrument remains at I_1. The stadia rod is moved to P-2.
4. Read the stadia rod at P-2. The foreshot is f = 0.50 feet, thus, the ground elevation at P-2 = 102.00 - 0.50 ft = 101.50 feet.
5. Move the instrument to I_2. The stadia rod remains at P-2 (which is TP1, the turning point). Read the backshot b.
6. Move the stadia rod to P-3. Read the foreshot f = 3.00.
7. Continue around the field determining the elevation of each measuring point at the piezometers and/or monitoring wells ending with the last foreshot at the starting point (P-1).
8. The calculated elevation of P-1 should be what it was assumed to be at the beginning, in this case 100.00 feet.
9. The degree of accuracy should be determined prior to the survey, and will depend on study needs. However, in most cases involving the calculation of groundwater elevations, the final foreshot should be within 0.01 feet of the starting point (P-1).

5.0 RESPONSIBILITIES

1. All personnel will legibly record data and observations (including phone conversations) in accordance with this SOP to enable others to reconstruct project events and provide sufficient evidence of activities conducted.

6.0 DEFINITIONS

1. *Observations & Remarks Form (O&R):* A pre-printed form, which contains mostly blank space for general note taking. The form typically prompts the user for the study or project designation, the Stone project number, the client or sponsor name, the total number of pages (page *n* of n) and requires a signature and date. The form is generally used to capture notes of one person when another, more specific forms is not available.
2. *Raw data* any laboratory worksheets, records, memoranda, notes, or exact copies thereof, that are the result of original observations and activities of a study and are necessary for the reconstruction and evaluation of the report of that study. In the event that exact transcripts of raw data have been prepared (e.g., tapes which have been transcribed verbatim, dated, and verified accurate by signature), the exact copy or exact transcript may be substituted for the original source as raw data. Raw data may include photographs, microfilm or microfiche

copies, computer printouts, magnetic media, including dictated observations, and recorded data from automated instruments.

7.0 REFERENCES

40 CFR Part 160 Good Laboratory Practice Standards, August, 1989.

8.0 TABLES, DIAGRAMS, FLOWCHARTS, AND VALIDATION DATA

None

9.0 AUTHORIZATION

Revised by: _____ Date: 11-29-05

Patrick Larsen, Project Geoscientist

Approved by: _____ Date: 11-23-05

Christopher T. Stone, President

10.0 REVISION HISTORY

Revision 1

1. Updated style to match SEI Style Guide – font and text. Reformatted using MS Word.
2. Added standardized section headers: 2.0 Policies, 3.0 Safety, 5.0 Responsibilities, 6.0 Definitions, 7.0 References, 8.0 Tables, Diagrams, Flowcharts and Validation data. Authorization moved to Section 9.0, andSection10.0 Revision History.

STANDARD OPERATING PROCEDURE

SEI-6.27.3

GROUNDWATER SAMPLING OF MONITORING WELLS

SOP Number: SEI-6.27.3
Revision Number: 3

Date Issued: 03/03/00
Date of Revision: 11/29/05

1.0 OBJECTIVE

Stone Environmental, Inc. utilizes a wide variety of sampling methods for the collection of groundwater samples from monitoring wells. This standard operating procedure outlines the sampling methods to be used for 12 volt electric submersible pumps, Grundfos pumps, bladder pumps, and bailers.

2.0 POLICIES

1. Personnel will legibly record data and observations in the field to enable others to reconstruct project events and provide sufficient evidence of activities conducted.
2. Field personnel will use a field logbook, observation and remark form (O&R) or other designated field form to record activities, measurements, and observations made in the field.

3.0 SAFETY ISSUES

1. Whenever visiting a hazardous or potentially hazardous site, it is important to read, follow and sign the site health and safety protocol (HASP).
2. Field visits may involve accessing remote areas. Health and safety concerns regarding these field visits are minimal however; slip, trip and fall hazards, and poisonous plant and dangerous animal, as well as getting vehicles stuck in remote areas do present considerable health and safety issues. To help ensure field staff's health and safety with remote areas, all field staff is to have daily communication with the project manager or another appropriate Stone Environmental, Inc. (Stone) employee.

4.0 PROCEDURES

4.1 Equipment and Materials

If using a dedicated electric submersible pump:

1. Electric submersible pump, tubing, and wiring (in place in well).
2. 12 volt power source

3. Connector cable with switch(optional)
4. Purge tubing (may be connected to calibrated buckets)
5. Replacement pump, wire, and connector kit

If using a bailer:

1. One dedicated bailer for each well is preferred. If a bailer is to be shared between wells, make sure it can be dismantled and thoroughly decontaminated in the field. Disposable bailers commonly have seams that can collect contaminants and are impossible to clean; these bailers should not be used in more than one well. Bailers must be constructed of materials that will not react with compounds to be analyzed.
2. Clean, inert string, not to be used in another well

If using a Grundfos Redi-Flo® Variable Pump assembly:

1. Grundfos Redi-Flo® Variable Pump assembly
2. Generator, ≥2500 watt with built in voltage regulator with extension cord or other power source
3. Dedicated sampling tube assembly (dedicated to each site or decontaminate between sites)
4. GPI industrial grade flow meter(see SEI SOP 5.24.*n*) with 100 ft. of tubing (optional)
5. Three, half gallon containers for decontamination procedure
6. Large tub or bucket to hold the pump and tubing/cable
7. Three containers large enough to completely immerse the Grundfos pump
8. Plastic garbage bags
9. Suspension hooks to keep tubing/cable off of the ground

If using a dedicated bladder pump:

1. Bladder pump system, including pump, tubing, and well cap
2. Oil-less air compressor compatible with the bladder pump system to be used (the compressor should be oil-less to prevent oil from collecting inside the controller and damaging it)
3. Air pressure controller for the air compressor

Equipment recommended regardless of the sampling method:

1. Protective gloves appropriate for site safety concerns and sample integrity
2. Sample containers for water quality measurements
3. Water quality meters with appropriate calibration solutions
4. Water level indicator
5. Calibrated purge buckets

6. Field notebook with protocol, applicable SOPs, study specific procedures, sampling forms, Chain of custody, and or other appropriate medium for recording field data
7. Sample containers and labels
8. Well keys
9. Calculator
10. Electrical tape
11. Clear packing tape
12. Distilled water
13. Alconox
14. Spray bottles for decontamination

4.2 Procedure

Sample Collection

Begin sample collection in locations least likely to be contaminated, and then proceed to areas more likely to be contaminated. Access the well(s).

When appropriate or necessary, all fittings, equipment, and receptacles used in the sampling process must be decontaminated in accordance with standard operating procedures for the decontamination of field equipment (see SOP 5.1.n).

Prior to measuring the water level, be sure to don appropriate, clean sampling gloves. Measure the water level in the well following the appropriate standard operating procedure for water level measurement (see SOP 6.2.n). If equipped with a dedicated pump and well head assembly, the water level indicator can be lowered into the well through the access hole in the well cap, so that the pump is not disturbed. Document use of the water level indicator on the appropriate, Use & Maintenance log following the instruments SOP.

Calculate the "well volume" - the volume of standing water in the well - using the following equation:

$$V_{gal} = (TD\text{-}WL)(\pi r^2)(CF) \qquad (1)$$

where:

V_{gal}	=	the well volume
r_{ft}	=	the radius of the well casing
π	=	3.14 (or as defined by the calculator)

TD_{ft} = the total depth of the well, measured from the top of the well casing

WL_{ft} = the water level in the well, measured from the top of the well casing

CF = conversion factor (cubic ft. to US gal. = 7.48gal/ft^3)

The $[(\pi r^2)(CF)]$expression in the well volume formula may already be calculated. For example, the attached sampling form has been calculated for a 2 inch diameter well.

Note that if the well is equipped with a dedicated pump, this equation does not take into account the volume displaced by the pump, so it will over-estimate the actual volume of water in the well. Document all well volume calculations on the sampling form or in another appropriate medium. Refer to SOP 4.5.*n* for more information on documentation of raw data. If appropriate, calculate 1, 3, and 5 well volumes for each well to be purged.

The standing water in the well has been out of direct contact with the soil and exposed to the open air. The effects of this on the quality of the water are difficult to determine, but to avoid any question of whether the sample is representative of true groundwater quality on the site, it is best to thoroughly purge the well before collecting a groundwater sample. Determine which type of sampling apparatus is to be used and follow the corresponding purging procedure from the following list.

Dedicated electric submersible pump:

1. Connect the purge tubing to the existing outlet extending above the well cap. Run the tubing from the outlet to the calibrated bucket.
2. Open the valve on the well sampling assembly (if one exists).
3. Connect the power supply to the pump leads using the switch box and connector cable and turn the switch on. If you do not have the switch box, connect the leads to complete the circuit when you are ready to begin pumping.
4. Confirm that water is flowing into the bucket via the purge tubing, if no water is flowing then turn the pump off and check connections.
5. Continue purging until complete as defined below.

Bailer:

1. Put on clean sampling gloves
2. Obtain a clean bailer. Do not allow the bailer to come into contact with surface soils on the site
3. Attach the inert string to the bailer. Make sure the knot is secure and that there is a sufficient length of string to reach the bottom of the well.
4. Lower the bailer into the well on the nylon string, and allow the bailer to fill with water.
5. Raise the bailer out of the well, coiling the string so as not to let it come into contact with the ground
6. Dump the bailer into one of the calibrated buckets.

7. Repeat steps 2-6 until purging is complete as defined below.

Grundfos Redi-Flo® Variable Pump:

1. Start the generator, allow it to warm up for at least 5 minutes, and check generator settings, 115 volts AC±10%, optimum setting of 120 volts without load.
2. Rinse pump with alconox and distilled water
3. Connect the sample tubing to the pump taking care not to let it come in contact with the ground. If the tubing is on a reel, it may be helpful to put a clean plastic bag under the reel to avoid contact with the ground.
4. Carefully lower the pump into the well taking care that is does not rest on the bottom.
5. Plug the Redi-Flo Variable Frequency Device (VFD) into the generator.
6. Using the mode selection dial, choose the pump model.
7. Turn the ten-turn speed dial fully counter clockwise.
8. Connect the pump motor lead to the VFD.
9. The frequency display should read 0.0, if it does not refer to the Redi-Flo Installation and Operating instructions.
10. If the unit has not been used for more than six months, allow a five minute warm up period before starting the pump.
11. If using the GPI industrial grade flow meter, connect the flow meter to the dedicated sample tubing and run the purge tubing down gradient of the well. If a flow meter is not used, calibrated buckets or some other purging method can be used.
12. Start the pump by switching the Redi-Flo VFD run/stop/reset switch to run
13. Adjust the pump to the desired speed using caution to preclude pumping the well dry. It may be useful to record the optimum pumping rate in hertz in the comments section of the applicable Monitoring Well Sampling form.
14. Purge the well until complete as defined below.

Dedicated bladder pump:

1. Connect the purge tubing to the existing outlet extending above the well cap.
2. Run the tubing from the outlet to the purge bucket.
3. Using the two air hoses, connect the compressor to the air pressure controller and the air pressure controller to the air tube inlet on the well cap. Set the controller to regulate the air pressure used for pumping and turn on the controller (if necessary/ applicable).
4. Start the air compressor, and if necessary/applicable, open the valve to allow the air to flow to the controller.

5. Confirm that water is flowing to the purge buckets via the purge tubing, if not, check all connections and turn the pump off if checking connections does not solve the problem.

6. Continue until purging is complete as defined below.

Track the volume purged. If you are using calibrated buckets, empty the buckets off-site down gradient of all wells in the study. Take care not to let the purge water flow or spill onto the ground around the well.

Remove a volume of water equal to or greater than 3-5 times the well volume. Water quality parameters may be monitored to assist in determining if the well has been adequately purged. The pH, temperature, and conductivity of the groundwater (and / or other parameters, depending on the requirements of the study), can be monitored while the well is purged using the following procedure:

1. After purging at least one well volume. Collect a sample of the purge water in a beaker or a clean container (other than the sample bottle) and measure and record the water quality parameters.

2. Continue to purge and collect water quality parameters at intervals of $\geq$ **0.5** well volumes.

3. If after **three** consecutive water quality parameter measurements **and** after purging a minimum of **three** well volumes, the parameters for temperature and conductivity vary less than 10% and the pH varies less than 0.1 unit, purging can be halted.

4. If any one of the parameters are outside of the range, successive measurements of water quality can than be made until the parameters stabilize per the above ranges. If the parameters do not stabilize after **five** well volumes have been purged, purging may be halted.

If water quality meters are used be sure to document their use on the appropriate, Use & Maintenance log following the instruments SOP. If calibrations are required, refer to the instruments SOP or the manufacturers instructions. Record the method of purging, the parameters monitored, if applicable, and the approximate volume of water purged. If any of the water quality meters are malfunctioning or are not used, always attempt to purge five well volumes. Regardless of whether water quality parameters are measured, a **minimum of 3 well volumes must always be purged.** The exception to this is if the well is purged dry.

Avoid purging the well completely dry (especially if sampling for volatiles). If the well is purged dry, allow the well to re-charge adequately enough to permit collection of a sample. **Note on the monitoring well sampling form if the well was purged dry** (purge volume may be less than 3 well volumes in this case).

Put on clean sampling gloves. Collect the groundwater sample in a clean bottle or other container appropriate for the analytical method to be used. The sample is collected by carefully removing the cap from the sample bottle, taking care not to touch the inside of the cap or bottle with your fingers, the discharge tube, or any other equipment. Do not put the cap down. Fill the sample bottle according to the requirements for the analysis (rinse as required by the protocol). Tightly cap the bottle. The sample container should be labeled, at a minimum, with the following information. (Generally, it is easier to attach the label or write on the container before collecting the sample. Condensation on the containers forms after sample collection, and reduces the likelihood of tape or ink sticking):

1. Study number(s) or project name

2. Sample identification (including the type of sample - groundwater)

3. Date of sample collection

Secure the label with a wrap of clear tape if applicable. Follow protocol or Quality Assurance Project Plan requirements for further label needs.

If appropriate to the study, the cap of the bottle can be secured with tape to prevent accidental loss of sample during storage and shipment.

Place the samples out of direct sunlight as soon as possible or into the appropriate storage devices such as a cooler or boxed container.

4.3 Documentation

A record of the sample collection must be made in the field at the time the sample is collected. The record should be made in a field notebook, a sample collection form, or other medium acceptable according to the study protocol or work plan. The information to be recorded must include the following:

1. Date and time of sample collection.
2. Name(s) of the personnel performing the sampling.
3. Sample location, well identification, and / or sample identification.
4. Project/study designation.
5. Water level prior to purging (equilibrium level).
6. Well volume calculations.
7. Volume purged from the well prior to sampling.
8. Method of sample collection.
9. Chemical parameters monitored in the field (if applicable).

4.4 Packing and Transportation

Package the samples in an insulated cooler or other sturdy container with ice or ice substitute if required. Send the samples to the laboratory for analysis as soon as is reasonable, but well within the maximum holding time for the sample allowing for time in transit. A completed Chain-of-Custody Form must accompany the sample set, in accordance with standard operating procedures for chain of custody forms.

4.5 Equipment Maintenance, Calibration and Decontamination

Follow Stone SOPs for specific equipment to determine the appropriate equipment, maintenance, calibration and decontamination procedures,

Stone does not maintain 12V electric submersible pumps. Should one fail, it will be replaced with a new one. Replacement of a pump should be noted in files and the number of the pump should be recorded.

4.6 Procedure for Decontamination of the Grundfos Pump and Reel

After use of this equipment this decontamination procedure must be followed prior to sampling another well. Ideally, decontamination will occur at the site before storing the equipment for any period of time.

1. Once the well has been purged and/or sampled, place the tubing/cable and pump into a large bucket lined with a clean garbage bag.
2. Leaving the reel attached to the well "walk" the bucket away from the well until the total length of tubing/cable that had been inside the casing is laid out for washing. In order to keep the tubing/cable off the ground after it has been cleaned it may be necessary to suspend the tubing/cable, with a series of hooks for example.
3. Spray the entire length of the tubing/cable with alconox being careful to coat each side thoroughly.
4. Spray the entire length again with distilled water until the alconox has been completely rinsed off.
5. Put on clean gloves.
6. Reel the tubing/cable up taking care not to let it come in contact with anything.
7. Place the pump into a container filled with alconox solution. Run the pump such that approximately two gallons of alconox "clear" the pump and associated tubing.
8. Place the pump into another container filled with distilled water and again run approximately two gallons through the system.
9. Place the pump in a third container and repeat step 9.
10. Unscrew the plug in the bottom of the pump and rinse well with distilled water. Fill the small space with distilled water and replace screw.
11. Replace the plug on the reel casing so that nothing can enter the tubing. Some distilled water may remain in the tubing that will be evacuated next time the pump is started.
12. Document the equipment has been decontaminated on the Use & Maintenance log.

5.0 RESPONSIBILITIES

1. All personnel will legibly record data and observations (including phone conversations) to enable others to reconstruct project events and provide sufficient evidence of activities conducted.

6.0 DEFINITIONS

1. *Observations & Remarks Form (O&R):* A pre-printed form, which contains mostly blank space for general note taking. The form typically prompts the user for the study or project designation, the Stone project number, the client or sponsor name, the total number of pages (page *n* of n) and requires a signature and date. The form is generally used to capture notes of one person when another, more specific forms is not available.

2. *Raw data* any laboratory worksheets, records, memoranda, notes, or exact copies thereof, that are the result of original observations and activities of a study and are necessary for the reconstruction and evaluation of the report of that study. In the event that exact transcripts of raw data have been prepared (e.g., tapes which have been transcribed verbatim, dated, and verified accurate by signature), the exact copy or exact transcript may be substituted for the original source as raw data. Raw data may include photographs, microfilm or microfiche copies, computer printouts, magnetic media, including dictated observations, and recorded data from automated instruments.

7.0 REFERENCES

40 CFR Part 160 Good Laboratory Practice Standards, August, 1989.

8.0 TABLES, DIAGRAMS, FLOWCHARTS, AND VALIDATION DATA

Attachment 1: Example Monitoring Well Sampling Form

9.0 AUTHORIZATION

Revised by: _____ Date: 11-29-05

Patrick Larsen, Project Geoscientist

Approved by: _____ Date: 11-23-05

Christopher T. Stone, President

10.0 REVISION HISTORY

Revision 1 and 2:

1. Reformatted
2. Added some items for decontamination to Section 2.0, #3
3. Minor word changes to Section 3.1.5
4. Deleted requirement for rinsing the sample bottles prior to collection.
5. Added Section 3.5 Procedures for Decontamination of the Grundfos Pump and Reel

Revision 3:

1. Updated style to match Stone Style Guide – font and text. Reformatted using MS Word.
2. Added standardized section headers: 2.0 Policies, 3.0 Safety, 5.0 Responsibilities, 6.0 Definitions, 7.0 References, 8.0 Tables, Diagrams, Flowcharts and Validation data. Authorization moved to Section 9.0, andSection10.0 Revision History.

Attachment I
Example Monitoring Well Sampling Form

MONITORING WELL SAMPLING FORM STONE ENVIRONMENTAL INC

Study Title	
Sponsor	
Protocol Number	
Study Director	
Stone Environmental, Inc. Study Number	

SITE ID			
Date			
SSP Followed			
Sampling Method	Whale Pump	Bailer	(Circle the appropriate one)
Sampling Personnel			
Weather			

Calculate Purge Volumes

Time of level measurement:	

Total Well Depth (btoc)		Depth to Water (btoc)		Height of Water Column		Well Water Volume
_____ feet	–	_____ feet	=	_____ feet	X 0.169 gal/feet	= _____ gallons

3 X Well Water Volume	gallons
5 X Well Water Volume	gallons

Time (HR:MIN)	pH (su)	Conductivity (mmho/cm)	Temperature (°C)	Cumulative Volume (gallons)

Total Volume Purged: _____ Gallons

Sample Number	Time Collected (HR:MIN)	Approx. Vol. (mL)	Comments

Signature of Sampler:		Date:	

Note: BTOC = Below Top of Casing

\\STONE_NT01\PROJ_DRV_G\PROJ\96-330\GLP\Forms\Sampling\Monitoring_Well.doc

STANDARD OPERATING PROCEDURE

SEI-6.34.1

PROCEDURE FOR SAMPLING GROUNDWATER MONITORING WELLS USING LOW STRESS (LOW FLOW) TECHNIQUE

SOP Number: SEI-6.34.1 Date Issued: 01/21/05
Revision Number: 1 Date of Revision: 1/10/13

1.0 OBJECTIVE

This Standard Operating Procedure (SOP) provides a framework for collecting groundwater samples that are indicative of mobile organic and inorganic loads at ambient flow conditions (both the dissolved fraction and the fraction associated with mobile particulates). The SOP emphasizes the need to minimize stress by low water-level draw downs, and low pumping rates (usually less than 1 liter/min) in order to collect samples with minimal alterations to water chemistry. This SOP is aimed primarily at sampling monitoring wells that can accept a submersible pump and have a screen, or open interval length of 10 feet or less. Samples thus obtained are suitable for analyses of ground water contaminants (volatile and semi-volatile organic analytes, pesticides, PCBs, metals and other inorganics), or other naturally occurring analytes.

2.0 POLICIES

It is the policy of Stone Environmental Inc. (Stone) that all field staff conducting this sampling read this SOP prior to acquiring samples. Refer to SEI 6.16.4 for sample handling, preservation and transportation procedures.

Stone personnel will legibly record data and observations in the field to enable others to reconstruct project events and provide sufficient evidence of activities conducted. Field personnel will use a field logbook, observation and remark form or other designated field form to record activities, measurements, and observations made in the field.

3.0 SAFETY ISSUES

The corporate Health and Safety Plan and the Site Health and Safety Plan specify the procedures to be followed and equipment to be used during site activities. The following is a brief and general overview of safety issues.

- Potential Safety Issues include:
    - Heavy equipment (drill rigs or direct push rigs) hazards
    - Overhead utility hazards

- Underground utility hazards
- Traffic/ motor vehicle hazards
- Hazards associated with exposure to various chemicals
- Slip, trip and fall hazards
- Pinch point hazards
- Compressed gas hazards
- Fire hazards from hot work (e.g., grinding)
- Gasoline hazards (filling generator)

- SEI staff and others under contract with SEI that may be present on-site will wear a hardhat whenever overhead hazards are present.
- Appropriate eye protection should be worn.
- Hearing protection shall be worn whenever the drill rig is actively advancing profiling equipment.
- Steel toed boots will be worn.
- SEI staff will read the site health and safety plan (HASP) prior to beginning a project.

Additional personal protective equipment shall be worn in accordance to the health and safety document.

4.0 PROCEDURES

Stabilization of indicator field parameters is used to indicate that conditions are suitable for sampling to begin. Achievement of turbidity levels of less than 5 NTU and stable draw-downs of less than 0.3 feet, while desirable, are not mandatory. Sample collection may still take place provided the remaining criteria in this procedure are met. If after four (4) hours of purging, indicator field parameters have not stabilized, one of the following three courses of action may be taken: a) discontinue purging, collect samples and provide full explanation of attempts to achieve stabilization (note: there is a risk that the analytical data obtained, especially metals and strongly hydrophobic organic analyses, may not meet the sampling objectives), b) continue purging until stabilization is achieved, c) discontinue purging, do not collect any samples, and record in study or project records that stabilization could not be achieved (documentation must describe attempts to achieve stabilization).

4.1 Equipment

1. Extraction device. Adjustable rate, submersible pumps should be used (for example, centrifugal or bladder pump constructed of stainless steel or Teflon.
2. Tubing. Fluorinated Ethyl Propylene (FEP) will be used when sampling is to include VOCs, SVOCs, pesticides, PCBs and inorganics. Stainless steel tubing may be used when sampling for VOCs, SVOCs, pesticides, and PCBs. However, it should be used with caution when

sampling for metals. The use of ¼-inch or ⅜-inch (inner diameter) tubing is preferred. This will help ensure the tubing remains liquid filled when operating at very low pumping

3. Slope Indicator or other water level measuring device. Water level measuring device(s), capable of measuring to 0.01 foot accuracy (electronic "tape", pressure transducer). Recording pressure transducers, mounted above the pump, are especially helpful in tracking water levels during pumping operations, but their use must include check measurements with a water level "tape" at the start and end of each record.
4. Graduated cylinder or other volumetric lab ware to determine purge volume.
5. Power source (generator, nitrogen tank, etc.) appropriate for pump. If a gasoline generator is used, it must be located downwind and at least 30 feet from the well so that the exhaust fumes do not contaminate the samples.
6. Indicator field parameter monitoring instruments (pH, oxidation-reduction potential (ORP), dissolved oxygen (DO), turbidity, specific conductance, and temperature). Use of a flow-through cell is required when measuring all listed parameters. Standards to calibrate the water quality instrument should be brought to the site for calibration prior to use. Refer to the manufacturer's operation manual for maintenance and calibration procedures for the water quality instrument and to SEI 10.7.1 operation of the YSI 600 XL.

4.2 Preliminary Site Activities

1. Check for security damage or evidence of tampering on each well, record pertinent observations.
2. Remove well cap and immediately measure VOCs at the rim of the well with a PID or FID instrument and record the reading in the logbook.
3. If the well casing does not have a reference point (usually a V-cut or indelible mark in the well casing), make one. Describe its location and record the date of the mark in the logbook.
4. Check newly constructed wells for the presence of NAPL before the initial sampling round. Note: Procedures for collection of LNAPL or DNAPL samples are not addressed in this SOP.

4.3 Purging and sampling procedures

Sampling wells in order of increasing chemical concentrations (known or anticipated) is preferred. If applicable, refer to the site Quality Assurance Project Plant (QAPP) for additional sample information including frequency, preservation methods, containers, and specific analytical methods.

4.3.1 Install Pump

Lower pump and any safety cables, tubing and/or electrical lines slowly (to minimize disturbance) into the well to the midpoint of the zone to be sampled. In most instances, the pump will be positioned in the middle of the well screen. For cases where the well screen straddles the water table, the pump should be positioned 2 feet above the bottom of the well.

4.3.2 Measure Water Level

Before starting pump, measure water level. If a recording pressure transducer is used, initialize starting condition. After recording the height of the water column in the well, determine the volume in gallons by the following formula:

Volume in gallons = (Height of water column $\times$ π*Radius^2) $\times$ 7.4805

When using the above formula, all measurements (height and radius) must be in feet.

4.3.3 Purge Well

4.3.3.1 Initial Low Stress Sampling Event

Start 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 draw-down (less than 0.3 feet). If the minimal draw-down that can be achieved is greater than 0.3 feet, continue purging until indicator field parameters stabilize.

Monitor and record water level and pumping rate every three to five minutes (or as appropriate) during purging. 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 fifteen minutes of pumping in order to help minimize purging time. During pump start-up, drawdown may exceed the 0.3 feet target and then "recover" as pump flow adjustments are made.

Using the appropriate volumetric container (one that is scalable relative to the volume to be purged), measure the volume of water purged at the same interval as field parameters are recorded (Section 4.3.4). If water is or believed to be contaminated, purge water must be containerized as specified in the site work plan. Purge volume calculations should utilize stabilized drawdown value, not the initial drawdown. Do not allow the water level to fall to the intake level (if static water level is above the well screen, avoid lowering the water level into the screen). The final purge volume should be greater than the stabilized drawdown volume plus the extraction tubing volume. This may not be possible in wells with low recharge rates.

Wells with low recharge rates may require the use of special pumps capable of attaining very low pumping rates (bladder, peristaltic) and/or the use of special techniques. If the recharge rate of the well is lower than the pump's minimum extraction rate, then continue to purge until the drawdown is near (but not below) the top of the well screen. Once the drawdown is within six (6) inches of the top of the well screen, discontinue purging. The well should be sampled as soon as the water level has recovered sufficiently to collect the appropriate volume needed for all anticipated samples (the intake should not be moved during this recovery period). In this case only, samples may be collected even if the well was not purged for four

(4) hours and the indicated field parameters did not stabilize (as instructed in Section 4.0).

4.3.3.2 Subsequent Low Stress Sampling Events

Prior to sampling, check intake depth and drawdown information from previous sampling event(s) for each well. Duplicate, to the extent practicable, the intake depth and extraction rate (use the final pump dial setting information) from previous event(s). Perform purging operations as above

4.3.4 Monitor Indicator Field Parameters

During well purging, monitor indicator field parameters (turbidity, specific conductance, pH, ORP, DO) every 3 to 5 minutes. Values are to be recorded on appropriate field forms or on Observation and Remark (O&R) forms. Note: during the early phase of purging, emphasis should be put on minimizing and stabilizing pumping stress, and recording those adjustments. Purging is considered complete and sampling may begin when all the above indicator field parameters have stabilized. Stabilization is considered to be achieved when three (3) consecutive readings, taken at three (3) to five (5) minute intervals, are within the following limits:

- Turbidity – 10% (if values are greater than 1 NTU)
- DO – 10%
- Specific conductance – 3%
- Temperature – 3%
- pH - +/- 0.1 units
- ORP - +/- 10 millivolts

Percentage difference should be calculated using the following formula:

(Absolute Value (Previous Value – Updated Value) ÷ Previous Value) × 100

All measurements should be obtained using a flow-through-cell. Transparent flow-through-cells are preferred, because they allow field personnel to watch for particulate build-up within the cell. This build-up may affect indicator field parameter values measured within the cell. If the cell needs to be cleaned during purging operations, continue pumping and disconnect cell for cleaning, then reconnect after cleaning and continue monitoring activities.

The flow-through-cell must be designed in a way that prevents air bubble entrapment in the cell. When the pump is turned off or cycling on/off (when using a bladder pump), water in the cell must not drain out. Monitoring probes must be submerged in the water at all times. If two flow-through-cells are used in series, the one with the dissolved oxygen probe should come first (this parameter is most susceptible to error if air leaks into the system).

4.3.5 Collect Water Sample

Water samples for laboratory analyses must be collected before water has passed through the flow-through-cell (use a by-pass assembly or disconnect cell to obtain the sample).

VOC samples should be collected first and directly into the sample containers. Sample water should be allowed to fill sample containers gently down the inside of the container with minimal turbulence.

During purging and sampling, the tubing should remain filled with water so as to minimize possible changes in water chemistry upon contact with the atmosphere. It is recommended that ¼-inch or ⅜-inch (inside diameter) tubing be used to help insure that the tubing remains water filled. If the pump tubing is not completely filled to the sampling point, use one of the following procedures to collect samples:

1. Add clamp, connector (Teflon or stainless steel) or valve to constrict sampling end of tubing;
2. Insert small diameter Teflon tubing into water filled portion of pump tubing, allowing the end to protrude beyond the end of the pump tubing, collect samples from small diameter tubing;
3. Collect non-VOC samples first, then increase flow rate slightly until the water completely fills the tubing, collect sample and record new drawdown, flow rate and new indicator parameter values.

The third technique (least favorable because VOC samples should be collected first) allows the drawdown in the well to remain constant while all other samples are being collected. If flow is increased to collect VOC samples, do not allow the drawdown in the well to drop below the top of the well screen. Note that any two, or all three, techniques may be used in conjunction to achieve the desired results.

Add preservative, as required by analytical methods, to samples immediately after they are collected if the sample containers are not pre-preserved.

Label each sample as collected. Samples requiring cooling (VOCs, organics, cyanide etc.) will be placed in a cooler with ice or refrigerant for delivery to the laboratory. Metal samples after acidification to a pH less than 2 do not need to be cooled.

4.3.6 Posting Sampling Activites

After collection of the samples, the pump tubing may be dedicated to the well for re sampling (by hanging the tubing inside the well), decontaminated, or properly discarded.

Before securing the well, measure and record the well depth (to 0.01 feet), if not measured during the monthly water level measurements. Note: measurement of total well depth is optional after the initial low stress sampling event. However, it is recommended if the well has a “silting” problem or if confirmation of well identity is needed.

Secure the well.

4.4 Decontamination

Refer to SEI -5.1.5 *Maintenance and Decontamination of Field Equipment* for further information regarding the decontamination of field equipment. Decontamination procedures specific to low stress sampling are described below.

Decontaminate sampling equipment prior to use in the first well and following sampling of each subsequent well. The pump will not be removed between purging and sampling operations. The pump and tubing (including support cable and/or electrical wires which are in contact with the well and/or groundwater) will be decontaminated by the procedure described below.

4.4.1 Procedure

The decontaminating solutions can be pumped from either buckets or short PVC casing sections through the pump or the pump can be disassembled and flushed with decontaminating solutions. It is recommended that detergents and isopropyl alcohol be used sparingly in the decontamination process and water flushing steps be extended to ensure that any sediment trapped in the pump is removed. The pump exterior, safety cables, electrical wires and other re-usable parts must be rinsed with the decontaminating solutions, as well. The procedure is as follows.

1. Flush the equipment with potable water.
2. Flush with non-phosphate detergent solution. If the solution is recycled, the solution must be changed periodically.
3. Flush with potable or distilled/deionized water to remove all of the detergent solution. If the solution is recycled, the solution must be changed periodically.
4. Flush with methanol (pesticide grade). If equipment blank data from previous sampling event show that the level of contaminants is insignificant, then this step may be skipped.

Flush with analyte-free water. The final water rinse must not be re-used for additional decontamination procedures.

4.5 Quality Assurance/Quality Control

QA/QC samples (i.e. trip and equipment blanks and field duplicates) will be collected in accordance to the site Quality Assurance Project Plan, if appropriate or as specified in the site work plan.

Quality control samples are required to verify that the sample collection and handling process has not compromised the quality of the groundwater samples. All field quality control samples must be prepared the same as regular investigation samples with regard to sample volume, containers, and preservation.

Collect samples in order from wells with lowest contaminant concentration to the highest. Collect equipment blanks after sampling contaminated wells and not after background wells.

4.6 Documentation

A record of the sample collection must be made in the field at the time the sample is collected. The record should be made in a field notebook, a sample collection form, or other medium acceptable according to the study protocol. The information to be recorded must, at a minimum, include the following:

- Date and time of sample collection;
- Name(s) of the personnel performing the sampling;
- Weather;
- Sample location, well identification, and / or sample identification;
- Project/study designation;
- Water level prior to purging (equilibrium level);
- Total Well Depth;
- Well volume calculations;
- Volume purged from the well prior to sampling;
- Observations (I.E. PID reading or presence of NAPL); and
- Equilibrated chemical parameters monitored in the field

5.0 RESPONSIBILITIES

Stone staff are required to take accurate and descriptive notes. Variations from this SOP should be noted on observations and remarks (O&R) forms along with data and personnel.

Field staff members are responsible for following and implementing all procedures outline within this SOP. Care shall be taken to avoid compromising sample and data integrity.

6.0 DEFINITIONS

1. *pH-* The log-normal activity (concentration) of Hydrogen Ion (H^+) and Hydroxide Ion (OH^-) in solution. Ranges from 0 (acidic) to 7 (neutral) to 14 (basic).
2. *Conductivity-* The ability of water to conduct electricity as a function of dissolve ionic species, suspended solids, and organic content. Usually measured in Siemans (S).
3. *Turbidity-* The amount of suspended solids in a liquid.
4. *EPA-* The United States Environmental Protection Agency
5. *VOC*-Volatile Organic Compound.
6. *SVOC*-Semi-volatile Organic Compound.
7. *Stabilization-* A condition that is achieved when all indicator field parameter measurements are sufficiently stable (as described in Subsection 4.3.4, "Monitor Indicator Field Parameters") to allow sample collection to begin.

7.0 REFERENCES

U.S. Environmental Protection Agency, Region 1, 2010, *Low Stress (low flow) Purging and Sampling Procedure for the Collection of Groundwater Samples from Monitoring Wells, Revision 3, January 19.* p-30

8.0 TABLES, DIAGRAMS, FLOWCHARTS, AND VALIDATION DATA

None

9.0 AUTHORIZATION

Revised by:_____ Date:_____

Joseph Schmidl, P.G, Senior Project Manager

Approved by:_____ Date:_____

Christopher T. Stone, President

10.0 REVISION HISTORY

None

STANDARD OPERATING PROCEDURE

SEI-6.45.0

Collection, Handling, and Preservation of Discrete Soil Samples

SOP Number: SEI-6.45.0
Revision Number: 0

Date Issued: 08/27/12
Date of Revision: NA

1.0 OBJECTIVE

The purpose of this procedure is to provide instruction for the collection, handling, and preservation of discrete soils for various analyses including, but not limited to: the potential presence of Volatile Organic Compounds (VOCs) along with representative samples for physical property analysis including porosity, bulk density, and fraction of organic carbon content (*foc*), Semi-Volatile Organic Compounds (SVOCs), Polycyclic Aromatic Hydrocarbons (PAHs), Metals, and Polychlorinated Biphenyls (PCBs).

VOC samples need to be obtained, logged, and preserved into methanol with a minimal loss of volatile analytes.

2.0 POLICIES

1. According to 40 CFR Part 160, Subpart E, Section 160.81 and the NELAC standards, a testing facility shall have standard operating procedures in writing setting forth study methods that management is satisfied are adequate to insure the quality and integrity of the data generated in the course of a study.
2. Personnel will legibly record data and observations in the field to enable others to reconstruct project events and provide sufficient evidence of activities conducted.

3.0 SAFETY ISSUES

1. If necessary and appropriate, a site-specific health and safety plan shall be created for each study site. A template for creating a proper health and safety plan is provided on the SEI network.
2. Care must always be taken when approaching a sampling location. Do not, under any circumstances, place yourself in danger to collect a sample.
3. If necessary and appropriate, all chemicals are required to be received with Material Safety Data Sheets (MSDS) or appropriate application label. These labels or MSDS shall be made available to all personnel involved in the sampling and testing.

4.0 PROCEDURES

4.1 Equipment and Materials

1. For VOCs: Pre-weighed VOA vials with screw cap and silicone septum containing 10ml of purge-and-trap grade methanol (MSDS, Attachment 1). Appropriate sample containers as needed for other analytes

2. For VOCs: Sample collection device for minimally disturbed delivery of sample such as:
    - Stainless steel or disposable plastic syringe with barrel cut-off and smaller than vial opening
    - Stone's custom made stainless steel soil samplers with removable plungers
    - Encore piston sampler

3. Stainless steel spoons and bowls

4. Hand auger or other manual soil sampling tool

5. Portable balance, accurate to 0.01 grams, and calibration weights for vial preparation

6. Decontamination supplies if reusing stainless steel sampling tools: distilled water, alconox, scrub brushes, buckets, methanol if specified in project work plan

7. Field notebook, sample collection form, observation and remark form, or other acceptable medium for recording field notes. Ballpoint, indelible pens

8. Portable photoionization detector (PID) with calibration gas

9. Hacksaw

10. Soil core caps of appropriate diameter for the drill tooling being used to retrieve cores

11. Plastic wrap and zip top bags

12. Shipping materials: coolers with ice, bubble wrap or other packaging materials, packing tape

13. Measuring tape

14. As necessary or as described in the Site HASP appropriate personnel protection equipment (PPE) such as gloves, hardhat, hearing protection and safety glasses (minimal).

15. Chain of custody records and custody seals

4.2 Vial Preparation for VOC Sample Collection

VOC samples will be collected in 40 mL clear glass VOA vials with Teflon-lined septa and screw caps. This sampling requires purge and trap grade methanol for sample preservation. Methanol should be ordered before the start of field work and vials should be prepared in the laboratory prior

to embarking into the field. If vials are prepared in the field it should be in a controlled environment (e.g. with an appropriate balance on a flat surface and in a room free of wind). The following outlines the steps necessary to prepare vials for VOC sample collection of soils:

1. Affix sample label and label each sample container sequentially (e.g. VOC-001, VOC-002, etc.). Label the lid of each sample vial with the same ID as written on the label. Affixing the sample label prior to the sampling event assures the weight of the label will be included in the tare weight of the vial.

2. Check calibration of the balance being used to weigh vials. Record this calibration check in the appropriate calibration logbook.

3. Weigh each empty vial with affixed label and record weights on appropriate forms.

4. Add 10ml of purge and trap grade methanol to each vial, screwing the cap back on each vial after filling it.

5. Re-weigh each vial once 10ml of purge and trap grade methanol has been added to accurately determine the weight and volume of methanol extract. Record these weights on appropriate forms.

6. Sample bottles will be stored at the laboratory in a freezer set between -10°C and -20°C until they are deployed into the field where the will be stored in a cooler with ice or fridge set to 4°C until samples are collected and are shipped to the laboratory.

Manufacturer, lot numbers, and other pertinent information of the methanol used to prepare vials will be recorded on appropriate forms. Recording this information enables Stone to track batches of methanol in the event contamination has partitioned into the methanol from various environmental sources.

4.3 Soil Core Retrieval, Handling, and VOC Sample Collection

Soil cores may be retrieved by several different methods, it is important to select a method that recovers soil cores representative of *in-situ* conditions and minimizes losses of VOCs. Examples of soil coring methods include, but are not limited to Geoprobe® Systems MC5, DT45, DT325/35, or DT22 coring tools, split spoon samplers, and AMS multi-stage soil core samplers. Refer to project specific protocols to determine which soil coring method will be utilized.

The rate of drilling and soil core retrieval shall not exceed the rate at which the sampler can collect samples and describe each core following SEI-6.42.*n Geologic Description of Unconsolidated Deposits*. This will prevent soil cores from being exposed to the atmosphere for extended periods of time prior to sampling, greatly reducing VOC losses.

Upon retrieval of soil cores the following steps shall be followed, working quickly to minimize the loss of VOCs:

1. Cores will be placed on a clean sampling surface, liners or spilt spoons shall be opened to reveal the core, and the core shall be photographed with labels indicating the site ID, borehole ID, depth interval, and recovery.

2. A portable PID will be utilized to screen the soil cores every 6 inches for the possible presence of VOCs. PIDs shall be calibrated daily prior to measurements according to manufactures instructions (SEI-6.19.*n*).

3. VOC sample frequency will follow site specific protocols. In general VOC samples will be collected from strata exhibiting the highest PID responses.

4. Using an appropriate sample device (see section 4.1 of this SOP), collect approximately 10-12 grams (g) of sample as soon as possible after the surface of the soil has been exposed to the atmosphere, generally within a few minutes at most. To the extent practical, samples should be obtained from undisturbed portions of the soil core.

5. Using the sample collection device, add the approximately 10-12g of sample to a pre-prepared sample vial containing purge-and-trap grade methanol.

6. Wipe any soil off the vial threads and immediately seal the vial with the septum and screw cap.

7. If project-specific work plan(s) specify, use a portable balance to weigh the sealed vial containing the sample (after either noting the laboratory-indicated weight or determining the tare weight of the vial in the field) to ensure 10-12g of sample was added. Samples will also be weighed upon receipt at the laboratory to determine sample mass.

8. Using the laboratory supplied label already fixed to the vials, label sample. Do not affix additional labels or custody seals to vials, as this will alter the tare weight.

9. Store samples on ice at 4ºC until delivered to the analytical laboratory.

10. A trip blank comprised of a blank methanol vial should be supplied by the laboratory, and shall be submitted back to the laboratory with each sample delivery group.

11. A separate 10-12g aliquot of soil should be collected for moisture content determination using the same sampling device and methods described above, and placed in an un-preserved container. Label the moisture content sample appropriately.

12. Upon completion of VOC sample collection soil cores shall be described for color, texture, consistence, structure, and moisture, and other parameters as called for in site specific protocols following SEI-6.42.*n*. Collect discrete soil samples for other analytical parameters.

13. Decontaminate reusable sampling equipment prior to reuse according to site specific procedures and/or SEI-5.1.*n Maintenance and Decontamination of Field Equipment.*

4.4 Physical Properties Sample Collection

Soil cores will be retrieved following procedures described in section 4.3 of this SOP. Refer to site specific protocols for sample frequency. However, in general, an effort will be made to obtain samples from different stratigraphic units where variations in stratigraphy as assessed during core logging.

An effort shall be made to collect physical property samples from lesser contaminated sections of coil cores as determined by PID screening.

Each sample will be a cylindrical disc of the same diameter as the soil core retrieved, with a height of approximately 4 to 6 inches. These sections of core will be obtained such that the *in-situ* moisture conditions are retained. Soil samples will be collected for physical property analyses (porosity, bulk density, and *foc*) in the following manner:

1. Select a section of soil core to be sampled
2. Use a hacksaw to cut through plastic core liners on both ends of the desired sample. If split spoons were used carefully roll the soil core onto a clean piece of plastic wrap and remove unwanted soil with a gloved hand or knife.
3. Wrap sample tightly in clean plastic wrap. Tape plastic wrap to secure.
4. If available, place proper size end caps on both ends of sample to secure soil. If not available use additional plastic wrap to secure the core sample.
5. Clearly label each sample with the sample ID, location, depth, and date. Secure label with packing tape and wrap in bubble wrap or other padding.
6. Place samples individually in zip top polyethylene bags.
7. Store samples on ice at 4ºC until delivered to the analytical laboratory.

4.5 Discrete Soil Sample Collection for Other Analytical Parameters

Discrete soil samples may be collected for other analytical parameters including, but not limited to, SVOCs, PAHs, Metals, and PCBs. These samples may be collected from soil cores retrieved from the subsurface using drilling techniques discussed in section 4.3 of this SOP or from surface or shallow soils collected using hand tools, such as a hand auger or AMS multi-stage soil core sampler. These discrete soil samples shall be collected following these steps:

1. Prior to fieldwork contact the laboratory conducting sample analysis and procure appropriate containers for the specific study. Container volume and amount of soil needed may vary by the analyses being conducted.
2. Using a stainless steel spoon or trowel collect the appropriate amount of soil needed for the analyses being performed into the appropriate sample containers provided by the laboratory.
3. Clearly label each sample with the sample ID, location, depth, and date. Secure label with packing tape and wrap in bubble wrap or other padding.
4. Store samples on ice at 4°C until delivered to the analytical laboratory.
5. Decontaminate reusable sampling equipment prior to reuse according to site specific procedures and/or SEI-5.1.*n Maintenance and Decontamination of Field Equipment*.

5.0 RESPONSIBILITIES

1. All personnel will legibly record data and observations (including phone conversations) in accordance with this SOP to enable others to reconstruct project events and provide sufficient evidence of activities conducted.

6.0 DEFINITIONS

1. *Observations & Remarks Form (O&R):* A pre-printed form, which contains mostly blank space for general note taking. The form typically prompts the user for the study or project designation, the SEI project number, the client or sponsor name, the total number of pages (page *n* of n) and requires a signature and date. The form is generally used to capture notes of one person when another, more specific forms is not available.

2. *Raw data* means any worksheets, records, memoranda, notes, or exact copies thereof, that are the result of original observations and activities of a study and are necessary for the reconstruction and evaluation of the report of that study. In the event that exact transcripts of raw data have been prepared (e.g., tapes which have been transcribed verbatim, dated, and verified accurate by signature), the exact copy or exact transcript may be substituted for the original source as raw data. Raw data may include photographs, microfilm or microfiche copies, computer printouts, magnetic media, including dictated observations, and recorded data from automated instruments.

3. *VOCs:* Volatile Organic Compounds

4. *SVOCs:* Semi-Volatile Organic Compounds

5. *PAHs*: Polycyclic Aromatic Hydrocarbons

6. *PCBs*: Polychlorinated Biphenyls

7. *PID*: Photoionization Detector

8. *MSDS*: Material Safety Data Sheet

9. *foc*: Fraction of Organic Carbon

7.0 REFERENCES

40 CFR Part 160 Good Laboratory Practice Standards, August, 1989.

8.0 TABLES, DIAGRAMS, FLOWCHARTS, AND VALIDATION DATA

None

9.0 AUTHORIZATION

Written by: _____ Date: _____

Lee Rosberg, Staff Scientist

Approved by: _____ Date: _____

Seth Pitkin, Vice President

10.0 REVISION HISTORY

Not applicable.

Attachment I
Methanol MSDS

STANDARD OPERATING PROCEDURE

SEI-6.46.0

GEOLOGIC DESCRIPTION OF UNCONSOLIDATED DEPOSITS USING THE UNIFIED SOIL CLASSIFICATION SYSTEM

SOP Number: SEI-6.46.0 Date Issued: 08/28/12
Revision Number: 0 Date of Revision: NA

1.0 OBJECTIVE

The objective of this procedure is to provide a standardized geological method of describing the nature of unconsolidated deposits, commonly referred to as soils or sediments, using the Unified Soil Classification System (USCS). A description of unconsolidated geologic deposits is often part of site investigations as a critical element in understanding the properties that govern the movement of water and other fluids, including contaminants. Samples of unconsolidated deposits are typically collected with a split-spoon sampler or other soil coring tools. These samples are then described to aid in interpretations about the geologic and hydrologic characteristics of the site. The characteristics of major importance for this type of description are: soil name, particle size disibution, gradation or plasticity, dry strength, dilatancy, toughness, color, odor, soil texture and structure, moisture content, relative density or consistency, cementation, relative permeability, local geologic name, and group symbol.

The USCS was developed for the US Army Corps of Engineers and Bureau of Reclamation for classifying soils for engineering purposes based on laboratory determination of particle size, liquid limit and plasticity index. It was first used to judge a soil's suitability as a subgrade for roads and airfields, but it is used today for most engineering applications of soil. It differentiates soils into three major divisions: coarse-grained, fine-grained and highly organic soils as shown in Attachment 1. Fine-grained soils are classified as those that will pass through a No. 200 U.S. standard sieve (0.075 mm). Organic material is a common component of soil but it has no size range. Each type of soil is given a two-letter designation based primarily on its particle-size distribution (texture), Atterberg limits, and organic matter content. Attachments 1 and 2 describe the USCS (reference).

2.0 POLICIES

1. The Staff Scientist will follow the USCS method of describing the nature of the unconsolidated deposits and will document their fieldwork in a field notebook for all site investigations.
2. Staff Scientists will be properly trained in site investigations prior to conducting a site investigation as documented in their training record on file at Stone Environmental, Inc.

3.0 SAFETY ISSUES

Staff following the methodology outlined in this SOP will read the site Health and Safety plan for the individual project, as well as appropriate Material Safety Data Sheets (MSDS).

4.0 PROCEDURES

4.1 Equipment

1. A notebook computer, field notebook, logging forms or other acceptable recording medium.
2. Indelible pens
3. A measuring rule in feet and tenths and hundreds of feet (not inches)
4. Measuring devices, such as engineers scale, electronic balances, electronic water level indicator, interface probe, etc.
5. A rule in mm for measuring particle sizes
6. A grain size key
7. Hand lens
8. Diluted Hydrochloric acid (HCl)
9. Knife or other appropriate tool for handling soil
10. Clean (new) poly/plastic sheeting or other material on which collected samples may be examined/preserved.
11. Appropriate personal protective equipment.
12. Sample containers, forms, labels, markers, preservatives and shipping materials.

4.2 Soil Description Procedures

4.2.1 Separating Significant Layers

Each distinct sediment layer in the split spoon or other type of sample should be described separately. Layers may be differentiated based on any of the characteristics listed in section 1.0. It may be most appropriate to describe layers less than 0.5 inches thick as laminae. In a situation where several laminae of similar properties are located in a sample, it may be best to describe them as a group. For example, 8 gray silty clay laminae between 5.5 and 6.5 feet, each about 1/4 inch thick. The following sections discuss each of the soil characteristics to be described.

4.2.1.1 Angularity

Describe the angularity of the sand (coarse sizes only), gravel, cobbles, and boulders, as angular, subangular, subrounded, or rounded in accordance with the criteria in Table 3 and Fig. 1, below. A range of angularity may be stated, such as: subrounded to rounded.

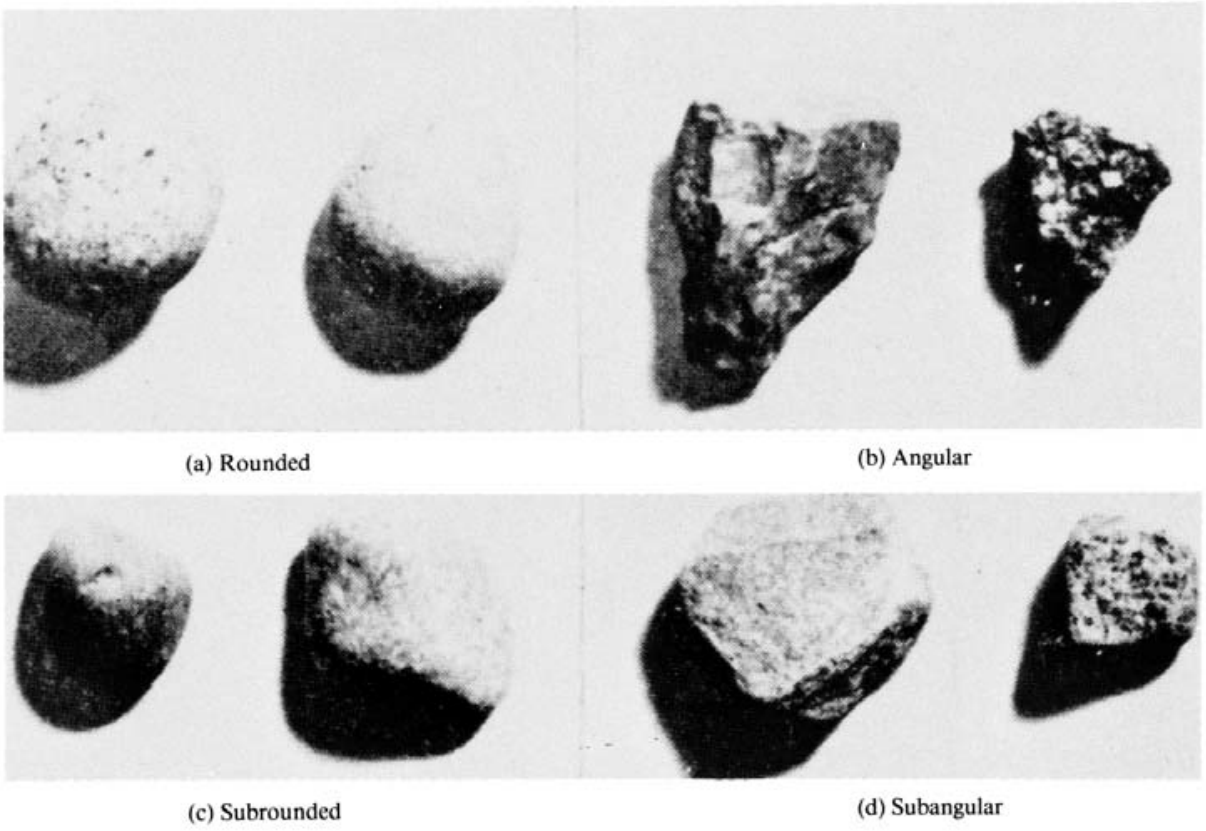
Table 3

Criteria for Describing Angularity of Coarse-Grained Particles (See Fig.1)

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

Figure 1

Typical Angularity of Bulky Grains



4.2.1.2 Shape

Describe the shape of the gravel, cobbles, and boulders as flat, elongated, or flat and elongated if they meet the criteria in Table 4 and Fig. 2, below. Otherwise, do not mention the shape. Indicate the fraction of the particles that have the shape, such as: one-third of the gravel particles are flat.

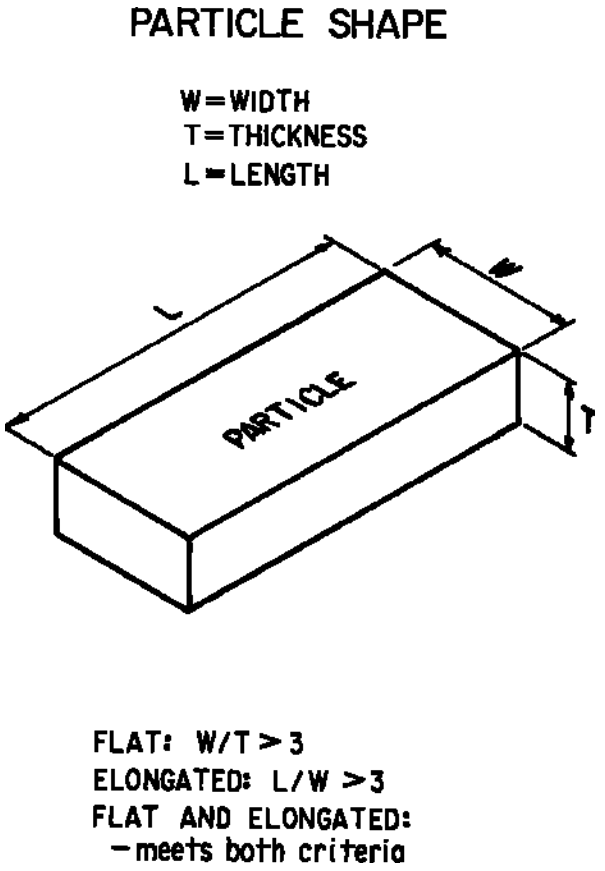
Table 4

Criteria for Describing Particle Shape (see Fig. 2)

The particle shape shall be described as follows where length, width, and thickness refer to the greatest, intermediate, and least dimensions of a particle, respectively.	
Flat	**Particles with width/thickness > 3**
Elongated	**Particles with length/width > 3**
Flat and Elongated	**Particles meet criteria for both flat and elongated**

Figure 2

Criteria for Particle Shape



4.2.1.3 Color

Describe the color. Color is an important property in identifying organic soils, and within a given locality it may also be useful in identifying materials of similar geologic origin. If the sample contains layers or patches of varying colors, this shall be noted and all representative colors shall be described. The color shall be described for moist samples. If the color represents a dry condition, this shall be stated on the soil boring log.

4.2.1.4 Odor

Describe the odor if organic or unusual. Soils containing a significant amount of organic material usually have a distinctive odor of decaying vegetation. This is especially apparent in fresh samples, but if the samples are dried, the odor may often be revived by heating a moistened sample. If the odor is unusual (petroleum product, chemical, and the like), it shall be described.

4.2.1.5 Moisture Condition

Describe the moisture conditionas dry, moist, or wet, in accordance with the criteria in Table 3, below.

Table 3

Criteria for Describing Moisture Condition

Description	Criteria
Dry	Absence of moisture, dusty, dry to the touch
Moist	Damp but no visible water
Wet	Visible free water, usually soil is below water table

4.2.1.6 HCL Reaction

Describe the reaction with HCl as none, weak, or strong, in accordance with the critera in Table 4, below. Since calcium carbonate is a common cementing agent, a report of its presence on the basis of the reaction with dilute hydrochloric acid is important.

Table 4

Criteria for Describing the Reaction with HCl

Description	Criteria
None	No visible reaction
Weak	Some reaction, with bubbles forming slowly
Strong	Violent reaction, with bubbles forming immediately

4.2.1.7 Consistency

For intact fine-grained soil, describe the consistency as very soft, soft, firm, hard, or very hard, in accordance with the criteria in Table 5, below. This observation is inappropriate for soils with significant amounts of gravel.

Table 5

Criteria for Describing Consistency

Description	Criteria
Very Soft	Thumb will penetrate soil more than 1 in. (25 mm)
Soft	Thumb will penetrate soil about 1 in. (25 mm)
Firm	Thumb will indent soil about 1⁄4 in. (6 mm)
Hard	Thumb will not indent soil but readily indented with thumbnail
Very Hard	Thumbnail will not indent soil

4.2.1.8 Cementation

Describe the cementation of intact coarse-grained soils as weak, moderate, or strong, in accordance with the criteria in Table 6, below.

Table 6

Criteria for Describing Cementation

Description	Criteria
Weak	Crumbles or breaks with handling or little finger pressure
Moderate	Crumbles or breaks with considerable finger pressure
Strong	Will not crumble or break with finger pressure

4.2.1.9 Structure

Describe the structure of intact soils in accordance with the criteria in Table 7, below.

Table 7

Criteria for Describing Structure

Description	Criteria
Stratified	Alternating layers of varying material or color with layers at least 6 mm thick; note thickness
Laminated	Alternating layers of varying material or color with the layers less than 6 mm thick; note thickness
Fissured	Breaks along definite planes of fracture with little resistance to fracturing
Slickensided	Fracture planes appear polished or glossy, sometimes striated
Blocky	Cohesive soil that can be broken down into small angular lumps which resist further breakdown
Lensed	Inclusion of small pockets of different soils, such as small lenses of sand scattered through a mass of clay; note thickness
Homogenous	Same color and appearance throughout

4.2.1.10 Range of Particle Sizes

For gravel and sand components, describe the range of particle sizes within each component as defined in 3.1.2 and 3.1.6. For example, about 20 % fine to coarse gravel, about 40 % fine to coarse sand.

The consistence of the sediment will be described whenever it remains evident in the split spoon sample. The sample may be compacted excessively or otherwise disturbed during sampling and the natural consistence may not be evident during examination. If the

consistence can be characterized, it will be described according to the terms and definitions listed in Attachment 4.

The key distinction for this parameter is the change from friable to firm. When handling a sample that is friable, it will take only slight pressure between the thumb and fingers to cause a clod or ped of the sediment to break apart. A sample that is firm will resist crushing to the point where it finally seems to explode when it is subjected to adequate pressure. A friable clod or ped will not explode or pop, it will simply fall apart when pressure is applied. The other descriptive classes, as show in Attachment 4, will be applied to add more information as to the degree of soil stability, or lack thereof.

4.2.1.11 Structure

Observations of structure will include a characterization of the orientation and continuity of contacts (e.g., sharp or gradational), angularity of grains (see Attachment 3), presence of fractures, cracks, partings or macropores and a qualitative description of the degree of sorting (poorly sorted means many different grain sizes, well sorted means uniform grain size). Additional description of such as features as crossbeds, grading, ripples and varves will aid in identifying the depositional environment.

4.2.1.12 Moisture

The soil moisture will be described on the basis of visual and manual field observations. The moisture class will be based on the percent water in the soil as follows:

Moisture Class	Moisture Content (%)	Field Description
Dry	0	No dampness is evident in sample.
Humid	1-25	Slight damp mark seen on hand when sample is squeezed.
Moist	26-75	Grading from humid to wet.
Wet	76-99	Water is released when sample is squeezed.
Saturated	100	Water drips freely from sample.

4.2.1.13 Concretions

When iron or manganese concretions are present in a soil sample, they will be noted as to their abundance and size. The use of dilute hydrochloric acid is helpful in identifying the presence of carbonate minerals, though its use is not required.

4.2.1.14 Other Parameters

The protocols or work plan for a given study may require the determination of other parameters, e.g., screening for the presence of volatile organic compounds with a photoionization detector at a hazardous waste investigation. In such cases, the protocol, work plan, additional SOPs, or other authority or regulation will describe the additional or alternate method of sediment evaluation.

4.3 Data Recording

The information required by this SOP will be recorded in a field book during the sampling process. Required information includes:

Project name

SEI study number

Site name

Well/Profile ID number

Name of geoscientist

Driller's name

Date of exploration

Attachment 5 presents a format for recording field data as well as examples of the descriptors to be used. All of the data fields must be addressed for each discrete layer being described. All measurements should be in feet and tenths of feet (not inches). The lengths of the segments of the particular sample being described must add up to the linear footage of recovery noted. In the interest of standardization, if less than complete recovery is achieved it should be assumed that the missing portion of the sample interval is at the bottom of the sample.

5.0 RESPONSIBILITIES

5.1 Principals/Group Officers/Group Leaders/Personnel

Any staff conducting these procedures are responsible for signing, dating, and managing all field observation forms.

6.0 DEFINITIONS

1. *Concretions* A nodular or irregular concentration of certain authigenic constituents of sedimentary rocks and tuffs; developed by the localized deposition of material from solution, generally about a nucleus. Harder than enclosing rock.
2. *Lamina (ae)* Unit layer or sheet of sediment in which the stratification planes are one centimeter or less apart. Laminae need not be parallel to bedding. When they are at an angle to the bedding planes the term "cross-lamination" may be used.

7.0 REFERENCES

Davis, Richard A. Jr., 1992, Depositional Systems: An Introduction to Sedimentology and Stratigraphy, 2nd Edition, Prentice Hall, New Jersey, 604 p.

Hunter/BCA. Standard Operating Procedure No. 6006, Identification of Soils

American Geological Institute, 1976, Dictionary of Geological Terms, Revised Edition, Anchor Press/Doubleday, Garden City, New York, 472 p.

Laboratory Manual for Hand Specimen Petrology, Department of Geology, University of Texas [Undated].

8.0 TABLES, DIAGRAMS, FLOWCHARTS, AND VALIDATION DATA

1. Table 1: Unified Soil Classification System Description #1
2. Table 2: Unified Soil Classification System Description #2
3. Table 3: Criteria for Describing Angularity of Coarse-Grained Particles
4. Table 4: Criteria for Describing Particle Shape
5. Figure 1:

9.0 AUTHORIZATION

Revised by: _____ Date:_____

Lee Rosberg, Project Scientist

Approved by:_____ Date:_____

Christopher T. Stone, President

10.0 REVISION HISTORY

Revision 1

1. Renumbered from Chapter SEI-10.11.1

Revision 2

1. Changed title from geologic description of unconsolidated deposits to geologic description of unconsolidated deposits using the Wentworth grain size scale
2. Added second paragraph of section 1.0
3. Updated Attachment 5 with current soil boring log

Several minor grammatical edits

Table 1

Unified Soil Classification System Description #1

Unified Soil Classification System; from American Society for Testing and Materials, 1985				
Major Divisions			Group Sym.	Group Name
Coarse Grained Soils–More Than 50% Retained On No.200 Sieve	Gravel–More Than 50% of Coarse Fraction Retained On No.4 Sieve	Clean Gravel	GW	Well-Graded Gravel, Fine to Coarse Gravel
			GP	Poorly-Graded Gravel
		Gravel With Fines	GM	Silty Gravel
			GC	Clayey Gravel
	Sand–More Than 50% of Coarse Fraction Passes No.4 Sieve	Clean Sand	SW	Well-Graded Sand, Fine to Coarse Sand
			SP	Poorly-Graded Sand
		Sand With Fines	SM	Silty Sand
			SC	Clayey Sand
Fine Grained Soils–More Than 50% Passes No. 200 Sieve	Silt And Clay Liquid Limit Less Than 50	Inorganic	ML	Silt
			CL	Clay
		Organic	OL	Organic Silt, Organic Clay
	Silt And Clay Liquid Limit 50 Or More	Inorganic	MH	Silt of High Plasticity, Elastic Silt
			CH	Clay of High Plasticity, Fat Clay
		Organic	OH	Organic Clay, Organic Silt
Highly Organic Soils			Pt	Peat

Table 2

Unified Soil Classification System Description #1

Unified Soil Classification System (USCS)			
	Millimeters	Inches	Sieve Size
Boulders	> 300	> 11.8	-
Cobbles	75 - 300	2.9 - 11.8	-
Gravel:			
Coarse	19 - 75	0.75 - 2.9	-
Fine	4.8 - 19	0.19 - 0.75	3/4" - No. 4
Sand:			
Coarse	2.0 - 4.8	0.08 - 0.02	No. 4 - No. 10
Medium	0.43 - 2.0	0.02 - 0.08	No. 10 - No. 40
Fine	0.08 - 0.43	0.003 - 0.02	No. 40 - No. 200
Fines:			
Silts	< 0.08	< 0.003	< No. 200
Clays	< 0.08	< 0.003	< No. 200

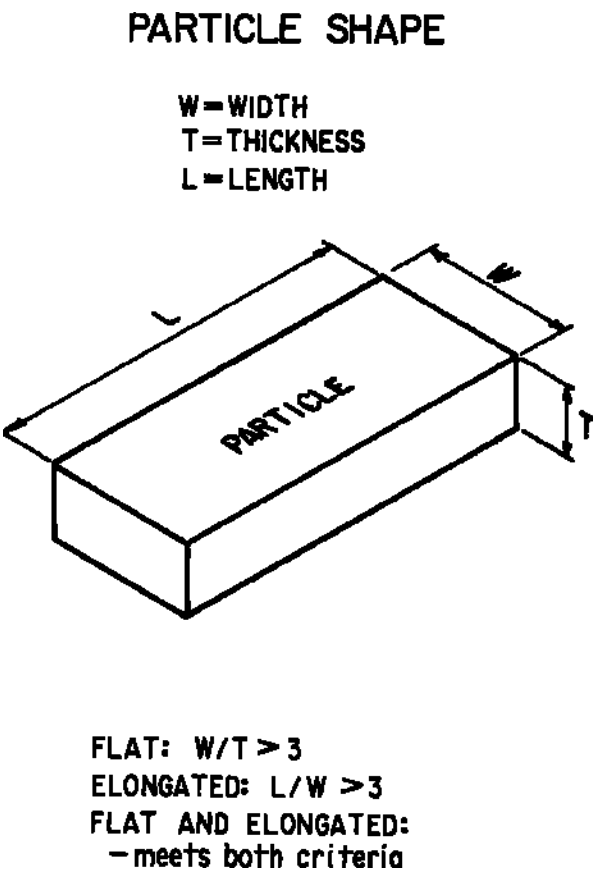
Table 4

Criteria for Describing Particle Shape (see Fig. 2)

The particle shape shall be described as follows where length, width, and thickness refer to the greatest, intermediate, and least dimensions of a particle, respectively.	
Flat	Particles with width/thickness > 3
Elongated	Particles with length/width > 3
Flat and Elongated	Particles meet criteria for both flat and elongated

Figure 2

Criteria for Particle Shape



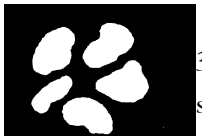
ATTACHMENT 3
ANGULARITY OF ROCK FRAGMENTS



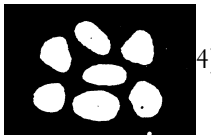
1) Angular: having sharp corners and edges, and therefore showing little or no effects of abrasion or wear.



2) Subangular: having edges and corners slightly rounded, so that wear is evident.



3) Subround: having most of the corners and edges worn down to smooth curves, and thus showing extensive abrasion.



4) Round: having all edges and corners smoothed off to gentle curves by prolonged wear.

ATTACHMENT 4
CONSISTENCE CLASSES

	Class	Description
Dry Soil:		
	dl —	loose- usually associated with sands
	ds —	soft- fragile
	dsh —	slightly hard- breaks easily
	dh —	hard- barely breakable with fingers
	deh —	extremely hard- can't break with fingers
Moist Soil:		
	ml —	noncoherent
	mvfr —	very friable- very easily broken apart
	mfr —	friable- easily broken apart
	mfi —	firm- resist crushing, "explode" when finally crushed
	mvfi —	very firm- barely breakable with fingers
Wet Soil:		
	wso —	nonsticky
	wss —	slightly sticky- soil sticks to one finger when squeezed and separated
	ws —	sticky- soil sticks to both thumb and finger, stretches slightly
	wvs —	very sticky- soil sticks strongly, resists separation of thumb and finger
	wsp —	slightly plastic- forms wire when rolled between hands, but breaks easily
	wp —	plastic- forms wire when rolled between hands, doesn't break easily
	wvp —	very plastic- forms a very durable wire when rolled between hands

ATTACHMENT 5
SOIL BORING LOG

STANDARD OPERATING PROCEDURE

SEI-6.43.3

GROUNDWATER PROFILING AND KPRO TESTING

SOP Number: SEI-6.43.3
Revision Number: 3

Date Issued: 08/13/02
Date of Revision: 8/22/12

1.0 OBJECTIVE

Groundwater profiling is conducted to assess the distribution of contaminants and hydrogeologic conditions of a given aquifer at a scale much smaller (several centimeters) than the conventional monitoring well system. This method uses the Waterloo^{APS} (Advanced Profiling System) to collect groundwater samples at multiple discrete depths as the profiler is advanced vertically through unconsolidated, saturated porous media at a given location. The Waterloo^{APS} is a direct push tool used to collect samples and other data at multiple depths within a single hole without withdrawing and decontaminating the tool between samples. The profiling system can generate the following data in addition to the collection of groundwater samples for analysis:

1) Index of hydraulic conductivity (I_k)
2) Depth to potentiometric surface (water table)
3) Specific conductance
4) pH
5) Dissolved oxygen
6) Oxidation/reduction potential
7) Temperature

The index of hydraulic conductivity provides a real-time indication of the relative changes in hydraulic conductivity which allows for decisions to be made on depths to stop driving the profiler and collect samples as well as to assess the hydro-stratigraphy across the site.

There are two types of profiler configurations: 1) a peristaltic pump configuration with a single downhole tube and 2) a gas drive pump configuration with three downhole tubes.

The peristaltic pump can be used when the depth to water is less than the suction limit (typically around 25 feet below ground surface). The gas drive pump system must be used when the depth to water is greater than 25 feet below ground surface (bgs).

2.0 POLICIES

An experienced, qualified SEI staff member will train all SEI staff using this SOP.

SEI staff will read the most current version of this SOP and other appropriate SOPs prior to starting fieldwork.

3.0 SAFETY ISSUES

The corporate Health and Safety Plan and the Site Health and Safety Plan specify the procedures to be followed and equipment to be used during site activities. The following is a brief and general overview of safety issues.

- Potential Safety Issues include:
    - Noise Levels
    - Heavy equipment (drill rigs or direct push rigs) hazards
    - Overhead utility hazards
    - Underground utility hazards
    - Traffic/ motor vehicle hazards
    - Hazards associated with exposure to various chemicals
    - Slip, trip and fall hazards
    - Pinch point hazards
    - Compressed gas hazards
    - Fire hazards from hot work (e.g., grinding)
    - Gasoline hazards (filling generator)
- SEI staff and others under contract with SEI that may be present on-site will wear a hardhat whenever overhead hazards are present.
- Appropriate eye protection should be worn
- Hearing protection shall be worn whenever the drill rig is actively advancing profiling equipment.
- Steel toed boots will be worn.
- SEI staff will read the site health and safety plan (HASP) prior to beginning a project.
- Additional personal protective equipment shall be worn in accordance to the health and safety document.

4.0 PROCEDURE

4.1 Equipment

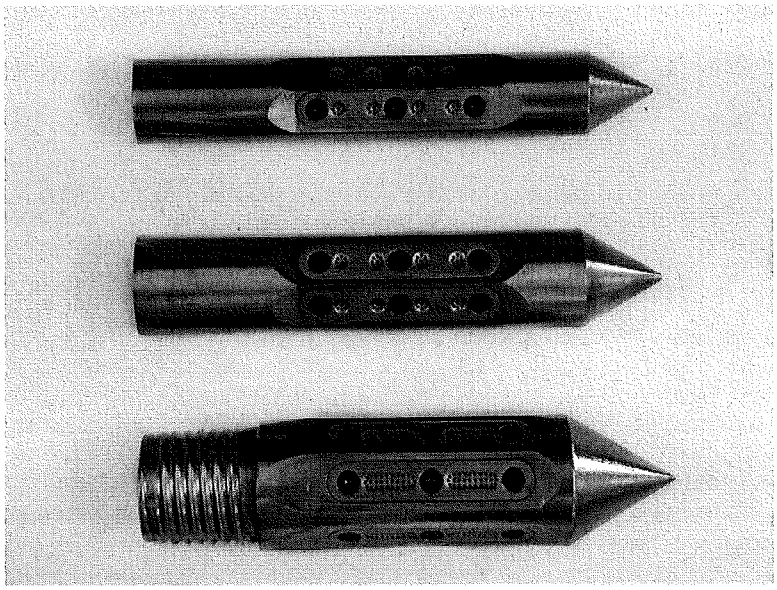
An equipment list is presented in Attachment 1. This list includes the equipment used for the peristaltic pump configuration and the gas drive pump configuration.

4.1.1 Drive Platform and Related Equipment

Any device that vibrates, hammers, or pushes drill tools can be used to advance the profiler. The type of drive platform selected is a function of the type of material to be sampled and the depth to which the profiler needs to be advanced. Typical drive platforms include GeoProbe 66 series probe rigs or similar rigs as well as larger conventional auger drill rigs with a breaker hammer mounted on the rig. A Geoprobe 8 series or equivalent is recommended to drive the 2.25" tip and rods. Field staff must ensure that the proper adaptors and subassemblies are provided for the particular drive platform to be used prior to taking to the field.

4.1.2 The Waterloo Profiler

This system is comprised of a 1.5, 1.75 or 2.25 -inch diameter profiling tip that is attached to 1.5 or 2.25-inch GeoProbe style rod or a quick thread style 1.75-inch diameter drill rod. The 1.5 and 2.25-inch drill rod is provided by others. The 1.75 inch rod is the standard rod size that Stone uses for profiling.



1.5, 1.75 and 2.25" Profiling Tips

The 1.5 and 1.75-inch profiling tips contain 4 removable plates, each with 4 screened sampling ports (16 total) that allow for the groundwater to pass into the tip. The 2.25-inch

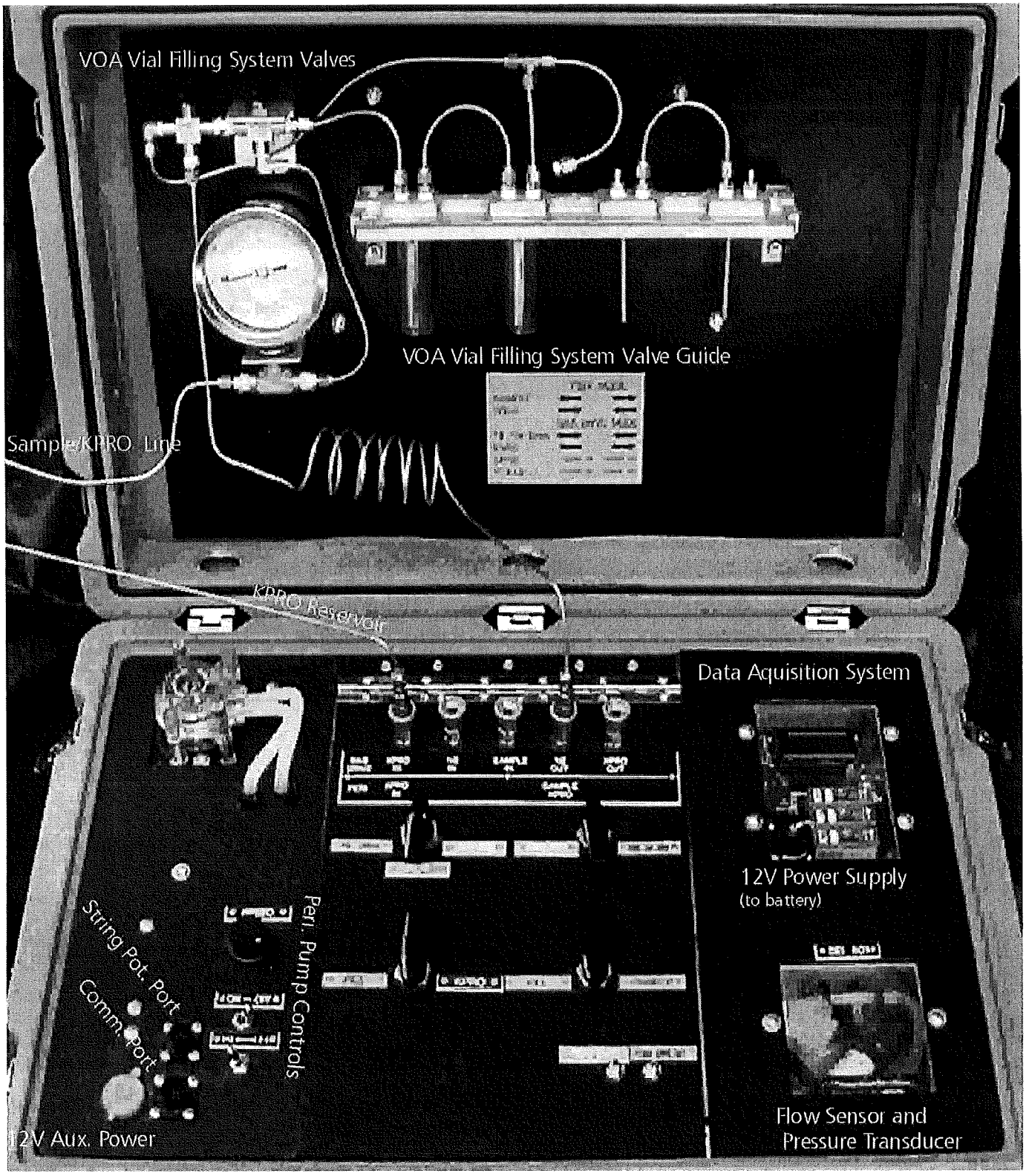
tip contains 6 plates. The plates have a slightly different design than the smaller tips in that the entire plate is more or less a sampling port. The groundwater is obtained using either a peristaltic pump or a gas-drive system (see Figure 4.1 for a schematic of these two profiler configurations).

4.1.3 Volatile Organic Analysis (VOA) Vial Filling System

The system contains as many as four vials connected in-series using stainless steel tubing and specially designed vial holders. The system is designed to mitigate losses of the volatile organic compounds (VOC) by ensuring that no exposure to the atmosphere occurs while the sample is collected. The bottle holders can accommodate up to four 40-mL VOA or 250-mL vials. The vial filling system has been incorporated into the SEI KPRO box. Although the KPRO box utilizes a peristaltic pump, the sample vials are located on the *down hole side of the pump* so that groundwater is pumped through the vials before going through the head of the pump.

4.1.4 KPRO Measurement and Data Acquisition System

The KPRO box consists of four valves, electronic enclosure, peristaltic pump and vial filling system (referred to as the sample jig). The valves can be oriented in a variety of ways in order to send water down and out of the profiling tip or to collect a groundwater sample in both peristaltic and gas drive modes. The following photo illustrates the box setup in peristaltic mode:

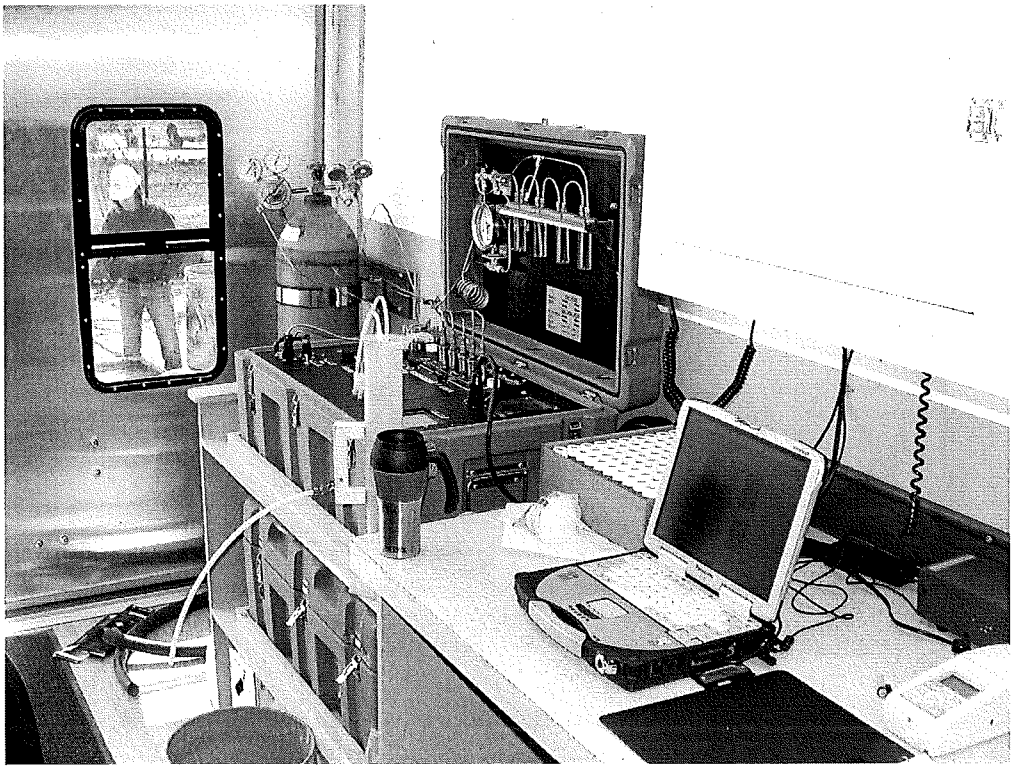


KPRO Box in Peristaltic Mode

The KPRO Measurement and data acquisition system consists of the following components:

- McMillan Model 102-4 (20-200mL/min) Flow Sensor
- Sensotec Model TGE/388-02TJA (0-50PSI) Pressure Transducer
- Celesco Model PT5DC-100-V62-UP-Z5-M6M String Potentiometer
- Analog Devices Acquisition System, includes signal conditioners and acquisition board. The removable container that houses the data acquisition system, flow sensor and pressure transducer is referred to as the "enclosure".
- GeoTech Model Geopump peristaltic pump
- Labview KPRO Software Program
- Notebook computer (Toughbook or other)
- USB-Serial Cable or Serial Socket Card
- USB mass storage unit for data back up
- AC/DC power inverter or 12V deep-cycle battery

The following photo illustrates how the entire system looks when it is set up in the truck:



4.1.5 *Sonde Water Quality Monitoring Systems*

Specific instruction regarding the use, calibration and maintenance of these systems can be found in SEI SOPs 5.25.0 (Hydro Lab MS-5), 5.27.0 (Troll 9000 and 9500) and 10.7.1 (YSI 600XL). The YSI 600XL, Troll 9000, Troll 9500, Hydro Lab MS-5, and Hach HQ30 Water Quality Monitoring Systems consists of the following components:

- pH/ORP Probe
- Conductivity/Temperature Probe
- Dissolved Oxygen Probe
- Custom Made Flow-Through Cell.
- 3/8th" Polyethylene tubing

4.1.6 *Record Keeping Materials*

- KPRO Calibration Log (dedicated to each profiling box).
- Groundwater Profiling Log (electronic and paper).
- Pens
- USB mass storage unit

4.2 General Procedure

The profiling procedure for the peristaltic pump mode and the gas drive mode consists of 6 main steps:

1. Set up of the KPRO box and associated equipment
2. Tip / Pump assembly
3. Adding rod and tubing
4. Drive the tool and acquire index of hydraulic conductivity data
5. Purge water from the system and obtain physico-chemical data from the Sonde water quality monitoring system
6. Collect samples and record data.

4.3 KPRO Box Set Up

4.3.1 *Set up common to both Peristaltic and Gas Drive Modes*

1. Open the KPRO box and support the top by using the built in wooden tab in the truck or the metal arm and screws that are located inside the KPRO box.

Plug the AC/DC inverter into the receptacle in the truck. Plug the power cable into the inverter and into the three pronged receptacle on the enclosure of the box.

2. Plug one end of the string potentiometer communication cable into the data port on the panel of the KPRO box and screw the other end into the string pot.
3. Connect the data cable to the Communication Port (COM port) on the KPRO box panel. Plug the other end of the cable into one of the serial ports on the computer or the USB – Serial Cable and secure it using the thumb screws on the cable.
4. Unpack the water quality sonde from its case. Unscrew the cal cup and screw on the flow through cell and connect the power and com cables to the sonde. Connect the com cable to one of the socket ports on the computer. Secure the sonde by putting it into the holder mounted on the counter. Connect a piece of poly tubing to the KPRO box out (peri or gas, depending on which method you are using) and the other to the bottom connection on the flow through cell. Connect a different piece of tubing to the other fitting on the flow cell. The other end of this tubing will go into a graduated cylinder. If you are using a stand-alone dissolved oxygen sensor, connect the "Out" tubing to the flow through cell of the DO meter, then use another length of tubing to connect to the sonde flow through cell.
5. Connect a male swage quick connect to a five foot length of stainless steel tubing and plug it into the female quick connect labeled KPRO In on the KPRO box. Connect the other end to the "out" port on the stainless steel KPRO reservoir using a reservoir quick connect fitting (black band).
6. Fill the stainless steel KPRO reservoir with analyte free water (distilled or spring water). Seal the top and close the pressure release valve.

4.3.2 Set up specific to Peristaltic Mode

1. Attach a low pressure regulator (0-500 psi) to the nitrogen cylinder.
2. Attach a five foot length of tubing to the "in" (white) port on the stainless steel KPRO reservoir using a quick connect fitting. Attach the other end to the outlet port on the nitrogen tank regulator. Open the valve on the nitrogen cylinder and set the regulator to 30 psi.
3. Attach a 20 foot length of stainless steel tubing to the inlet side of the SwageLok® fitting at the bottom of the pressure/vacuum gauge on the top panel. Tighten firmly. This line is the KPRO / sample line and is referred to as the harness.
4. On the KPRO box plug the male quick connect that is attached to the sample jig to the "KPRO Out" female quick connect on the KPRO box (Second from the right).

4.3.3 Set up specific to Gas Drive Mode

1. The gas drive system utilizes two regulators attached to the cylinder by a "T" adaptor. One of the regulators is low pressure (0-500) and the other is high pressure (0-4000).
2. Attach a five foot length of tubing to the "in" (white) port on the stainless steel KPRO reservoir using a quick connect fitting. Attach the other end to the outlet port on the low pressure nitrogen tank regulator. Open the valve on the nitrogen cylinder and set the regulator to 30 psi.
3. Connect a five foot length of tubing to the high pressure regulator. Attach a quick connect to the other end and insert into the "N2 In" female quick connect on the KPRO box. Set the regulator to 80 psi.
4. On the KPRO box plug the male quick connect that is attached to the sample jig to the "Sample In" female quick connect on the KPRO box (middle connection).
5. The harness for gas drive mode consists of three stainless steel tubes which are labeled with colored zip ties. Plug the KPRO line male quick connect (blue) into the "KPRO Out" quick connect on the KPRO box and plug the Nitrogen line (red) quick connect into the "N2 Out" quick connect. The Sample line (yellow) does not have a quick connect. This line connects to the 1/8" Swage fitting on the pressure gauge.
6. Attach a one end of a length of plastic tubing (FEP or other) to the "Peri Out" port located at the bottom right of the KPRO box and put the other end into a plastic 1L graduated cylinder. Attach a similar length of tubing to the "Gas Drive Out" port and connect this to the bottom port on the flow cell of the water quality sonde.
7. Inside each KPRO box is a vent tube, which is a length of poly tubing attached to a quick connect. Connect this tube to the port located on the outside front of the KPRO box.

4.4 Tip and Pump Assembly for Peristaltic and Gas Drive Modes

The 1.5 and 1.75-inch profiling tips have 4 plates and the 2.25-inch tip have 6. Behind the plate is a removable mesh screen. The size of the inner screen can be altered to prevent fines from entering the profiler or to maximize flow. Screen mesh sizes range from 40 (coarse) to 120 (fine). 80 size screen is usually the default screen size and can be changed depending on site geology. For peristaltic mode either the 1.5, 1.75 or 2.25-inch diameter tips can be used. With gas drive mode only the 1.75 or 2.25-inch tips can be used.

4.4.1 Peristaltic Mode

A small diameter tube (typically ¼-inch outside diameter fluorinated ethylene propylene, or FEP) is fitted into the threaded hole in the center of the profiler tip by means of a ¼- inch

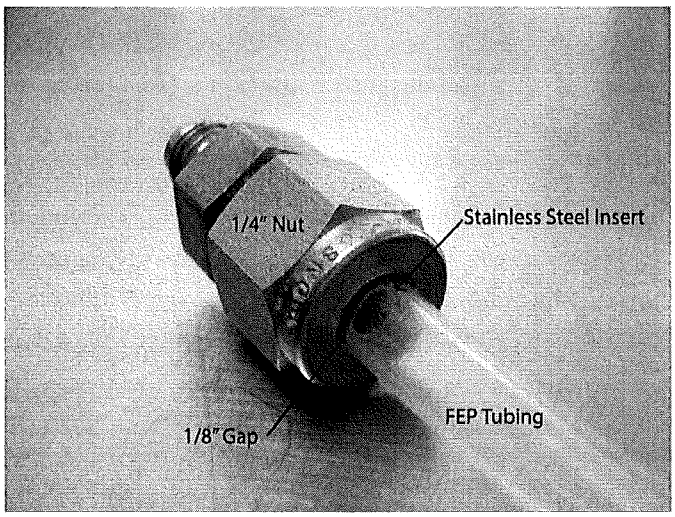
NPT thread to ¼- inch SwageLok® fitting. The following procedure will outline the procedure for assembling the tip and adding rod and drive hardware.

1. Wrap the NPT threads of the ¼- inch NPT to ¼- inch SwageLok® fitting with Teflon tape and thread into the tip. With the tip locked into a vise, tighten the fitting as tight as you can with a 9/16" socket by hand. Most profiling tips already have this fitting installed and if so, there is no need to remove it.

2. Each tip has plates that are attached to the tip via T-25 torx plus screws. Each plate has sampling ports. Under each plate is a mesh screen (40-120 slot). The smaller the slot size the larger the opening is in the screen. The most common screen size used is 80 slot. If there are no plates on the tip first place the mesh screen into the space for the plate making sure to line up the screw holes. Put on the plate and tighten the screws as tight as possible using only a T handle screwdriver or similar tool. Do not use an impact driver as it will over tighten the screws. If you are using a tip that already has the plates installed, double check that the screws are tight.

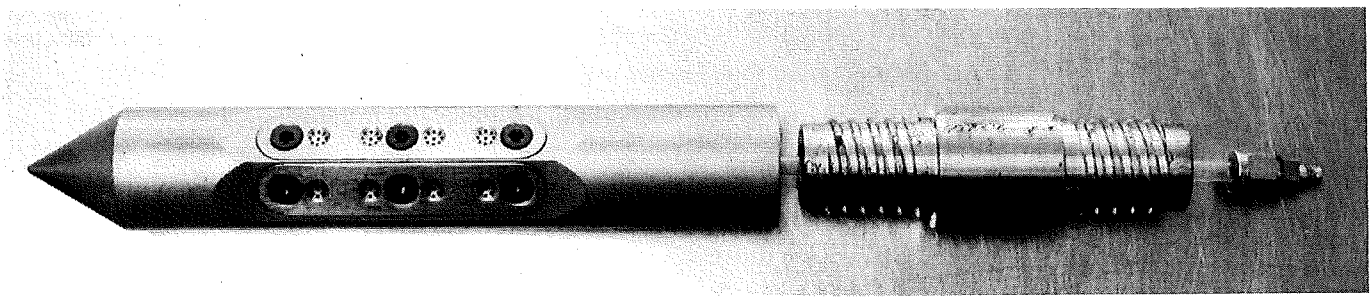
3. Cut a 6-8" length of FEP tubing and insert a steel barb into each end.

4. For this step it is best to lock the tip into a vise. On the end of the FEP that will attach to the tip, slide a ¼- inch SwageLok ® nut onto the FEP and then slide on a ¼" nylon ferrule. Insert the end of the FEP tubing into the ¼" fitting on the tip making sure that it is inserted as far as it can go. Using a specialized $^{9}/_{16}$" open ended socket, tighten the ¼" nut until there is approximately $^{1}/_{8}$" vertical gap between the top of the steel insert and the nut as shown in the following photo:



5. For the other end of the FEP, slide on another ¼" SwageLok nut and ¼" nylon ferrule. Insert the end of the FEP into a ¼ "to $^{1}/_{8}$" SwageLok® reducer. Using a ½" and $^{9}/_{16}$" wrench, tighten the nut as before. Be careful to keep the FEP seated tightly into the fitting while tightening.

When using the 1.5" tip, a male/male adaptor must be screwed into the tip after the FEP tubing is attached to the ¼- inch fitting on the tip. This allows the rod pin to be up so that the GeoProbe drive / slip cap can be utilized. After the adaptor is attached, then the ¼ inch to $^1/_8$ inch SwageLok® reducer can be attached. The inside diameter of the adaptor is not large enough to accommodate the reducer so it has to be fitted after the adaptor is attached.



6. Gently pull on the reducer after the fittings have been attached to make sure that it is attached properly. If the fittings are over tight then the FEP can snap; if they are too loose than the FEP can pull out of the fitting.
7. A five-foot length of 1.75-inch diameter rod is slipped over the stainless steel tubing with the pin end down and is threaded into the profiler tip. When using the 1.5 or 2.25-inch tip the rod is threaded pin up. This union should also be "stepped on" using pipe wrenches to ensure that it does not vibrate loose. A sacrificial knock out tip should be fitted with two o-rings and the shank of the tip wrapped with Teflon® tape to minimize the movement of silt into the bottom of the tip. Electrical tape should only be used to affix the knock out tip to the profiler if toluene is not an analyte of concern at the site.
8. The top drive cap is then threaded into the top of the 5-foot 1.75-inch rod with the stainless steel tubing protruding through the center of the drive cap. The slotted slip cap adapter is then placed over the drive cap and fitted into the bottom of the hammer on the drive platform. When using the 1.5 or 2.25-inch tip there is no threaded drive cap. Because the rods are threaded pin up, a one piece GeoProbe style slotted drive cap is used instead.

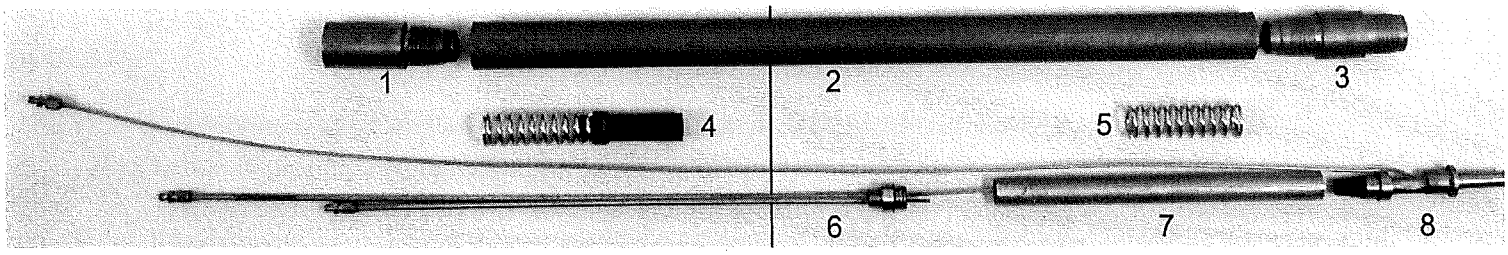
4.4.2 Gas Drive Mode

1. Wrap the NPT threads of the ¼- inch NPT thread to ¼- inch SwageLok® fitting with Teflon tape and thread into the tip. With the tip locked into a vise, tighten the

fitting as tight as you can with a $^{9}/_{16}$" socket by hand. Most profiling tips already have this fitting installed and if so, there is no need to remove it.

2. Each tip has plates that are attached to the tip via T-25 torx plus screws. Each plate has four sampling ports. Under each plate is a mesh screen (40-120 slot). The smaller the slot size the larger the opening is in the screen. The most common screen size used is 80 slot. If there are no plates on the tip first place the mesh screen into the space for the plate making sure to line up the screw holes. Put on the plate and tighten the screws as tight as possible using only a T handle screwdriver or similar tool. Do not use an impact driver as it will over tighten the screws. If you are using a tip that already has the plates installed, double check that the screws are tight.

3. Instead of FEP tubing, the gas drive system utilizes a ¼" stainless steel tube ("piston"). To attach the piston, slip a ¼" swage nut and ¼" steel ferrule onto one end of the piston. Insert this end into the ¼" fitting on the tip. It is best to have the tip locked in a vise for this step. Slip the open ended $^{9}/_{16}$" socket over the piston and tighten the nut as tight as you can.



The above photo illustrates the components of the gas drive pump.

1. Pump Housing – Top
2. Pump Housing – Middle
3. Pump Housing – Bottom
4. Top Spring with plastic bushing
5. Bottom Spring
6. Pump Body - top. Sample line is the middle tube and nitrogen is the short
7. Pump Body – middle
8. Pump Body – bottom with reed valve. The KPRO line is attached.

4. The gas drive pump body has three parts, the bottom (which has the KPRO line), the middle and top (which has the sample and nitrogen lines). Look into the narrow part of the pump bottom from the bottom. The piston on the tip will

eventually be inserted into this end of the pump. There should be two very small rubber o-rings seated into two slots. If they are present, it is a good idea to replace them. If the o-rings get worn or dry out you will not get a proper seal between the piston and pump.

5. Pry the o-rings out with an angled pick and discard. Rinse out the slots with water. Replace the o-rings using the same pick.

6. Place an o-ring at the base of the threads of the top and bottom pump pieces and wrap the threads with Teflon® tape.

7. Trim a reed valve and place it onto the stub on the top of the pump bottom. In order for the reed valve to fit inside the middle pump piece, the flange of the valve must be trimmed. NOTE: There are two types of reed valves, one for depths shallower than 150' and one for profiles deeper than 150'. The storage container for the valves should be labeled as to which valve is which. Using a reed valves designed for shallow depths in a deeper profile could result in a catastrophic failure of the valve. If the container is not labeled assume the valve us for lesser pressures.

8. Thread the middle pump body onto the top and bottom pump pieces. Lock the middle of the pump into a vise and tighten the top and bottom pump pieces with a $^{9}/_{16}$" open ended wrench until tight. The o-rings should squeeze out of the joint slightly.

9. Slide the plastic bushing and spring over the three stainless steel tubes so it rests snug on the top of the pump. Make sure that the KPRO line is located into the slot on the bushing.

10. The pump housing is comprised of three pieces: the middle 2.4-foot section of rod (with female threads on both ends), the bottom which has two male threads, and the top which has one male and one female thread. The 2.25-inch pump housing has the same parts but the female – male goes at the bottom and the male –male fitting is at the top. Insert the bottom spring into one end of the middle pump housing. Apply Teflon paste or tape to the male threads on all of the pump housing pieces as well as the profiling tip and first drill rod. This will prevent silt from migrating into the pump housing. Screw in the bottom housing piece so that the extended end is inside the pump. Make sure that the spring is at this end of the pump. Insert the pump into the housing. Screw on the top housing piece.

11. Take the profiler tip and slide the ¼-inch stainless steel tube up into the receptacle in the pump bottom. Screw the tip onto the pump housing.

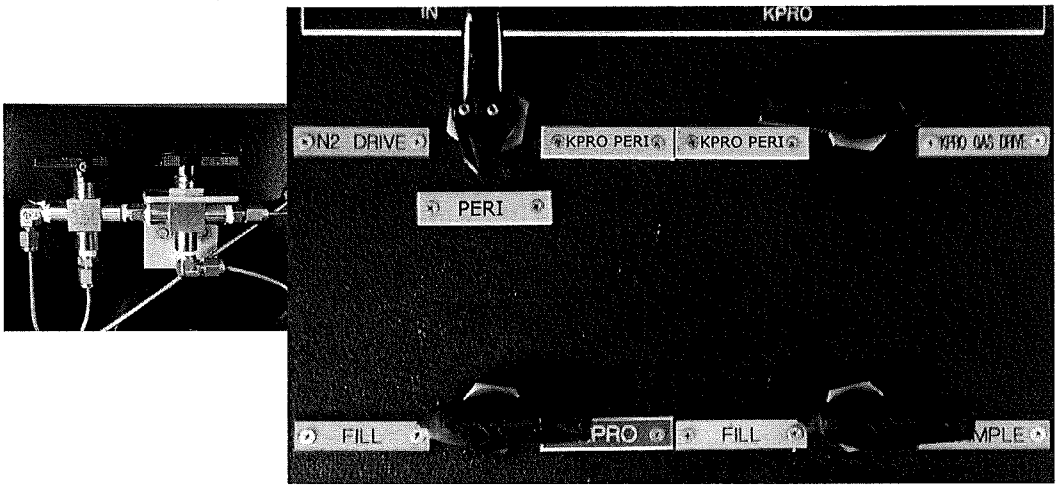
12. With the pump in a vertical position and the tip on the ground, grasp the three stainless steel lines and push them down. You should feel the springs move up and down smoothly. If they don't, the pump must be taken out of the housing and

checked. It is very important that the springs are free to move. If they cannot, the vibration of the hammer will damage the pump.

13. **Pressure testing the Gas Drive Pump:** The pump must be pressure tested before it is used to make sure that the o-rings and the reed valve have been installed properly. It is usually a good idea to pressure test the pump after collecting the equipment blank or decontaminating the KPRO box since the pump is attached and full of water. The following steps will lead you through this:

    a. Connect the pump to the tubing harness (all three tubes). You will notice that the three lines coming out of the pump are at three different lengths. The difference in length is the only way to be able to differentiate between them. The KPRO line is the longest, the nitrogen line is the shortest and the sample line is in the middle.

    b. Arrange the valves according to the following photo to start filling the pump:



    c. When a steady stream of water is flowing out of the Peri Out tubing on the KPRO box then the pump is full. Turn the two valves on the sample jig so that they are closed (facing the back of the box). Turn the bottom left valve 90 degrees to shut off the flow. Turn the top left valve to N2 Drive. The pressure on the gauge will rise to where it is set on the regulator. Make sure the pressure is set to around 100 psi or the expected working pressure.

    d. Disconnect the N2 Out quick connect on the KPRO box and watch the pressure gauge. If the needle on the gauge is steady then the pump is holding pressure. If the pressure drops then there is a leak. Check that all of the fittings are tight and refill and pressurize the pump. If that does not work, there is a

leak in the pump itself (either an o-ring or reed valve) or possibly there is a leaky valve in the KPRO box.

e. **Testing the Bottom O-rings.** If there is not a tight seal between the small o-rings at the bottom of the pump and the piston, KPRO water will not be able to flow into the tip, which will result in inaccurate IK data, sampling of the water inside of the drilling rods and a clogged tip. With the pump pressurized, turn on the flow by turning the bottom left valve on the KPRO box to KPRO. You should see water flowing out the tip. Grasp the pump just above the tip and invert the pump at a 45 degree angle so that your hand blocks the flow of KPRO water. If you see water flowing out of the pump housing then it is possible that the o-rings are leaking and they need to be checked.

NOTE: A more reliable way to determine if there is a leak is to check for air bubbles flowing through the vials while collecting the equipment blank. Double check that all of the fittings on the harness, KPRO box and pump are tight. If there are still bubbles then the o-rings need to be checked. Refer to section 4.9.3 on equipment blank procedures.

14. Add a 20-foot length of stainless steel tubing to each tube fitting leading from the pump.
15. Slide a 5' drill rod over the stainless steel tubing and screw it into the pump.
16. Tighten all joints by firmly by standing on the pipe wrench. To do this put one pipe wrench so that it is resting on the ground. Place the middle of the pump into the wrench. Put the other wrench on the tip and stand on it, making sure that the other end of the rod is weighted down (have a helper stand on it). This will tighten the tip and the bottom pump housing piece. Repeat for the top of the pump and rod.
17. The top drive cap is then threaded into the top of the 5-foot rod with the three pieces of stainless steel tubing protruding through the center of the drive cap. The slotted slip cap adapter is then placed over the drive cap and fitted into the bottom of the hammer on the drive platform.

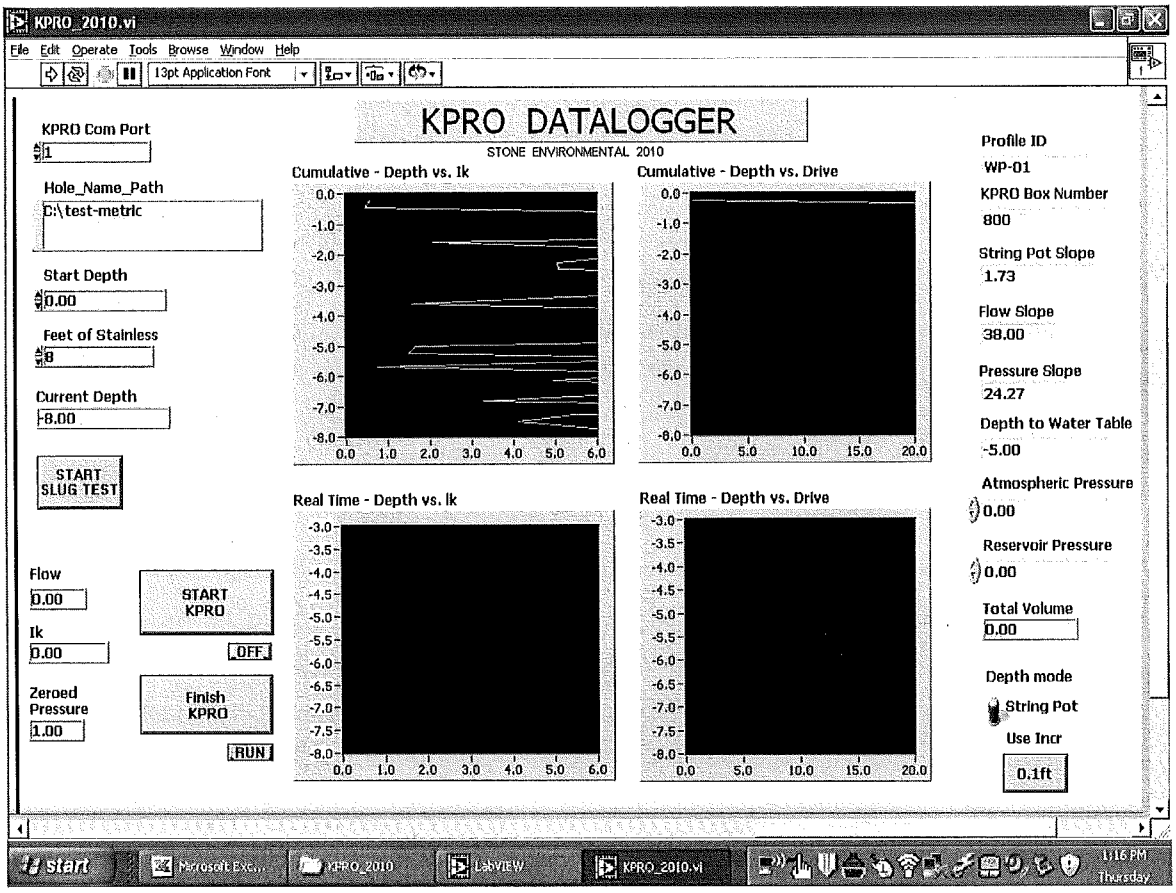
4.5 KPRO Software Calibration and Startup

4.5.1 Calibration

The KPRO box should be calibrated at the beginning of a new project and once weekly (or every 5 days if there is a longer shift). If an individual sensor or entire electronics enclosure

is replaced then there should be a recalibration. The calibration is saved on the field computer, so if the KPRO box was calibrated with a different computer then it needs to be recalibrated.

1. At this point the computer should be plugged in and booted up. If not, do so now.
2. Find the KPRO program icon on the desktop or located in the C:\KPRO_2010 folder. The program is named "KPRO_2010.vi". Double click on the icon. The Labview software will load and the KPRO software will look like this:



KPRO Software

3. Make sure that the correct COM port is provided for the KPRO box before starting the program and correct it if necessary. The text box for the COM port is located at the top left of the above figure. The COM port located at the back of the computer is COM1.

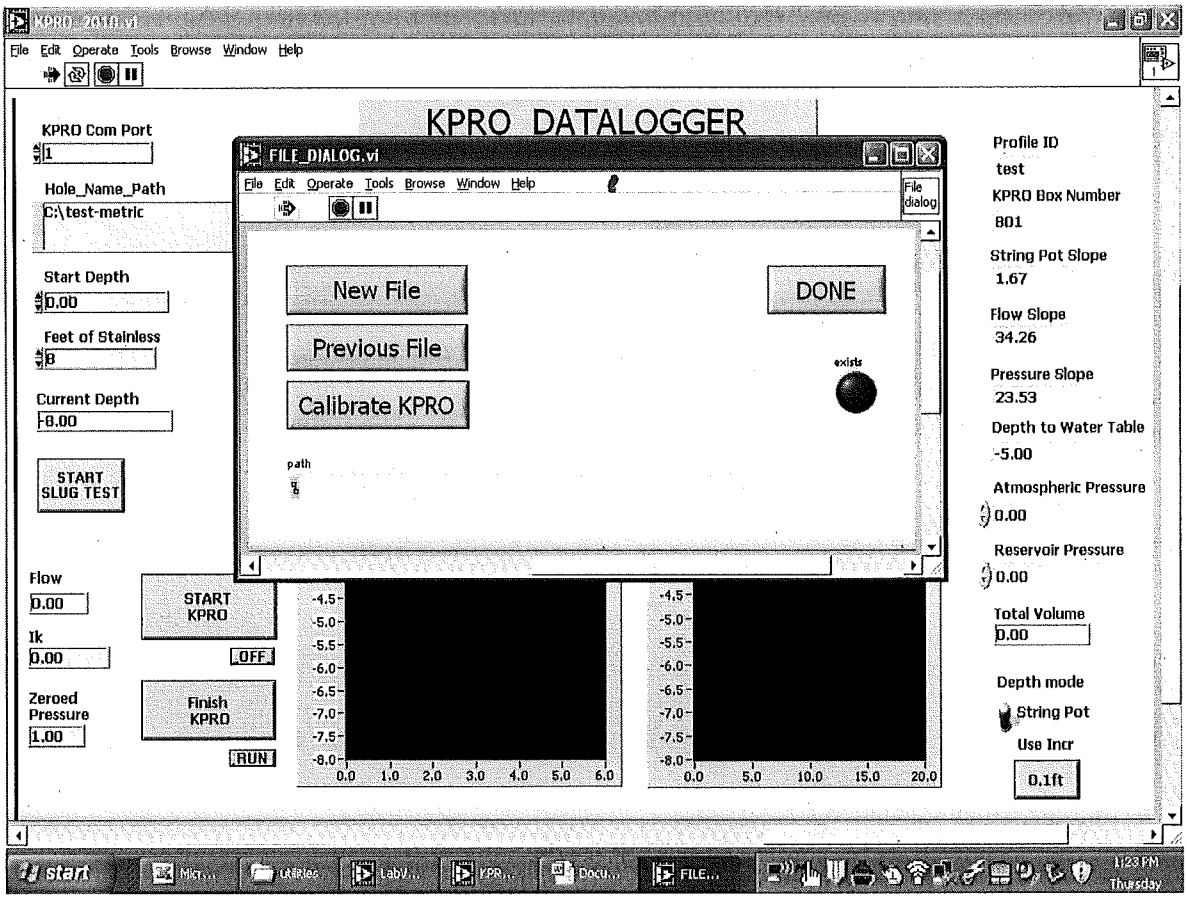
   When using the USB-Serial cables navigate to Control Panel /System/Device Manager and scroll down to Ports. The cable will be listed as "Prolific USB-Serial" and the com port assignment will be listed. For the socket cards, click on the icon for the socket driver located in the taskbar to see which COM port numbers have been assigned to the

socket card (On the figure above it is the symbol fourth from the left at the bottom right of the screen).

4. Start the KPRO program by clicking on the right arrow icon on the top left menu bar (above the COM port text box). There is no need to enter any data before starting the program (other than the COM port for the KPRO box), as the program will populate the fields or prompt you to enter a value.

5. A window opens and prompts you to enter the number of the KPRO sample box that you are using (800 through 809). Enter the number and select the enter button.

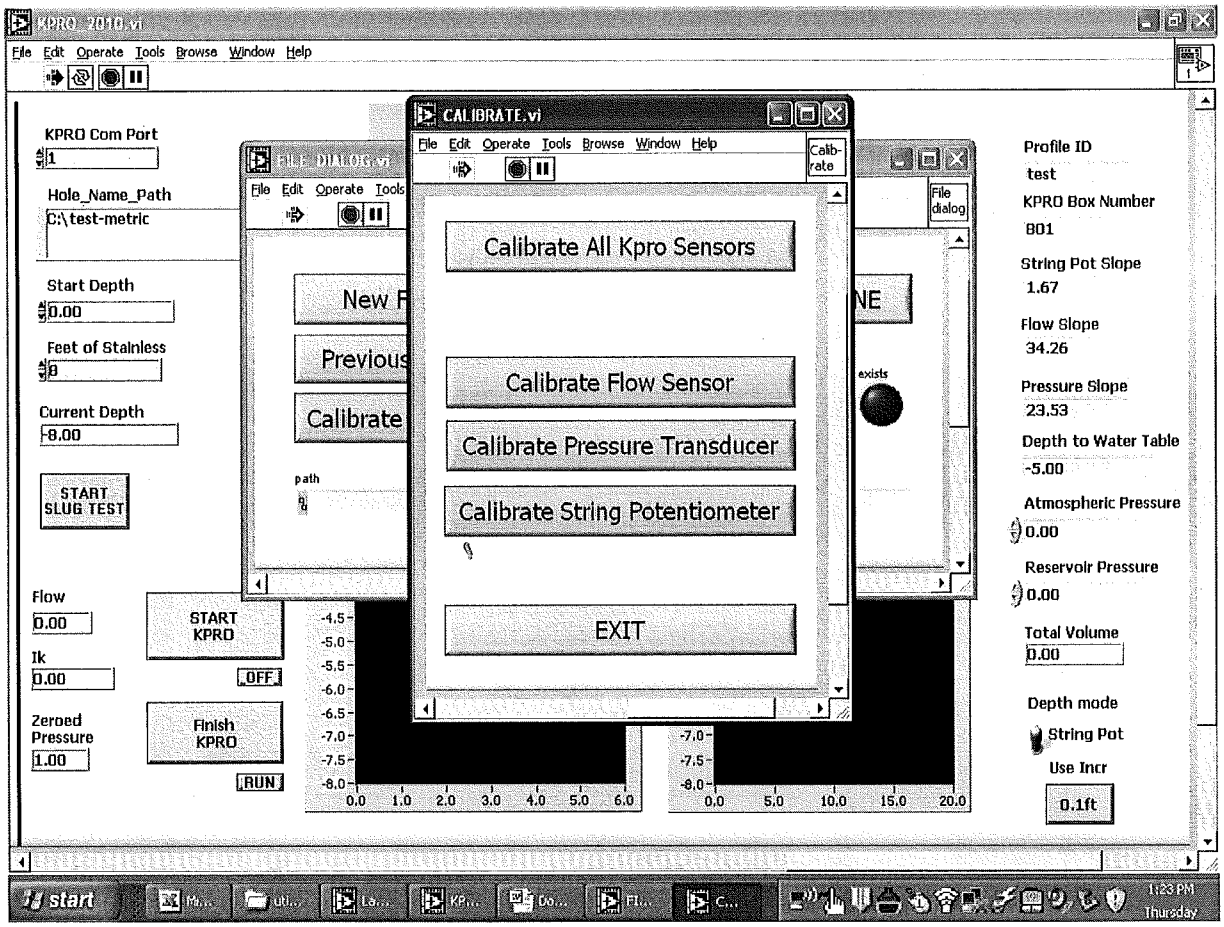
6. A window opens and prompts you to enter the profile ID. Enter the ID and select the enter button. The following screen will load.



Startup Screen

7. Select CALIBRATE KPRO. Make sure that there is at least a 20' foot length of stainless steel tubing attached to the profiling box when calibrating. This will ensure accurate calibration of the flow and pressure. Using a smaller piece of tubing can adversely affect the validity of the calibration.

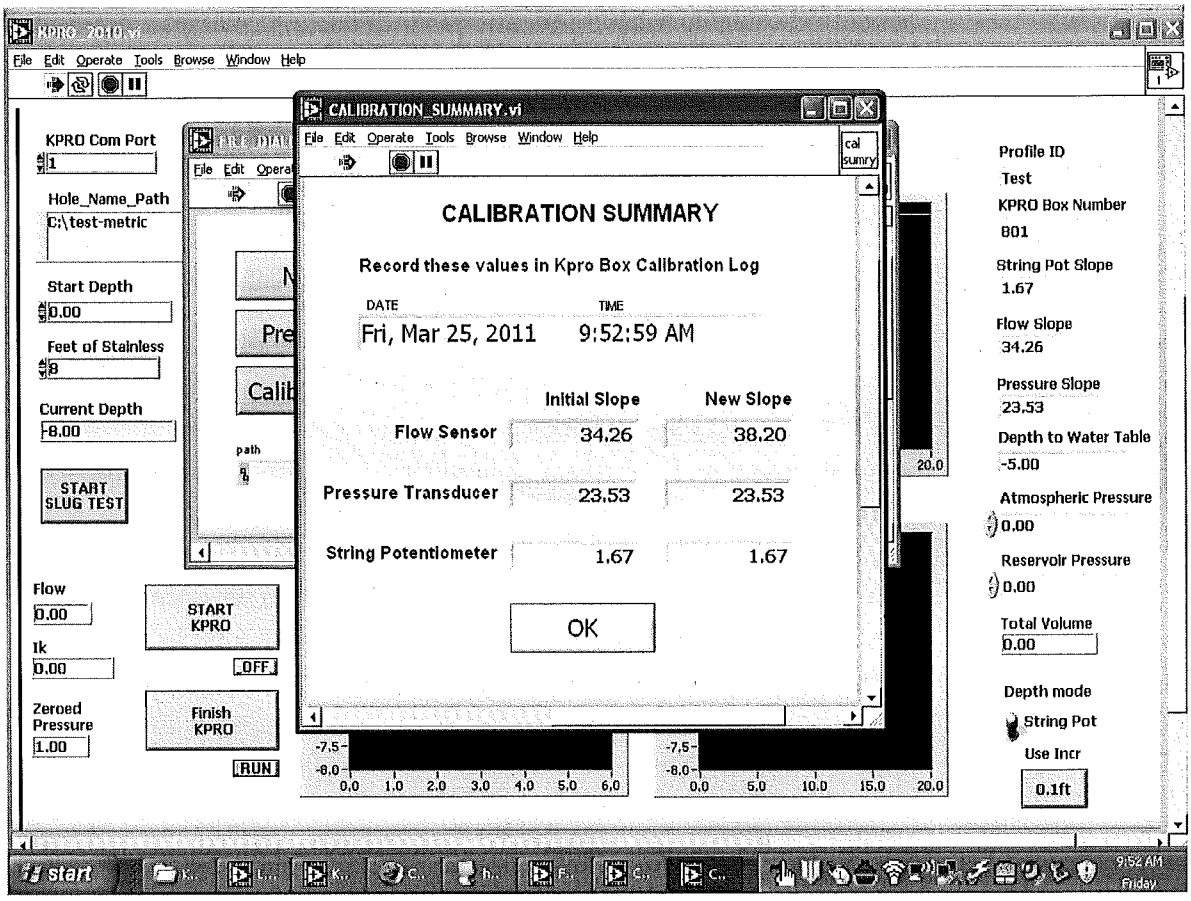
8. The next screen allows you to calibrate all of the sensors or each individual sensor (string pot, flow and pressure).



Calibration

Select the "Calibrate All KPRO Sensors" or the button corresponding to each individual sensor (if you do not need to calibrate all of the sensors). The program will then lead you through the calibration. Follow the directions on the screen to calibrate the flow, pressure and string pot. After calibration is complete, if you feel that one or more of the sensors was not calibrated correctly, you can repeat the entire calibration or just repeat the calibration for an individual sensor. If you decide to recalibrate one or all of the sensors, be aware that the program will use the original slopes and not the newly calibrated slopes for the recalibration.

9. After calibration, a summary screen will open.



Calibration Summary

Record the initial and new slopes in the calibration log (yellow book in the KPRO box) for the KPRO box you are using. Slope values should be within the following parameters: string pot 1.6-1.8, flow 32-38, and pressure 22-28. (There are a few string pots that have longer cables than normal and as a result will have a slope value double the normal value, usually around 3.2-3.6). If the slopes are off from these values, recalibrate. If that does not work, you may need to change out the pressure transducer, flow meter or string pot. Select the OK button to exit this screen.

Note: The program records the slope values in two files located in the C:\KPRO_2010\Output_Files folder. The file named "slopes.txt" is used by the KPRO program to import the slopes the next time it is started and after calibration. The other file, "cal-slopes-record.txt" is used only as a record of the calibration (it records date, time and KPRO box number as well as the slopes). Do not delete the files or any of the data in them or move them to a different folder. **Additional Note:** If the calibration slopes are off due to a calibration error, simply go through the calibration process again. If you are having problems calibrating, see the KPRO Problem Solving Guide for more

information. It may be necessary to delete bad slope values from the slopes.txt file in order to calibrate the box. The software uses the old slopes to calculate the new slopes, so if the slope that the software is using is way off, it may not be possible to get a good new slope using this value. If this seems to be the case, open the slopes.txt file and delete the entire line of bad data. Save and close the file.

4.5.2 Startup

1. After calibration or after clicking on the start button, the startup screen will load (Figure 4.5-2). If you are starting a new profile, select NEW, and if you are continuing a profile select PREVIOUS. There may be a path to the file you want to use already displayed in the box at the bottom of the screen. You still need to select the PREVIOUS button and navigate to the file. When starting a new profile, after selecting NEW, navigate to the C:\Projects\Project Name\kpro_raw folder and name and save the file. Name the file the same name as the profile ID. There is no need to add a file extension (.txt, .doc) as the program will add its own. The program will then create a file by adding "_KPRO.txt" to the end of the filename. For example "WP-01" becomes "WP-01_KPRO.txt". If you selected PREVIOUS, after navigating to and selecting the file, the green indicator light next to the word "exists" will be illuminated. Make sure to select the filename with no extension when selecting an existing file (for example, select "WP-01" and not "WP-01_KPRO.txt"). Select DONE. You will now begin the software startup procedure.

2. The first window that appears is for setting the atmospheric pressure. Follow the instructions on screen to set the pressure. If there are only zeroes in the atmospheric readout, there is a problem with the connection between the KPRO box and the computer (incorrect COM port, bad cable) or the slopes file has been corrupted. Refer to the KPRO Problem Solvers Guide for more information. Record the atmospheric pressure on the Groundwater Profile Log. The atmospheric pressure reading should be close to 34 feet of water. If the reading is higher or lower by a few feet, the pressure transducer needs to be calibrated. This value can also change depending on where you are profiling (high or low elevations, Europe, etc). It is a good idea to check the local atmospheric pressure.

3. The next window is for setting the KPRO vessel pressure. Follow the instructions on screen to set the pressure. If the pressure is too high, turn the handle on the regulator counter-clockwise and release some pressure from the KPRO vessel by using the valve on the top of the vessel. If the pressure is too low, turn the handle on the regulator clockwise and allow pressure to stabilize. After adjusting the pressure make sure that the pressure has stabilized and is not falling or climbing before clicking the OK button. Record the pressure on the Groundwater Profile Log.

4. The next window will prompt you to enter the start depth. If you are continuing a profile, the last recorded depth will be provided. You can use this depth or enter a different depth. When entering a depth you can enter a positive or negative value; the program will make it negative regardless of the sign that was entered.

5. The next window will prompt you to enter the height of the sample box above the profile location. Measure the height from the ground to the pressure transducer and enter it. When profiling out of T5, T6, T7 or T8 the height is 7 feet. When profiling out of T9, the height is 5 feet. The value will be different if you are profiling out of a different vehicle or other situation. If the ground between the truck and profile location has a significant slope, this also must be accounted for.

6. The next window will prompt you to enter the depth to water. If you are continuing a profile, the last recorded depth to water will be provided. You can use this depth or enter a different depth. If you do not know the depth to water, you must make an educated guess. You can enter a more accurate depth later. When entering a depth you can enter a positive or negative number. Make sure you enter the depth to water from ground surface, not from the height of the KPRO box.

7. The last window will prompt you to zero the pressure to the ground at the profile location. Make sure that the graduated cylinder is placed on the ground as close to the profile location as possible. Follow the instructions on screen and select OK. The setup is now complete and the profiling can begin.

8. **Recording Data** - Navigate to and open the Microsoft Excel "Groundwater Profiling Log with Plot" template. This file should be located in the C:\Forms folder or in the file template in the Projects folder. Rename the spreadsheet to reflect the profile name (e.g. Groundwater Profiling Log _WP-01.xls). Select the tab labeled "Groundwater Profiling Log". Fill in the top section of the log. The bottom section of the log will be empty at first but will be populated with data for each separate groundwater sample. The following photo shows the log after sample data has been entered:

GROUNDWATER PROFILE LOG

STONE ENVIRONMENTAL INC

Client: ERM (Started / Completed)

Profile Location: APS-018

Dates: 5/27/2010 5/28/2010

KPRO Box Serial # / Acquisition Laptop: 808/ Rasc7

Gas Drive or Peri Pump: Gas

Location: Plainville, CT

Troll Serial #: o2k0934 YSI

Atmospheric Pressure: 34.89

SEI #: 092193-R

Drilling Contractor: Platform

KPRO N_2 Pressure (set via P transducer): 70.14

Sampler(s): VLD

Average Depth to Water: -17.28

Gas Drive Pump N_2 Pressure: 80-100

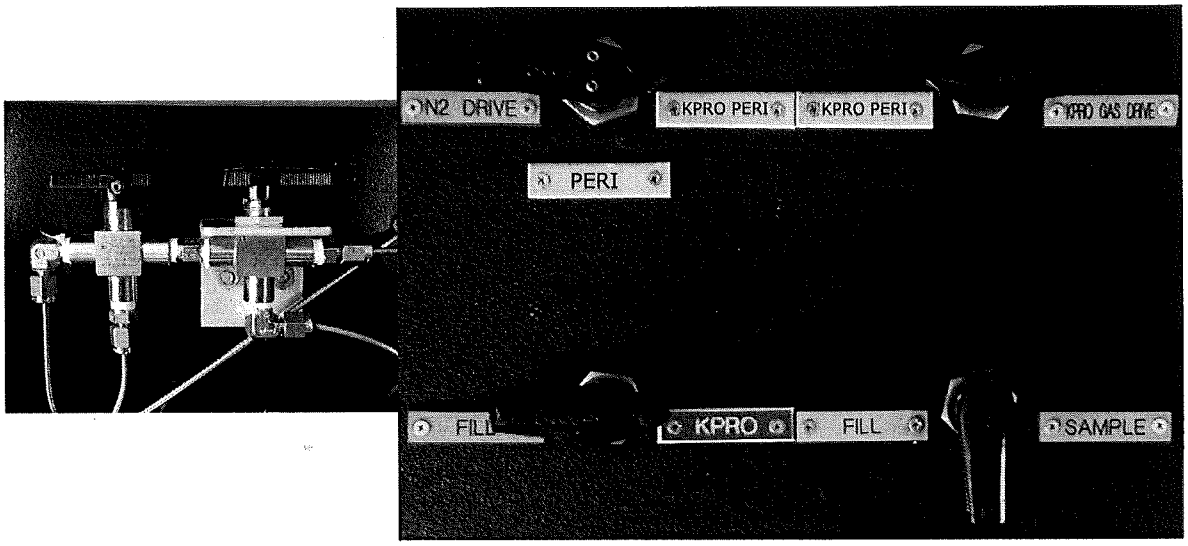
				PHYSICOCHEMICAL PARAMETERS				
Depth (ft)	Date / Time	Volume Purged (mL)	Head (ft)	SC (uS/cm)	DO (PPM)	pH	ORP (mV)	COMMENTS
-18.00	5/27/10 14:40	900	-17.20	519	8.00	6.50	63	
-26.70	5/27/10 15:30	850	-17.10	305	6.40	5.70	96	
-34.00	5/27/10 16:11	1300	-17.15	184	7.20	6.00	96	
-41.00	5/27/10 16:38	1000	-17.20	202	7.50	6.30	83	
-48.00	5/28/10 8:05	900	-17.70	212	6.30	6.40	38	
-55.00	5/27/10 8:43	1000	-17.20	140	7.30	6.16	53	
-63.00	5/28/10 9:36	910	-17.20	483	0.50	5.75	57	
-70.00	5/28/10 10:15	850	-17.30	763	0.46	5.90	65	
-77.00	5/28/10 10:50	900	-17.40	664	3.14	5.94	77	
-84.00	5/28/10 12:02	1000	-17.35	646	4.65	7.70	34	
-91.00	5/28/10 12:39	1000	-17.30	779	4.30	7.90	22	

Groundwater Profile Tab

4.6 Drive the Profiler/Acquire I_k data for both Peristaltic and Gas Drive Modes

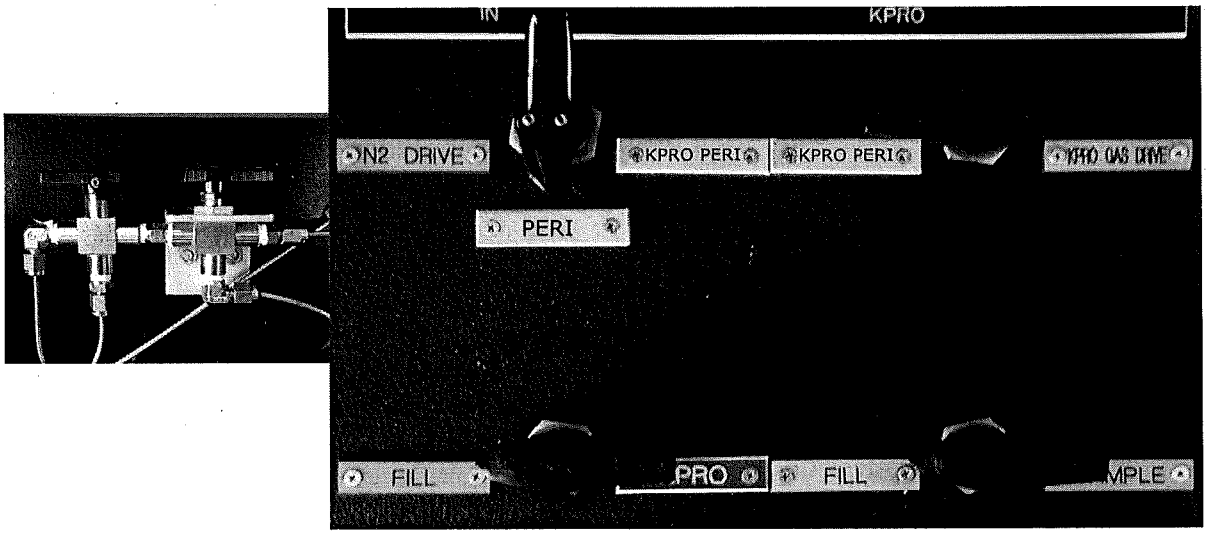
1. Attach the harness to the tube or tubes sticking up out of the top of the profiler rod at the drive platform. Place the string pot on the ground next to the drill rig and weight it down with a pipe wrench or the rod puller. Pull out the string and attach it to head of the drill dig.

2. **Peristaltic Mode**: turn on the KPRO flow by arranging the valves as shown below:



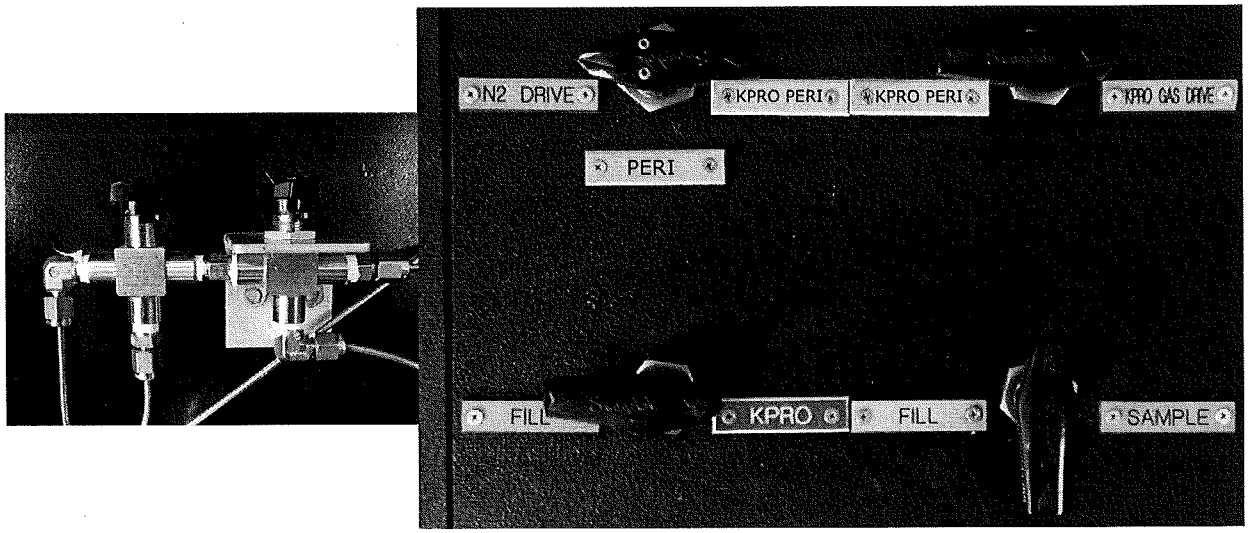
Peri Mode - Drive

Gas Drive: fill the pump by arranging the valves per the photo below:



Gas Drive Fill Pump

When the pump is full and a steady stream of water is flowing out of the Peri Out, lock off the pump (turn the valves on the sample jig so they face the back of the box), pressurize and turn on the flow per the following photo (make sure to lock off the pump first).



Gas Drive - Driving

3. With the flow on, put the profiling tip / rod assembly into the pre-cleared hole or so that the tip rests vertically on the ground surface. Have the driller lower the head of the probe so that the anvil of the probe rests on the slip cap. At this point the driller will adjust the probe so that the drill rod and probe are straight and level. **Note:** Make sure that the driller has broken through any surface concrete or asphalt. Driving through either of those will damage the tip.

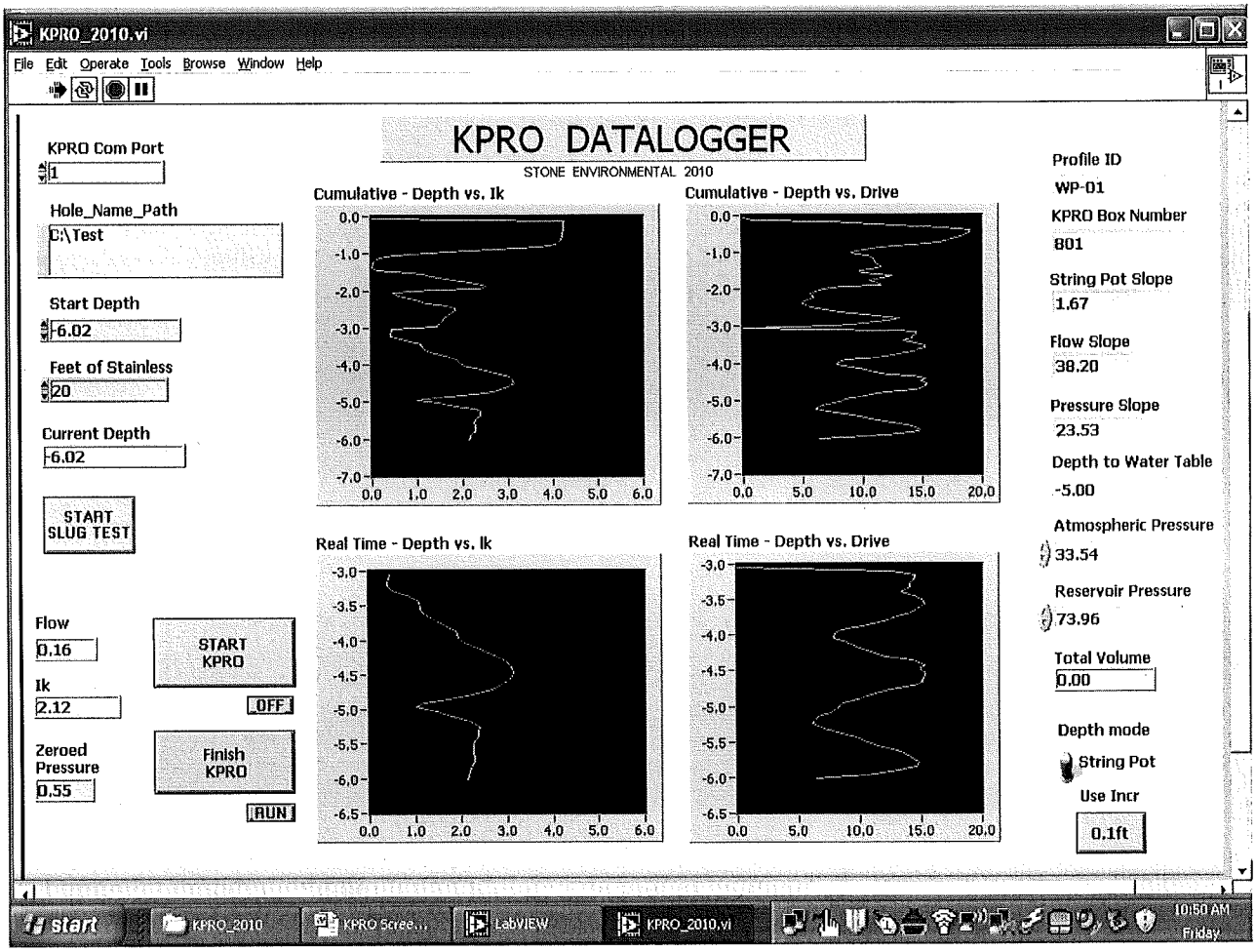
4. When the flow value stabilizes click on the Start KPRO bottom at the bottom of the KPRO software. Make sure that the string put is extended and the head of the drill rig is resting fully on the slip cap before starting the software. The software will not read the depth correctly if it is started before the string pot is extended.

5. A window will open asking the depth to water. If this is the first drive, the depth provided is the depth entered during the startup procedure. If this is your second drive and the head measurement collected at the first sample location is different from the value originally entered, a new depth can be entered here. Enter a new depth or select OK. A pop up window will open showing the feet of stainless steel tubes. Set the feet of SS tubing to the correct feet of tubing used, including the 20' harness.

6. Signal to the rig operator that you are ready to commence driving the profiler. Just before the profiler begins to move, hit the Go button on the software.

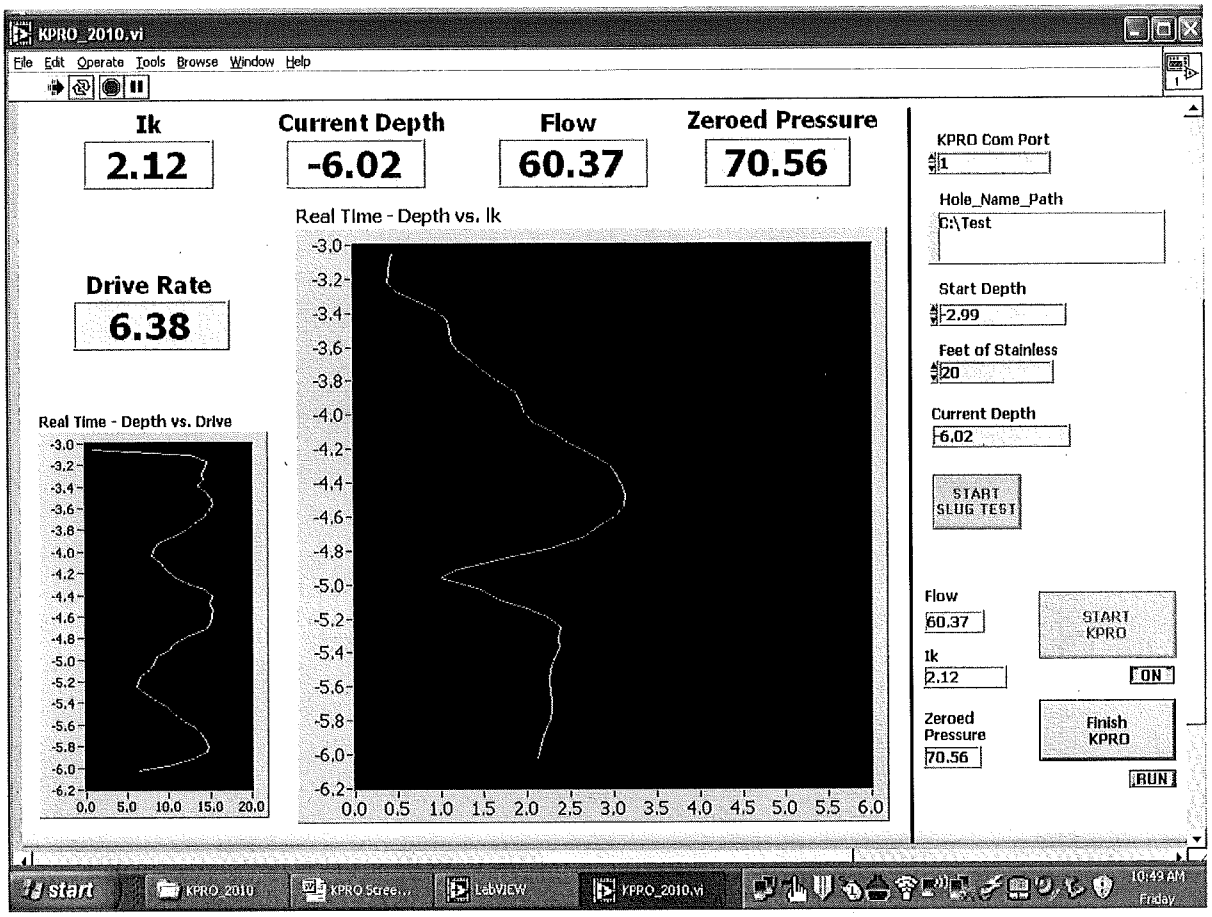
   Notes on the string pot: 1. The KPRO software only records data while the string pot is moving. 2. If during a drive the driller needs to lift the probe, there is no need to stop the KPRO software. The software will only record data once the string pot has moved past the last recorded depth.

7. Observe the I_k plot on the screen as the tool is advanced as shown in the following figure:



KPRO Software Screen While Driving

The current I_k , flow and pressure are displayed at the bottom left of the screen. The I_k for the current drive is displayed in real time in the graph at the lower left. The cumulative I_k for the entire profile is displayed in the graph at the top left. The drive rate (feet/minute) for the current drive is displayed in the bottom right graph, and the cumulative drive rate is displayed in the top right graph. A larger I_k plot can be seen by scrolling the software screen to the left. The screen looks like this:



KPRO Software Screen While Driving

Notes on I_k: I_k can range from 0 to 6+. Fine grained (silts and clay) or compacted sediment will have an I_{kv} towards the lower end, and sands, gravels and looser material towards the middle to higher end. The software will correct the Ik for depth using a head loss equation, so I_k should remain relative for the profile regardless of how much stainless tubing is attached (even though the flow is lower and the pressure is higher). I_k is only calculated and recorded by the software if the string pot is moving. In some cases the flow will drop after hammering has stopped, which results in a lower I_k. Since the string pot is not moving this new I_k value is not displayed on the screen and is not recorded. In this case take note of the flow, because the flow reading on the software is constantly updating, whether or not the string pot is moving. I_k behavior when tooling advance has stopped should be recorded on the "Profile Info" tab of the Groundwater Profiling Log (see below).

8. When you reach a zone that you wish to sample, signal to the rig operator that he/she should stop. When he/she stops press and hold the Finish KPRO button down until the icon (located below the button) changes from Run to Done.

9. **Determining whether to sample or not:** Observe the flow. If the flow drops significantly it may be an indication that the sample interval will not yield a timely sample. If the flow stays the same or increases, then it is probably a good interval to sample. In the Groundwater Profiling Log there is a "Profile Info" tab. The purpose of this worksheet is to record additional data regarding the driving and sampling of the profiler.

I_k and Sample Attempt Log

STONE ENVIRONMENTAL INC

Client:	ERM			Profile Location:	APS-018
Date:	5/27/2010	KPRO Box Serial #:	808/ Rasc7	Gas Drive or Peri Pump:	Gas
Location:	Plainville, CT	Troll Serial #:	o2k0934 YSI	Atmospheric Pressure:	34.89
SEI #:	092193-R	Drilling Contractor:	Platform	N_2 Pressure (set via P tranducer):	70.14
Sampler(s):	VLD	Depth to Water:	-17.28	Gas Drive Pump N_2 Pressure:	80-100

Depth (ft)	I_K Behavior Type		Unsuccessful Sample Attempt		Hole Termination Type		COMMENTS
-8.00	No change when hammer stops	3		#N/A		#N/A	
-13.00	No change when hammer stops	3		#N/A		#N/A	
-18.00	No change when hammer stops	3		#N/A		#N/A	
-23.00	No change when hammer stops	3	Could not produce water	4		#N/A	
-26.70	IK increase when hammer stops	2		#N/A		#N/A	
-28.00	No change when hammer stops	3		#N/A		#N/A	
-33.00	IK decrease when hammer stops	1	Yield deemed too slow	6		#N/A	
-38.00	No change when hammer stops	3		#N/A		#N/A	
-41.00	No change when hammer stops	3		#N/A		#N/A	
-48.00	No change when hammer stops	3		#N/A	ROP dropped below threshold	9	

Profiling Info Tab

Each time the hammer is stopped (to add rod, sample, determining whether or not to sample), the I_k Behavior column must be filled in. Record the depth and behavior type from the drop down menu.

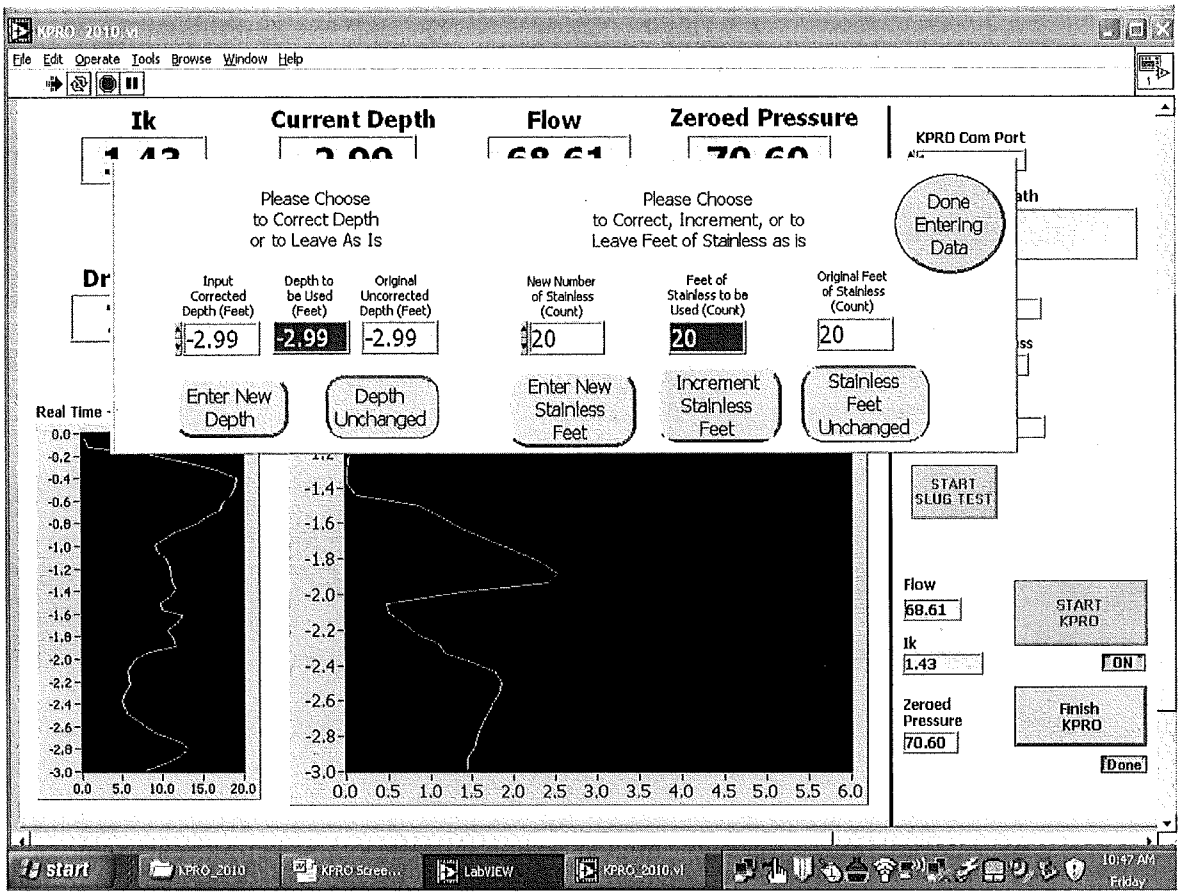
It will take experience to get a feel for which intervals will yield fast samples and which will not. The I_k and flow are a good indication as to which samples will be fast or slow but remember that the flow decreases with the amount of stainless tubing that is

attached. So at shallower depths a flow of 60 ml/min may be slow but at a depth of 60 feet it may be fast.

10. Turn off the KPRO flow by turning the lower left valve from the red KPRO label 90 degrees to an off position.

11. Measure the stick up from the ground surface to where the drive cap abuts the top of the 1.75-inch rod, or to the shoulder if using the 1.5 of 2.25-inch rod. Subtract the stick up from the total length of rod in the ground including tip and/or pump. For peristaltic, the tip length used is 0.4 feet. For gas drive, the length of the tip and pump is 3.2', so add this to the length of rod in the ground.

12. Compare the measured value to the depth reading on the screen:



Correct Depth

If the measured depth is different than the depth given in the "Original Uncorrected Depth" box, enter the correct depth into the "Input Corrected Depth" box and click on the "Enter New Depth" button. You can also change the feet of stainless if required. If no other data needs to be corrected, click on the "Done Entering Data" button on the software.

13. **Measuring Hydraulic Head:** Observe the Zeroed Pressure text box. When this value equilibrates, record the value on the field form along with the depth (Attachment 2). The water in the stainless steel tubing will equilibrate to the head of the groundwater when the flow is turned off. Turn on the flow and leave it on for 30 seconds. Turn off the flow and again observe the Zeroed Pressure and wait for equilibration. If this value matches the first value, the head measurement is valid. If it is different, repeat turning the flow on / off until two successive head readings are obtained.

    NOTE: When the water table is at a depth of more than 25' the head value is not valid. When using gas drive mode this is often the case. If so do not record the head value.

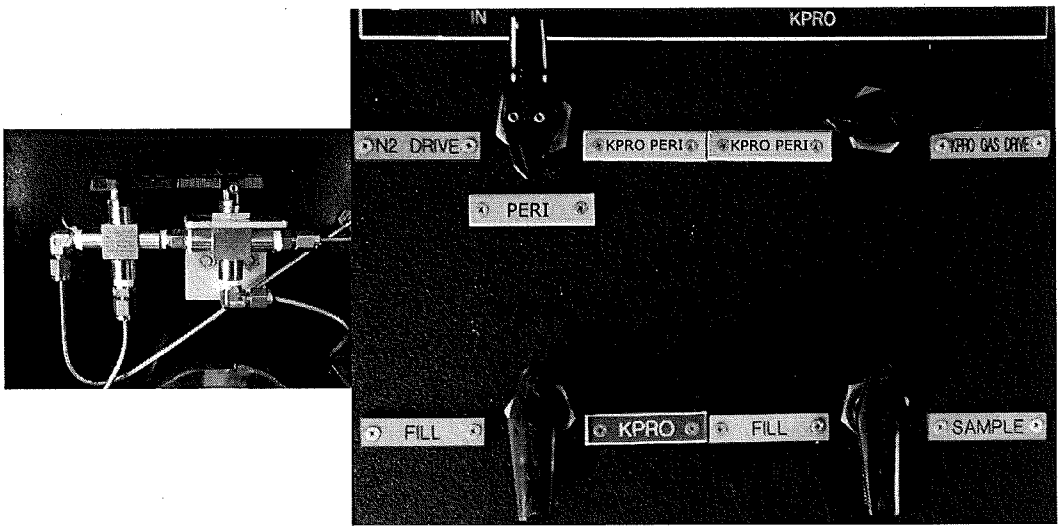
14. Double click on the appropriate Sonde water quality monitoring system icon on the notebook computer desktop (Ecowatch for the YSI, Win-Situ for the Troll, and HydraS3 LT for the Hydrolab). Instructions on how to operate the specific software for each Sonde can be found in SEI SOP 5.25.0 (Hydro Lab MS-5), 5.27.0 (Troll 9000 and 9500) and 10.7.1 (YSI 600XL).

15. **Missing a sampling interval.** It may be required to pull the profiler up to sample an interval that was passed on the way down. To do this first make sure that the "Finish KPRO" button has been clicked. For Peri mode make sure that the flow is on, and in gas drive mode make sure that the pump is pressurized and the flow is on. Determine at what interval you would like to sample . Hold a measuring stick next to the drill rod as the driller pulls back on the rod and have the operator stop when the depth is achieved. Check the flow on the software to be sure that the interval will yield a timely sample. It is also possible to watch the flow on the KPRO software as the driller pulls the rod up. When the flow increases, tell the driller to stop. Measure the stickup and record in the paper log that the profiler was pulled up and record the new depth. Before your next drive you will have to correct the depth in the KPRO software. To do this do a "fake" drive by starting and stopping the software as you would during a drive but correct the depth to the new depth. You can also mark the rod before pulling it back and then drive the rod back to that depth (with the flow on) before starting the program.

4.7 Purging the Profiler and Sample Collection

4.7.1 Purging in Peristaltic Mode

1. Turn on the peristaltic pump and set the +/- selector to –
2. Set the valves shown below.



Peri Mode Purging

3. Observe the vacuum gauge. A vacuum should build toward -30 inches of mercury
4. Observe water flowing through the bottles. The water will be clear at first but should become silty. This is because there is some KPRO water still in the tubing.
5. If vacuum does not build and water does not flow it is possible that there is a leak in the system. Refer to the Problem Solver's Guide on how to proceed.
6. Observe the volume of water in the graduated cylinder into which the purge water discharges. On the Groundwater Profiling Log, select the tab "Sample 1". The top section of the sheet except the date should be populated with data that was entered on the first tab. Record the time, cumulative purge volume and physio-chemical parameters in the Excel spreadsheet after every 50-100 mL of sample discharges to graduated cylinder. This data is used as a benchmark for future readings and is not recorded in the field form (paper log).
7. If the sample purge rate is slow (less than 10 ml/min) then it may be necessary to surge the pump. Sometimes silt can get smeared on the outside of the profiling tip or can be drawn into the tip. Surging the pump can loosen up the silt to increase the flow. Reverse the flow of the pump by turning it off, then switching the directional switch. Turn the pump back on and observe the pressure gauge. Once the gauge maxes out, turn off the pump and reverse again. Repeat this two to three times. Continue to purge the sample. If the purge rate is still slow, it may be necessary to drive the profiler to a better sampling interval. If no sample was collected, record this in the Profile Info tab of the log.

8. When all of the physico-chemical parameters stabilize on two successive readings (separated by 50-100 mL, indicated by yellow in the Microsoft Excel worksheet) record the depth, time, head value, total volume purged and the values of the physico-chemical parameters on the paper field form (only record the final line of stabilization parameters). The paper log serves as a backup in case there is a problem with the electronic Groundwater Profiling Log. Put any character in the column labeled "Final ?" (located to the left of the depth column) next to the last row of data. This tells the spreadsheet to display that row of data on the "Groundwater Profiling Log" tab. The following photo illustrates what the data looks like for a sample that has equilibrated.

PARAMETER EQUILIBRATION LOG

STONE ENVIRONMENTAL INC

Client: ERM — Profile Location: APS-018

Date: 5/27/2010 — KPRO Box Serial #: 808/ Rasc7 — Gas Drive or Peri Pump: Gas

Location: Plainville, CT — Troll Serial #: o2k0934 YSI — Atmospheric Pressure: 34.89

SEI #: 092193-R — Drilling Contractor: Platform — KPRO N_2 Pressure (set via P tranducer): 70.14

Sampler(s): VLD — Depth to Water: -17.2818182 — Gas Drive Pump N_2 Pressure: 80-100

				PHYSICOCHEMICAL PARAMETERS								
Depth (ft)	Head (ft)	Time	Volume Purged (mL)	SC (uS/cm)	SC %Change	DO (PPM)	DO %Change	pH	pH %Change	ORP (mV)	ORP %Change	COMMENTS
-41.00	-17.20	16:27	300	136		5.50		6.30		81		
		16:29	480	159	16.9	6.60	20.0	6.30	0.0	77	4.9	
		16:32	650	183	15.1	8.00	21.2	6.36	1.0	78	1.3	
		16:34	800	197	7.7	8.20	2.5	6.35	0.2	80	2.6	
		16:36	900	201	2.0	7.60	7.3	6.33	0.3	83	3.8	
		16:38	1000	202	0.5	7.50	1.3	6.30	0.5	83	0.0	

Sample Log Showing Equilibrated Sample

Notes on Purging the Sample: Make sure to purge at least 500ml before collecting a sample. The first few physico-chemical readings may indicate that the sample has equilibrated but that is because the tubing contains the analyte free water and there is still water from the previous sample interval in the flow through cell of the water quality sonde. The volume of the flow cell is usually 100-200 ml, so it will take some time for the groundwater from the current interval to replace the water in the flow cell from the previous sample.

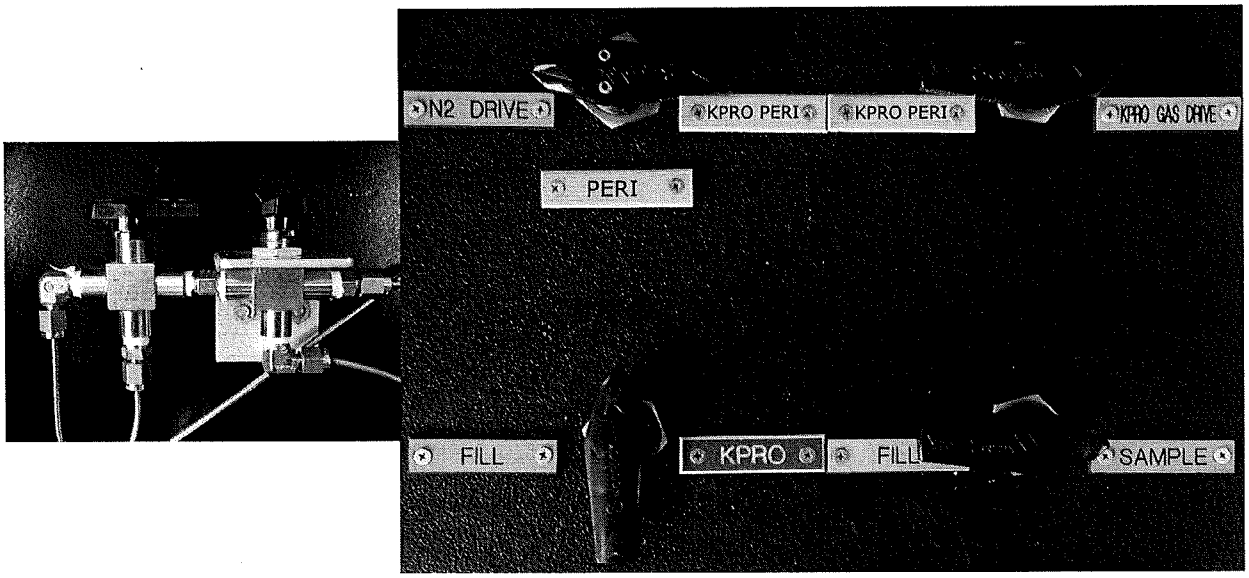
4.7.2 Collect Sample in Peristaltic Mode

1. Turn the top right valve on the sample jig so that it is locked off (pointing towards the back of the box).

2. Reverse the direction of the pump by changing the toggle switch on the box panel from minus (-) to plus (+).
3. Run the pump backward for 2 to 3 seconds
4. Shut off the pump
5. Remove the sample bottle on the left.
6. Remove the bottle on the right.
7. If there is headspace in the tops of the vials (this is normal), hold a vial cap under the right bottle holder and run the pump in reverse slowly. Water will flow into the vial cap. Transfer this water from the vial cap to the vials until the headspace is gone and cap the vial. Turn the vial upside down and tap the vial lightly on the palm of your hand. If there are any air bubbles present, you must uncap the bottle and repeat the headspace procedure.
8. Label the vials appropriately. Note the time of sampling on the field form.
9. Replace the vials with new vials. Prepare to drive as before after adding tubing and rod as necessary.

4.7.3 Purging in Gas Drive Mode

1. At this point the pump is already pressurized from driving. Arrange the valves per the following photo:



Pressurized Gas Drive Pump Ready to Purge

Turn the top right valve on the sample jig slowly to the right. This allows the nitrogen to push the water up from the pump and through the sample jig. The pressure on the gauge will drop and stabilize as the water flows.

2. Observe the pressure gauge. Just before the pump body is completely emptied, the pressure will start to rise. When you observe the rise, turn the top left valve on the KPRO box 90 degrees clockwise, which will stop the flow of nitrogen and vent the gas out of the pump (you will hear a hissing sound).

3. Watch the pressure gauge and when the pressure drops below zero, lock off the right valve on the sample jig and spin the top left valve on the KPRO box clockwise to Peri. Make sure that the peristaltic pump is on. At this point the pump is depressurized and is filling with groundwater. These three steps complete one pump purge, or "whack".

4. Observe the volume of water in the graduated cylinder into which the purge water discharges. Record the time, cumulative purge volume and physico-chemical parameters in the Excel spreadsheet after every purge of sample discharges to graduated cylinder. This data is used as a benchmark for future readings and is not recorded in the field form (paper log). The first whack will be larger than most because the pump and lines are already filled with water. If after the second whack the amount of sample discharged is less than 80 ml then you need to wait longer between evacuations (3-4 minutes). If after waiting 5-6 minutes the pump evacuation yields less than 50 ml, it may not be prudent to collect a sample at that location. Check with the client oversight to see if they want you to continue sampling or drive to a better sampling interval. If the volume is significantly larger (150-200ml) you can evacuate the pump more often (1-2 minutes). If no sample was collected, record this in the Profile Info tab of the log.

   Note on purge volume: The volume of the gas drive pump is 100 ml. The volume of the stainless steel tubing is 0.8 ml/ foot, and there are two lines (the sample and gas lines) that are full of water (water from the previous sample and water used to fill the pump). For example, if you have 60 feet of tubing attached, the volume would be 100ml + (2 *60*0.8), for a total of 196 ml. When purging a sample this water will be purged first so the physico-chemical parameters may appear to be stable. It is common to see the parameters rise (previous sample water left in pump), then fall (analyte free water used to fill the pump) before starting to equilibrate (fresh groundwater).

5. After the previous whack, allow some time (as explained in step 4) for the pump body to fill such that you receive at least 50 mL of sample on subsequent evacuation (Step 4).

6. To purge the pump turn the top left valve on the KPRO box to N2 and watch the pressure gauge. When the pressure crosses zero, turn the top right valve on the sample

jig to the right. You are now back at Step 1. Follow the rest of the steps to complete the whack and record the parameters.

7. When all of the physico-chemical parameters stabilize on two successive readings (indicated by yellow in the Microsoft Excel worksheet) record the depth, time, head value, total volume purged and the values of the physico-chemical parameters on the paper field form. The paper log serves as a backup in case there is a problem with the electronic Groundwater Profiling Log. Put any character in the column labeled "Final ?" next to the last row of data. This tells the spreadsheet to display that row of data on the "Groundwater Profiling Log" tab.

4.7.4 Collect Sample in Gas Drive Mode

1. Arrange the valves as you would for purging the pump but when the pressure builds do not open the valve on the sampling jig.
2. Remove the sample bottle on the left.
3. Hold the sample vial under the straw for the first vial holder on the sample jig. Slowly open the right valve on the sample jig. Water will flow out of the straw and into the vial. When you have a mounding of water on the top of the vial, shut the valve and cap the vial. Repeat this process for subsequent vials. Check for bubble by inverting the vial and lightly tapping it on the palm of your hand. If there are bubbles, uncap the vial and fill again.
4. Label the vials appropriately. Note the time of sampling on the field form.
5. Replace the vials with new vials.
6. Prepare to drive as before after adding tubing and rod as necessary by filling and pressurizing the pump. Refer to section 4.6 above for the procedure.

4.8 How to Determine the End of the Profile

There are several ways to determine the end of a profile. Some profiles have a predetermined depth set by the client. The end of the profile is most often determined by the Drive Rate. As the profiler is advanced into the ground the drive rate will decrease as the skin friction of the rod in the ground increases. There are also stratigraphic layers that are more compact than others (clays, tills) that can slow the drive rate. It is important not to drive the profiler once the drive rate gets below 1.5-2 ft per minute for the 1.5 and 1.75-inch rods and 0.5-1.0 ft per min for the 2.25-inch rods. If the rods, tips and drive hardware are hammered on too much they can fatigue and break, crack or bend. The skin friction can also become too great so that the profiling rods cannot be pulled back out of the ground. Consult with the driller / probe operator during profiling to determine when to stop drilling.

It is also possible to drill into bedrock or other subsurface objects (boulders, cobbles, concrete chunks or slabs, etc.). In this case the drive rate can drop very quickly. Stop hammering immediately and discus with the driller and client how to proceed.

Be sure to note on the paper field log as well as the Profile Info tab in the Groundwater Profiling Log how the profile was completed.

4.9 Decontamination

The only decontamination step required between samples in the same hole is to flush the line with analyte free water (distilled or spring water) while driving to the next depth. There are required decontamination procedures for the tip and pump, drill rod, stainless steel tubing and KPRO box that need to be completed at the end of a profile. Some sites and clients may require that all the down-hole tooling be decontaminated using a steam cleaner. If this is the case remove the plates and screens from the tip and take the pump out of the housing so that all of the parts can be cleaned. Usually the drillers will provide the steam cleaner and will clean everything.

4.9.1 Tip and Pump Decontamination

The tip and pump need to be disassembled and cleaned prior to reuse. For the tip, remove the plates and screens. The screws can be reused if they are not rusted or stripped. It may be required to use an impact driver to remove the screws if they are stuck. Rinse out the inside of the tip as well as the screens and plates with Alconox and water to remove all sediment. Scrub the tip and plates with a firm brush or use a pipe cleaner to lean the inside of the tip. Carefully check the plates to make sure that the sampling ports are clear. Use a pick to remove any sediment from the sampling port holes. Remove and replace the FEP tubing. The piston for the gas drive system can be reused.

For the pump: unthread the tip from the pump housing and unthread the top and bottom housing pieces. Take the pump body out of the housing. Use a brush and bucket of Alconox and water to clean the inside and outside of the housing pieces and pump as well as the springs and plastic bushing. Disassemble the pump body and discard the o-rings, reed valve and Teflon tape. Rinse out the inside of the pump with Alconox and water and then clean water. Check the small o-rings at the narrow end of the pump bottom and replace if necessary.

4.9.2 Drill Rod and Stainless Steel Tubing Decontamination

Upon completion of the sampling and grouting of the hole, the drill rods should be cleaned by steam cleaning, pressure washing or by washing with Alconox and water. It is important to clean the inside, outside and threads of the rod. The stainless steel tubes should have any grout, drilling mud or soil removed from the outside using a steam cleaner, pressure washer or Alconox and water. The inside should be rinsed with analyte free water (distilled or spring water). It is very important that before the stainless steel tubing is reused that it is checked for clogs by using a garden sprayer with an adaptor.

4.9.3 KPRO Box Decontamination and Equipment Blank Procedure

The KPRO box should be decontaminated at the end of each profiling location to make sure that there is no possibility of cross contamination between profiles.

1. The four o-rings located at the top of each bottle holder on the sample jig should be replaced at the completion of the profile. Remove any vials from the sample jig and discard them. Remove the o-rings located at the top of each bottle holder with a straight or slightly angled pick, being careful not to score the threads on the plastic bottle holder. Alconox and water should be squirted up into the top of each bottle holder before the new o-rings are installed.
2. After changing out the o-rings on the sample jig, the box and jig should be flushed out with at least 1L of Alconox and water and 1L of clean water. This can be done in conjunction with the collection of an equipment blank (as required).
3. For peristaltic mode, connect a clean profiling tip to the KPRO box via the harness. Place the tip into a clean vessel containing Alconox and water (the vessel needs only to be as deep as to make sure all of the ports on the tip are submerged). Screw four new vials into the sample jig and arrange the valves on the box to the same orientation for sample collection. Turn on the peristaltic pump. Top off the water in the vessel as the water is pumped through the box. Once you have pumped 1L of Alconox and water, place the profile tip into a second clean vessel containing only clean, analyte free water and pump 1L of clean water through the box.
4. For gas drive mode, connect all three stainless steel tubes from the pump to the corresponding tubes on the harness and place the tip of the pump in a clean vessel containing Alconox and water. Screw four new vials into the sample jig and arrange the valves on the box to the same orientation for sample collection in peristaltic mode. Disconnect the N2 Out quick connect from the KPRO box. Disconnect the Sample In quick connect and connect it to the N2 Out quick connect and turn on the peristaltic pump. Top off the water in the vessel as the water is pumped through the box. Once you have pumped 1L of Alconox and water, place the profile tip / pump into a second clean vessel containing only clean, analyte free water and pump 1L through the box.
5. If an equipment blank is required, simply collect the required number of voa vials from the sample jig as you would if you were collecting a groundwater sample using the peristaltic mode. If no equipment blank is required, the decontamination process is complete.
6. Record on the paper Groundwater Profile Log that the box has been decontaminated with 1L of Alconox and water and 1L of clean water. Record the date, time and name of the equipment blank (if required).

4.10 Profile Field Data Deliverables

One of the biggest advantages to using the Waterloo ^{APS} is that field data can be quickly processed in the field and given to the client in a matter of minutes. At the end of the profile the raw data needs to be processed so that it can be imported into either Origin to provide a field log to the client or into gINT.

The data should be processed as soon as possible after finishing a profile; either during the decontamination of the KPRO box or during the collection of the first few samples of the following profile. All efforts should be made to get the data to the client by the end of the working day unless otherwise directed. These are the steps:

1. Open the kpro_raw.txt file associated with the profile. Select All (shortcut - Control A) and Copy All (Control C).
2. In the Groundwater Profiling Log Excel workbook, select the Processed Ik tab. Select the top left cell and Paste (Control V) the raw data.
3. This is where alterations or corrections can be made to the data. You can change the depth to water, feet of stainless or other parameters that the software uses to calculate the I_k. If you pulled the profiler up you can delete duplicate data as well.
4. After any corrections are made, scroll to the two far right columns of data; "Corrected Depth to Water" and "New Ik". Both of these columns have data in the first row only. Highlight the data cell and double click on the small black square at the bottom right corner of the cell. This will populate the entire column with data. Do this for both columns.
5. Copy the entire New Ik column. Right click on the top row of the Ik column and select Paste Special – Values. This will overwrite the I_k that was generated by the software in real time with a corrected Ik based on any changes that were made to the data. Even if no changes were made to the data this step should be done anyway.
6. Select the Ik Plot tab. Follow the instructions on the screen to plot the Ik data. Carefully check the plot for any errors or bad data. Bad data can be generated a variety of ways and should be deleted from the plot. Examples of bad data are: driving the profiler with the flow off (flow is zero), driving the profiler without starting the software (no data is recorded), not stopping the software after a drive, entering the wrong depth after a drive, etc. If any of these things happen, delete or change the bad data from the Processed Ik tab. Leave a blank row where the data was deleted so that the Ik Plot does not connect the data with a straight line.

   There are other cases where after adding a length of stainless tubing the flow drops so there is a horizontal line on the plot. This data must be shifted up or down to match the previous data. Consult the Data Manager or Project Manager on how to proceed.

7. Open the Origin file named "Physchem_hole_id.opj". This file should be located in the Origin folder in the project directory or in the Forms folder. Save the file to reflect the profile name (Pyschem_WP-01.opj). If you are using gas drive mode and did not collect head measurements, select the file "Physchem_No_Head_hole_id.opj".

8. Select and copy all the data on the Processed Ik tab on the Groundwater Profiling Log. On the Origin file select the Ik record sheet from the bottom of the screen. Select the top left cell and paste the data. It may take a few seconds to paste all of the data.

9. Back on the Groundwater Profiling Log click on the Groundwater Profiling Log tab. Select all the data and paste it into the Physchem sheet in the Origin file.

10. Click on the Layout sheet on the bottom of the Origin file. Check the plots to make sure that the scale is correct and all the data is displayed properly. If the scale needs to be changed select the PhysPlot sheet on the Origin plot and select the axis of the graph you want to change. Select the Scale tab to change the range of values. It is important that all of the plots from a particular site are consistent. Finally, save the file ad a PDF.

11. Provide the client with a copy of the Origin plot by copying the PDF file to a data stick or printing out a hard copy. Double check the Groundwater Profiling Log and make sure that all of the fields on the Groundwater Profiling tab, Profile Info tab and processed Ik tab are completed. At the end of the day email the data (only the Groundwater Profiling Logs and a scan or photo of the field form) to the Project Manager and/or Data Manager and back up the entire project folder to a data stick.

12. Metric Data – There are versions of all of the electronic field logs in metric (either as depth in meters or elevation). There is also a version of the KPRO software that displays the depth in meters but does not record the depth in meters to the kpro_raw file. Before importing the data to the Origin plot it needs to be converted from feet to meters in the Processed Ik tab. To do this insert a blank column to the right of the Corrected Depth column after all of the changes or corrections have been made to the data. In the first cell of the new column create a formula that divides the Corrected Depth by 3.281. (=C2/3.281 where C2 is the cell of the depth you want to convert). Copy this formula to the entire column. Select and copy the entire column and right click on the Corrected Depth column and select Paste Special – Values. Delete the column where the data was copied from.

4.11 Cold Weather Operation

During the winter months when temperatures fall below freezing special precautions need to be taken to protect and successfully operate the Waterloo^{APS} equipment, KPRO box, and associated equipment.

4.11.1 Exposed Stainless Steel Tubing and Rods

While attempting to collect a groundwater samples with below freezing temperatures a cold weather harness may be required. The cold weather harness is a combination of heat tape and pipe insulation wrapped around the stainless steel tubing exiting the KPRO Box. In extreme cold the rod stickup and stainless steel tubing exposed between the cold weather harness and the ground may need to be heated using either a brush-burner or torpedo heater. Flexible duct work can be used to cover the stainless steel and exposed rod to direct the heat from the torpedo heater and to help insulate.

4.11.2 Leaving the site overnight

If freezing conditions will occur overnight the KPRO Box needs to be winterized. The enclosure should be removed and stored where temperatures will not fall below freezing. The KPRO box and cold weather harness should be drained of water:

1. Disconnect the stainless steel tubing harness from the down-hole tubing.
2. Remove the enclosure and replace with a short piece of stainless steel tubing with a female quick connect at each end or leave the enclosure in place.
3. Disconnect the nitrogen "in" (white) KPRO vessel quick connect.
4. Remove the water "out" (black) KPRO vessel quick connect.
5. Turn the regulator dial counter clockwise so that regulator is off. Attach the stainless steel piece that was connected to the "out" (black) quick connect to the low pressure regulator on the nitrogen tank.
6. In the peristaltic pump mode set up the valves on the box as if you were in drive mode and slowly turn the knob on the regulator clockwise slowly until the smallest amount of nitrogen is flowing. If the pressure is too high the flow meter in the enclosure can be damaged. After a few seconds you should see water being pushed out of the harness. After all of the water has been pushed out turn off the nitrogen regulator.
7. In gas drive mode you need to blow out the KPRO box as well as all three of the harness lines. To blow out the nitrogen line, simply turn the 5 way valve to the N2 Out and wait for all of the water to be pushed out of the nitrogen line on the harness. To blow out the sample line (middle tube on the harness), arrange the valves as you would if you were filling the pump. Disconnect the N2 In and KPRO in quick connects ont eh KPRO box. Connect the N2 In to the KPRO In. Slowly turn the knob on the regulator clockwise slowly until the smallest amount of nitrogen is flowing and wait for all of the water to be pushed out of the sample line. To blow out the KPRO line, set up the valves on the box as if you were in drive mode and slowly turn the knob on the regulator clockwise slowly until the smallest amount of nitrogen is flowing. If the pressure is too high the flow meter in the enclosure can be damaged. After a few seconds you should

see water being pushed out of the harness. After all of the water has been pushed out turn off the nitrogen regulator.

The KPRO enclosure, KPRO vessel, "out" (black) quick connect, Sonde water quality monitoring system, and calibration solutions should be stored where temperatures will not fall below freezing.

4.12 Other Profiling Configurations

It is possible to set up the KPRO box on a table, cart, tailgate or other surface in order to profile in locations that cannot be accessed with the truck. In instances such as this it may be necessary to power the equipment using a battery. Do not use a standard car battery as repeated draining and charging will diminish its capacity over time. It is better to use a deep cycle RV or similar battery that is designed to power devices and not for starting engines. It is possible to power the KPRO box (including peristaltic pump), field computer and water quality sonde off of a battery for an entire 10 hour day as long as the battery is charged at night. The KPRO box can be connected directly to the battery because it already is 12V. The computer and sonde need to be connected to a DC to AC power inverter that is connected to the battery.

5.0 RESPONSIBILITIES

1. It is the responsibility of the individual employee to read SOPs and document training associated with the area of work they are performing.
2. It is the responsibility of the individual employee to follow SOPs covering activities in his/her work area or to identify a deviation from the written SOP.
3. All personnel will legibly record data and observation to enable others to reconstruct project events and provide sufficient evidence of activities conducted.
4. All personnel will label each page with the date, the signature of the person taking notes (initials may also be appropriate), and the page number. All notes, signatures, and other observations should be entered in the field at the time the notes are taken.
5. SEI field staff should take care to ensure proper data management and integrity of samples.

6.0 DEFINITIONS

KPRO: Hydraulic Conductivity (K) Profiling

7.0 REFERENCES

None

8.0 TABLES, DIAGRAMS, FLOWCHARTS, AND VALIDATION DATA

Diagrams and Attachments:

1. Schematic of Waterloo Profiler Tips and Gas Drive Pump
2. Attachment 1: Groundwater Profiling and KPRO Testing Equipment List
3. Attachment 2: Groundwater Profiling Field Form

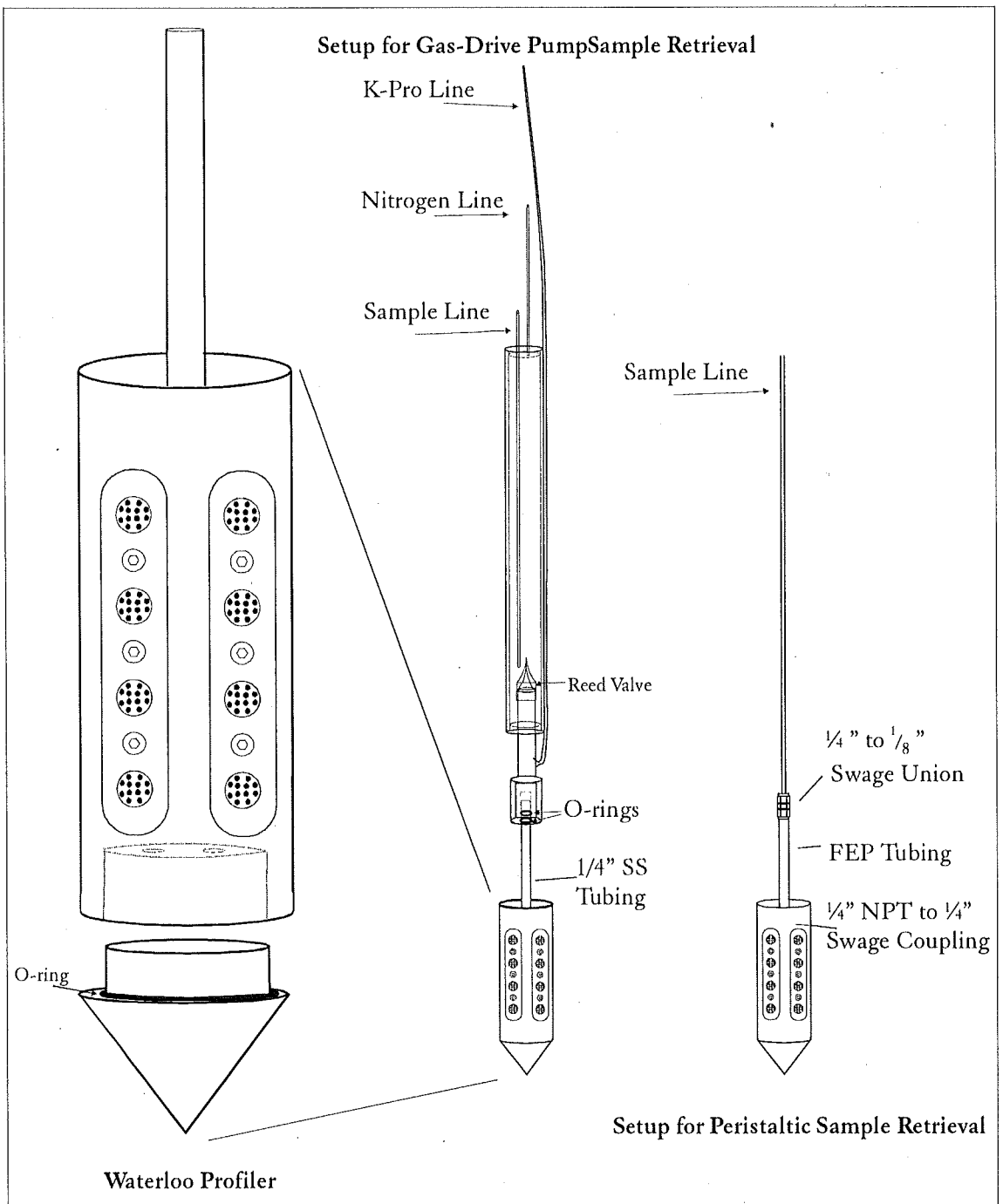


FIGURE 4.1: Schematic of Waterloo profiler with Gas-Drive and Peristaltic Retrieval Systems
SOP #10-5-0: *GROUNDWATER PROFILING AND KPRO TESTING*

Source: I:\\GLP\SOP\SEI_SOPs\CH10_EnvDrill\current\10-5-0 Figure 4.1
7-10-02 dtv

STONE ENVIRONMENTAL INC

9.0 AUTHORIZATION

Revised by: _____ for _____ Date: 9/24/12

Vincent DeLeone, Waterloo APS Service Leader

Approved by: _____ Date: 24 AUG 2012

Seth Pitkin, Vice President

10.0 REVISION HISTORY

Revision 1:

1. Modified Attachment 1: Groundwater Profiling and K-Pro Testing Equipment Checklist.
2. Modified Attachment 2: Groundwater Profiling Log

Revision 2:

1. ADD trade name APS Advance Profiling System to Waterloo.
2. Under Section 4, changed startup and calibration procedures for the KPRO software to reflect the new KPRO-2006 revision.

Revision 3:

1. Renumbered to take out of laboratory SOP location.
2. Changed and added text
3. Changed the order of steps
4. Replaced photos of valve orientation
5. Deleted old KPRO box photos
6. Added photos of KPRO software
7. Added text for Data Processing
8. Replace KPRO checklist
9. Update Problem Solver
10. Update quick reference Guide
11. Added text and figures about the 2.25" tip and rod

ATTACHMENT 1

GROUNDWATER PROFILING AND KPRO TESTING
EQUIPMENT CHECKLIST

PROFILING EQUIPMENT

- _ Profiler Tips (Sac-tip or Integral)
  - _ Sacrificial Tip
  - _ O-rings for Sac Tip
  - _ SS Profiler Screens (4 per tip)
  - _ SS Allen Screws for Screen Plates
- _ 5' x 1.75" Drill Rod (2 X max. anticipated depth)
- _ 2' x 1.75" Drill Rod (1)
- _ Swivel pull
- _ Geoprobe Pull Clamp for 1.75" Rod
- _ Drive Cap (3-4)
- _ Slip Cap (3)
- _ $^1/_8$" SS Tubing
- _ SS Caps for SS Tubing
- _ Extra $^1/_8$" SS Compression Fittings
- _ Stainless steel mesh for tips
  - o 80 X 80
  - o 60 X 60
  - o 100X 100
- _ 1½" Geoprobe X 1¾" Rope crossover (for hooking to grout pump swivel)

KPRO TESTING BOX AND RELATED EQUIPMENT

- _ String Pot
  - _ Com Cable (2)
  - _ Quick Clamp
- _ Compressed N_2 Tank (Large(T) for Gas drive, Small(Q) for peri-sampling)
- _ Dual phase regulator (0-60 psi) for K-pro
- _ Double Pressure Regulator Set (1, 0-60 psi; 1, 0-600 psi), if gas drive
- _ Pressure Vessel (2)
- _ Pressure Vessel quick connects
  - o White-N_2 side (2)
  - o Black-Water side (2)
- _ $^1/_8$" SS Tubing Harness
  - _ $^1/_8$" swage to quick connect (peri/gas) (2+spare)
  - _ $^1/_8$"swage to quick connect (gas drive) (5+spare)
- _ KPRO Box
  - _ Com Cable (2)
  - _ Power Cable (2)
- _ Regulated Power Supply
- _ 1 Liter Graduated Cylinder (plastic)
- _ Field Computer (2)
  - _ Serial Port Card, PCMCIA
  - _ Power Adapter
  - _ CD-Drive
  - _ Floppy Drive
  - _ Extra Battery and Charger
  - _ USB Data Stick
  - _ External Keyboard and Mouse, USB
  - _ USB Hub (optional)
  - _ Field Software CD
  - _ Current Templates
  - _ Blank CD's
- _ Color Printer
  - _ Com Cable (parallel or USB)
  - _ Power Cable
- _ Printer Paper
- _ 5 Gallon Pails for Purge Water/Decon (2)
- _ Water Quality Sonde
  - _ Com Cable
  - _ Flow Cell
  - _ extra D-cell Batteries (Troll)
  - _ power cable (YSI)
  - _ Calibration Standards
  - _ DO membrane maint. kit (Troll)
- _ Field Book/Field Forms

GAS DRIVE PUMP ASSEMBLY

- _ 2.4' x 1.75" female – female drill rod (3-4)
- _ male – male pin (bottom) (3-4)
- _ male – female pin (top) (3-4)
- _ Springs (2 per pump)
- _ Nylon Bushing (1 per pump)
- _ Pump Bottom
- _ Pump Body
- _ Pump Top
- _ Reed Valves (bag of 50)
- _ O-rings for Pump Body
- _ O-rings for Pump Bottom
- _ ¼" SS Tubing (10)
- _ ¼" NPT male to ¼" compression fitting
- _ ¼" Ferrules, SS

PERI MODE TIP ASSEMBLY

- _ FEP Tubing (1/4" OD X 3/16 ID)
- _ ¼" NPT male to ¼" compression fitting
- _ Ferrules, Nylon
- _ Barbed inserts for FEP Tubing, SS (3/16 OD)
- _ ¼" compression to $^1/_8$" compression fitting

SAMPLE COLLECTION

- _ Quatro-rings for Sample Jig
- _ 40mL VOA Vials (Eagle Pitcher) (2-3 cases)
- _ Vial Labels
- _ Silicon tubing for Peri Pump (10 feet)
- _ Coolers and Ice

DECONTAMINATION EQUIPMENT

- _ Alconox
- _ Squirt Bottles
- _ Garden Spray Bottle (3 gallon)
- _ Plastic Sheeting
- _ Contractor Garbage Bags

AUXILIARY EQUIPMENT

- _ Lumber Crayons
- _ 100' Engineers Tape
- _ Electrical Tape
- _ Ratchet Straps and Bungee Cords
- _ Teflon Tape
- _ Colored Zip Ties
- _ Halogen Work Lights
  - _ Extra Bulb
- _ Chair
- _ Fan (optional)
- _ Padlock with Long Hasp
- _ Coffee Maker
  - _ Green Mountain Coffee French Roast Beans
  - _ Grinder (if whole beans used)
  - _ Filters
  - _ Coffee Cups
- _ Paper Towels
- _ Broom
- _ Ground Fault Circuit Interrupt
- _ Extension Cords
- _ Land line for Circuit panel
- _ Surge Protector

PERSONAL PROTECTIVE EQUIPMENT/SAFETY EQUIPMENT

- _ Nitrile Gloves (2 cases)
- _ Cotton liner Gloves (4-6 pairs)
- _ Leather Work Gloves
- _ Safety Glasses
- _ Hardhats (1 per person + spare)
- _ Hearing Protection

- _ Tyvek Suits
- _ Emergency Eyewash
- _ First Aid Kit
- _ Compressed Air Horn
- _ Respirator and Cartridges
- _ Spill Kit
- _ Spare absorbent Pads
- _ Fire Extinguisher
- _ Flashlight

HAND TOOLS AND POWER EQUIPMENT

- _ $^{7}/_{16}$" Wrenches (4)
- _ Pipe Wrenches, 2, 24"
- _ Open Ended Wrenches, Standard, Full Set
- _ Screw Drivers, Regular and Phillips
- _ Impact Wrench
- _ Oil Filter Wrench (suitable to
- _ Allen Wrenches, Standard and Metric
- _ Crescent Wrench
- _ Socket Set, Standard, Full Set
- _ 9/16" Socket, Open Ended for removing fittings from Profile Tip
- _ Taps for Profiler Tip Screws
- _ Tap Handle
- _ Torx Bits, T-25
- _ Picks, Full Set
- _ Pliers, Standard, Needle Nose, and Channel Lock
- _ Vise Grips
- _ Wire Strippers
- _ Electrical Multi-meter
- _ Soldering Iron
- _ Tin Snips
- _ SS Screen Hole Punch
- _ Files
- _ Utility Knife
  - _ Extra Blades
- _ Tubing Cutter
- _ Sledge Hammer, 3lb
- _ Butane Lighter
- _ Electric Drill
- _ Drill Bits, Full Set
- _ Carbide Bit for Reaming SS Tubing
- _ Angle Grinder

ADDITIONAL WINTER GEAR

- _ Pipe Insulation
- _ Heat Tape
- _ Propane Heaters
- _ Kerosene Heaters

SWAGE BOX

- _ Ferrules
  - -1/4" Nylon
  - -1/8" Stainless Steel
  - -1/4"Stainless Steel
- _ Extra Profiler Tip Plates (4)
- _ O-Rings for K-pro Reservoir (large and small)
- _ Hose Clamps
- _ 1/8" Elbows
  - -Barb/NPT fittings
  - -Compression/NPT fittings
- _ Spare Pressure Transducer
- _ Spare Flow Meter
- _ ¼" to ¼" Barb Plastic-NPT
- _ ¼" to ¼" Barb Plastic T's
- _ 1/4 " in-line Filters
- _ Spare 5-way valve
- _ Spare 3-way valve
  - -¼" NPT
  - -1/8" NPT
- _ 1/8" SS tube Caps
- _ Extra Regulators
  - - 100 psi
  - - 300psi
- _ Extra pressure gauge
- _ Screws for tip (3/8" #10/32, flathead T-25)
- _ Quick disconnect for stainless (4)

DOT REQUIRED EQUIPMENT

_ Driver's Log Notebook
_ Spare Bus Fuses
_ Flares/Reflective Triangles
_ Fire Extinguisher in Cab
_ Windshield Wiper Fluid
_ Flashlight

VEHICLE MAINTENANCE EQUIPMENT

_ Generator Maintenance Log
_ Truck Maintenance Log
_ Owners/Shop Manual (Generator and Truck)
_ Oil Pan
_ Spare Oil (Generator and Truck)
_ Spare Oil Filters (Generator and Truck)
_ Antifreeze
_ Oil Filter Wrench (Generator and Truck)
_ Battery terminal brush
_ Oil Funnel

RENTAL TRUCK GEAR

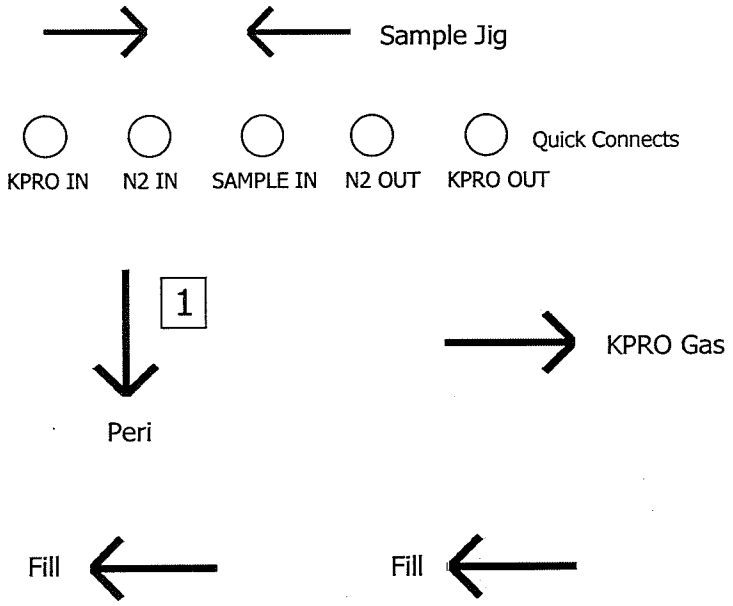
(In addition to the equipment listed above)

ATTACHMENT 2

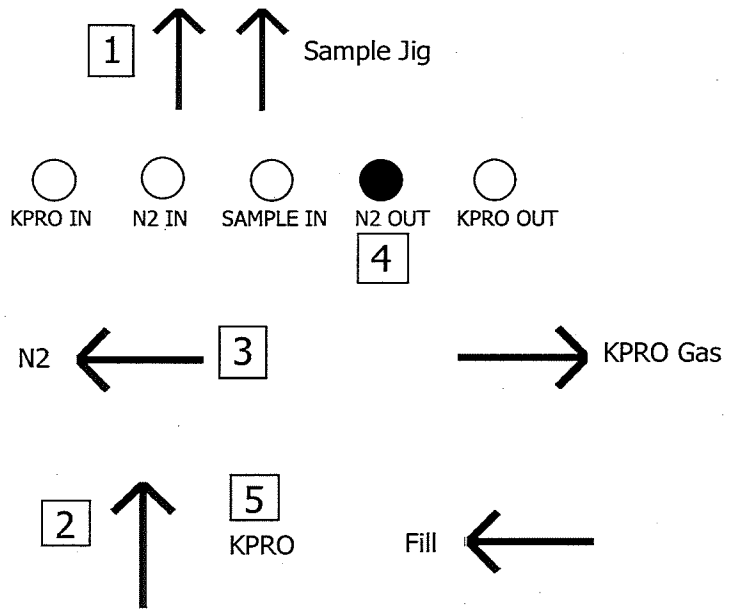
KPRO Quick Reference Guide

Gas Drive Quick Reference Guide

Filling For Drive / Pressure Testing the Pump



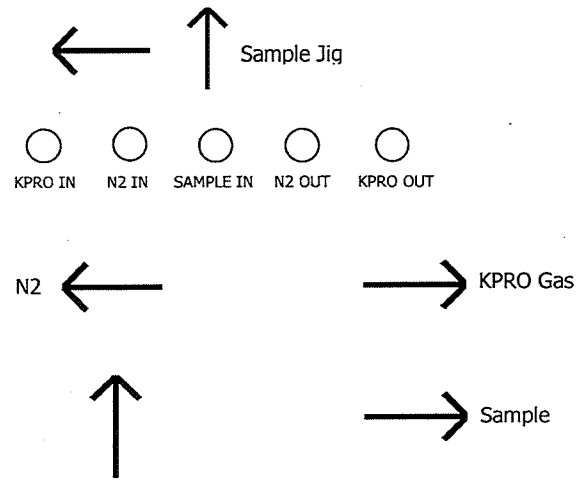
1. Connect the pump to the harness.
2. Arrange the valves as shown. Always turn the 5-way valve clockwise (the top left valve on the bottom, labeled as 1).
3. Make sure that the regulator on the nitrogen tank that supplies the nitrogen to the "N2 IN" is set to the pressure the pump will be used at (to find the pressure, halve the depth, so for 400 feet set the regulator at 200 psi).
4. Turn on the peristaltic pump.
5. The pump is now filling with KPRO water. When the pump is full, you will see water flowing out of the "PERI OUT" tube located at the bottom right hand corner of the KPRO box. This can take several minutes depending on depth. The deeper you are, the longer it will take.
6. Let the water flow out until there are no air bubbles and you get a steady stream.



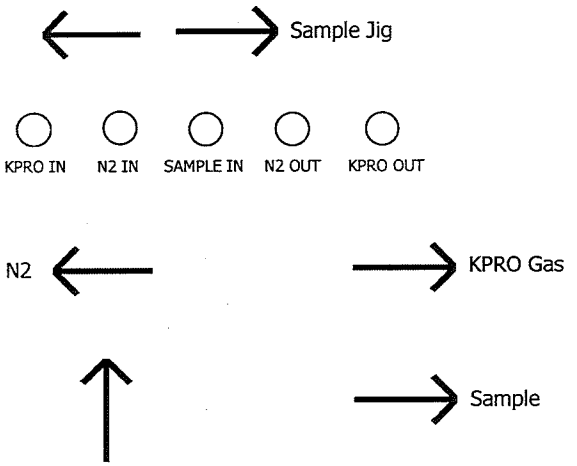
1. Lock off the valves on the sample jig by turning them towards the back (perpendicular to the tubing).
2. Turn off the flow by turning the bottom left valve one quarter turn (which may be to the right or left depending on how the valve was installed.
3. Turn on the nitrogen by turning the top left valve one quarter turn clockwise. You will see the pressure on the gauge rise to the approximate pressure set on the regulator.
4. Disconnect the "N2 OUT" quick connect from the KPRO box. (Second from the right). Observe the pressure gauge. The pressure should drop slightly, then hold. This means that the pump is holding pressure. If the pressure drops significantly then there is a leak. Double check that the fittings connecting the harness to the pump are tight. If this doesn't work, then double check the O-rings and reed valve. If there is still a leak it could be the 5-way valve. Check the Problem Solving Guide for troubleshooting tips./
5. If the pump holds pressure, reconnect the N2 out and turn the flow on by turning the bottom left valve one quarter turn clockwise to "KPRO".

Sampling / Purging The Pump

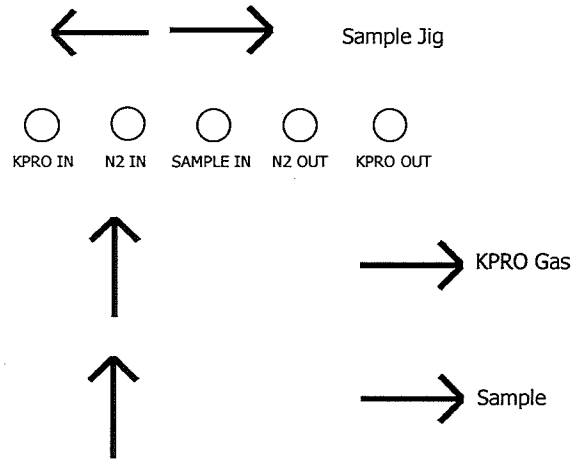
1. After driving to the sample depth and hitting the "Finish" button on the KPRO software, arrange the valves as shown below by turning the left valve on the sample jig to the left and the bottom right valve one half turn clockwise to "Sample". Make sure the Peri pump is on. The Peri pump will help the sample to come in faster at depths up to around 200 feet. Samples deeper do not require the Peri pump to be on.



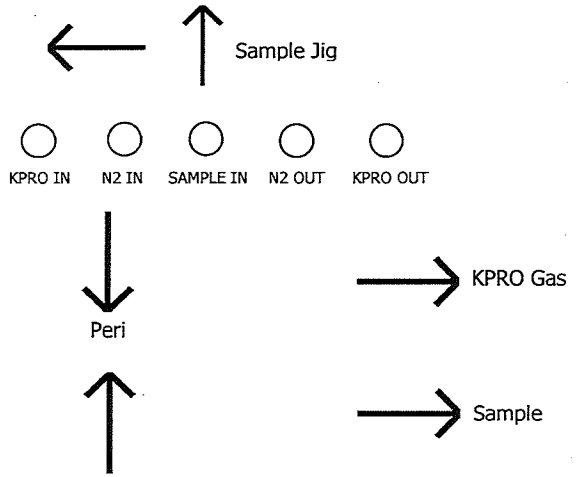
2. Since the pump is already pressurized, turn the right valve on the sample jig so it is facing to the right. This will allow the nitrogen to push the water up from the pump through the sample jig. You should see the VOAs begin to fill. The pressure on the gauge will drop and stabilize.



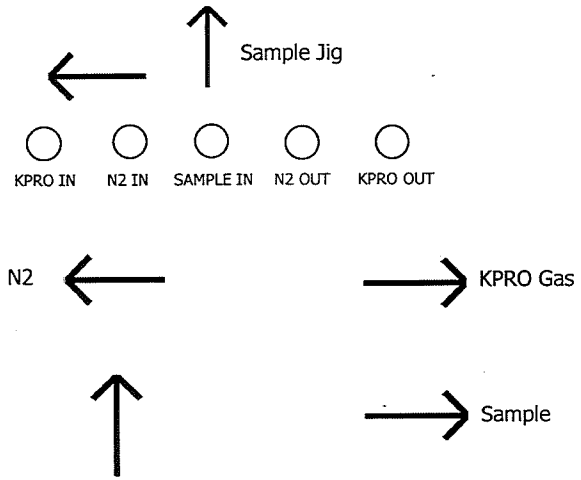
3. Keep a close eye on the pressure gauge. When the pressure gauge starts to move up, turn the top left valve a quarter turn clockwise. This will vent the nitrogen, which will make a hissing sound.



4. After venting the nitrogen, the pressure gauge will drop. When it crosses zero, turn the right valve on the sample jig towards the back (locked off). Wait for all of the gas to vent (the hissing sounds will dissipate) and turn the top left valve one half turn clockwise so it is on "Peri". Record the physchem values.



5. After waiting three minutes (or more or less, depending on how much water was purged during the previous whack) turn the top left valve one quarter turn clockwise to "N2". This will send nitrogen down to pressurize the pump. This is called a "whack". Watch the pressure gauge. When it moves up past zero, turn the right valve on the sample jig to the right. Go back up to step 3 above and follow the sequence.



6. Once the physchem parameters have stabilized and the sample vials have been collected, the pump needs to be filled and pressurized again for driving. Go back up to that section for instructions.

Some notes:
After driving, the stainless steel lines and pump are filled with KPRO water. This water needs to be purged before formation water will appear. To calculate how much KPRO water is in the tubing and pump, multiply the amount of stainless by two, then by 0.8, then add 100. (This accounts for the volume of the tubing going down, coming back up and the pump. There is 0.8 ml per foot of tubing). The first whack should produce almost all of this volume of water.
A good whack should yield 50-60 ml minimum. If you do not get this volume after waiting 3 minutes, wait longer (4 minutes, then 5, etc). If you get more volume than 50-60, do the whacks more often (2 minutes). If you do not get 50-60 ml after waiting 5 or 6 minutes, you may want to drive to a better sampling interval.

If after the first whack you have purged the volume of KPRO that is in the pump and tubing, and after waiting longer between whacks you get nitrogen coming back up instead of water, chances are the sampling interval will not yield a sample. To double check, refill the pump with KPRO water and start the purging/sampling process again.

If you have just added stainless and notice that the flow is a lot lower than it was at the end of the previous drive, you may have a blocked or kinked piece of stainless. Before driving, disconnect the stainless and blow it out with water to make sure all the lines are clear.

Please see the Problem Solving Guide if you have problems that aren't listed here, or if you can't figure it out call the office or somebody to help.

ATTACHMENT 3

Profiling Problem Solvers Guide

KPRO Trouble Shooting Guide

Version 1.0 Updated 2-26-08 by VLD

Software / Electrical Issues

Symptom	Possible Problem	Solution
Kpro Software Will Not Recognize Box	COM port set incorrectly in Software	There is a text box labeled "Kpro Com Port" on the Kpro software. Make sure that the number matches that of the com port where the Kpro box is plugged in. If it is plugged into the Com port on the back of the computer it is Com 1. If it is plugged into the socket card, click on the socket icon on the toolbar at the bottom right corner of the computer screen to determine which Com ports the computer has assigned to the socket card. A fast way to determine if the box is reading is to start the Kpro software and select "Calibrate" when the dialogue box pops up. Select one of the three choices (Flow, Pressure, String Pot). If the red light in the enclosure comes on and blinks and there are numbers other than zero for the readings, you have a good connection.
	Wiring harness inside Kpro box not connected/ or is loose	Double check that the wiring harness inside the Kpro box is securely connected to the enclosure.
	Computer assigns higher com port for serial card	On the newer tough book laptop computers (Rasc 6,7 and 8), sometimes the socket cards are given Com ports of 9 and or 15 and 16. The Kpro software does not like these higher numbers. Click on the socket card icon at the bottom right corner of the computer screen. Change the com ports of the socket card to Com 2 and 3. If the Kpro box is connected to the socket card, make sure you change the number in the software. If for some reason the socket card will not change the com ports, connect the Kpro box to Com 1 and the water quality sonde to Com 9.
	Bad data cable	After checking all of the above, the data cable may be broken. Double check the connections and maybe try another cable.

	Bad data cable connector	Check the connection plugs on the left-side of the Kpro box. All of the pins should be sticking out about the same distance. If not try using pliers to gently pull out any sunken in pins to the same level as the others. If this is not the case or does not work, if the truck has a spare wiring harness connect that to the enclosure and plug the data and string pot cables directly to it. If there is no spare harness and the truck has another Kpro box, swap out the boxes.
	Computer is not reading the serial card (dual or single)	Insert the serial com card and click on the socket card icon on the lower right corner of the computer screen. If the text box says "no socket card detected" then the driver needs to be installed. On the software disk with each computer there is a socket driver folder with a read me text file in it. Open the text file and follow the instructions.
No power to enclosure	Blown Fuse	The fuses are located inside the top compartment of the enclosure to the right of the LED voltage readout. Remove and inspect the fuses.
	Bad Power cable	Check that the ends of the power cable are securely fastened to the power inverter. If they are and the inverter is on, try replacing the power cable.
	Bad Power inverter	The on/ off switch on the inverter will light up when it is on.
	Broken Enclosure	If after trying all of the above and the enclosure still will not power up, then the enclosure may be broken. You can either swap out the enclosure or the entire Kpro box.
String Pot Malfunction	String pot reads intermittently	Make sure that the connection form the cable to the string pot is tight and that the cable is properly connected to the Kpro box. If they are, try swapping out the cable. If that doesn't work try swapping out the string pot. Also make sure that the wire that extends out of the string pot is not getting hung up on the drill rig or something else (wrench or pulling plug used to weight it down).
	String pot not reading at all	Make sure that the cable is properly connected to the string pot and the box. If there is still no reading, try swapping out the cable and/ or the string pot.
	String pot not reading during drive	Make sure that the string pot is extended before pressing the "Start Kpro" button on the software. If the button is pressed when the string pot is retracted, it will not read.

	String pot reading long/ short	Recalibrate the string pot. If that does not work and the string pot it not off by that much (+/- .5') then correct the depth at the end of the drive or when you stop to collect a sample. If it is off by more than that, try swapping out the string pot, then recalibrate. The string pot slope should be around 1.65 (+/ - 0/2). If it is more or less than that, swap out the cable or string pot.
Flow Meter Issues	Flow seems too high/ or low	Make sure that all of the connections inside the enclosure are tight. Flow out with one piece of stainless attached should be between 170-270. The flow slope should be close to 38 (+/- 2). Recalibrate the flow. If after recalibrating the slope is above or below this, try replacing the flow meter and recalibrating. You can also do a manual check of the flow by timing how long it takes to fill 100ml. If the flow rate matches what the software is reading and it is too high or low, change out the flow meter.
	No flow	Double check that the quick connects that go to the enclosure are connected. Make sure that the bottom left valve is on "Kpro". Check Kpro reservoir for water. In Peri mode, make sure that the top left and right valves are in the "Kpro Peri" position. For gas drive make sure that the top right valve is in "Kpro Gas Drive" position.
	Flow out but flow meter does not read any flow	Make sure that the flow meter is properly connected (electronics cable and plumbing). Make sure that the Kpro software is on and is connected properly. If it is, then replace the flow meter.
Pressure Transducer	Pressure seems too high/ or low	First, make sure that there are no leaks in the enclosure. When checking atmospheric pressure, the pressure should be between 32 and 36. If it is higher or lower, recalibrate the pressure transducer. The pressure slope should be around 22-28. If it is higher or lower than this and the transducer is still reading way off, replace the pressure transducer.
Calibration Problems	KPRO software folder in wrong directory	Make sure that the KPRO_2006 folder is located in the root directory of the C drive (*C:\Kpro_2006). There is an output file that gets written to and if the folder is in the wrong place, the software does not know where to find it.

	Slopes do not change, unrealistic numbers	If the slopes were calibrated with a bad cable, flow meter or pressure transducer, the program can come up with some crazy slopes (-16777 for string pot). The program uses the old slope to calculate the new slope, so it cant create a proper slope based on some crazy unreal number. After replacing the bad component, go to C:\Kpro_2006\Output Files\slopes.txt. Open the file and delete the bad slopes from the file. Make sure that the cursur is at the end of the line with the slopes when you save the file, otherwise the Kpro software will read in from the next line, which is blank, and will give a "NAN" (Not A Number) in place of the slope. Save and close the file, then try calibrating.

Note on calibration: If you calibrate and want to recalibrate right away due to a bad calibration, the program will NOT use the freshly calibrated slopes, but will use the slope that was read in when the program first started. So if you think the newly re-caled slopes are bad, simply recalibrate. If you think that the slopes the program are using are bad, go in and delete the bad slopes from the text file according to the directions above.

KPRO Box Issues

General Peristaltic Pump Problems

Symptom	Possible Problem	Solution
Peri pump not working - no power	Loose connection / bad switch	Make sure that the wiring harness is connected, the power cord is connected, all other connections are good. Some peristaltic pumps are connected to the power supply via a cigarette lighter plug located inside the Kpro box. Make sure that this connection is tight and that there is no corrosion/ and or dirt on the plug. Double check that the connections from the wiring harness to the pump are tight and there is no corrosion on the connectors and switches. If all the connections and switches appear to be fine, then the problem is probably in the wiring harness. In this case you will need to replace the harness or bypass the harness with a spare, or use a different Kpro box.
Pump head will not spin but pump motor is on	Broken spindle	There is a plastic piece inside the pump that has a slot in it that the shaft from the pump head fits into. Over time the slot can get worn and break off. If this happens you need to open up the pump and replace the spindle, or replace the entire pump.

Peristaltic Pump Repair / Replacement		If there is time, remove all the tubing and electrical connections and detach the old pump from the black plastic piece that holds it in the box. Connect the new pump to the black plastic piece and rewire the switches to connect to the switches on the kpro box and reconnect the tubing. A quicker solution is to disconnect the tubing and electrical connection and remove the old peri (black plastic piece and all) and put in the new pump by laying it in the open space in the box and connecting the tubing and electrical. When changing the tubing in the pumps do not tighten the pump head down with a screwdriver, these are meant to be finger tight, using a screwdriver causes the plastic framework within the pump to fatigue and break. None of the fittings for the pump between the black plastic and the switches/pump body are water tight which has lead to corrosion and switch failure. In an effort to extend the life of this equipment using a little silicon sealant at these points is a good idea, if you are working on a box that does not have this please apply a small amount between the 2 parts, any questions please feel free to call me.
Peristaltic Mode		
Cannot pull vacuum	Upper left valve on box not on peri	Should be on "Kpro Peri" during drive, need to switch to "Peri" when pulling sample.
	Worn out silicon tubing	The silicon tubing located inside the head of the pump gets worn and needs to be replaced. Remove the four screws and remove the top half of the head assembly. Replace the silicon with a new piece cut to the same length. Replace the head and only finger tighten the screws.
	Peri pump on reverse	Make sure that the peri pump is going in the right direction. If while the pump is on and there is a positive pressure reading on the gauge, stop the pump, reverse direction and restart the pump.
	Check if problem is in sample jig or other stainless	Turn the valves on the sample jig so that they face one another, therefore bypassing the jig. If the pressure drops and a vacuum is achieved then there is a leak in the sample jig.
	If leak is in sample jig: Loose fittings	Double check that all of the swage fittings on the sample jig are tight.

	Loose Vials / Bad vials	Make sure that the sample vials are tight. They should not be excessively tight. If there is still a leak, replace the vials. Sometimes the threads on the vials can be slightly off and wont fit correctly in the bottle holder.
	No o-rings or bad o-rings	There are special quad o-rings that are located in the bottle holder. Double check that there are o-rings in there and that they are in good condition (not dried out, cracked or cut).
	Sample straw too close to vial bottom	On some of the sample jigs, the straw that extends down into the sample vial is too long and will come in contact with the bottom of the vial. Sometimes this will result in the vial bottom breaking. Other times it will block the straw and water will not be able to pass through. If it looks as if the straw is too long, try inserting an additional o-ring in the bottle holder. If this does not work, file down the straw or cut a small amount off the bottom with a tubing cutter or grinder.
	Plastic bottle holder is worn out / scarified	The threads of the bottle holder can become scarified from people not being careful when removing the o-rings with a pick. If after trying all of the above, the bottle holder may need to be replaced. There are screws located on the under side of the plastic piece (hex screws). Once the holder has been removed, inspect the inside of the threads and if they are scratched and gauged then replace the plastic piece.
	If leak is not in sample jig:	
	Leaky fittings	If it is determined that the leak is not in the sample jig, then you may have a leaky fitting on the harness or down hole. Double check that all the fittings on the Kpro box, harness and stainless are tight.

Gas Drive Mode

Symptom	**Possible Problem**	**Solution**
No inflow of sample water	No Flow during whack. Pressurize pump then open sample jig, but no water flows.	Make sure the bottom right valve is on "sample". Make sure that there are no kinks in the tubing coming out of the box and going to the water quality sonde's flow cell. Also sample straw in jig could be too close to the bottom of the sample vial and is preventing flow. Try loosening the vials to see if you get flow.

Gas bubbles when filling pump for drive	Leaky 5-way valve	If the 5-way valve (the top left valve on the Kpro box) is leaking, nitrogen can leak through. Tighten the valve by first removing the know with a hex wrench. Remove the metal plate. If you see metal shavings, it is possible that the pump has been tightened before and needs to be replace. Insert needle nosed pliers into the two holes on the valve and turn. If the valve still leaks, it needs to be replaced.
Silicon tubing pops off peri pump	Leaky 5-way valve	See above. Another test to determine if the valve is leaking is to pop off the silicon tubing and put your finger over the peri intakes and remove it after a second or two. If you hear a pop, then gas is leaking through the valve. Another way is that with the valve pointed to "Peri", place the vent tubing into some water. If there are bubbles coming out of the tubing, the valve is leaking.
	Replacing 5-way valve	Disconnect the "N2 in" quick connect from the Kpro box. Before removing the valve, take note of which way the fittings are pointing on the under-side of the valve. Is is helpful to make a drawing showing how they are oriented. Remove the knob of the valve with a hex wrench. There is a large nut/ ring on the top-side of the valve that can be removed using a crescent wrench. Disconnect the valve (there are 5 connections). Remove the fittings from the valve, and re-use them if you can. Some of the plastic fittings can become worn out and break, so do not reuse these (there should be new ones in the Swage box). Before installing the fittings on the new valve, wrap the threads with teflon tape. It is much easier to install the fittings onto the valve before putting the valve into the box. Orient the fittings the same way that they were on the old valve, making sure that they are tight. This will make re-installation much easier. It is also easier to re-attach all of the connections before putting the valve through the hole in the plastic and securing the nut/ ring on the top side of the valve.

Profiling Problems

Peristaltic Mode

Symptom	Possible Problem	Solution

Head values seem odd	FEP is broken or disconnected	If you know you are in the water table, and based on previous head readings the head value seems wrong (i.e. a previous head reading was -10, and now it is -1) the FEP tubing could be broken. Slightly pull on the stainless steel tubing coming out of the rods. If you feel resistance, then the tubing is still intact.
	Loose fitting	If the FEP is still intact, there could be a loose fitting that is leaking. To test this, pour water down the rods and watch the pressure reading on the Kpro software. If the pressure rises, there is a leak and the tooling needs to be tripped out and replaced.
	Silty layer	If the profiling tip is in lower-Ik zone, it can take a while for the head value to settle, and it still may settle at a value different than the previous head value (usually a few feet above). Wait a few minutes and record the head value once it stabilizes. Orient the valves and turn on the peri pump to start pulling a sample. If you do not get a sample, the profiler needs to be advanced to a higher Ik zone.
No flow out	Valves in wrong position	Make sure that the valves on the sample jig are pointing towards each other, then that the top left and right valves on the box is pointing towards "Kpro Peri", and that the bottom left valve is turned towards "Kpro".
	Tip is clogged	After attempting to pull a sample, orient the valves for flow and turn on the Kpro. If the flow was good before pumping and is now zero, then the tip became clogged during pumping. Try to advance the profiler, as sometimes the vibration will loosen the soil in the tip and can free it up. If after a few feet there is still no flow, trip out the tooling and replace the tip.
	Kpro reservoir is empty	Make sure there is water in the Kpro reservoir. Disconnect the line going from the nitrogen regulator to the reservoir before opening the top.
Good flow out, no water back	Tip is slightly encased with soil	Develop the formation by alternately flowing Kpro water out the tip and pumping, or by alternating the direction of the peri pump for a few seconds. Make sure to turn the pump off before switching between forward and reverse because it can damage the pump.
	Not deep enough in water table	The profile tip should be at least 6" into the water table.
	Not pulling vacuum	Check previous section listed above
	FEP is broken or disconnected	Check previous section listed above
	Stainless is broken or has sheared off	If the drill rod has broken off it will shear the stainless steel. Pull on the stainless to see if it is still connected.

Pull in very dirty sample	Water from drilling rods is being pulled in	If the sample water is excessively silty and a lot of silt has filled up the sample vials, then there is a chance that the FEP tubing is broken. Check FEP - see above.
Start to get good sample return, then nothing	Tip is clogged	Develop the formation by alternately flowing Kpro water out the tip and pumping, or by alternating the direction of the peri pump for a few seconds. Make sure to turn the pump off before switching between forward and reverse because it can damage the pump.

Gas Drive Mode

Symptom	**Possible Problem**	**Solution**
No flow out tip	Tip is clogged / smeared	The profile tip can become clogged after pulling in a sample that contains fine particles such as silty or fine sand. If after pulling a sample the flow out the tip is lower than before pulling the sample, try advancing the tip a few feet. If during the drive the flow is good but drops off when the hammer is stopped, there is a good chance that the tip is clogged and must be changed. When profiling through a clay layer, it is possible that clay will smear on the tip and cover the sampling ports.
	Kpro line is clogged	See the clogged lines section below
No flow out tip - deep profiling	Rubber inner tube still on tip	When profiling using a drill rig where the drill rods are lowered down into casing filled with drilling mud, it is necessary to put a rubber inner tube over the profile tip to prevent it from clogging. The tip must be advanced at least 1-1.5 feet for the inner tube to be pushed back to expose the sampling ports on the tip.
Cannot fill pump for drive	Nitrogen pressure set to high - reed valve failure	Make sure that the pressure regulator is set at the appropriate pressure for the depth. the conversion is 1 psi = 2.3 feet of water (So for a depth of 200 feet, the regulator should be set around 90 psi, at 500 feet at 220 psi). If the pressure is set to high the reed valve will fail.

	Leaky fitting / reed valve, reed valve silted up	Make sure that the fittings on the harness are tight. The pump will not fill if the reed valve located in the pump is not closing correctly due to silt or a defective valve. Sometimes it is possible to "shock" the reed valve by first making sure that the valves on the sample jig are facing each other, then connecting the "sample out" line to the "N2 out" line and turning the valve on the top right of the box to "N2". This will send nitrogen down the sample line (the sample line inside the pump ends above the reed valve) hopefully mobilizing any silt, or by forcing the valve to close. If this does not work, then the pump needs to be removed from the hole and the reed valve must be replaced.
Cannot pressurize pump	Leaky fitting / reed valve, reed valve silted up / pressure set to high	See above.
		See above.
Pump loses pressure during drive	Leaky fitting / reed valve, reed valve silted up / pressure set to high	See above.
Good flow out, no sample back	Tip is clogged	Sometimes while driving the vibration from the hammer will allow flow out, but when the hammer stops flow will drop. If the flow goes to zero or very low after the hammer is stopped, the tip could be clogged or the formation is tight. You need to either pull out the tooling or drive further.
	Kpro line is loose or came unscrewed	If the flow out is high enough where you think a sample can be pulled but you get no water back when whacking, the Kpro line could be broken of unscrewed. While driving, the tip will become clogged if no water is flowing out. The Kpro water will flow out the open stainless giving the illusion of high Ik. Another clue to this is if you cannot pull a sample and the Ik is high and didn't vary at all over a large depth.
Small sample return (small volume whacks)	Lines are switched	If you are well into the water table and the Ik is high and the whacks are small (less than 50 ml) then the stainless steel lines could be reversed. If there are two lines that are very close in length (usually the sample and nitrogen lines) a short piece of stainless can be added to sample line to separate them.
Sparge sample	Tip is clogged	The pump volume is approximately 120 ml and there is approximately 0.8 ml per foot of stainless. If after purging the appropriate amount of water to fill the pump and stainless the sample sparges, refill the pump, pressurize the pump and turn on

		the Kpro to see if the tip is clogged.
	Ik is too low to sample	Wait longer between whacks. If you purge the volume of the lines and pump and do not see formation water (usually silty) advance the profiler.
	Not venting nitrogen when needle moves	If excess nitrogen gets into the pump the sample will sparge. Be sure to vent the nitrogen as soon as the needle on the gauge tips up. If it sparges, try doing smaller whacks by venting the nitrogen before the needle tips up. If the sparge is too much try refilling the pump and starting over.

Clogged Lines

1. Before attempting to unclog any lines, turn the valves on the sample jig so that they are facing each other, thereby bypassing the sample jig.

2. If you think a line is clogged, the first and fastest thing to check is the harness. The harness can become plugged if it touches the ground while adding stainless. Try disconnecting the harness from the stainless lines and turn on the nitrogen and kpro and see if the lines are clear. You can check the sample line by putting the valves on fill, or plugging the sample line into the kpro out and turning the kpro on.

Determining if a line is clogged

Line	Problem	Solution
Kpro line	No flow - pump is full and pressurized, flow bottoms out to zero before driving.	Depressurize the pump by venting the nitrogen and turn the valves on the sample jig in. Turn on the Kpro and look at the flow on the Kpro software. Since the pump is de-pressurized and pressure in the pump is less than the pressure outside the tip, Kpro water should flow up into the pump at about 10-20 ml/min. If the flow is still zero, the Kpro line is clogged. See the "Unclogging lines" section below.

Sample Line	Pump does not fill	Turn the left valve on the sample jig so that it faces the wall (closed) if the needle on the pressure gauge does not drop, the line is probably clogged. To double check, plug the sample line into the Kpro out and turn on the Kpro. Watch the flow on the computer. If the flow is zero, then the line is clogged. See the "Unclogging lines" section below.
Nitrogen Line	Pump does not fill	Plug the nitrogen out line into the Kpro out and turn on the Kpro. Watch the flow on the computer. If the flow is zero, then the line is clogged. See the "Unclogging lines" section below.
Unclogging Lines	Kpro Line	1) Isolate zero-headspace sampling apparatus by pointing the two valves handles, located above pressure gauge, inward towards each other. 2) Place "KPRO out" quick connect into "Nitrogen Out" quick connect slot. 3) Turn nitrogen/peri valve to "Gas Drive" position, which will allow nitrogen flow down the KPRO line and out into the formation. 4) Listen to gas drive regulator to determine if regulator stops delivering flow, as indicated by the regulator becoming silent. If regulator continues to make "hissing noise", then the KPRO line is free of any obstructions. 5) If the obstruction cannot be removed via the nitrogen pressure, the tool must be removed from the ground and inspected to determine where the clog is present.
	Nitrogen Line and Sample Line	1) Isolate zero-headspace sampling apparatus by pointing the two valves handles, located above pressure gauge, inward towards each other. 2) Turn nitrogen/peri valve to "Gas Drive" position and disconnect the sample line from where it enters the pressure gauge. This will allow nitrogen flow down the nitrogen line and up the sample line. 3) Listen to gas drive regulator to determine if regulator stops delivering flow, as indicated by the regulator becoming silent. If regulator continues to make a "hissing noise" and nitrogen flow is observed at the effluent of the sample line, then the nitrogen line, the gas drive pump and the sample line are free of any clogs. If regulator stops delivering flow, as indicated by the regulator becoming silent, then either the sample line, the nitrogen line, or the

gas drive pump may be clogged.

4) If the obstruction cannot be removed via the nitrogen pressure, the tool must be removed from the ground and inspected to determine where the clog is present.

Water Quality Sondes

Symptom	Possible Problem	Solution
Sonde is not recognized by the computer	Wrong Com Port in Software	Make sure that the com port assigned in the software is the same one that the sonde is connected to. Most of the software will only go up to Com 9.
	Power not connected/ batteries are dead	The YSI and Hach sondes have a power supply. The Trolls have batteries. To change the batteries first unscrew/ disconnect the data cable and then unscrew the top of the sonde.
	Cable is broken	If after trying the first two steps listed above there is still no connection between the sonde and the computer, the data cable connecting the sonde to the computer could be faulty. Try a different cable.
Water quality parameter values are way off	Computer/ Sonde needs to be restarted or recalibrated.	Sometimes the sondes can read strange numbers (i.e. pH of 100, DO of 300). The first thing to try is to restart the computer. If this does not work try recalibrating the sonde. It is better to calibrate/ check each probe individually rather than using an all in one cal solution (such as Quick Cal). If the sonde is still off, look below.
	Individual probes need to be changed.	From time to time the individual probes on the sondes can be faulty. Each troll/ YSI case should have extra probes. Swap out the probe and check/ and or recalibrate. Make sure when checking/ calibrating that the probes are fully immersed in the cal solution. Also, it is very important to store the probes in the correct manner. Never store the probes dry, even overnight. At the end of the day, take the sonde out of the flow cell and store it in the screw on cap filled with pH 4 solution For long term storage (longer than a week) remove the individual probes and store them in the appropriate solution (check the manual for the particular sondes for the correct way to do this).

	Bad Cal Solutions	Calibration solutions can go bad if they have been opened for a while (especially conductivity). Always write the date opened on any bottle of solution when it is opened for the first time. If you think they solution may be old, recheck or calibrate with fresh solution.
	Water Temperature - DO calibration	When calibrating or checking the DO using the water saturated air method, make sure that the water you are using is relatively warm. If you are checking the sonde first thing on a cold morning with water that was in the truck, it will take longer for the water to vaporize in the flow cell and therefore the calibration will take longer or won't happen at all. Try heating the water in a cup using a space heater prior to calibration. Make sure to record the DO value and temperature in the log book for the sonde after calibration or check.
Values do not change	Clogged flow cell - silted up probes	Make sure that the sonde is kept vertical, otherwise sediment can build up in the flow cell and coat the probes.

Calibration Notes for Hach Hydrolab

1. There is a reference solution for the PH/ORP probe that needs to be changed monthly (not sure if that is only in use or always) it is very easy to do following the instructions in the manual.
2. When you calibrate the PH on these units it is IMPORTANT to calibrate the PH 7 standard first followed by either the 4 or 10 or both as the calibration method uses the 7 as zero and calculates the slope with the 4/10.

Torpedo/ Salimander Heaters

********** UNPLUG THE HEATER WHEN YOU ARE TROUBLESHOOTING IT EXCEPT IN AN ATTEMPT TO START IT************

Symptom	Possible Problem	Solution
Unit will not start / shuts off	Not plugged in / no power Fuel	1. Make sure the unit has Power. 2. Check your fuel source for diesel treatment; they do not fire quite the same as a diesel engine and the treatment in the fuel may not be working for the heater. Possible solutions include more additive, a different additive and using kerosene.

Starter element is bad	3. Look inside the unit (FROM THE REAR) as it tries to start, if there is not a red glow inside then the element must be replaced, parts available at Home Depot.
Air filter is plugged / clogged.	4. Check the air filter on the back of the unit: This can become iced up or plugged with dust/dirt. Clean it!
Fuel filter	5. If the temperature isn't far below freezing and the fuel is not in question you may try replacing the fuel filter. This can be found by removing the control panel (2 Philips screws above the controls) it is a white inverted cone with a black tube attached to it sticking out of the fuel tank. To remove the filter you most pull out the rubber bushing it sits in; failure to do this will remove the fuel pickup tube from the bottom of the filter and cause the unit to only be able to utilize 10% of the fuel tank capacity (the first 10) replacement filters can be purchased at Home Depot.
Fuel line	6. Check the fuel pickup line mentioned above.

Other issues:

1. If the unit runs for a while then shuts off repeatedly it is likely 2 or 6 above.
2. Sometimes the unit will start with the top cover off and not with it on, I have come across this while troubleshooting and it is the result of improper fuel supply pressure, this can be corrected with the pressure gauge from the manufacturer but they are too costly to have in every truck. For a temporary solution you can restrict the airflow from the back with a piece of cardboard to get it to fire then remove the cardboard for operation, doing so will cause the fuel supply to spread further and interact properly with the igniter.
3. They get very hot, allow them to cool after use before placing them in a truck.
4. While in operation they are a serious hazard and must be used in such a manner as to not melt nearby objects (people, drill rigs etc.) keep in mind that the heat from these is dangerous for more than 10 feet.
5. Do not fuel them while in use.
6. Do not fuel them to the very top, the caps have a vent that will leak if not perfectly level.

DO NOT USE GASOLINE IN THEM !!!

STANDARD OPERATING PROCEDURE

SEI-6.1.6

COLLECTION OF SOIL SAMPLES FOR PRELIMINARY SITE CHARACTERIZATION

SOP Number: SEI-6.1.6
Revision Number: 6
Date Issued: 10/26/92
Date of Revision: 11/18/05

1.0 OBJECTIVE

Soil samples may be collected to supplement field observations made during an initial visit to a site. The procedure below provides the essential elements in collection of soil samples from study sites. The analytical results will furnish preliminary information regarding the general physical or chemical characteristics of the soils on the site.

2.0 POLICIES

1. According to 40 CFR Part 160, Subpart E, Section 160.81, a testing facility shall have standard operating procedures in writing setting forth study methods that management is satisfied are adequate to insure the quality and integrity of the data generated in the course of a study.
2. Personnel will legibly record data and observations in the field to enable others to reconstruct project events and provide sufficient evidence of activities conducted.

3.0 SAFETY ISSUES

1. If necessary and appropriate, a site-specific health and safety plan shall be created for each study site. A template for creating a proper health and safety plan is provided on the SEI network.
2. Care must always be taken when approaching a sampling location. Do not, under any circumstances, place yourself in danger to collect a sample.
3. If necessary and appropriate, all chemicals are required to be received with Material Safety Data Sheets (MSDS) or appropriate application labels. These labels or MSDS shall be made available to all personnel involved in the sampling and testing.

4.0 PROCEDURES

4.1 Equipment and Materials

1. Appropriate soil collection device such as a hand auger or geoprobe.
2. Appropriate soil sample containers (e.g., ziplock plastic bags, wide mouth glass or high-density polyethylene (HDPE) jars).
3. Field notebook, sample collection form, or other acceptable medium for recording field notes. Ballpoint, indelible pens.
4. Shipping materials.
5. Sample Labels.
6. Chain of Custody forms.
7. Measuring tape.
8. As necessary or as described in the Site HASP appropriate personnel protection equipment (PPE) such as gloves.
9. Rubber mallet and/or other appropriate hand tools.
10. Plastic sheeting for laying out soil samples for characterization.
11. If necessary, SEI-SOP:6.17.n, or Munnsell Color Charts.
12. If necessary, bentonite chips.
13. Distilled water or "purified" bottled water.
14. Alconox or other mild detergent.
15. Appropriate shipping container.

4.2 Sample Collection

Prior to use, field personnel will understand and have read any instructions associated with the soil sample collection device and inspect the device for cleanliness and any defects.

Once at the designated sampling location, a borehole will be excavated using a hand auger or other appropriate sampling device. As the borehole is excavated, the soil sample will be collected from the

desired depth interval. Instances that require undisturbed samples should utilize an acetate sleeve, which should be capped immediately after removing the sleeve from the boring device. Top and bottom should be clearly marked on the sleeve.

If appropriate, samples will be removed from the auger and placed on plastic sheeting or other suitable material for visual characterization analysis and segregation.

The sample will then be placed into a sample container, which will then be sealed and labeled with at least the following information, but not limited to:

1. Test system or Site Identification.
2. Study identification or Project Identification (maybe study number).
3. Nature of the study.
4. Date of collection.
5. Name or initials of the sample collector.
6. Sample location or project name.
7. Sample identification number.

The collection of each sample will be recorded in a field logbook or on a specified sample collection form. Information to be recorded will include the following but may not be limited to:

1. Test system or Site Identification.
2. Study identification or Project Identification (maybe study number).
3. Nature of the study.
4. Date of sample collection.
5. Name of personnel collecting and describing the sample or site characterization.
6. Sample identification.
7. Sample location.
8. Sample depth.
9. Sample description, including texture, color, moisture content (dry, moist, or wet), mottles (if present), and other observable features.

Due to field conditions, if necessary, item 9 above may be performed at a later date. The color description should be performed in light similar to natural sunlight (if possible), and every attempt should be made to preserve field moisture conditions. If descriptions are performed at a time other than during field collection, this should be noted on the soil description sheet. The sampling device requires decontamination with Alconox and DI rinse between borings.

If laboratory analysis is necessary, the sample containers will be placed in a secure and appropriate shipping container and shipped to a soil characterization laboratory for designated analysis via an appropriate approved carrier. Samples may need to be chilled or frozen.

Chain of custody forms will be filled out and signed prior to shipment to the laboratory and included in the shipping container with the samples, according to standard operating procedures and/or protocol instructions.

After soil sample collection is complete, the soil borings may need to be backfilled according to the site work plan. Typically, the soil boring will be backfilled with bentonite chips and hydrated with distilled or "purified" bottle water.

5.0 RESPONSIBILITIES

1. All personnel will legibly record data and observations (including phone conversations) in accordance with this SOP to enable others to reconstruct project events and provide sufficient evidence of activities conducted.

6.0 DEFINITIONS

1. *Study* means any experiment at one or more test sites, in which a test substance is studied in a test system under laboratory conditions or in the environment to determine or help predict its effects, metabolism, product performance (pesticide efficacy studies only as required by 40 CFR 158.640) environmental and chemical fate, persistence, or residue, or other characteristics in humans, other living organisms, or media. The term "study" does not include basic exploratory studies carried out to determine whether a test substance or a test method has any potential utility.

7.0 REFERENCES

40 CFR Part 160 Good Laboratory Practice Standards, August, 1989.

8.0 TABLES, DIAGRAMS, FLOWCHARTS, AND VALIDATION DATA

None

9.0 AUTHORIZATION

Revised by:_____ Date:_____

Patrick Larsen, Project Geoscientist

Approved by:_____ Date:_____

Christopher T. Stone, President

10.0 REVISION HISTORY

Revision number 1:

Unavailable, under old numbering scheme.

Revision number 2:

1. Reformatted under new numbering scheme.

Revision number 3:

1. Reformatted
2. Change sampling procedure in section 3

Revision number 4:

1. Corrected section numbering.
2. Minor wording edits.
3. Remove documentation of Atime@ sample collected.
4. Add GLP requirements for sample identification and variation of labeling per protocol.
5. Add flexibility to describe samples at a time later than collection if noted.

Revision number 5:

1. Updated style to match SEI Style Guide – font and text. Reformatted using MS Word.
2. Added standardized section headers: 2.0 Policies, 3.0 Safety, 5.0 Responsibilities, 6.0 Definitions, 7.0 References, 8.0 Tables, Diagrams, Flowcharts and Validation data. Authorization moved to Section 9.0, andSection10.0 Revision History.
3. Under equipment added appropriate soil collection device (geoprobe).
4. Changed plastic sample bags to appropriate soil sample containers.
5. Removed note on items in italics. All items should be on labels and in field notebooks, regardless of GLP.
6. Other minor wording edits such as changing box to shipping container.

Revision number 6:

1. Under materials added reference to Munnsell® Color Charts and/or SOP SEI-6.17.n, also bullets for distilled water and bentonite chips.
2. In Section 4.2 added need for soil boring backfilling.

STANDARD OPERATING PROCEDURE

SEI-5.29.0

USE, MAINTENANCE AND CALIBRATION OF THE MultiRAE IR MULTI-GAS MONITOR, PGM-54

SOP Number: SEI-5.29.0
Revision Number: NA

Date Issued: 02/04/2011
Date of Revision: NA

1.0 OBJECTIVE

The MultiRAE IR is a programmable multi-gas monitor designed to provide continuous exposure monitoring of carbon monoxide, toxic gases, oxygen and combustible gases for workers in potentially hazardous environments. The MultiRAE IR Multi-Gas Monitor may be worn on a belt to allow for hands-free use. The MultiRAE IR gives real time measurements and activates alarm signals whenever the exposure exceeds preset limits. Prior to factory shipment, the MultiRAE IR is preset with default alarm limits and the sensors are pre-calibrated with standard calibration gas. However, the user should calibrate the instrument before the first use. After the monitor is fully charged and calibrated, it is ready for immediate operation.

2.0 POLICIES

1. The MultiRAE Multi-Gas Monitor should be calibrated each day before use. Maintenance, corrective actions, and calibration should be noted in the "Maintenance and Calibration Log".
2. Personnel will legibly record data and observations in the field to enable others to reconstruct project events and provide sufficient evidence of activities conducted.
3. Field personnel will use a field logbook, observation and remark form (O&R) or other designated field form to record activities, measurements, and observations made in the field.
4. Any change in entries shall be made so as not to obscure the original entry. The recorder will initial corrections.
5. Field personnel will read this SOP prior to operating the Multi-Gas Monitor.

3.0 SAFETY ISSUES

1. Whenever visiting a hazardous or potentially hazardous site, it is important to read, follow and sign the site health and safety protocol (HASP).

2. By itself, use of the MultiRAE IR Multi-Gas Monitor poses no risk to human health, however, if elevated levels of hazardous gases are detected by the Multi-Gas Monitor, it may be necessary to don personnel protective equipment or vacate the area as described in the site's HASP.

3. Field visits may involve accessing remote areas. Health and safety concerns regarding these field visits are minimal however; slip, trip and fall hazards, dangerous flora and fauna, as well as getting vehicles stuck in remote areas do present considerable health and safety issues. To help ensure field staff's health and safety with remote areas, all field staff is to have daily communication with the project manager or another appropriate Stone Environmental, Inc. (SEI) employee.

4.0 PROCEDURES

4.1 Introduction

The MultiRAE IR combines a PID (Photoionization Detector) and Carbon Dioxide (CO2) sensor with O2, LEL, and one specific toxic gas sensor in one compact monitor with sampling pump. Although there are numerous types of PIDs available, all PIDs are based on the following principle:

> A sampling pump continuously draws air sample into the ionization chamber. The ionization chamber is usually constructed as a small cavity in front of the UV lamp. The electrode is made of a wire mesh so that the high energy UV light can shine through the mesh and excite the gas molecules when they pass across the face of the UV window. The ions collected on the electrode are measured by an electrometer.
>
> A single chip microcomputer is used to control the operation of the lamp, alarm buzzer, LED, sampling pump, power supply and electrometer. It measures the electrometer readings and calculates the gas concentrations based on calibration to a known reference gas. An LCD display is used to show the readings. The user interacts with the unit though three keys (one operation key and two programming keys) on the front panel.
>
> A rechargeable lithium-ion battery pack powers the unit. A built-in charging unit allows the batteries to be charged directly from an AC power source with the included power connection.

The MultiRAE IR Multi-Gas Monitor also contains sensors to monitor Carbon Dioxide, Toxic Gas (VOCs), Oxygen, and Combustible Gas.

4.2 General Considerations

The MultiRAE IR is adversely affected by both damp and windy conditions, as well as temperature. Ensure that the MultiRAE IR is used with its probe and a clean sample filter at all times. Replace the filter frequently. Refer to the operating manual to determine the lowest allowable operating temperature and humidity.

The MultiRAE IR Multi-Gas Monitor has 3 operating modes: Text, Display, and Programming. In "programming" mode, which is the default setting, the user can perform all the functions of the other modes. In addition, the user can get into calibration mode, perform calibrations, and change any of the parameters in

the calibration mode. For more of an explanation about the "text" and "display" modes, see the Operation Manual.

4.2.1 Equipment

- MultiRAE IR Multi-Gas Monitor Operation and Maintenance Manual
- Calibration gas and regulator
- Tedlar bag for calibration gas collection
- Poly tubing for connection tedlar bag to regulator and monitor
- Extension wand and tubing
- Serial port computer connection cable
- Spare sample filters
- Phillips head and flathead screwdriver
- Maintenance and Calibration Log

4.2.2 Operation-Turning the MultiRAE IR Multi-Gas Monitor ON and OFF

The MultiRAE IR Multi-Gas Monitor has only three controls: two programming keys and one operation key. To **TURN ON** the MultiRAE IR Multi-Gas Monitor, press the [MODE] key. The audio buzzer will beep once and the display will show "ON!" and then "Multigas Monitor Ver n.nn" to indicate the software version number. The monitor will then go through each sensor socket to check if a valid sensor is installed. Next, the monitor displays the preset alarm limits for each sensor, battery voltage, shut off voltage, user mode, alarm mode, datalog mode available data storage memory (in hours), datalog mode and datalog interval (in seconds). Then after a set delay time of several seconds, the display shows the instantaneous reading of the gas concentrations and is ready to monitor gases.

To **TURN OFF** the MultiRAE IR Multi-Gas Monitor, press and hold the [MODE] key for 5 seconds. Monitor will beep once per second during power-down sequence with a countdown timer showing the number of remaining seconds. The message "Off!" flashes on the LCD display and the display will go blank, indicating that the monitor is turned off.

Turning off the MultiRAE IR Multi-Gas Monitor with the AC charger in place will result in a display of "Charging.." or "Battery Charged" and indication of battery voltage. This indicates that the monitor is off, but the charger circuit is now active.

- *Programming Mode*

The three keys perform a different set of functions during the programming mode as summarized below:

[MODE]	Exit Menu when pressed momentarily or exit data entry mode when pressed and held for 1 second.
[Y/+]	Increase numerical value for data entry or conform (yes) for a question.
[N/-]	Decrease numerical value for data entry or deny (no) for a question.

To enter programming mode (which is the default mode), press and hold down both [MODE] and [N/-] keys for three seconds. At this point, the display should show "Enter Password = 0000". Use the programming keys to enter the password "0000", then hold [MODE] for one second. Refer to section 4.3 in the manual for help with this step.

4.2.3 Calibration

For maximum safety, the accuracy of the MultiRAE IR Multi-Gas Monitor should be calibrated before each day's use by exposing the sensors to a known concentration calibration gas. Refer to section 4.4 in the Operation Manual for a more thorough explanation.

In programming mode (the default mode), the user may calibrate the sensors in the MultiRAE IR Multi-Gas Monitor. This is a two-point calibration process using "fresh air" and a standard reference gas. First, "fresh air" containing 20.9% oxygen and no detectable VOC, toxic or combustible gases is used to set the zero point for each sensor. Then a standard reference gas, containing a known concentration of a given gas, is used to set the second point of reference (also known as span gas). The two-point calibration procedure is described below.

NOTE: "Fresh Air" calibration zeros only the LEL, VOC, and toxic gas sensors, and spans the O_2 sensor. The CO_2 sensor is spanned and zeroed in two sub-menus of the Single Sensor calibration. For better accuracy, zero calibration should be performed before span calibration of all sensors. The following table shows the structure of the calibration sub-menu:

Calibration Sub-menu
Fresh Air Calibration?
Multiple Sensor Calibration?
Single Sensor Calibration?
Modify Span Gas Value?
Change LEL/VOC Span Gas?

To perform a "fresh air" calibration, move to a clean air environment and follow the steps below. If unsure of the ambient air's purity, a charcoal filter should be used.

- *Fresh Air Calibration (Zeroing the sensors)*

To enter Calibration mode, hold down the [MODE] and [N/-] key for three seconds.

1. "Calibrate Monitor?" Press [Y/+] to perform calibration. The first sub-menu shows: "Fresh Air Calibration?"
2. Leave the monitor in an area free of any detectable vapor.

3. Press the [Y/+] key to start "fresh air calibration" of the monitor. The display shows "zero... in progress" followed by the name of each sensor and the message "zeroed". The display should show a reading of "20.9" for the oxygen sensor and "0.0", or a very small number, for all other sensors.
4. After about a five second pause, the display will show the message "Zero Cal Done!" and move to the next sub-menu "Multiple Sensor Calibration?"

- *Multi Sensor Calibration*

This function simultaneously determines the second point of calibration curves for multiple sensors in the monitor. Mixed standard reference gases are needed to perform this procedure (and should be included in the case). The standard reference gas needs to be transferred into a tedlar sample bag. To do this, ensure the regulator is in the off position (righty-tighty!) and screw onto the standard gas cylinder. Use a short section of poly tubing to connect the regulator to the tedlar bag. Open the valve on the tedlar bag, then slowly open the valve on the regulator, allowing the tedlar bag to fill with the standard gas. Once the tedlar bag is full (tedlar bag should NOT be taught), turn off the valve on the regulator. Next, tighten the valve on the tedlar bag. Finally remove poly tubing from either end.

Now, attach the MultiRAE IR Multi-Gas Monitor to the tedlar sample bag containing the standard gas. Continuing from Step 4 above, the display shows "Multiple Sensor Calibration?".

5. Press [Y/+]. The display shows all the preselected gases for calibration and an "OK?" question. Press [Y/+] to accept the sensor selections and start the calibration, or [N/-] to modify the sensor selection (see manual section 4.4.2 for further instructions).
6. Turn on the valve of the tedlar bag to start the flow of the "span" gas (standard gas). Display shows "Apply Mixed Gas" and will wait for the calibration gas to reach the sensor. When the calibration gas has reached the sensor the display will show "calibration in progress... 75" with the countdown timer showing the number of remaining seconds while the monitor performs the calibration. When the timer reaches zero (0), the display shows the name of each sensor, the message "cal'ed" and the calibrated values for each gas. If no gas has reached the sensor after 75 seconds, the display will show "No Gas flow..." and abort the calibration. The readings should be close to the span gas values (These values are printed on the label of the calibration gas and are reprinted below). After about 5 seconds pause, the display will show the message "Span Cal Done! Turn Off Gas".
7. Turn off the flow of gas. Disconnect the calibration adapter from the MultiRAE IR Multi-Gas Monitor. If all sensors calibrated correctly, the MultiRAE IR Multi-Gas Monitor is ready to use.
8. If the sensor failed calibration, the sensor name and an error message "failed, continue?" will appear and the failed sensor may be calibrated individually. Press [N/-] or [MODE] to abort the calibration and move to the next sub-menu. Press [Y/+] to continue to the next sensor calibration. In either case, the calibration data

for the current sensor are not changed. For further information about calibrating an individual sensor, refer to Step 6 in section 4.4.2 of the Operation Manual.

Calibration Gas (aka "Span Gas") components and concentrations (there are other types, so make sure via the label that these are what you have) are listed in the table below:

Gas	Concentration
Carbon Monoxide (CO)	50 ppm
Carbon Dioxide (CO_2)	5,000 ppm
Methane (CH_4)	2.5% (50% LEL)
Oxygen (O_2)	20.9%
Nitrogen (N_2)	BALANCE

4.2.4 Changing Alarm Limits

Alarm limits for the MultiRAE IR Multi-Gas Monitor can be modified. These should be left at the default settings unless the user has special needs. If these limits need to be modified, refer to section 4.5 in the Operations Manual.

4.2.5 Data-logging

The MultiRAE IR Multi-Gas Monitor has the ability to calculate and store the gas readings as specified intervals. The user can review these stored readings or change datalog setup from the programming mode. Users can also program additional datalog options in PC and down load them to the monitor. See sections 4.6 and 5.0 in the Operation Manual for details on programming data-logging options.

4.2.6 Maintenance

The MultiRAE IR Multi-Gas Monitor requires minimal maintenance besides battery charging and calibration. If any sensor, PID Lamp, or other errors occur, refer to the Operation Manual. A trouble-shooting guide is included in the Operation Manual in section 7.2.

Detailed maintenance must be performed by a qualified technician and usually requires the shipping of the MultiRAE IR Multi-Gas Monitor to the manufacturer. Stone typically sends the MultiRAE IR Multi-Gas Monitor to the manufacturer annually for lamp cleaning and general maintenance – or more frequently as needed. Call the manufacturer for shipping instructions prior to sending the MultiRAE IR Multi-Gas Monitor.

4.2.7 Documentation

A record of calibrations and any maintenance performed on the PID must be made in the Maintenance and Calibration log.

If used in the field to screen soil samples, PID values are recorded onto soil boring logs (see attached). If screening is being conducted of miscellaneous soil samples, such as those from surface samples, values should be recorded on "Observations and Remarks (O&R)" forms.

4.3 Collection of Soil Samples for Headspace Screening

Fill a clean jar or baggie approximately half full using the stainless steel spoon with a "consistent amount" of soil. Take special care to ensure that sample and container are not cross-contaminated from previous sample by rinsing spoon between samples with distilled water. It is also important to ensure that the sample is not frozen prior to analysis.

Seal top of jar with two layers of aluminum foil, or insure that bagged is properly sealed. Secure aluminum foil with either a rubber band or screw lid that fits jar. If using screw lid, do not include solid top with rubber gasket included with new mason jars.

Allow VOC concentrations to develop in the headspace of the sample container for five minutes. During this headspace development period, the sample should be shaken vigorously for a total of one minute.

Immediately pierce the aluminum foil seal (or un-zip an approximate one-inch section of baggie) with the probe of PID and note the highest (peak) reading, and sustained reading. Take care not to submerge PID tip into soil.

Record time of sample collection and peak value as ppm onto appropriate form (Soil Boring Log or O&R).

4.4 Documentation

A record of the soil screening must be made in the field at the time the peak sample headspace concentration is read from the PID. It may also be helpful to record the weather or any site-specific conditions such as excessive heat or cold that may affect the readings from the PID. Be sure that field notes are properly labeled to the project number and date.

5.0 RESPONSIBILITIES

1. SEI personnel must calibrate the MultiRAE IR Multi-Gas Monitor prior to use. Calibrations should be recorded in the Maintenance and Calibration Log.
2. It is the responsibility of SEI field staff to make sure that the MultiRAE IR Multi-Gas Monitor is in proper working order prior to bringing it onto a site.
3. SEI personnel must read this SOP prior to using the MultiRAE IR Multi-Gas Monitor. After reading the SOP, personnel should update their personnel training folder.

6.0 DEFINITIONS

1. *EPA* the U.S. Environmental Protection Agency.

2. *Observations & Remarks Form (O&R):* A pre-printed form, which contains mostly blank space for general note taking. The form typically prompts the user for the study or project designation, the SEI project number, the client or sponsor name, the total number of pages (page *n* of n) and requires a signature and date. The form is generally used to capture notes of one person when another, more specific forms is not available.
3. *PID*: Photoionization Detector
4. *PPM*: Parts per million.
5. *Quality Assurance Unit (QAU)* any person or organizational element except the study director, designated by testing facility management to perform the duties related to quality assurance of the studies.
6. *Raw data* any laboratory worksheets, records, memoranda, notes, or exact copies thereof, that are the result of original observations and activities of a study and are necessary for the reconstruction and evaluation of the report of that study. In the event that exact transcripts of raw data have been prepared (e.g., tapes which have been transcribed verbatim, dated, and verified accurate by signature), the exact copy or exact transcript may be substituted for the original source as raw data. Raw data may include photographs, microfilm or microfiche copies, computer printouts, magnetic media, including dictated observations, and recorded data from automated instruments.

7.0 REFERENCES

1. Vermont State Agency of Natural Resources, Waste Management Division "Agency Guidelines for Petroleum Contaminated Soils and Debris" August 1996.
2. MultiRAE IR Multi-Gas Monitor, PGM-54, Operation and Maintenance Manual, Rev. B. December, 2001

8.0 TABLES, DIAGRAMS, FLOWCHARTS, AND VALIDATION DATA

None

9.0 AUTHORIZATION

Revised by: _____ Date: 2/5/10

Willapa J. Waterstrat, Project Scientist

Approved by: _____ Date: 2-4-2011

Christopher T. Stone, President

10.0 REVISION HISTORY

1. Not applicable.

STANDARD OPERATING PROCEDURE

SEI-5.30.2

USE, CALIBRATION, AND MAINTENANCE OF THE YSI MODEL 600XL MULTI-PARAMETER WATER QUALITY MONITORING SYSTEM (TEMPERATURE, SPECIFIC CONDUCTANCE, pH, OXIDATION REDUCTION POTENTIAL)

SOP Number: SEI-5.30.2
Revision Number: 2

Date Issued: 08/13/02
Date of Revision: 7/12/12

1.0 OBJECTIVE

The YSI Model 600XL Water Quality Monitoring System is used to monitor the temperature, specific conductance, pH, and oxidation-reduction potential (ORP) of water flowing through the system's flow-through cell. These parameters are measured to gain knowledge of the quality of the water and to ensure that a representative sample is being collected. To ensure the accuracy of the measurements, each of the probes in the system must be calibrated and have periodic and preventative maintenance.

2.0 SUMMARY OF METHOD

At Stone Environmental, Inc. the YSI 600 XL is most often used in the low-flow sampling of groundwater. The YSI is equipped with a low-volume flow through cell though which water is pumped at a specified rate. Data produced by the YSI 600 XL in the form of Specific Conductance, pH, Oxidation-Reduction Potential and Temperature is recorded at predetermined intervals until parameters stabilize. For a description of how the YSI 600 XL is incorporated within the K-Pro groundwater profiling program (SOP SEI-10.5.*n*) or sampling program (SOP SEI-6.27.*n*). For details regarding low-flow sampling methodology, refer to SOP SEI-6.34.0. This SOP serves as a reference for use, maintenance and calibration of the YSI 600 XL.

3.0 DEFINITIONS

1. *Oxidation-Reduction Potential* (ORP): The ability for a compound to gain electrons (Reduce) or lose electrons (Oxidize).
2. *pH:* Negative log rhythm of Hydrogen Ion (H^+) activity in solution. Ranges from 0 (acidic) to 7 (neutral) to 14 (basic).
3. *Conductivity:* The ability of water to conduct electricity as a function of dissolve ionic species, suspended solids, and organic content. Usually measured in Siemans (S) or microsiemans (μS).

4.0 INTERFERENCES

The manufacturer reports no interferences with any of the sensors. The presence of HS^- in sample water can lead to a greatly shortened membrane life on the dissolved oxygen (DO) sensor. As such, Stone uses stand-alone DO meters to measure this parameter when using the YSI 600XL.

5.0 SAFETY ISSUES

1. SEI staff will read the site health and safety plan (HASP) prior to beginning a project.
2. To avoid accidental eye contact with standard solutions, gloves and eye protection should be worn when calibration is performed.
3. Field visits may involve accessing remote areas. Health and safety concerns regarding these field visits are minimal, however, hazards such as slip, trip and falls, poisonous plant and dangerous animals, as well as getting vehicles stuck in remote areas, do present considerable health and safety issues. To help ensure field staff's health and safety in remote areas, all field staff are to have daily communication with the project manager or another appropriate SEI employee.
4. All chemicals are required to be received with a Material Safety Data Sheets (MSDS). MSDS shall be made available to all personnel involved in the sampling and testing.

6.0 EQUIPMENT AND SUPPLIES

The YSI 600XL system consists of the following components:

- YSI 600 XL Sonde body
- Power cable (either AC or DC depending on type of power source)
- Communication Cable
- Laptop computer with EcoWatch software
- YSI Model 6567 pH/ORP Probe
- YSI Model 6560 Conductivity/Temperature Probe
- YSI Custom Made 50 mL Flow-Through Cell
- Use, Calibration and Maintenance Log book

Ancillary equipment for calibration, maintenance and general use includes:

- Sensor removal tool
- Calcium Lime Rust (CLR) cleaner
- Cotton swabs
- Scissors
- Isopropyl Alcohol
- Dust Off or equivalent compressed air
- Scotch Guard scrub pad.
- Stopcock grease
- 3/8th" silicon tubing
- 1/4th " LDPE tubing
- ¼" to ½" hose clamps
- Graduated Cylinder
- Purge bucket

7.0 REAGENTS AND STANDARDS

Standards for use in calibration of the YSI 600 XL are purchased as specific solutions and come from the manufacturer with a traceable lot number. These solutions include the following:

- pH calibration solution (e.g. pH 4.02 and 7.0)
- Conductivity calibration solution (within range expected in field)
- ORP solution or powdered standard

ORP solution generally comes as Eh standard. Eh, or Redox Potential, is equal to ORP + 200 mV. Therefore, to determine the ORP value for the standard, subtract 200 mV from the Eh value. Alternatively, a powdered ORP standard can be used. Mix one sachet of powder with 100ml of distilled water and ensure all powder has dissolved. This yields a 250mV solution.

When a Sonde needs to be re-calibrated, personnel should ensure that calibration standards are appropriately labeled (as described in SOP SEI-4.7.*n*) and within the expiration criteria. The lot number and expiration date for each standard used for recalibration should be noted within the Use, Calibration and Maintenance Log and in the field notes as necessary and appropriate.

8.0 PROCEDURES

8.1 General Considerations

To effectively use the YSI model 600 multi-parameter water-quality monitoring system, it is advised that users refer to the user manual in addition to this SOP. Due to the volume of this manual (>300 pages), it is not included as an attachment to this SOP. Digital copies of the manual can be found on the Stone network at L:\RASC\Reference Materials\YSI Manual or in the same directory as this SOP (See Footer).

When YSI is used in conjunction with the K-Pro sampling apparatus, do not allow HCl^- from pre-preserved VOA vials to pass through the YSI flow cell. Pre-rinse all vials that contain preservative before sampling. Failure to do this can result in corrosion of the probes. HCl^- or other preservative should be added after the sample has been collected.

To prolong the lifespan of individual probes on the sonde, appropriate storage conditions should be implemented if the unit will not be used for a period of two weeks or more. Please see Table 1 for further detail on storage requirements.

8.2 Operation

Assemble the system according the manufacturer's instructions (Section 2 of Instruction Manual). Be sure all seals and o-rings are in good condition before assembly, and repair or replace them if necessary. To prevent sample water from coming in contact with internal electrical circuitry stopcock grease should be applied in a thin film along the o-ring of each sensor prior to inserting it into the sonde. Applying grease to the threads of the sensors is not necessary and can result in cross threading of the sensor in the sonde body.

The YSI 600 XL is designed to work along with EcoWatch software. Once this program is initialized, the user should open communication between the YSI and the program by selecting

which Communications Port is being used for the YSI under the "Sonde" toolbar heading. To determine which comport is in use, check hardware system settings under the laptop control panel. After communication has been established, the user should type "Menu" at the # prompt. This will bring up the main menu. Select the option desired from this menu and follow directions as they appear on the screen. The YSI can be set to take measurements at a desired interval. To prolong sensor life, it is recommended by the manufacturer that the interval between readings be set to 10 seconds.

8.3 CALIBRATION

Sondes should be calibrated before each use. If calibration is required more than once per day, a solution may be used more than once, provided it is not returned to its original container.

8.3.1 Temperature/Conductivity

The temperature and conductivity probes are deployed as a combination probe. The temperature probe does not require calibration according to the manufacturer (Section 4.2 of Instruction Manual). However, it is suggested that the temperature probe be verified with an independent thermometer before use. The calibration of the conductivity cell should be checked before use at each test site and at least weekly when in use at the same site. If the reading is within 10% of the correct value no calibration is required. If the reading differs by more than 10% of the correct value the probe must be re-calibrated. Specific calibration instructions can be found in Section 4.2 of the Instruction Manual. Note the details of each calibration check in the system's Use, Calibration and Maintenance Log.

8.3.2 pH/ORP

Calibration of the pH/ORP combination probe should be checked before use at each site, and at least weekly when in use at the same site, following the manufacturer's instructions (Section 4.2 of Instruction Manual). If the reading is within 10% of the correct value no calibration is required. If the reading differs by more than 10% of the correct value the probe must be re-calibrated. Note the details of each calibration in the system's Use, Calibration and Maintenance Log.

8.4 Maintenance

8.4.1 600XL Multi-Parameter Probe

Record all maintenance in the Use, Calibration and Maintenance Log (See Attachment 1 for an example entry). Each time the system is used, note the date of use and the personnel present in the Use, Calibration, and Maintenance Log for the system.

The 600XL multi-probe requires little maintenance. The unit may be cleaned using a mild soap solution, and stubborn stains (i.e. inorganic salt deposits) may be removed using either

a solution of 10% hydrochloric acid or a solution of Calcium Lime Rust (CLR) cleaner (or a similar stain remover). This should be done every other week when in heavy use.

The unit is water resistant, but care should be taken to ensure that water does not get into the main body of the unit where it can damage the internal circuitry. If not all probes are deployed with the unit; make sure the probe inserts are covered with the appropriate screw-in plugs. If water does get into the unit, disassemble it, wipe any excess water, and allow the unit to air dry - do not use hot air. Compressed air can be used to expedite drying.

Unlike other multi-parameter probes, the 600 XL has no internal batteries and therefore no power maintenance. Power cables for AC and DC are supplied with the unit.

Please refer to Table 1 for long-term storage conditions for each probe. Long term is this instance is any period of time that the probe will not be used that is two weeks or longer.

8.4.2 *Flow-Through Cell*

Clean the flow-through cell and tubing as needed using an appropriate solution as discussed in Section 8.4.1. If microbial growth becomes an issue when storing the specific conductance sonde in the flow through cell with a moistened paper towel, the probe can be stored dry. Replace o-rings on as-needed bases. Note all o-ring changes and cleaning in the Use, Calibration and Maintenance Log for the unit.

8.4.3 *Temperature/Conductivity Combination Probe*

The temperature/conductivity combination probe requires very little maintenance. If deposits are present, they can be removed using a 1 to 1 CLR solution and a scotch pad. Refer to Section 7.2 of Instruction Manual or Table 1 for further instructions regarding long- and short-term storage. Note all cleaning and reconditioning in the Use, Calibration and Maintenance Log for the unit. If the probe cannot be re-conditioned, it must be replaced.

8.4.4 *pH/ORP Combination Probe*

The pH/ORP combination probe is a fully enclosed unit and therefore requires very little maintenance. If deposits are present on the glass bulb they must be removed using appropriate solutions as discussed in Section 8.4.1. Scotch Guard pads may be used to clean build-up of deposits from both the pH and ORP probes. Refer to Section 7.2 of Instruction Manual or Table 1 for further instructions regarding the long- and short-term storage of this unit. Note all cleaning and reconditioning in the Use, Calibration and Maintenance Log for the unit. If the probe cannot be re-conditioned, it must be replaced.

Cleaning the unit is done using the appropriate solution(s) as discussed in Section 8.4.1. Refer to Section 7.2 of Instruction Manual or Table 1 for further instructions regarding long

and short-term storage. Note all cleaning and reconditioning in the Use, Calibration and Maintenance Log for the unit. If the probe cannot be re-conditioned, it must be replaced.

8.5 USE, CALIBRATION AND MAINTENANCE LOG

Any use, calibration, or maintenance performed on any part of the YSI 600XL system must be noted in the designated Use, Calibration and Maintenance Log (Attachment 1). Indicate whether the action performed was calibration, routine maintenance (such as replacing reference solutions or o-rings), or non-routine maintenance to correct a failure or malfunction of any part of the system (including replacement of probes). Note the date on which the action is taken, and describe the action taken. Be sure to initial and date each entry.

Each uniquely assigned Use, Calibration and Maintenance logbook should be kept with the instrument to which it was assigned at all times.

9.0 RESPONSIBILITIES

1. It is the responsibility of the individual employee to read SOPs and document training associated with the area of work they are performing.
2. It is the responsibility of the individual employee to follow SOPs covering activities in his/her work area or to identify a deviation from the written SOP.
3. All personnel will legibly record data and observation to enable others to reconstruct project events and provide sufficient evidence of activities conducted.
4. All personnel will label each page with the date, the signature of the person taking notes (initials may also be appropriate), and the page number. All notes, signatures, and other observations should be entered in the field at the time the notes are taken.

10.0 REFERENCES

YSI Model 600XL Multi-parameter Water Quality Monitoring System Operations Manual, YSI Incorporated.

11.0 TABLES, DIAGRAMS, FLOWCHARTS, AND VALIDATION DATA

Table 1: Long-Term Storage Matrix for YSI 600 XL

Probe/ISE	Long Term[1]	Short Term	Comments
pH/ORP	Remove probe from Sonde and place in pH 4.0 Solution	Moist towel in cal cup	
Spec Conductivity	Clean probe with CLR and place in cal cup with moist towel	Moist towel in cal cup	Can also be stored dry for long term.
DO	Remove probe from sonde, inspect for electrode and membrane health and place in 2M KCl solution[2]	Moist towel in cal cup	Always store with membrane and electrolyte in place. At the end of long-term storage, always install a new membrane with fresh

			electrolyte.

[1] Long term should be considered any period longer than 2 weeks.

[2] 2 Molar KCl solution is prepared by adding 149 grams of granular KCl to 1 liter of distilled water.

12.0 AUTHORIZATION

Authored by: _____ Date: 8/16/12

Daniel T. Voisin, Staff Geoscientist

Approved by: _____ Date: 8-16-12

Christopher T. Stone, President

13.0 REVISION HISTORY

Revision Number 1

1. Added Table 1: Storage matrix
2. Added text to Section 8.0- Procedures
3. Updated equipment list in Section 6.0
4. Added text to Section 8.3
5. Added text to Section 8.4
6. Added text to Section 8.5
7. Re-Formatted to Analytical SOP in accordance with SEI SOP 3.1.8

Revision Number 2

1. Removed all information about D.O. sensor, since YSI no longer has a D.O. sensor. The SOP for the HACH stand-alone D.O. sensor that is usually used with the YSI is 5.28.0 (WJW, 2/7/11)
2. Minor grammatical changes.
3. Updated calibration instructions
4. Changed number from 10.7.2 to 5.30.2.

ATTACHMENT 1
Example YSI 600 Calibration and Maintenance Log

Calibration and Maintenance Log - YSI Model 600 Water Quality Monitoring System

Model ID:_____
Date:_____ Dates of Use: From _____ to _____
User Name(s):_____
SEI Project I.D.:_____

Calibration Record

Parameter	Expected Meter Value	Actual Meter Value	Comments/Corrective Action
pH	7.00		
Lot#/Exp. Date			
	4.02		
Lot#/Exp. Date			
ORP			
Lot#/Exp. Date			
DO			
Lot#/Exp. Date			
Conductivity			
Lot#/Exp. Date			

Maintenance Record

Comments/Corrective Action

Check One

Routine Maintenance: _____ Non-Routine Maintenance: _____

Annual Inspection: _____ Calibration: _____

Other: (Specify) _____ Use: _____

Signature(s) _____

_____

STONE ENVIRONMENTAL INC

STANDARD OPERATING PROCEDURE

SEI-5.22.2

MAINTENANCE AND CALIBRATION OF THE TROLL SP4000 AND MINITROLL SSP-100 DATALOGGER

SOP Number: SEI-5.22.2
Revision Number: 2

Date Issued: 05/14/99
Date of Revision: 02/24/04

1.0 OBJECTIVE

This standard operating procedure (SOP) sets forth in sufficient detail the methods, materials, and schedules to be used in the maintenance and calibration for Troll SP4000 and MiniTroll SSP-100 datalogger. It also describes the methods to be followed to ensure that the Troll datalogger and pressure transducer are kept in good working order. The Troll datalogger generates data, and thus must meet the requirements of 40 CFR part 160 subpart D.

2.0 POLICIES

1. According to 40 CFR Part 160, Subpart D, Section 160.61, Equipment used in the generation, measurement, or assessment of data and equipment used for facility environmental control shall be of appropriate design and adequate capacity to function according to the protocol and shall be suitable located for operation, inspection, cleaning, and maintenance.
2. Personnel will legibly record data and observations in the field to enable others to reconstruct project events and provide sufficient evidence of activities conducted.

3.0 SAFETY ISSUES

1. If necessary and appropriate, a site-specific health and safety plan shall be created for each study site. A template for creating a proper health and safety plan is provided on the SEI network.
2. If necessary and appropriate, all chemicals are required to be received with a Material Safety Data Sheets (MSDS) or appropriate application label. These labels or MSDS shall be made available to all personnel involved in the sampling and testing.

4.0 PROCEDURES

4.1 Equipment and Materials

1. Troll Intelligent Probe and associated accessories and RS232 kit (Environmental Datalogger/Pressure transducer), Model SP4000 and/or MiniTroll SSP-100, manufactured by In-Situ Inc. of Laramie, Wyoming.

2. Cable for connecting the probe to an external computer. Appropriate interface adapters.

3. If necessary, external power pack, adapter and cord for connecting the probe to an electrical wall socket. Input: 105 - 130 volts AC, 60 HZ; Output: 15 volts DC, 500 mA.

4. If necessary, external power adapter and cord for connecting the probe to an automobile cigarette lighter (12 volt DC).

5. Communications software: Win-Situ Data Acquisition Software Version 2.1 and Win-Situ 2000 Version 3.70 (for Troll SSP-100).

6. Operators Manuals.

    a. Troll SP4000 and MiniTroll SSP-100.

    b. Win-Situ Data Acquisition Software Version 2.1.

7. Computer with Win-Situ Software installed.

Prior to use in the field, the user should obtain (if available), read and understand the manufacturer's operation manual.

4.2 Decontamination

The probe shall be tripled rinsed with distilled or deionized water and wiped off after use and if necessary, prior to use. Further decontamination and cleaning procedures may be called for in special situations or outlined in approved protocols or work plans. This will be documented in field notes or in an appropriate logbook.

4.3 Calibration

Both probes were certified as calibrated by the manufacturer prior to shipment to SEI. Calibration documents will be kept on file.

The calibration should be verified by comparing readings to hand measurements or other independent measurements. The frequency of the field check will be based on the use of the pressure transducer.

Generally this should be done annually unless the equipment is not used for an extended period of time.

4.4 General and Annual Maintenance

Individual users are responsible for the cleaning, verifying, repair, and maintenance of the instrument.

Routine inspection and maintenance schedules vary for each piece of equipment. Typically there are minor maintenance needs each piece of equipment will need to undergo prior to use in the field (such as cleaning or conditioning). Always consult the manufacturer's instructions for general maintenance.

Specific per use maintenance needs for the Troll Dataloggers include, but are not limited to:

1. Inspect for physical damage.
2. Check receptacle pins for debris.
3. Inspect cables.
4. Alconox rinse.
5. Distilled water rinse.
6. Clean probe with mild detergent.

The Troll datalogger shall be stored in it's case, along with the accessories and cables. The cable should be stored on a reel to prevent kinks in the cable. Care should be given to keep the instrument from dust and contamination.

All maintenance, repairs, and calibrations are to be documented on an equipment maintenance log or other appropriate medium. Follow the checklist provided on the equipments maintenance log for regular use maintenance needs. Any maintenance must include documentation of whether the maintenance was routine and followed the SOP or not.

Equipment logs shall be brought to the field for documenting use and calibration. The logs will be returned to the office after each field use and filed in the equipment records filing cabinet.

In the event of failure due to breakage or loss of parts, an attempt will be made to repair or replace the necessary parts by the field personnel who discover the malfunction. All repairs will be documented in field notes and/or on a non-routine maintenance log. If the instrument is rendered "out of service" or "broken", it should be tagged as such. If further repair is necessary, return the instrument to the manufacturer following proper shipping procedures.

Non-routine repairs must include documentation of the nature of the defect, how and when the defect was discovered, and any remedial action taken in response to the defect.

Replace the batteries as necessary and appropriate in accordance with the manufacturer's instructions Document the battery replacement on appropriate log forms. The battery pack is replaceable in the field.

Annual maintenance is outlined in the annual maintenance checklist for each piece of equipment. All annual maintenance shall be conducted in no more than 13 month increments after the initial use of the equipment.

5.0 RESPONSIBILITIES

1. All personnel will legibly record data and observations (including phone conversations) in accordance with this SOP to enable others to reconstruct project events and provide sufficient evidence of activities conducted.

2. Prior to use and after use, all equipment will be appropriately cleaned, decontaminated and calibrated (if necessary) in accordance with the manufacturer's instructions and this SOP.

6.0 DEFINITIONS

1. *Decontamination* – Procedures followed to ensure cross contamination does not occur between sampling points or that potential contamination of equipment does not pose a hazard to sampling personnel.
2. *EPA* the U.S. Environmental Protection Agency.
3. *FIFRA* the Federal Insecticide, Fungicide, and Rodenticide Act as amended.
4. *Maintenance* – Actions performed on equipment to standardize and/or correct the accuracy and precision of a piece of equipment to ensure that the equipment is operating within the manufacturer's specifications and standard values.
5. *Quality Assurance Unit (QAU)* any person or organizational element except the study director, designated by testing facility management to perform the duties related to quality assurance of the studies.
6. *Study* means any experiment at one or more test sites, in which a test substance is studied in a test system under laboratory conditions or in the environment to determine or help predict its effects, metabolism, product performance (pesticide efficacy studies only as required by 40 CFR 158.640) environmental and chemical fate, persistence, or residue, or other characteristics in humans, other living organisms, or media. The term "study" does not include basic exploratory studies carried out to determine whether a test substance or a test method has any potential utility.

7.0 REFERENCES

40 CFR Part 160 Good Laboratory Practice Standards, August, 1989.

8.0 TABLES, DIAGRAMS, FLOWCHARTS, AND VALIDATION DATA

None

9.0 AUTHORIZATION

Revised by: Michael Nuss Date: 2/24/04

Michael Nuss, Staff Scientist

Approved by: Date: 2-24-04

Christopher T. Stone, President

10.0 REVISION HISTORY

Revision number 1:

1. Changed Title.
2. Removed sections 4.0 (measurement procedure) and 5.0 (maintenance/repairs).
3. Added section 4.0 (General and Annual Maintenance).
4. Minor editing.
5. Reformatted.

Revision number 2:

1. Added reference to the MiniTroll SSP-100 and the Win-Situ 2000 Software v.3.70 throughout text.
2. Minor wording edits in Section 1.0, Objective.
3. Updated style to match SEI Style Guide – font and text. Reformatted using MS Word.
4. Added standardized section headers: 2.0 Policies, 3.0 Safety, 5.0 Responsibilities, 6.0 Definitions, 7.0 References, 8.0 Tables, Diagrams, Flowcharts and Validation data. Authorization moved to Section 9.0, andSection10.0 Revision History.
5. Deleted section on logs being given to the QAU.
6. Other minor wording edits

STANDARD OPERATING PROCEDURE

SEI-6.49.0

PROCEDURE FOR COLLECTION OF INDOOR AIR SAMPLES FOR TO-15 VOLATILE ORGANIC ANALYSIS USING SUMMA CANISTERS

SOP Number: SEI-6.49.0
Revision Number: 0

Date Issued: 03/22/13
Date of Revision: NA

1.0 OBJECTIVE

The objective of this standard operating procedure (SOP) is to provide reference for collecting indoor air samples that are representative of volatile organic compounds (VOCs) concentrations within the indoor air. Sampling locations and methodology must be selected to achieve the objectives of the study and the data required. Project planning details should be provided to the sampler prior to sampling event.

Health and safety of the sampling personnel is of utmost importance; therefore, prior to sampling a site specific health and safety plan or equivalent task analysis should be developed and reviewed by the sampler prior to sampling.

2.0 POLICIES

1. According to the Corporate Quality Management Plan (QMP), Stone shall have standard operating procedures in writing setting forth study methods that management is satisfied are adequate to insure the quality and integrity of the data generated in the course of a study.
2. Personnel will legibly record data and observations in the field to enable others to reconstruct project events and provide sufficient evidence of activities conducted.

3.0 SAFETY ISSUES

1. A site-specific health and safety plan (HASP) shall be created for the site. A template for creating a proper health and safety plan is provided on the Stone network. If a HASP was created by a company other than Stone, then staff must review and sign off that they have reviewed the site specific HASP.
2. Care must always be taken when approaching a sampling location. Do not, under any circumstances, place yourself in danger to collect a sample.

4.0 PROCEDURES

4.1 Equipment and Materials

1. Photoionization detector (PID) equipped with a 10.6 eV bulb to determine risk of exposure to sampling personnel, as required in the HASP and to screen sample locations.

2. Appropriate personnel protective equipment as specified in the HASP.

3. For Summa Canister Method (TO-15 Analysis):

    a. Certified pre-cleaned, 6-Liter Summa Canisters and laboratory canister chain of custody (COC) form(s).

    b. Laboratory instructions and shipping material, if provided.

    c. Flow controllers calibrated for the desired collection period, usually 8 hour for commercial/industrial properties and 24 hour for residential properties.

4. Pre-Sample Site Inspection Form

5. Field notebook, sample collection form (see Attachment 1), or other acceptable medium for recording field data. Ball point pens.

6. Open ended wrenches to match fittings on Summa canister and flow controller. Typically, these fittings take two 9/16" wrenches.

4.2 Pre-sample collection inspection

Prior to deploying canisters for sample collection, the structure should be inspected for conditions and contents that may affect the presence, distribution or magnitude of VOCs in indoor air. The inspection should include:

1) Occupant information, including:
    a) Contact information
    b) Number of occupants
    c) Age of occupants
2) Owner or Landlord contact information
3) Building Characteristics, including:
    a) Use type (e.g., residential or industrial)
    b) Number of floors, age, tightness

c) Materials and methods of construction

4) Airflow characteristics and patterns

5) Bottom floor construction and entry points for soil vapor

6) Heating, venting and air conditioning

7) Occupancy

8) Factors that may influence air quality

9) Site Utilities

10) Presence of VOC containing materials (See Section 8 of Attachment 1 for list)

Findings of the site inspections should be recorded on the field form provided as Attachment 1 of this SOP.

4.3 Sample Collection Procedure

Read and understand all laboratory instructions prior to sample collection, if provided.

Summa canisters should be deployed in an open area at a height of three (3) feet above the floor surface. Record canister and flow controller serial number on label, field form (Attachment 2) and chain of custody.

Open canister and record initial vacuum on to the field form. Do not linger in the area of the canister.

Following the sample collection period, shut the canister and record the final vacuum. If no vacuum remains (gauge reads zero) the flow controller is faulty and the sample collection period is uncertain; the sample should be abandoned and a new sample collected.

Following successful sample collection, remove the flow controller and install compression fitting plugs on the canister. All observations are recorded in to field forms. Samples are to be packaged for shipping and the chain of custody form is completed.

4.4 Retrieving Quality Control Samples

Duplicate samples for either split confirmation analyses or field duplicates will be collected in the same manner as described in Section 4.3.

To establish background concentration of site contaminants of concern a sample is collected of outdoor ambient air. Deploy a canister in a secure upwind location at least 50 feet away from any structures. Sample collection procedures for background samples should mimic indoor air quality samples..

4.5 Sample Packing and Transportation

Pack samples in the containers that the canisters arrived in and seal with packing tape. Samples should be sent to the laboratory as soon as is reasonable, but within the maximum holding time for the sample. Chain of custody forms will be filled out and signed prior to shipment to the laboratory and included in the shipping container with the samples, according to standard operating procedures (SEI-4.2.n) and/or protocol and work plan instructions.

5.0 RESPONSIBILITIES

1. All personnel will legibly record data and observations (including phone conversations) in accordance with this SOP to enable others to reconstruct project events and provide sufficient evidence of activities conducted.

6.0 DEFINITIONS

1. *EPA* the U.S. Environmental Protection Agency.
2. *Maintenance* – Actions performed on equipment to standardize and/or correct the accuracy and precision of a piece of equipment to ensure that the equipment is operating within the manufacturer's specifications and standard values.

7.0 REFERENCES

Corporate Quality Management Plan, September 2012.

New York Department of Health, Center for Environmental Health, Bureau of Environmental Exposure Investigation, *Guidance for Evaluating Soil Vapor Intrusion in the State of New York*, October 2006.

8.0 TABLES, DIAGRAMS, FLOWCHARTS, AND VALIDATION DATA

Attachment 1: Indoor Air Quality Questionnaire and Building Inventory

Attachment 2: Indoor Air Sample Collection Form

9.0 AUTHORIZATION

Authored by: _____ Date: 3/21/13

Daniel Voisin, Project Scientist II

Approved by: _____ Date: 3-21-13

Christopher T. Stone, President, Management

10.0 REVISION HISTORY

ATTACHMENT 1: INDOOR AIR QUALITY QUESTIONNAIRE AND BUILDING INVENTORY

Stone Environmental, Inc.

Indoor Air Quality Questionnaire & Building Inventory

This form must be completed for each structure involved in indoor air testing.

Preparer's Name: _____ Date/Time Prepared: _____

Preparer's Affiliation: _____ Phone No: _____

Purpose of Investigation: _____

Project # _____ Project Title: _____

1. OCCUPANT

Interviewed: Y/N

Last Name: _____ First Name: _____

Address: _____

County: _____

Home Phone: _____ Office Phone: _____

Number of Occupants/persons at this location _____ Age of Occupants _____

2. OWNER OR LANDLORD: (check if same as occupant___)

Interviewed: Y/N

Last Name: _____ First Name: _____

Address: _____

County: _____

Home Phone: _____ Office Phone: _____

3. BUILDING CHARACTERISTICS

Type of Building: (Circle appropriate response)

Residential	School	Commercial/Multi-use
Industrial	Church	Other: _____

If the property is residential, type? (Circle appropriate response)

Ranch	2-Family	3-Family
Raised Ranch	Split Level	Colonial
Cape Cod	Contemporary	Mobile Home
Duplex	Apartment House	Townhouses/Condos
Modular	Log Home	Other: _____

If multiple units, how many? _____

If the property is commercial, type?

Business Type(s) _____

Does it include residences (i.e., multi-use)? Y/N If yes, how many? _____

Other Characteristics:

Number of floors _____ Building age _____

Is the building insulated? Y/N How air tight? Tight / Average / Not Tight

4. AIRFLOW

Use air current tubes or tracer smoke to evaluate airflow patterns and qualitatively describe:

Airflow between floors

_____

_____

_____

Airflow near source

_____

_____

_____

Outdoor air infiltration

_____

_____

_____

Infiltration into air ducts

_____

_____

_____

5. BASEMENT AND CONSTRUCTION CHARACTERISTICS (Circle all that apply)

a. Above grade construction:	Wood frame	Concrete	Stone	Brick
b. Basement type:	Full	Crawlspace	Slab	other _____
c. Basement floor:	Concrete	Dirt	Stone	other _____
	Uncovered	Covered	Covered with	_____
d. Concrete floor:	Unsealed	Sealed	Sealed with	_____
e. Foundation walls:	Poured	Block	Stone	other _____
	Unsealed	Sealed	Sealed with	_____
f. The basement is:	Wet	Damp	Dry	Moldy
	Finished	Unfinished	Partially finished	
g. Sump present?	Y / N			
h. Water in sump?	Y / N / not applicable			

Basement/Lowest level depth below grade: _____ (feet)

Identify potential soil vapor entry points and approximate size (e.g., cracks, utility ports, drains)

_____

_____

6. HEATING, VENTING, and AIR CONDITIONING (Circle all that apply)

Type of heating system(s) used in this building: (note primary)

Hot air circulation	Heat pump	Hot water baseboard	
Space heaters	Steam radiation	Radiant floor	
Electric baseboard	Wood stove	Outdoor wood boiler	other _____

Primary type of fuel used is:

Natural Gas	Fuel Oil	Kerosene
Electric	Propane	Solar
Wood	Coal	

Domestic hot water tank fueled by: _____

Boiler/furnace located in: Basement Outdoors Main floor Other _____

Air conditioning: Central Air Window units Open windows None

Are there air distribution ducts present? Y / N

Describe the supply and cold air return ductwork, and its condition where visible, including whether there is a cold air return and the tightness of duct joints. Indicate the locations on the floor plan diagram.

_____

_____

_____

_____

7. OCCUPANCY

Is basement/lowest level occupied? Full time Occasionally Seldom Almost Never

Level	General Use of Each Floor (e.g., family room, bedroom, laundry, workshop, storage)
Basement	
1st floor	
2nd floor	
3rd floor	
4th floor	

8. FACTORS THAT MAY INFLUENCE INDOOR AIR QUALITY

Is there an attached garage?	Y / N	
Does the garage have a separate heating unit?	Y / N	
Are petroleum-powered machines or vehicles stored in the garage (e.g., lawnmower, atv, car)	Y / N / NA	Please specify _____
Has the building ever had a fire?	Y / N	When? _____
Is a kerosene or unvented gas space heater present?	Y / N	Where? _____
Is there a workshop or hobby/craft area?	Y / N	Where & Type? _____
Is there smoking in the building?	Y / N	How Frequently? _____
Have cleaning products been used recently?	Y / N	When & Type? _____
Have cosmetic products been used recently?	Y / N	When & Type? _____
Has painting/staining been done in the last 6 months?	Y / N	Where & When? _____
Is there new carpet, drapes, or other textiles?	Y / N	Where & When? _____
Have air fresheners been used recently?	Y / N	When & Type? _____
Is there a kitchen exhaust fan?	Y / N	Where vented? _____

Is there a bathroom exhaust fan? Y / N Where vented? _____

Is there a clothes dryer? Y / N Vented outside? _____

Has there been a pesticide application? Y / N When & Type? _____

Are there odors in the building? Y / N

If yes, please describe: _____

Do any of the building occupants use solvents at work? Y / N
(e.g., chemical manufacturing or laboratory, auto mechanic or auto body shop, painting, fuel oil delivery, boiler mechanic, pesticide application, cosmetologist)

If yes, what type of solvents are used? _____

If yes, are their clothes washed at work? Y / N

Do any of the building occupants regularly use or work at a dry-cleaning service? (Circle appropriate response)

Yes, use dry-cleaning regularly (weekly) No

Yes, use dry-cleaning infrequently (monthly or less) Unknown

Yes, work at a dry-cleaning service

9. WATER AND SEWAGE

Water Supply: Public Water Drilled Well Driven Well Dug Well Other: _____

Sewage Disposal: Public Sewer Septic Tank Leach Field Dry Well Other: _____

10. RELOCATION INFORMATION (for oil spill residential emergency)

a. Provide reasons why relocation is recommended: _____

b. Residents choose to: Remain in home Relocate to friends/family Relocate to hotel/motel

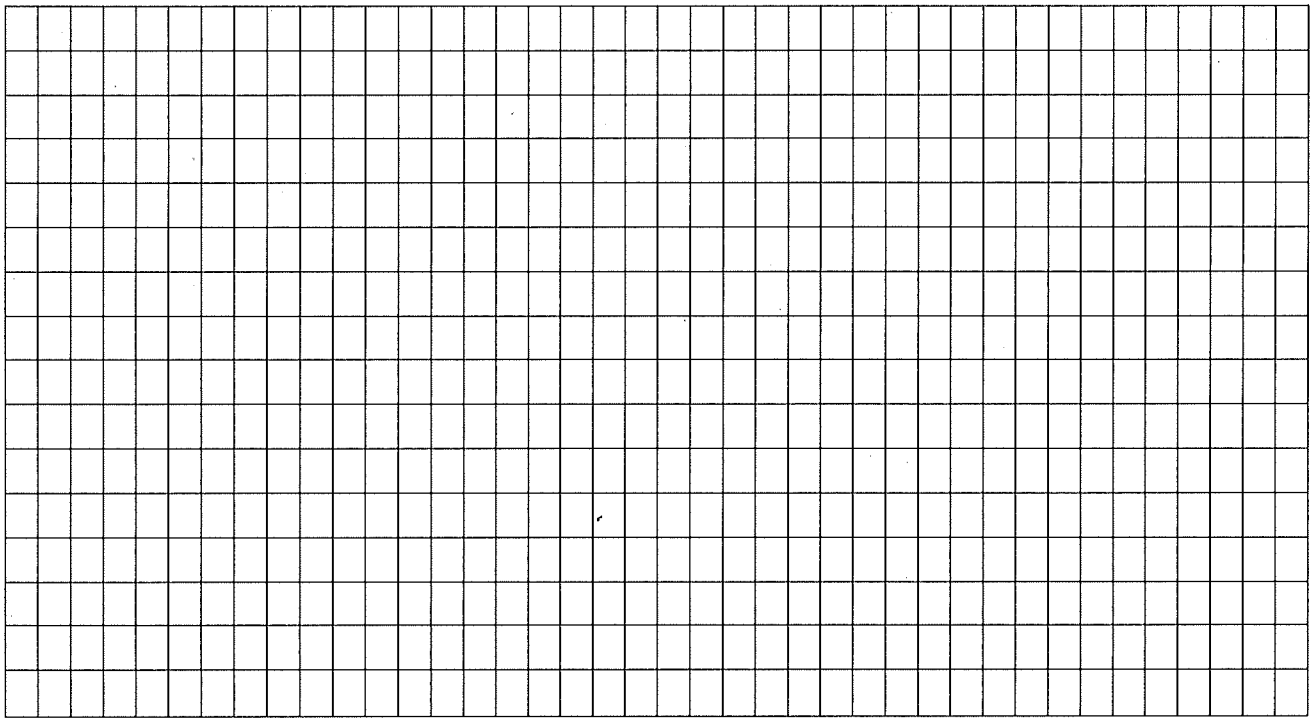
c. Responsibility for costs associated with reimbursement explained? Y / N

d. Relocation package provided and explained to residents? Y / N

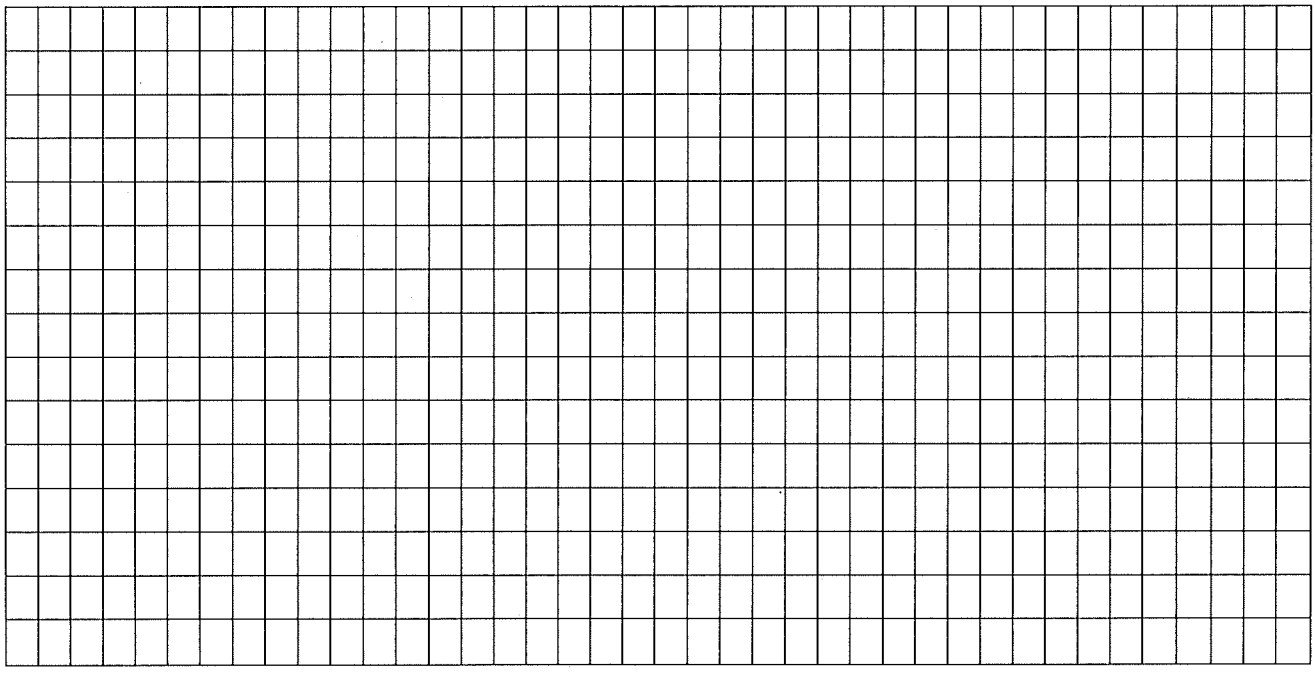
11. FLOOR PLANS

Draw a plan view sketch of the basement and first floor of the building. Indicate air sampling locations, possible indoor air pollution sources and PID meter readings. If the building does not have a basement, please note.

Basement:



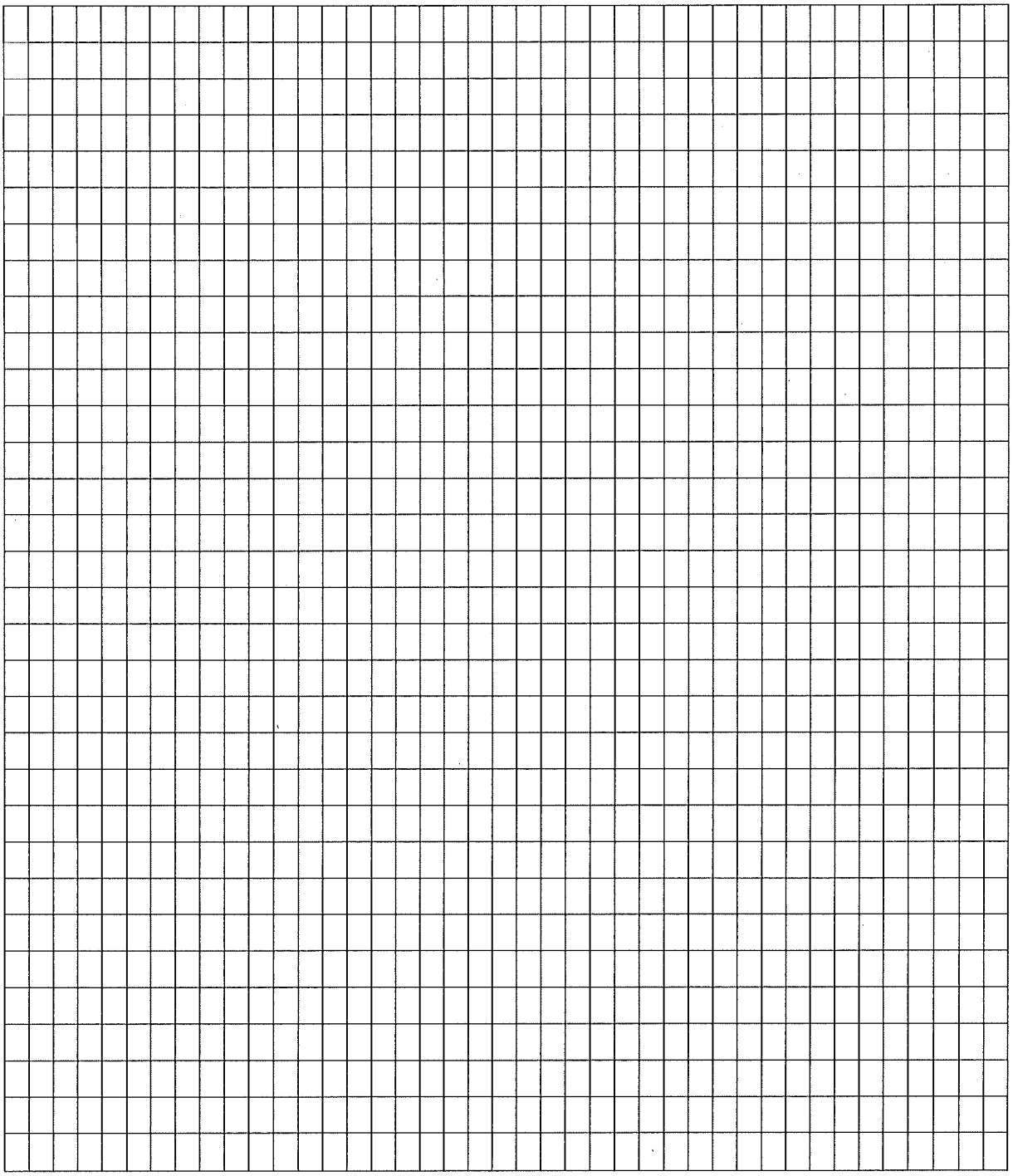
First Floor:



12. OUTDOOR PLOT

Draw a sketch of the area surrounding the building being sampled. If applicable, provide information on spill locations, potential air contamination sources(industries, gas stations, repair shops, landfills, etc.), outdoor air sampling location(s) and PID meter readings.

Also indicate compass direction, wind direction and speed during sampling, the locations of the well and septic system, if applicable, and a qualifying statement to help locate the site on a topographic map.



ATTACHMENT 2: INDOOR AIR SAMPLE COLLECTION FORM

Indoor Air Sampling Form

Sampler: _____ Method: _____ Project #: _____

Sample ID	Date	Time Started	Time Ended	Sampling Height (ft)	Summa Canister ID	Vacuum at start	Vacuum at end	Flow Rate	Sample Type	COC #

Notes:

STANDARD OPERATING PROCEDURE

SEI-6.36.1

PROCEDURE FOR COLLECTION OF SOIL GAS SAMPLES FOR VOC ANALYSIS

SOP Number: SEI-6.36.1
Revision Number: 1.

Date Issued: 06/22/07
Date of Revision: 03/6/2013

1.0 OBJECTIVE

The objective of this standard operating procedure (SOP) is to provide reference for collecting soil gas samples that are representative of Volatile Organic Compounds (VOCs) within the vadose zone. Sampling locations and methodology must be selected to achieve the objectives of the study and the data required.

Health and safety of the sampling personnel is of utmost importance.

2.0 POLICIES

1. According to 40 CFR Part 160, Subpart E, Section 160.81, a testing facility shall have standard operating procedures in writing setting forth study methods that management is satisfied are adequate to insure the quality and integrity of the data generated in the course of a study.
2. Personnel will legibly record data and observations in the field to enable others to reconstruct project events and provide sufficient evidence of activities conducted.

3.0 SAFETY ISSUES

1. A site-specific health and safety plan (HASP) shall be created for the site. A template for creating a proper health and safety plan is provided on the Stone network. If a HASP was created by a company other than Stone, then staff must review and sign off that they have reviewed the site specific HASP.
2. All locations proposed for collecting soil gas samples will be pre-marked and cleared for utilities by Digsafe.
3. Care must always be taken when approaching a sampling location. Do not, under any circumstances, place yourself in danger to collect a sample.

4.0 PROCEDURES

4.1 Equipment and Materials

1. Photoionization detector (PID) equipped with a 10.6 eV bulb to determine risk of exposure to sampling personnel, as required in the HASP and to screen sample locations.

2. .A $CO/CO_2/O_2$ meter.

3. Appropriate personnel protective equipment as specified in the HASP.

4. For Tedlar Bag Method (8260 Analysis):

    a. Certified pre-cleaned, 1.6 liter Tedlar bags complete with influent fitting and extraction septa.

    b. One, 4-liter Pelican® case ("lung box") with barbed fittings to attach to two Tedlar bags and the effluent line to the pump (for splits).

    c. One, low flow air sampling pump capable of pumping rates between 100 and 200 ml/minute. Recommended pumps are SKC 224 Series or Buck VSS Series. Alternatively, a 12-volt Gast pump may be used in conjunction with a flow controller.

    d. If using a Gast pump, one, 12-volt deep cycle battery

5. For Summa Canister Method (TO-15 Analysis):

    a. Certified pre-cleaned, 6-Liter Summa Canisters

    b. Flow controllers calibrated for a 30-minute collection period or as specified within the Site Investigation Workplan.

6. For sub-slab samples:

    a. Sample Probe Installation

        1) Electric Rotary Hammer Drill with $3/8^{th}$ inch OD drill bit

        2) 1/4" Outer diameter fluorinated ethylene propylene (FEP) tubing of sufficient length to reach between the target depth and surface equipment.

        3) Well sand (#1 Silica)

        4) Modeling clay

7. For moderate depth samples using the AMS Soil Gas Probe:

    a. 2" SDS-Max Bosch Combination Hammer Drill

    b. AMS Soil Gas Kit, including:

        1) Drive head

        2) Hollow drive rods

        3) Solid pre-probe rob and solid tip.

        4) Soil gas retracta-tip.

        5) Off-road jack for retracting tooling

        6) Sufficient FEP tubing to extend from target depth to surface and reach sampling canister.

        7) Modeling Clay

8. For Deep Sample Intervals using the Geoprobe Post Run Tubing (PRT) soil gas sampling tool):

    a. 5-foot 1.5" outer diameter direct push rod of sufficient quantity to reach the target depth and allow for redundancy

    b. PRT expendable points

    c. PRT expendable point holder

    d. PRT expendable point holder o-rings

    e. PRT adapter

    f. PRT adapter o-rings

    g. Drive cap for 1.5" direct push rod

    h. Auxiliary rods and point popper

    i. Sufficient FEP tubing to extend from target depth to surface and reach sampling canister.

9. Leak Testing Equipment

    a. Compressed Helium cylinder (small Q size)

    b. Regulator

c. 5 gallon pail equipped with 2 bulk head connectors (one to connect to helium, one to connect to sample line.

d. Helium leak detector

e. Tygon tubing (1/4 inch inner diameter) to plumb between both the regulator and pail and sample port bulk head fitting and He leak detector

10. Field notebook, sample collection form (see Attachment 1), or other acceptable medium for recording field data. Ball point pens.

11. Decontamination equipment

    a. Alconox, scrub brushes and containment pails

    b. Spring Water

    c. Propane torch

    d. Rod brushes suitable for cleaning the inside of the direct push rod.

12. Bentonite chips for backfilling

13. Surface patch to match pre-existing conditions.

4.2 Soil Gas Sample Port Installation

The method selected for installation of the soil gas sample probe will depend on the desired depth and study objectives. Shallow samples may be installed in turf or gravel surfaces using hand tools or through an existing slab using a rotary hammer drill equipped with a 3/8" diameter drill bit. The annular space between the FEP tubing installed within the borehole and surrounding material should be infilled with silica sand. In all circumstances, the sample train is sealed from the atmosphere to prevent short circuiting. Commonly used materials to create this seal include modeling clay or hydrated bentonite.

Permanent soil gas sample ports may be used in instances where routine monitoring of soil gas concentrations require periodic sample collection. Installation of permanent soil gas sample ports fall out of the scope of this SOP.

Installation of deeper soil gas sample ports require the use of either the Geoprobe PRT system or the AMS Soil Gas Probe.

4.2.1 Sample Port Installation using the Geoprobe PRT System

Decontaminate all direct push tooling, point holders and adapters in accordance with the Site Investigation workplan. Equip direct push rod with PRT point holder and sacrificial knockout tip. Advance PRT to desired depth using percussion implement. Make sure drive rod is sufficiently low as to not hinder leak testing.

Set-up soil gas probe according to the following methodology:

1. Connect ¼" FEP tubing to PRT adapter, cap other end using a clamped piece of silicon tubing.
2. Pull back on direct push rod.
3. Seal void between rod and surface material using hydrated bentonite.
4. Insert auxiliary rods with point popper. Push auxiliary rod down to remove expendable tip from point holder. Remove auxiliary rods.
5. Slide PRT adapter and tubing into the direct push rod. While applying down-ward pressure, rotate tubing counterclockwise (reverse thread) to tighten adapter to PRT knock out tip holder.

4.2.2 Sample Port Installation using the AMS Soil Gas Tip

1. Connect ¼" FEP tubing to Retracta-Tip Soil Gas probe.
2. String FEP Tubing through hollow AMS Rod of sufficient length to achieve desired depth.
3. Connect Retracta-Tip to drive rod using vise grips, taking care to avoid damaging the threads.
4. Connect drive head to top of first section of drive rod.
5. Advance probe to approximately 5 inches from ground surface; remove drive head and attach next rod.
6. Repeat Step 5 until desired depth is achieved.
7. Retract drive rod approximately 4 inches to expose sample ports.
8. Seal rod to ground surface using either hydrated bentonite or modeling clay.

4.3 Sample Interval Screening and Sample Train Purge

Upon installation of the soil gas port using any of the aforementioned methods, connect the tubing to the PID. Allow for sufficient time for air within the sample tubing and borehole to be purged based on an estimated flow rate for the PID of 200 mL/min. Continue to purge entrained air until 1 to 3 volumes of the sample train are expunged.

Using $CO/CO_2/O_2$ meter, record CO, CO_2, and O_2 values on the field form.

4.4 Leak Testing

A leak testing procedure will be conducted at each of the sampling locations to demonstrate that the sampling system does not allow for short circuiting to occur between the ambient air and the sampling train.

The leak test procedure includes:

1) Turn on helium detector to allow the unit to warm up.

2) Purge Sample Train by connecting PID to sample side of the leak testing pail using Tygon tubing.

    1. Allow unit to draw sample train air for 4 minutes.

    2. Record peak value on field form.

3) Measure $CO/CO_2/O_2$ with $CO/CO_2/O_2$ meter and record value on field form.

4) Connect helium detector to the sample side of leak testing bucker

5) Connect the sample train tubing to the sample side of the leak test pail

6) Connect helium regulator to open port of leak test pail

7) Allow helium detector to purge sample train of ambient air (5 minutes)

8) Turn on helium until gas is heard escaping from the bottom of the pail

9) Check for leaks in sample train, as indicated by positive readings on the helium detector.

If no helium is present it can be concluded that no short circuiting has occurred. If helium is found in the sample train, the sample train should be checked for loose fittings and the modeling clay seal inspected for leaks. The procedure should be repeated until it is demonstrated that no short circuiting

has occurred. If needed, the soil gas borehole can be re-drilled; however, to avoid short circuiting of ambient air, the prior borehole should be filled with hydrated bentonite.

Following successful leak testing, plug sample line with a knotted piece of silicon tubing to prevent loss of soil gas contaminant mass.

4.5 Sample Collection - Tedlar Bag Method

The Following sections detail the methodology used when collecting soil gas samples in to 1 liter Tedlar bags.

4.5.1 Calculate Flow Rate

To calculate flow rate for the pump, affix a calibrated bubble-flow meter to the effluent (positive) side of the pump and turn on the pump. Using a stopwatch, record the time in seconds required for a bubble to reach the 100 mL mark on the bubble flow meter. Divide this time by 100, and take the inverse. This will result in the pump flow rate in milliliters per second.

Using the calculated flow rate, determine the time needed to fill the 1.6 Liter Tedlar bags.

It is important to continuously check the flow rate during each of the sampling events; although the pump may be calibrated to a certain flow rate, the variable resistance (depending on soil types) can cause a change in the actual flow rate. The pumps can be adjusted during the sampling event to provide the desired flow rate.

4.5.2 Sample Collection Procedure

Following successful leak testing and flow setting, disconnect the sample tubing from the leak testing apparatus and connect it to the Vacuum Box. Install a new Tedlar bag and open the bag valve. Close box and begin collecting sample. Record start time on field logs.

Periodically inspect the bag through the window in the top of the vacuum box. Once full, stop pump, record final time, and close valve on Tedlar bag. Label bag and store in a cooler and transport to onsite laboratory as soon as possible. Disconnect vacuum box and repeat at next location.

4.6 Sample Collection Procedure – Summa canisters

Following successful leak testing and flow setting, to collect soil gas samples using Summa canisters, connect the sample train to the Summa canister using swage fittings. Open canister and record initial vacuum on to the field form. Following the sample collection period, shut the canister and record the final vacuum. If no vacuum remains (gauge reads zero) the flow controller is faulty and sample collection period is uncertain; the sample should be abandoned and a new sample collected. If, following the collection period the vacuum reading remains high (above 20 inches of Hg vacuum)

either the sub slab conditions restrict soil gas flow or the flow controller is faulty. In either instance, field staff should record the observation and continue to collect the sample until the vacuum gauge reads less than 10 inches of Hg.

Following successful sample collection, observations are recorded in to field forms, samples are packaged, and the chain of custody form is completed.

4.7 Retrieving Quality Control Samples

Duplicate samples for either split confirmation analyses or field duplicates will be collected using two Tedlar bags or Summa canisters filled simultaneously using "T" fittings.

Field blanks to determine background contamination levels are collected through all sampling apparatus at the ground surface.

4.8 Sample Packing and Transportation

Pack samples in an insulated cooler or other sturdy container with refrigerant, if required. Samples for onsite analyses should be sent to the onsite laboratory as soon as possible. Samples for offsite analyses should be sent to the laboratory as soon as is reasonable, but within the maximum holding time for the sample. Chain of custody forms will be filled out and signed prior to shipment to the laboratory and included in the shipping container with the samples, according to standard operating procedures (SEI-4.2.n) and/or protocol and work plan instructions.

5.0 RESPONSIBILITIES

1. All personnel will legibly record data and observations (including phone conversations) in accordance with this SOP to enable others to reconstruct project events and provide sufficient evidence of activities conducted.

6.0 DEFINITIONS

1. *Soil Gas* – Interstitial air within the vadose zone.
2. *Decontamination* – Procedures followed to ensure cross contamination does not occur between sampling points or that potential contamination of equipment does not pose a hazard to sampling personnel.
3. *EPA* the U.S. Environmental Protection Agency.
4. *Maintenance* – Actions performed on equipment to standardize and/or correct the accuracy and precision of a piece of equipment to ensure that the equipment is operating within the manufacturer's specifications and standard values.

7.0 REFERENCES

40 CFR Part 160 Good Laboratory Practice Standards, August, 1989.

8.0 TABLES, DIAGRAMS, FLOWCHARTS, AND VALIDATION DATA

Attachment 1: Soil Gas Sample Collection Form

Attachment 2: Soil gas sampling – PRT system operation (from Geoprobe Systems®)

9.0 AUTHORIZATION

Revised by:_____ Date: 3/7/13

Daniel Voisin, Project Scientist

Approved by:_____ Date: 3-7-13

Christopher T. Stone, President, Management

10.0 REVISION HISTORY

Revision 1

1. Leak test procedure added.
2. Included sample collection methodology and equipment/materials when using Summa Canisters
3. Included sample collection methodology and equipment/materials when using AMS Soil Gas Probe
4. Included sample collection methodology using temporary soil gas probe.
5. Changed title.
6. Reorganized document.
7. Minor edits to text describing retrieval of quality control samples.
8. Included procedure for sample interval screening and sample train purge. Added the use of a $CO/CO_2/O_2$ meter to screening procedure.

ATTACHMENT 1: SOIL GAS SAMPLE COLLECTION FORM

Soil Gas Sample Collection Record

Soil Gas Point ID: _____

Project Name: _____ Project #:_____

Site Location: _____ Date: _____

Weather Conditions: _____

Rain within last 24 hours? (y/n): _____Amount: _____

Sampler(s): _____

1. SAMPLE LOCATIONS AND COLLECTION METHODOLOGY INFORMATION

Description of soil gas point location: _____

Soil Gas point installation method: _____

Sample depths (ft): _____

Sample collection method: _____

2. SAMPLE INFORMATION:

PID Reading: _____(ppm v/v) CO Reading: _____

CO_2 Reading: _____ O_2 Reading: _____

Sample Name	Sample Time	Laboratory	Chain of Custody #	Container Type #	Collection Period	Flow Rate (mL/min)	Analysis Required

Comments:

_____

_____

_____

_____

_____

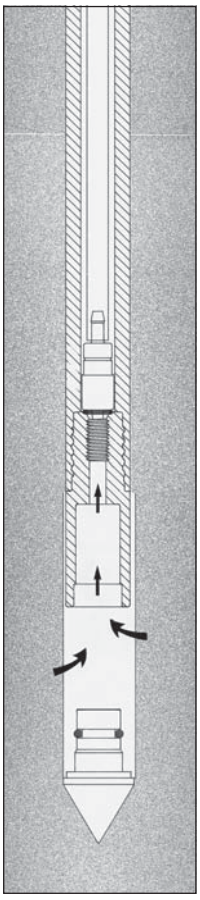
Signature: _____ Date: _____

ATTACHMENT 2: SOIL GAS SAMPLING – PRT SYSTEM OPERATION (FROM GEOPROBE SYSTEMS®)

Soil Gas Sampling – PRT System Operation

from Geoprobe Systems®

www.geoprobe.com
1-800-436-7762



Soil Gas Sampling using the Post-Run Tubing (PRT) System.

Soil Gas Sampling — PRT System Operation

Basics

Using the Post-Run Tubing System, one can drive probe rods to the desired sampling depth, then insert and seal an internal tubing for soil gas sampling. The usual Geoprobe probe rods and driving accessories and the following tools are required:

- PRT Expendable Point Holder
- PRT Adapter
- Selected PRT Tubing

Preparation

1. Clean all parts prior to use. Install O-rings on the PRT Expendable Point Holder and the PRT adapter.

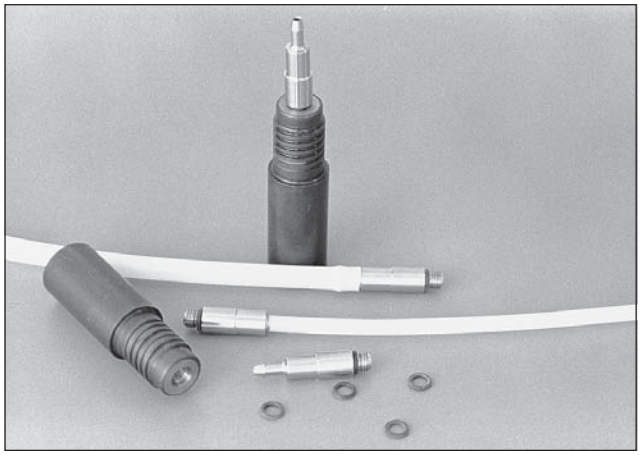
2. Inspect the probe rods and clear them of all obstructions.

3. TEST FIT the adapter with the PRT fitting on the expendable point holder to assure that the threads are compatible and fit together smoothly.

   NOTE: PRT fittings are left-hand threaded.

4. Push the adapter into the end of the selected tubing. Tape may be used on the outside of the adapter and tubing to prevent the tubing from spinning freely around the adapter during connection – especially when using Teflon tubing **(Figure 1)**.

   REMEMBER: The sample will not contact the outside of the tubing or adapter.



PRT SYSTEM PARTS
PRT Expendable Point Holder, PRT Adapters, Tubing, and O-rings.

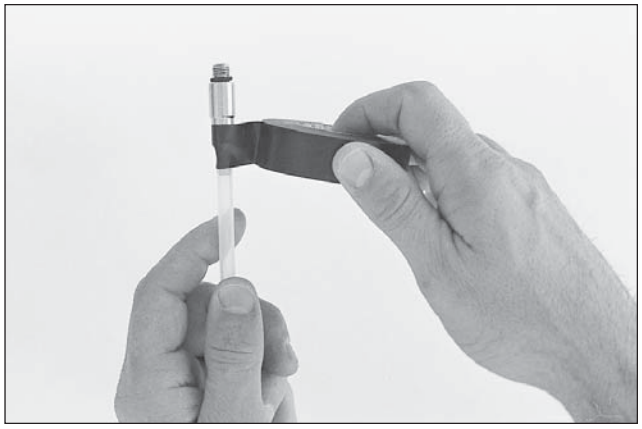


Figure 1. Securing adapter to tubing with tape. NOTE: Tape does not contact soil gas sample.

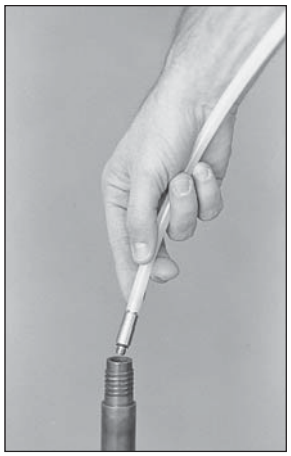


Figure 2. Insertion of tubing and PRT adapter.

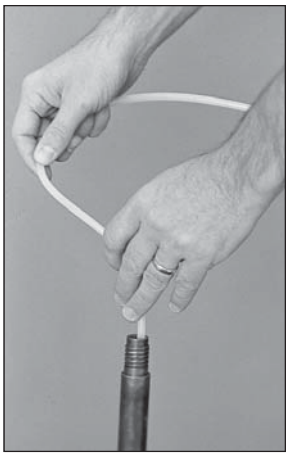
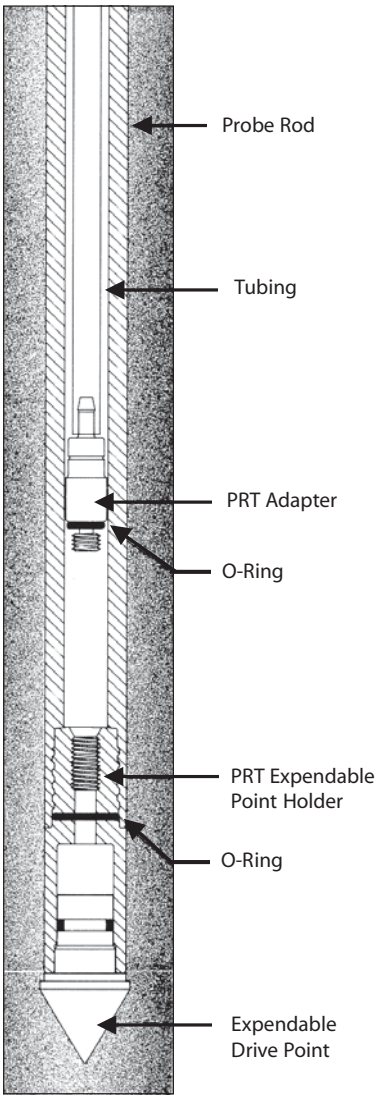


Figure 3. Engaging threads by rotating tubing.



A cross section of probe rods driven to depth and then retracted to allow for soil gas sampling. The PRT adapter and tubing are now fed through the rods and rotated to form a vacuum-tight connection at the point holder. The result is a continuous run of tubing from the sample level to the surface.

Probing

Drive the PRT tip configuration into the ground. Connect probe rods as necessary to reach the desired depth. After depth has been reached, disengage the expendable point by pulling up on the probe rods. Remove the pull cap from the top probe rod, and position the Geoprobe unit to allow room to work.

Connection

1. Insert the adapter end of the tubing down the inside diameter of the probe rods **(Figure 2)**.

2. Feed the tubing down the rod bore until it hits bottom on the expendable point holder. Allow about 2 ft. (610 mm) of tubing to extend out of the hole before cutting it.

3. Grasp the excess tubing and apply some downward pressure while turning it in a counterclockwise motion to engage the adapter threads with the expendable point holder **(Figure 3)**.

4. Pull up lightly on the tubing to test engagement of the threads. (Failure of adapter to thread could mean that intrusion of soil may have occurred during driving of probe rods or disengagement of drive point.)

Sampling

1. Connect the outer end of the tubing to the Silicone Tubing Adapter and vacuum hose (or other sampling apparatus).

2. Follow the appropriate sampling procedure for collecting a soil gas sample **(Figure 1)**.

Removal

1. After collecting a sample, disconnect the tubing from the vacuum hose or sampling system.

2. Pull up firmly on the tubing until it releases from the adapter at the bottom of the hole. (Taped tubing requires a stronger pull.)

3. Remove the tubing from the probe rods. Dispose of polyethylene tubing or decontaminate Teflon tubing as protocol dictates.

4. Retrieve the probe rods from the ground and recover the expendable point holder with the attached PRT adapter.

5. Inspect the O-ring at the base of the PRT adapter to verify that proper sealing was achieved during sampling. The O-ring should be compressed. This seal can be tested by capping the open end of the point holder applying vacuum to the PRT adapter.

6. Prepare for the next sample.



Figure 1. Taking a soil gas sample for direct injection into a GC with the PRT system.

STANDARD OPERATING PROCEDURE

SEI-5.29.0

USE, MAINTENANCE AND CALIBRATION OF THE MultiRAE IR MULTI-GAS MONITOR, PGM-54

SOP Number: SEI-5.29.0
Revision Number: NA

Date Issued: 02/04/2011
Date of Revision: NA

1.0 OBJECTIVE

The MultiRAE IR is a programmable multi-gas monitor designed to provide continuous exposure monitoring of carbon monoxide, toxic gases, oxygen and combustible gases for workers in potentially hazardous environments. The MultiRAE IR Multi-Gas Monitor may be worn on a belt to allow for hands-free use. The MultiRAE IR gives real time measurements and activates alarm signals whenever the exposure exceeds preset limits. Prior to factory shipment, the MultiRAE IR is preset with default alarm limits and the sensors are pre-calibrated with standard calibration gas. However, the user should calibrate the instrument before the first use. After the monitor is fully charged and calibrated, it is ready for immediate operation.

2.0 POLICIES

1. The MultiRAE Multi-Gas Monitor should be calibrated each day before use. Maintenance, corrective actions, and calibration should be noted in the "Maintenance and Calibration Log".
2. Personnel will legibly record data and observations in the field to enable others to reconstruct project events and provide sufficient evidence of activities conducted.
3. Field personnel will use a field logbook, observation and remark form (O&R) or other designated field form to record activities, measurements, and observations made in the field.
4. Any change in entries shall be made so as not to obscure the original entry. The recorder will initial corrections.
5. Field personnel will read this SOP prior to operating the Multi-Gas Monitor.

3.0 SAFETY ISSUES

1. Whenever visiting a hazardous or potentially hazardous site, it is important to read, follow and sign the site health and safety protocol (HASP).

2. By itself, use of the MultiRAE IR Multi-Gas Monitor poses no risk to human health, however, if elevated levels of hazardous gases are detected by the Multi-Gas Monitor, it may be necessary to don personnel protective equipment or vacate the area as described in the site's HASP.

3. Field visits may involve accessing remote areas. Health and safety concerns regarding these field visits are minimal however; slip, trip and fall hazards, dangerous flora and fauna, as well as getting vehicles stuck in remote areas do present considerable health and safety issues. To help ensure field staff's health and safety with remote areas, all field staff is to have daily communication with the project manager or another appropriate Stone Environmental, Inc. (SEI) employee.

4.0 PROCEDURES

4.1 Introduction

The MultiRAE IR combines a PID (Photoionization Detector) and Carbon Dioxide (CO2) sensor with O2, LEL, and one specific toxic gas sensor in one compact monitor with sampling pump. Although there are numerous types of PIDs available, all PIDs are based on the following principle:

> A sampling pump continuously draws air sample into the ionization chamber. The ionization chamber is usually constructed as a small cavity in front of the UV lamp. The electrode is made of a wire mesh so that the high energy UV light can shine through the mesh and excite the gas molecules when they pass across the face of the UV window. The ions collected on the electrode are measured by an electrometer.
>
> A single chip microcomputer is used to control the operation of the lamp, alarm buzzer, LED, sampling pump, power supply and electrometer. It measures the electrometer readings and calculates the gas concentrations based on calibration to a known reference gas. An LCD display is used to show the readings. The user interacts with the unit though three keys (one operation key and two programming keys) on the front panel.
>
> A rechargeable lithium-ion battery pack powers the unit. A built-in charging unit allows the batteries to be charged directly from an AC power source with the included power connection.

The MultiRAE IR Multi-Gas Monitor also contains sensors to monitor Carbon Dioxide, Toxic Gas (VOCs), Oxygen, and Combustible Gas.

4.2 General Considerations

The MultiRAE IR is adversely affected by both damp and windy conditions, as well as temperature. Ensure that the MultiRAE IR is used with its probe and a clean sample filter at all times. Replace the filter frequently. Refer to the operating manual to determine the lowest allowable operating temperature and humidity.

The MultiRAE IR Multi-Gas Monitor has 3 operating modes: Text, Display, and Programming. In "programming" mode, which is the default setting, the user can perform all the functions of the other modes. In addition, the user can get into calibration mode, perform calibrations, and change any of the parameters in

the calibration mode. For more of an explanation about the "text" and "display" modes, see the Operation Manual.

4.2.1 Equipment

- MultiRAE IR Multi-Gas Monitor Operation and Maintenance Manual
- Calibration gas and regulator
- Tedlar bag for calibration gas collection
- Poly tubing for connection tedlar bag to regulator and monitor
- Extension wand and tubing
- Serial port computer connection cable
- Spare sample filters
- Phillips head and flathead screwdriver
- Maintenance and Calibration Log

4.2.2 Operation-Turning the MultiRAE IR Multi-Gas Monitor ON and OFF

The MultiRAE IR Multi-Gas Monitor has only three controls: two programming keys and one operation key. To **TURN ON** the MultiRAE IR Multi-Gas Monitor, press the [MODE] key. The audio buzzer will beep once and the display will show "ON!" and then "Multigas Monitor Ver n.nn" to indicate the software version number. The monitor will then go through each sensor socket to check if a valid sensor is installed. Next, the monitor displays the preset alarm limits for each sensor, battery voltage, shut off voltage, user mode, alarm mode, datalog mode available data storage memory (in hours), datalog mode and datalog interval (in seconds). Then after a set delay time of several seconds, the display shows the instantaneous reading of the gas concentrations and is ready to monitor gases.

To **TURN OFF** the MultiRAE IR Multi-Gas Monitor, press and hold the [MODE] key for 5 seconds. Monitor will beep once per second during power-down sequence with a countdown timer showing the number of remaining seconds. The message "Off!" flashes on the LCD display and the display will go blank, indicating that the monitor is turned off.

Turning off the MultiRAE IR Multi-Gas Monitor with the AC charger in place will result in a display of "Charging.." or "Battery Charged" and indication of battery voltage. This indicates that the monitor is off, but the charger circuit is now active.

- *Programming Mode*

The three keys perform a different set of functions during the programming mode as summarized below:

[MODE]	Exit Menu when pressed momentarily or exit data entry mode when pressed and held for 1 second.
[Y/+]	Increase numerical value for data entry or conform (yes) for a question.
[N/-]	Decrease numerical value for data entry or deny (no) for a question.

To enter programming mode (which is the default mode), press and hold down both [MODE] and [N/-] keys for three seconds. At this point, the display should show "Enter Password = 0000". Use the programming keys to enter the password "0000", then hold [MODE] for one second. Refer to section 4.3 in the manual for help with this step.

4.2.3 Calibration

For maximum safety, the accuracy of the MultiRAE IR Multi-Gas Monitor should be calibrated before each day's use by exposing the sensors to a known concentration calibration gas. Refer to section 4.4 in the Operation Manual for a more thorough explanation.

In programming mode (the default mode), the user may calibrate the sensors in the MultiRAE IR Multi-Gas Monitor. This is a two-point calibration process using "fresh air" and a standard reference gas. First, "fresh air" containing 20.9% oxygen and no detectable VOC, toxic or combustible gases is used to set the zero point for each sensor. Then a standard reference gas, containing a known concentration of a given gas, is used to set the second point of reference (also known as span gas). The two-point calibration procedure is described below.

NOTE: "Fresh Air" calibration zeros only the LEL, VOC, and toxic gas sensors, and spans the O_2 sensor. The CO_2 sensor is spanned and zeroed in two sub-menus of the Single Sensor calibration. For better accuracy, zero calibration should be performed before span calibration of all sensors. The following table shows the structure of the calibration sub-menu:

Calibration Sub-menu
Fresh Air Calibration?
Multiple Sensor Calibration?
Single Sensor Calibration?
Modify Span Gas Value?
Change LEL/VOC Span Gas?

To perform a "fresh air" calibration, move to a clean air environment and follow the steps below. If unsure of the ambient air's purity, a charcoal filter should be used.

- *Fresh Air Calibration (Zeroing the sensors)*

To enter Calibration mode, hold down the [MODE] and [N/-] key for three seconds.

1. "Calibrate Monitor?" Press [Y/+] to perform calibration. The first sub-menu shows: "Fresh Air Calibration?"
2. Leave the monitor in an area free of any detectable vapor.

3. Press the [Y/+] key to start "fresh air calibration" of the monitor. The display shows "zero... in progress" followed by the name of each sensor and the message "zeroed". The display should show a reading of "20.9" for the oxygen sensor and "0.0", or a very small number, for all other sensors.
4. After about a five second pause, the display will show the message "Zero Cal Done!" and move to the next sub-menu "Multiple Sensor Calibration?"

- *Multi Sensor Calibration*

This function simultaneously determines the second point of calibration curves for multiple sensors in the monitor. Mixed standard reference gases are needed to perform this procedure (and should be included in the case). The standard reference gas needs to be transferred into a tedlar sample bag. To do this, ensure the regulator is in the off position (righty-tighty!) and screw onto the standard gas cylinder. Use a short section of poly tubing to connect the regulator to the tedlar bag. Open the valve on the tedlar bag, then slowly open the valve on the regulator, allowing the tedlar bag to fill with the standard gas. Once the tedlar bag is full (tedlar bag should NOT be taught), turn off the valve on the regulator. Next, tighten the valve on the tedlar bag. Finally remove poly tubing from either end.

Now, attach the MultiRAE IR Multi-Gas Monitor to the tedlar sample bag containing the standard gas. Continuing from Step 4 above, the display shows "Multiple Sensor Calibration?".

5. Press [Y/+]. The display shows all the preselected gases for calibration and an "OK?" question. Press [Y/+] to accept the sensor selections and start the calibration, or [N/-] to modify the sensor selection (see manual section 4.4.2 for further instructions).
6. Turn on the valve of the tedlar bag to start the flow of the "span" gas (standard gas). Display shows "Apply Mixed Gas" and will wait for the calibration gas to reach the sensor. When the calibration gas has reached the sensor the display will show "calibration in progress... 75" with the countdown timer showing the number of remaining seconds while the monitor performs the calibration. When the timer reaches zero (0), the display shows the name of each sensor, the message "cal'ed" and the calibrated values for each gas. If no gas has reached the sensor after 75 seconds, the display will show "No Gas flow..." and abort the calibration. The readings should be close to the span gas values (These values are printed on the label of the calibration gas and are reprinted below). After about 5 seconds pause, the display will show the message "Span Cal Done! Turn Off Gas".
7. Turn off the flow of gas. Disconnect the calibration adapter from the MultiRAE IR Multi-Gas Monitor. If all sensors calibrated correctly, the MultiRAE IR Multi-Gas Monitor is ready to use.
8. If the sensor failed calibration, the sensor name and an error message "failed, continue?" will appear and the failed sensor may be calibrated individually. Press [N/-] or [MODE] to abort the calibration and move to the next sub-menu. Press [Y/+] to continue to the next sensor calibration. In either case, the calibration data

for the current sensor are not changed. For further information about calibrating an individual sensor, refer to Step 6 in section 4.4.2 of the Operation Manual.

Calibration Gas (aka "Span Gas") components and concentrations (there are other types, so make sure via the label that these are what you have) are listed in the table below:

Gas	Concentration
Carbon Monoxide (CO)	50 ppm
Carbon Dioxide (CO_2)	5,000 ppm
Methane (CH_4)	2.5% (50% LEL)
Oxygen (O_2)	20.9%
Nitrogen (N_2)	BALANCE

4.2.4 Changing Alarm Limits

Alarm limits for the MultiRAE IR Multi-Gas Monitor can be modified. These should be left at the default settings unless the user has special needs. If these limits need to be modified, refer to section 4.5 in the Operations Manual.

4.2.5 Data-logging

The MultiRAE IR Multi-Gas Monitor has the ability to calculate and store the gas readings as specified intervals. The user can review these stored readings or change datalog setup from the programming mode. Users can also program additional datalog options in PC and down load them to the monitor. See sections 4.6 and 5.0 in the Operation Manual for details on programming data-logging options.

4.2.6 Maintenance

The MultiRAE IR Multi-Gas Monitor requires minimal maintenance besides battery charging and calibration. If any sensor, PID Lamp, or other errors occur, refer to the Operation Manual. A trouble-shooting guide is included in the Operation Manual in section 7.2.

Detailed maintenance must be performed by a qualified technician and usually requires the shipping of the MultiRAE IR Multi-Gas Monitor to the manufacturer. Stone typically sends the MultiRAE IR Multi-Gas Monitor to the manufacturer annually for lamp cleaning and general maintenance – or more frequently as needed. Call the manufacturer for shipping instructions prior to sending the MultiRAE IR Multi-Gas Monitor.

4.2.7 Documentation

A record of calibrations and any maintenance performed on the PID must be made in the Maintenance and Calibration log.

If used in the field to screen soil samples, PID values are recorded onto soil boring logs (see attached). If screening is being conducted of miscellaneous soil samples, such as those from surface samples, values should be recorded on "Observations and Remarks (O&R)" forms.

4.3 Collection of Soil Samples for Headspace Screening

Fill a clean jar or baggie approximately half full using the stainless steel spoon with a "consistent amount" of soil. Take special care to ensure that sample and container are not cross-contaminated from previous sample by rinsing spoon between samples with distilled water. It is also important to ensure that the sample is not frozen prior to analysis.

Seal top of jar with two layers of aluminum foil, or insure that bagged is properly sealed. Secure aluminum foil with either a rubber band or screw lid that fits jar. If using screw lid, do not include solid top with rubber gasket included with new mason jars.

Allow VOC concentrations to develop in the headspace of the sample container for five minutes. During this headspace development period, the sample should be shaken vigorously for a total of one minute.

Immediately pierce the aluminum foil seal (or un-zip an approximate one-inch section of baggie) with the probe of PID and note the highest (peak) reading, and sustained reading. Take care not to submerge PID tip into soil.

Record time of sample collection and peak value as ppm onto appropriate form (Soil Boring Log or O&R).

4.4 Documentation

A record of the soil screening must be made in the field at the time the peak sample headspace concentration is read from the PID. It may also be helpful to record the weather or any site-specific conditions such as excessive heat or cold that may affect the readings from the PID. Be sure that field notes are properly labeled to the project number and date.

5.0 RESPONSIBILITIES

1. SEI personnel must calibrate the MultiRAE IR Multi-Gas Monitor prior to use. Calibrations should be recorded in the Maintenance and Calibration Log.
2. It is the responsibility of SEI field staff to make sure that the MultiRAE IR Multi-Gas Monitor is in proper working order prior to bringing it onto a site.
3. SEI personnel must read this SOP prior to using the MultiRAE IR Multi-Gas Monitor. After reading the SOP, personnel should update their personnel training folder.

6.0 DEFINITIONS

1. *EPA* the U.S. Environmental Protection Agency.

2. *Observations & Remarks Form (O&R):* A pre-printed form, which contains mostly blank space for general note taking. The form typically prompts the user for the study or project designation, the SEI project number, the client or sponsor name, the total number of pages (page *n* of n) and requires a signature and date. The form is generally used to capture notes of one person when another, more specific forms is not available.
3. *PID*: Photoionization Detector
4. *PPM*: Parts per million.
5. *Quality Assurance Unit (QAU)* any person or organizational element except the study director, designated by testing facility management to perform the duties related to quality assurance of the studies.
6. *Raw data* any laboratory worksheets, records, memoranda, notes, or exact copies thereof, that are the result of original observations and activities of a study and are necessary for the reconstruction and evaluation of the report of that study. In the event that exact transcripts of raw data have been prepared (e.g., tapes which have been transcribed verbatim, dated, and verified accurate by signature), the exact copy or exact transcript may be substituted for the original source as raw data. Raw data may include photographs, microfilm or microfiche copies, computer printouts, magnetic media, including dictated observations, and recorded data from automated instruments.

7.0 REFERENCES

1. Vermont State Agency of Natural Resources, Waste Management Division "Agency Guidelines for Petroleum Contaminated Soils and Debris" August 1996.
2. MultiRAE IR Multi-Gas Monitor, PGM-54, Operation and Maintenance Manual, Rev. B. December, 2001

8.0 TABLES, DIAGRAMS, FLOWCHARTS, AND VALIDATION DATA

None

9.0 AUTHORIZATION

Revised by: _____ Date: 2/5/10

Willapa J. Waterstrat, Project Scientist

Approved by: _____ Date: 2-4-2011

Christopher T. Stone, President

10.0 REVISION HISTORY

1. Not applicable.

C: LABORATORY STANDARD OPERATING PROCEDURES

Title: Particle Size Analysis (ASTM D 2217 and D422-63)

Approval Signatures:

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SOP REVIEW FORM

SOP Number	Revision	Effective Date:	Title
GR-GT-006	5	03/17/10	Particle Size Analysis

Review Statement:

My signature signifies that I reviewed and compared the above referenced SOP against current bench practice.

			Revision Needed	
Date	Reviewer	Job Title	Yes[1]	No
04/03/12	Christopher Calloway	Dept Manager		X
04/03/12	Eric Lee	Analyst		X

[1] *List the section for revision and the the reason using the revision summary page and attach to this cover sheet.*

QA Use Only:

X The SOP was reviewed and does not require revision. Attach this form to the SOP.

____ The SOP Revision will be made with a Change in Progress Attachment (CIPA).

____ The SOP Revision will be released as a new version of the SOP.

____ The SOP Revision requires method validation or demonstration of capability

____ The SOP Revision does not reqire method validation or demonatration of capability.

____ The SOP revison affects other SOPs that must now also be revised (List SOPs)

Kristin Daigle
QA Signature

4/3/2012
Date

Comments:

1.0 Scope and Application

This SOP describes the laboratory procedure for the determination of particle size distribution in soils.

2.0 Summary of Method

A portion of sample is soaked in a dispersing agent then partitioned into separate portions, material retained on a #10 sieve and material passing the #10 sieve. The material retained on the #10 sieve is dried to constant weight then passed through a large size sieve stack; the material retained on each sieve is measured and recorded. Material passing the #10 sieve is subject to hydrometer analysis then passed through a small size sieve stack, the material retained on each sieve is measured and recorded. All measurements, large and small sieves and hydrometer readings and the hygroscopic moisture are used to establish the particle size distribution of the sample.

This SOP is based on the following reference methods:

- ASTM Standard D 2217 – 85 (Rapproved 1998) "Standard Practice for Wet Preparation of Soil Samples for Particle-Size Analysis and Determination of Soil Constants", ASTM International, West Conshohocken, PA 2003, DOI: 10.1520/C0033-03, www.astm.org

- ASTM Standard D 422-63 (Rapproved 2007) "Standard Test Method for Particle-Size Analysis of Soils", ASTM International, West Conshohocken, PA 2003, DOI: 10.1520/C0033-03, www.astm.org

NOTE: ASTM D2217 was method was withdrawn without replacement by ASTM in 2007. A withdrawn standard is an ASTM standard that has been discontinued by the ASTM Sponsoring Committee responsible for the standard.

If the laboratory has modified the procedure from the reference method(s) a list of modifications will be provided in Section 16.0.

3.0 Definitions

Not Applicable

4.0 Interferences

Not Applicable

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1 Specific Safety Concerns or Requirements

None

5.2 Primary Materials Used

Not Applicable

6.0 Equipment and Supplies

Catalog numbers listed in this SOP are subject to change at the discretion of the vendor. Analysts are cautioned to be sure equipment used meets the specification of this SOP.

- Top-Loading Balance, capable of weight measurement to 0.01 g
- Mechanical Stirring Device and Dispersion Cup
- Thermometer: Accurate to 0.5°C
- Mortar and Rubber Tipped Pestle
- Sedimentation Cylinder(s) 1000 mL
- Hydrometer: ASTM 151H in specification E 100.
- Sieves, of the following size(s): Gilson Company, Inc. or equivalent
  3.0” (75.00 mm)
  2.0” (50.00 mm)
  1.5” (37.50 mm)
  1.0” (25.00 mm)
  3/4" (19.00 mm)
  3/8” (9.50 mm)
  # 4 (4.75 mm)
  #10 (2.00 mm)
  #20 (850.0 um)
  #40 (425 um)
  #60 (250.0 um)
  #80 (180.0 um)
  #100 (150.0 um)
  #200 (75.0 um)
- Drying Oven with temperature range of 60-110°C
- Stainless Steel Spatulas & Spoons
- Metal & Bristle Brushes
- Ro-Tap Sieve Shaker, W. S. Tyler or equivalent.
- Timing Device with second hand and capable of counting up to 25 hours

7.0 Reagents and Standards

- Reverse Osmosis (RO) water: In-House System
- Sodium Hexametaphosphate: ELE International or equivalent.

Sodium Hexametaphosphate Solution: Add 120 g of sodium hexametaphosphate and 2940 g of reagent water to a 1-gallon bottle. Add a stir rod to the container and place on a stir plate. Mix the solution until it is homogeneous. Assign an expiration date of 30 days from the date made

unless the parent reagent expires sooner in which case use the earliest expiration date. Store the prepared solution at ambient temperature.

8.0 Sample Collection, Preservation, Shipment and Storage

The laboratory does not perform sample collection so these procedures are not included in this SOP. Sampling requirements may be found in the published reference method.

Listed below are minimum sample size, preservation and holding time requirements:

Matrix	Sample Container	Minimum Sample Size	Preservation	Holding Time	Reference
Solid	Glass Jar w/ Teflon Lid	500 g	None	None	ASTM D422-63

Unless otherwise specified by client or regulatory program, after analysis, samples and extracts are retained for a minimum of 30 days after provision of the project report and then disposed of in accordance with applicable regulations.

9.0 Quality Control

Not Applicable

10.0 Procedure

10.1 Equipment Calibration

Check the calibration of the balance on each day of use prior to use using at least 2 Class S weights that bracket the range of use. Record in the logbook designated for this purpose.

Check the temperature of the drying oven(s) each day of use, prior to use. Record in the logbook designated for this purpose.

NOTE: The QA Manager or her designee checks the calibration of liquid in glass thermometers annually against a NIST-traceable thermometer following the procedures given in laboratory SOP BR-QA-004. Electronic / digital thermometers that are battery-operated are checked quarterly using the same procedure.

Calibrate the hydrometers every two years following the procedure given in BR-GT-008.

Calibrate the sieves 6 months following the procedure given in BR-GT-008.

Calibrate the Ro-Tap sieve shaker every 12 months following the procedure given in BR-GT-008.

10.2 Hygroscopic Moisture Determination

Label an aluminum pan with the Lab ID for each sample. Tare the balance, weigh each pan and record the weight measurement in the spreadsheet.

Mix the sample with a stainless steel spatula. Measure at least 10-15 g of each sample into the labeled aluminum pan and record the weight of sample in the spreadsheet.

Place the pan + sample in an oven maintained at a temperature of 110ºC and dry the sample for at least 16 hours. Reweigh each pan and record the weight measurement in the spreadsheet.

Percent solids are calculated using the equation given in Section 11.0.

10.3 Sample Preparation

Use the calculated percent solids and the sample characteristic for each sample to determine the amount needed for analysis using Table 2. For example, if the calculated percent solids for a sample are 50% and the sample characteristic is sand, use 200 g for analysis. If there is an insufficient amount of sample available, initiate a nonconformance memo (NCM) and contact the PM for further instruction.

Place a 1000 mL plastic beaker on the balance and tare the balance. Weight the amount of sample for analysis and record the weight in the bench sheet.

Add 125 mL of sodium hexametaphosphate solution to each beaker. Stir to mix and soak the sample in this solution for 16 hours

10.4 Sample Partition

Rinse the sample slurry into a dispersion cup using reagent water. Fill the dispersion cup ½ full with reagent water and place the cup on the blender to mix for one minute.

NOTE: Some samples may not be amendable to using the blender examples include but not limited to large gravel, sands, or organic material. If the sample is not amenable, initiate a NCM to notify the PM of the anomaly and proceed to the next step without blending the sample.

Place a #10 sieve on a 1000 mL graduated cylinder. Pour the sample through the sieve. Rinse the dispersion cup with reagent water and pour the rinse through the sieve. Repeat until transfer is complete. Bring the volume in the graduated cylinder to 1000 mL with reagent water. Cover the cylinder with a rubber stopper and equilibrate the sample to ambient temperature in preparation for hydrometer analysis.

Label a medium size aluminum dish with the sample's LAB ID then transfer the sample material that was retained on the #10 sieve to the dish. Place the aluminum dish in the drying oven set at 110 ± 5° C and dry the sample material for at least 16 hours or until constant weight. Set aside for sieve analysis.

10.5 Hydrometer Analysis

Prepare a hydrometer rinse bath by adding 1000 mL of reagent water to a 1000 mL graduated cylinder

Record the hydrometer ID and start time on the worksheet. Set the timer for the elapsed time and perform each task as listed in Table 1: Hydrometer Reading Table.

To shake the cylinder, rotate the flask up and down for one minute approximating at least 60 turns. One turn down and one turn up equals two turns.

To take a hydrometer reading, gently insert the hydrometer into the graduated cylinder and wait ~ 20 seconds. Read the hydrometer from the top of the meniscus to the nearest 0.0005. Enter the reading on the worksheet. After each reading, clean the hydrometer by twisting and dropping the hydrometer into the hydrometer rinse bath.

Insert a temperature probe into the cylinder to the same depth used for the hydrometer reading. Read the temperature to the nearest 0.5°C and enter the temperature measurement on the worksheet. Rinse the temperature probe in the hydrometer rinse bath.

Repeat the above process taking hydrometer readings every 2, 5, 15, 30, 60, 240 and 1440 minutes as per Table 1 then proceed to small sieve analysis.

10.6 Sieve Analysis

Inspect the sample material in the aluminum pan and record a description of the non-soil material (e.g.- sticks, grass, wood, plastic), hardness of material and shape of material in the worksheet.

Hardness qualifiers include hard, soft or brittle. Shape qualifiers include well rounded, rounded, subrounded, subangular, and angular.

Large Sieves

Weigh the 3/4", 3/8", #4 and #10 sieves and enter the weight measurements in the worksheet as the tare weight.

Stack the sieves then transfer the sample material from the aluminum dish to the sieve stack. If the sample material is less than 30 g, manually shake the sieve stack for 2 minutes. If the sample material is greater than 30 g, place the sieve stack into the Ro-tap machine and shake the sieve stack for 10 minutes.

Weigh each sieve and record these measurements in the worksheet.

Small Sieves

Transfer the sample from the graduated cylinder to a #200 wet wash sieve. Wash the sample through the #200 sieve until the water runs clear then transfer the material retained on the sieve to a 250 mL glass beaker labeled with the sample's LAB ID.

Place the beaker in the drying oven and dry at a temperature of 110ºC for at least 16 hours. After 16 hours, remove the beaker from the oven and allow it to cool.

Gently mix the dried contents of the beaker with a rubber-tipped pestle to break any soil aggregates that may have formed during the drying stage.

Tare the balance and weigh the sieve stack sized between #20 and #200 and record the tare weights.

Transfer the sample to the sieve stack and ensure complete transfer. Use hair or wire brushes to clean the beaker. Place the sieve stack on the Rotap machine and shake for ten minutes.

Weigh each sieve and record these measurements in the worksheet.

11.0 Calculations / Data Reduction

11.1 Calculations

Sample Used (SU): Dry Preparation

$$SU = (pan + dry\ sample\ -\ pan) - (pan + non\ -\ soil\ material\ -\ pan) \otimes HMCF$$

Where:
HMCF = Hygroscopic moisture correction factor.

Sieve Analysis (Percent Finer = PF)

Large Sieves:

3 inch: PF = 100-100 (Sieve and Sample (3 inch) - Sieve (3 inch))/SU*

2 inch: PF = PF (3 inch) - 100(Sieve and Sample (2 inch) - Sieve (2 inch))/SU and so on through the #10 Sieve.*

Small Sieves:

#20: PF = PF(#10) - 100(mass passing #10/sample mass (Hyd))*(sieve and sample (#20) - sieve(#20))/sample used*

#40: PF = PF (#20) - 100(mass passing #10/sample mass (Hyd))*(sieve and sample (#40) - sieve (#40))/sample used and so on up through #10 sieve.*

Hydrometer Analysis

Particle size, Micron

1000*sqrt [930*viscosity/980*(SG-1))*(effective depth/time)]

Viscosity at sample temperature, poises
Effective Depth, cm = 16.29-264.5*(actual Hydrometer reading - 1) above equation for effective depth based on equation found with table 2 in method, in which 16.29 = 0.5*(14.0-67.0/27.8)+10.5 and 264.5 = (10.5-2.3)/0.031
Time, minutes = Time of hydrometer reading from beginning of sedimentation
Sqrt - square root
SG - Specific Gravity of soil
Viscosity - is the resistance of a liquid to flow
Percent Finer (PF):

PF = Constant*(actual hydrometer reading - hydrometer correction factor - 1)

Where:
Constant = (100,000/W)*SG/(SG-1)
W = (Total sample used *sample used for hydrometer analysis*HMCF)/Amount of total sample

passing #10 sieve
Hydrometer Correction = slope*sample temperature + Intercept
Slope = ((low temp. reading -1)-(high temp. reading -1)/(low temp. - high temp.))
Intercept = (low temp. reading -1) - (low temp. * slope)

11.2 Data Reduction

11.2.1 Primary Data Review

Review project documents such as the Project Plan (PP), Project Memo or any other document/process used to communicate project requirements to ensure those project requirements were met. If project requirements were not met, immediately notify the project manager (PM) to determine an appropriate course of action.

Upload the batch information into LIMS and complete the batch editor and worksheet. Initiate NCMs for any anomalies observed during the preparation process. Set the status of the batch to 1st level review.

11.2.2 Secondary Data Review

Review project documents such as the Project Plan (PP), Project Memo or any other document/process used to communicate project requirements and verify those project requirements were met. If project requirements were not met, immediately notify the project manager (PM) to determine an appropriate course of action.

Check the batch editor and worksheet to verify the batch is complete and any outages are documented with an NCM along with the results of any corrective actions taken. Set the status of the batch to second level review.

11.2.3 Lab Complete

Review the batch, run QC checker as appropriate and set the status to lab complete.

11.2.4 Data Reporting

Sample results are reported from the laboratory's LIMS system using the formatter specified by the Project Manager.

11.2.5 Data Archival

Data are stored in the laboratory's LIMS system.

12.0 Method Performance

Not Applicable

13.0 Pollution Control

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide

by the policies in Section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

14.0 Waste Management

Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to BR-EH-001. The following waste streams are produced when this method is carried out.

- Solid Waste-Satellite Container: Solid Waste 5 Gallon Plastic Bucket (inside fume hood)
- Liquid Waste- 55 gallon poly drum

15.0 References / Cross-References

- ASTM Standard D 2217 – 85 (Reapproved 1998) "Standard Practice for Wet Preparation of Soil Samples for Particle-Size Analysis and Determination of Soil Constants", ASTM International, West Conshohocken, PA 2003, DOI: 10.1520/C0033-03, www.astm.org

- ASTM Standard D 422-63 (Rapproved 2007) "Standard Test Method for Particle-Size Analysis of Soils", ASTM International, West Conshohocken, PA 2003, DOI: 10.1520/C0033-03, www.astm.org

16.0 Method Modifications

D2217: The laboratory performs sample portioning after soaking the solution in the dispersing agent because the dispersion agent helps break up aggregates associated with clay and sediments.

D422: The laboratory does not always use the recommended amount of sample for analysis because sufficient sample volume is not always received.

17.0 Attachments

- Table 1: Hydrometer Reading Table (For up to 12 Sedimentation Cylinders)
- Table 2: Percent Solids Table for Weight Determination for D422.

18.0 Revision History

BR-GT-006, Revision 6:

- Title Page: Updated approval signatures
- All Sections: Removed references to dry preparation by ASTM D421; Added procedure for wet preparation.
- Attachments: Inserted Percent Solids Table

Table 1: Hydrometer Reading Table (For up to 12 Sedimentation Cylinders)

Elapsed Time (hr:min)	Task	Cyl. No.	Actual Time (min)	Elapsed Time (hr:min)	Task	Cyl. No.	Actual Time (min)
0:00	Shake	1		1:01	Read	10	5
0:01	**Place**	**1**		**1:02**	Shake	11	
0:01	**Shake**	**2**		**1:03**	Place	11	
0:02	Place	2		1:04	Read	9	15
0:03	Read	1	2	1:05	Read	11	2
0:04	Read	2	2	1:06	Read	7	31
0:06	Read	1	5	1:07	Read	3	58
0:07	Read	2	5	1:08	Read	11	5
0:08	Shake	3		**1:09**	Shake	12	
0:09	**Place**	**3**		**1:10**	Place	12	
0:09	**Shake**	**4**		1:11	Read	10	15
0:10	Place	4		1:12	Read	12	2
0:11	Read	3	2	1:13	Read	4	63
0:12	Read	4	2	1:14	Read	8	32
0:14	Read	3	5	1:15	Read	12	5
0:15	Read	4	5	1:18	Read	11	15
0:16	Read	1	15	1:19	Read	9	30
0:17	Read	2	15	1:21	Read	5	60
0:20	Shake	5		1:25	Read	12	15
0:21	Place	5		1:26	Read	10	30
0:23	Read	5	2	1:27	Read	6	59
0:24	Read	3	15	1:33	Read	11	30
0:25	Read	4	15	1:34	Read	7	59
0:26	Read	5	5	1:41	Read	12	31
0:27	Shake	6		1:42	Read	8	60
0:28	Place	6		1:52	Read	9	63
0:30	Read	6	2	1:53	Read	10	57
0:31	Read	1	30	2:06	Read	11	63
0:32	Read	2	30	2:07	Read	12	57
0:33	Read	6	5	4:17	Read	1	256
0:34	Shake	7		4:18	Read	2	256
0:35	Place	7		4:19	Read	3	250
0:36	Read	5	15	4:20	Read	4	250
0:37	Read	7	2	4:21	Read	5	240
0:38	Read	3	29	4:22	Read	6	234
0:39	Read	4	29	5:00	Read	7	265
0:40	Read	7	5	5:01	Read	8	259
0:41	Shake	8		5:02	Read	9	253
0:42	Place	8		5:03	Read	10	247
0:43	Read	6	15	5:04	Read	11	241
0:44	Read	8	2	5:05	Read	12	235
0:47	Read	8	5	24:01	Read	1	1440
0:48	Shake	9		24:02	Read	2	1440
0:49	Place	9		24:03	Read	3	1434
0:50	Read	7	15	24:04	Read	4	1434
0:51	Read	9	2	24:05	Read	5	1424
0:52	Read	5	31	24:06	Read	6	1418
0:54	Read	9	5	24:07	Read	7	1412
0:55	Shake	10		24:08	Read	8	1406

0:56	Place	10		24:09	Read	9	1400
0:57	Read	8	15	24:10	Read	10	1394
0:58	Read	10	2	24:11	Read	11	1388
0:59	Read	6	31	24:12	Read	12	1382
1:00	**Read**	**1**	**59**				
1:00	**Read**	**2**	**58**				

Source: Laboratory Prepared Reference Document

Table 2: Percent Solids Table for Weight Determination for D422.

Percent Solid Table

Quantities of sample (in grams) to be utilized in Wet method version of ASTM D854 and D422

% Sol	Spec Grav	Hydrometer Slt/Cl	Slt/Snd	Snd	Snd/Gr
	25	**50**	**75**	**100**	**200**
1	2500	5000	7500	10000	20000
2	1250	2500	3750	5000	10000
3	833	1667	2500	3333	6667
4	625	1250	1875	2500	5000
5	500	1000	1500	2000	4000
6	417	833	1250	1667	3333
7	357	714	1071	1429	2857
8	313	625	938	1250	2500
9	278	556	833	1111	2222
10	250	500	750	1000	2000
11	227	455	682	909	1818
12	208	417	625	833	1667
13	192	385	577	769	1538
14	179	357	536	714	1429
15	167	333	500	667	1333
16	156	313	469	625	1250
17	147	294	441	588	1176
18	139	278	417	556	1111
19	132	263	395	526	1053
20	125	250	375	500	1000
21	119	238	357	476	952
22	114	227	341	455	909
23	109	217	326	435	870
24	104	208	313	417	833
25	100	200	300	400	800
26	96	192	288	385	769
27	93	185	278	370	741
28	89	179	268	357	714
29	86	172	259	345	690
30	83	167	250	333	667
31	81	161	242	323	645
32	78	156	234	313	625
33	76	152	227	303	606
34	74	147	221	294	588
35	71	143	214	286	571
36	69	139	208	278	556
37	68	135	203	270	541
38	66	132	197	263	526
39	64	128	192	256	513
40	63	125	188	250	500
41	61	122	183	244	488
42	60	119	179	238	476
43	58	116	174	233	465
44	57	114	170	227	455
45	56	111	167	222	444
46	54	109	163	217	435
47	53	106	160	213	426
48	52	104	156	208	417
49	51	102	153	204	408
50	50	100	150	200	400

% Sol	Spec Grav	Hydrometer Slt/Cl	Slt/Snd	Snd	Snd/Gr
	25	**50**	**75**	**100**	**200**
51	49	98	147	196	392
52	48	96	144	192	385
53	47	94	142	189	377
54	46	93	139	185	370
55	45	91	136	182	364
56	45	89	134	179	357
57	44	88	132	175	351
58	43	86	129	172	345
59	42	85	127	169	339
60	42	83	125	167	333
61	41	82	123	164	328
62	40	81	121	161	323
63	40	79	119	159	317
64	39	78	117	156	313
65	38	77	115	154	308
66	38	76	114	152	303
67	37	75	112	149	299
68	37	74	110	147	294
69	36	72	109	145	290
70	36	71	107	143	286
71	35	70	106	141	282
72	35	69	104	139	278
73	34	68	103	137	274
74	34	68	101	135	270
75	33	67	100	133	267
76	33	66	99	132	263
77	32	65	97	130	260
78	32	64	96	128	256
79	32	63	95	127	253
80	31	63	94	125	250
81	31	62	93	123	247
82	30	61	91	122	244
83	30	60	90	120	241
84	30	60	89	119	238
85	29	59	88	118	235
86	29	58	87	116	233
87	29	57	86	115	230
88	28	57	85	114	227
89	28	56	84	112	225
90	28	56	83	111	222
91	27	55	82	110	220
92	27	54	82	109	217
93	27	54	81	108	215
94	27	53	80	106	213
95	26	53	79	105	211
96	26	52	78	104	208
97	26	52	77	103	206
98	26	51	77	102	204
99	25	51	76	101	202
100	25	50	75	100	200

Title: Density in Soils by Drive Cylinder Method (ASTM D2937-04)

Approval Signatures:

William S. Cicero
Laboratory Director

Christopher G. Callahan
Department Manager

Kirstin L. McCracken
Quality Assurance Manager

Bryce E. Stearns
Technical Director

Dan W. Helfrich
Health & Safety Coordinator

Approval Date: June 21, 2010

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SOP REVIEW FORM

SOP Number	Revision	Effective Date:	Title
BR-GT-018	6	06/21/10	Density in Soils by Drive Cylinder Method

Review Statement: 5 KLD 11-12-3-12

My signature signifies that I reviewed and compared the above referenced SOP against current bench practice.

Date	Reviewer	Job Title	Revision Needed	
			Yes[1]	No
11/28/12	Chris Cullen	Dept Manager		X

[1]: *List the section for revision and the the reason using the revision summary page and attach to this cover sheet.*

QA Use Only:

X The SOP was reviewed and does not require revision. Attach this form to the SOP.

_____ The SOP Revision will be made with a Change in Progress Attachment (CIPA).

_____ The SOP Revision will be released as a new version of the SOP.

_____ The SOP Revision requires method validation or demonstration of capability

_____ The SOP Revision does not reqire method validation or demonatration of capability.

_____ The SOP revison affects other SOPs that must now also be revised (List SOPs)

_____

_____

Kristie Daigle
QA Signature

11/28/12
Date

Comments:

1.0 Scope and Application

This standard operating procedure (SOP) describes the laboratory procedure for the determination of in-place density of natural, inorganic soil by the drive cylinder method.

This procedure is not suitable for organic soils that can compress upon sampling, very hard natural soils, heavily compacted soils, soils with low plasticity or soils with large amounts of gravel. The procedure is not recommended for friable soils or soft, highly plastic, non-cohesive, saturated or other soils which are not easily deformed.

2.0 Summary of Method

A representative portion of sample is removed from the sample collection tube and dried in an oven maintained at a temperature of 110°C for 16 hours. The moisture content of the sample is determined and expressed as a percent of the oven-dried mass. Density is subsequently calculated by dividing the value obtained for oven-dried mass by the determined volume of the drive cylinder.

This procedure is based on the following reference method:

- ASTM Standard D D2937-04 "Standard Test Method for Density of Soil in Place by the Drive-Cylinder Method. ASTM International, West Conshohocken, PA 2003, DOI: 10.1520/C0033-03, www.astm.org

If the laboratory has modified the procedure from the reference method(s) a list of modifications will be provided in Section 16.0.

3.0 Definitions

Not Applicable

4.0 Interferences

This method is not appropriate for samples that can be "altered" during sampling, shipping and/or processing.

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1 Specific Safety Concerns or Requirements

None

5.2 Primary Materials Used

Table 1 lists those materials used in this procedure that have a serious or significant hazard rating along with the exposure limits and primary hazards associated with that material as identified in the MSDS. **NOTE: This list does not include all materials used in the method.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

6.0 Equipment and Supplies

Catalog numbers listed in this SOP are subject to change at the discretion of the vendor. Analysts are cautioned to be sure equipment used meets the specification of this SOP.

- Oven, 110°C (+/-5°C), Barnstead LC Oven Model# 3513 or equivalent.
- Top loading balance, Mettler Model# PB3002 or equivalent.
- Aluminum measuring pans, Fisher Scientific or equivalent.
- Stainless steel spatulas and spoons, Fisher Scientific or equivalent.
- Ruler/meter stick with millimeter increments
- Heat shield gloves / Oven Tongs, Fisher Scientific or equivalent.

7.0 Reagents and Standards

Not Applicable

8.0 Sample Collection, Preservation, Shipment and Storage

The laboratory does not perform sample collection. Sample collection procedures are provided in this SOP for guidance only. The laboratory recommends that all samples be collected in accordance with a client field and sampling plan. Immediately after collection the sample(s) should be cooled and stabilized with packing material or wax to prevent shifting during transport.

Holding times are not applicable. Unless otherwise specified by client or regulatory program, after analysis samples are held for a minimum of 30 days and then disposed of in accordance with applicable regulations.

9.0 Quality Control

Not Applicable

10.0 Procedure

10.1 Calibration and Standardization

Check the calibration of the balance on each day of use prior to use using at least 2 Class S weights that bracket the range of use. Record in the logbook designated for this purpose.

Check the temperature of the drying oven(s) each day of use, prior to use. Record in the logbook designated for this purpose.

10.2 Sample Preparation/Analysis

Clean the outside of the sample collection tube then remove the end caps, wax any other protective material from each end of the sample collection tube. Remove any loose material from the collection tube. If the sample extends beyond the tube, trim the end of the sample so that it is equal to the length of the tube.

Measure the diameter of the sample tube and enter this value into the LIMS worksheet.

If sample fills the entire tube, measure the length of the tube and enter this value into the LIMS worksheet. If sample does not fill the entire tube, measure the length of the tube and measure the length of empty space in the tube, then subtract the length of empty space. Enter this value into the LIMS worksheet.

Weigh the tube and sample and enter this value into the LIMS worksheet.

Empty the sample into a large pan then clean the tube with a spatula to ensure all sample is taken into account. Weigh the clean tube and enter the weight measurement into the LIMS worksheet.

Label a clean aluminum pan with the sample ID and weigh the pan and enter this weight measurement into the LIMS worksheet.

Thoroughly mix the sample to homogenize with a spatula or spoon. Measure at least 100 g of the sample into the pan, or if practical, use the entire sample. Enter this weight measurement into the LIMS worksheet.

Place the sample into an oven maintained at 110°C and allow the sample to dry for a minimum of 16 hours. After this time period has elapsed, remove the pan from the oven and re-weigh. Enter this weight measurement into the LIMS worksheet.

The LIMS method calculate the moisture content, sample length, sample volume and in-place density using the equations give in Section 11.0.

11.0 Calculations / Data Reduction

11.1 Calculations

Percent Moisture Content

Moisture Content, % = [(A-B) * 100]/B

Where:
A = pan + wet sample, g
B = pan + dry sample, g

Sample length:

$L=L_1-(L_2+L_3)$ mm

Where:
L = sample length
L_1 = length of sample tube
L_2 = average recovery, top
L_3 = average recovery, bottom

Sample volume:

$V = [(\pi(D/2)^2)L]$

Where:
V = sample volume (calculated)
D = sample tube diameter
L = sample length

In-place Density:
$= m_1/V$ g/cm^3 (dry)

$= m_2/V$ g/cm^3 (wet)

Where:
= calculated dry density
m_1 = mass of sample, dry
m_2 = mass of sample, wet
m_3 = mass of sample tube
V = sample volume

11.2 Data Review

Primary Review

Review project documents such as the Project Plan (PP), Project Memo or any other document/process used to communicate project requirements and verify those project requirements were met. If project requirements were not met, immediately notify the project manager (PM) to determine an appropriate course of action.

Enter the batch information into LIMS and complete the batch editor and worksheet for each extraction and cleanup batch performed. Initiate NCMs for any anomalies observed during the process. Set the status of each batch to 1st level review.

Secondary Data Review

Review project documents such as the Project Plan (PP), Project Memo or any other document/process used to communicate project requirements and verify those project requirements were met. If project requirements were not met, immediately notify the project manager (PM) to determine an appropriate course of action.

Check the batch editor and worksheet to verify the batch is complete and any outages are documented with an NCM along with the results of any corrective actions taken. Set the status of each batch to second level review.

Run the QC Checker, review the deliverable and set the job to lab complete.

12.0 Method Performance

12.1 Training Requirements

Any employee that performs any portion of the procedure described in this SOP must have documentation in their employee training file that they have read this version of this SOP.

13.0 Pollution Control

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

14.0 Waste Management

Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to BR-EH-001. The following waste streams are produced when this method is carried out.

- Solid Waste- Satellite Container: 5 Gallon Plastic Bucket.

15.0 References / Cross-References

- ASTM Standard D D2937-04 "Standard Test Method for Density of Soil in Place by the Drive-Cylinder Method. ASTM International, West Conshohocken, PA 2003, DOI: 10.1520/C0033-03, www.astm.org

- Corporate Environmental Health and Safety Manual (CW-E-M-001)

16.0 Method Modifications

None

17.0 Attachments

None

18.0 Revision History

Revision 5:
- Title Page: Updated approval signatures.
- Section 4.0: Inserted a statement saying this method is not appropriate for all soil samples.
- Section 10.2: changed minimum weight for water content from 50 g to 100 g.

MEASUREMENT OF ANALYTES USING THE KONELAB ANALYZER

Approvals (Signature/Date):

Andrea Teal — March 23, 2005
Andrea Teal — Date
Quality Assurance Manager

Benjamin Gulizia — March 23, 2005
Benjamin Gulizia — Date
Laboratory Director/Lead Technical Director

Ernest B. Walton — March 23, 2005
Ernest Walton — Date
Health & Safety Manager / Coordinator

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SOP REVISION / REVIEW SUMMARY

SOP Number: GE01:03.18.05:1
Review Date: March 18, 2005
Effective Date: April 18, 2005

Review / Revision Type:
Analytical/procedural revisions
- Revision # change. Completed SOP Training Forms are required.

Summary of Changes:
- ***Complete re-write.***
- Added several new analyses to be analyzed by this method: nitrate, nitrite, total phosphorus, TKN, hydrazine, tannin & lignin, and hexavalent chromium
- Revised the SOP title
- Revised format to be consistent with current STL Savannah SOP format and NELAC requirements
- Revised safety information
- Added quality control, method performance, preventative maintenance, and waste management information

SOP Number: GE01:03.18.05:1A
Review Date: August 5, 2005
Effective Date: August 5, 2005

Review / Revision Type:
Minor text, grammatical, and/or formatting changes
- Alpha character revision, notification is not required. SOP training is not required.

Summary of Changes:
- Minor typographical changes made to Section 1.0, and 9.0. Separated method and SOP references for total cyanide and free cyanide.

Approval:

Quality Assurance Manager: Andrea Teal Date: August 5, 2005
Andrea Teal

SOP Number: GE01:03.18.05:1B
Review / Effective Date: March 14, 2007

Review / Revision Type:
Minor text, grammatical, and/or formatting changes
- Alpha character revision, notification is not required. SOP training is not required.

Summary of Changes:
- Minor grammatical changes.
- Revised all referenced SOP titles to be consistent with current revisions.

Approval:

Quality Assurance Manager: Andrea Teal Date: March 14, 2007
Andrea Teal

SOP Number: GE01:03.18.05:1C
Review / Effective Date: March 14, 2007

Review / Revision Type:
Minor text, grammatical, and/or formatting changes
- Alpha character revision, notification is not required. SOP training is not required.

Summary of Changes:
- Updated method references for Chloride, Cyanide, Orthophosphate, and Cyanide.

Approval:

Quality Assurance Manager: Andrea Teal Date: February 14, 2008
Andrea Teal

SOP Number: GE01:03.18.05:1D
Review Date: December 31, 2008
Effective Date: February 19, 2009

Review / Revision Type:
Minor text, grammatical, and/or formatting changes
- Alpha character revision, notification is not required. SOP training is not required.

Summary of Changes:
- Included Ammonia Distillation information form SOP GE15. SOP GE15 is now obsolete.
- Removed references to cyanide and hydrazine as these are no longer performed using this procedure.
- Updated nitrate soil preparation procedures to match current lab practice.
- Removed reference to SOP AN99: *Definitions, Terms, and Acronyms*. This SOP is now obsolete. Definitions are now contained in the glossary in the Laboratory Quality Manual.
- Revised all referenced SOP titles to be consistent with current revisions.
- Removed raw data from Attachments.

Approval:

Quality Assurance Manager: Andrea Teal Date: December 31, 2008
Andrea Teal

SOP Number: GE01:03.18.05:1E
Review Date: January 4, 2011
Effective Date: January 4, 2011

Review / Revision Type:
Minor text, grammatical, and/or formatting changes
- Alpha character revision, notification is not required. SOP training is not required.

Summary of Changes:
- Revised referenced methods to match those performed.

Approval:

Quality Assurance Manager: Andrea Teal Date: January 4, 2011
Andrea Teal

SOP Number: GE01:03.18.05:1F
Review Date: February 11, 2011
Effective Date: February 11, 2011

Review / Revision Type:
Minor text, grammatical, and/or formatting changes
- Alpha character revision, notification is required. SOP training is not required.

Summary of Changes:
- Clarified pH requirement for hexavalent chromium to reflect 1.5-2.5 as specified in SM3500-Cr B (On-line Edition) and EPA 7196A. This SOP change is administrative in nature as the laboratory has previously adopted this range.
- Added note regarding sulfate determination using average readings as opposed to reading taken from replicate with highest turbidity value.

Approval:

Quality Assurance Manager: Andrea Teal Date: February 11, 2011
Andrea Teal

SOP Number: GE01:03.18.05:1G
Review Date: April 24, 2012
Effective Date: April 24, 2012

Review / Revision Type:
Minor text, grammatical, and/or formatting changes
- Alpha character revision, notification is required. SOP training is not required.

Summary of Changes:
- Incorporated the current ammonia micro-distillation procedure. This SOP change is administrative in nature as the laboratory has previously adopted this range.
- Tannin and Lignin procedures were removed as this procedure is no longer performed.

Approval:

Quality Assurance Specialist: Ammie L. Martin Date: April 24, 2012
Ammie Martin
Designee for: Andrea Teal, Quality Assurance Manager / Interim Environmental Health and Coordinator

1.0 SCOPE AND APPLICATION

This SOP describes the procedures used by TestAmerica Savannah to determine the concentration of various analytes in water and soil using the Konelab autoanalyzer. The following analytes may be determined:

ANALYTE	REFERENCE METHOD	APPLICATION NOTE/ SOP SUMMARY
Ferrous iron	SM3500-Fe-D (20th), SM3500-Fe-B (online)	Appendix A
Chloride	EPA 325.2, EPA 9251, SM4500-Cl-E	Appendix B
Sulfate	EPA 375.4, EPA 9038	Appendix C
Ortho-Phosphate	EPA 365.1, SM4500-P-F	Appendix D
Ammonia	EPA 350.1, SM4500-NH3-B (prep), SM4500-NH3-G, COE 3_154 (soil prep)	Appendix E
Total Phosphorus	EPA 365.4	Appendix G
TKN	EPA 351.2	Appendix H
Hexavalent Chromium	EPA 7196A, SM3500-Cr-D (20th), SM3500-Cr-B (online)	Appendix I

The routine analytes, reporting limits (RLs), method detection limits (MDLs), and precision and accuracy limits associated with these methods are listed in the LIMS Method Limit Groups (MLGs).

2.0 SUMMARY OF METHOD AND DEFINITIONS

2.1 The SOP Appendices contain the method parameters required to perform the analysis. The appendices also contain a summary of the "chemistries" (reagents, procedures, wavelengths, etc.) used to measure the target analyte.

2.2 Instrument Design, Data System, and Basic Operation

2.2.2 Instrument Design

The analytical components (analyzer and autosampler) of the Konelab Model 20 Analyzer are contained in a single unit equipped with casters, which allows the unit to be moved easily. The dimensions of the analyzer are 80cm wide, 120 cm high, 79 cm deep, and it weighs 150 kg.

No plumbing is required for this instrument as there are reservoirs for both reagent water as well as a waste container. Reagents are stored on the autosampler and are kept at a temperature of approximately 10 degrees below ambient. The reagents can be loaded in 10, 20, or 60mL containers. The sampler monitors the volume of reagents and warns the analyst when the reagent is running out.

The samples and reagents are dispensed into disposable, acrylic multicell cuvettes that hold 12 separate analyses. The maximum volume of sample used is 120uL with the maximum total volume of sample and reagent of 250uL. The accuracy of the sampler is accomplished through the use of an inline GC-type syringe that is driven by micro-stepping motors. During the incubation period(s), the sample cuvettes are maintained @37°C.

The measurement system is a single channel interference filter photometer with beam splitting reference. The color wheel can be configured with up to 15 different filters. Fiber optic cabling

transfers the light signal from the source to the detector. The lamp is a halogen lamp. The lamp can be tested by the use of a water blank. This test checks the stability of the lamp at all 15 filter settings as well as testing the optical purity of all 12 positions of a cuvette. The detection step can be set up to treat all standards and samples in the same time fashion, that is, the time between reagent addition and absorbance readings will be the same for all sample and standard positions.

2.2.2 Data System

The data system is a separate unit and is placed near the analyzer on a separate desk or counter. The software system is based on the Windows NT operating system. The hardware interface can be either RS232 or an Ethernet connection and can be integrated to both transmit and receive information from a LIMS.

Programmable instrument parameters include:

Volume of sample (maximum of 125uL)
Type and order of reagent(s) addition
Time of sample/reagent mixing
Dilutions
Track reagents lot numbers/expiration dates.
Wavelength
Background absorbance
Timing of absorbance readings

QC checks can be programmed into the methods as needed, including initial and continuing control standards, blanks, and upper and lower limits on absorbance. If samples or standards exceed these control limits, the data are flagged.

The data may be printed on the day of analysis or archived and retrieved as needed. The data can be sorted by test or by sample ID, and the results are stored on the hard drive and can also be archived on a CD-ROM.

2.2.3 Operation:

The instrument analyzes samples in the following general order:

Sample is dispensed into the cuvettes according to the test method (maximum sample volume is 125uL)
The reagents are added to the sample and mixed
The sample is incubated
The sample absorbance is measured (if absorbance exceeds calibration range, sample is diluted as defined in the test method and reanalyzed. If the dilution exceeds the maximum range, the data are flagged and the analyst must prepare dilution for sample)

The calibration of the instrument can be linear, nonlinear, or bias correction. The calibration can be accomplished through separate calibrator samples or with a series of dilutions made automatically from an onboard standard.

Tests may be chosen individually, or groups of analyses can also be set up. For example, a test can be set up for nitrate that will automatically choose to run nitrate+nitrite and nitrite. The difference between the two tests will be calculated and the nitrate will be reported. Test methods may include multiple analyses that are analyzed on the same type of preserved sample. For example, chloride, phosphate, nitrite, and sulfate may be included in one Konelab method and analyzed from the same sample portion placed in the autosampler

Samples are loaded into racks that hold 14 samples each. Racks may be removed and replaced with more samples as the analyses are finished. The sampler holds up to 6 racks of 14 samples.

The racks can be fitted to hold sample containers from 0.5mL up to 10mL. Two separate wash stations are used to minimize sampler carryover. The probe is flow through and externally rinsed. Rush samples can be added into one of 5 stat positions.

Instrument and software maintenance tests are an integral part of the analytical system. The test programs evaluate items such as temperature, dispensing ratios, both accuracy and precision of the dispenser, and photometric noise.

2.3 Definitions

Definitions – Refer to TestAmerica Savannah's *Quality Assurance Manual* (QAM) for a complete listing of applicable definitions.

3.0 SAFETY

Employees must abide by the policies and procedures in the analytical SOPs, TestAmerica Environmental Health and Safety Manual (EHSM), the TestAmerica Savannah Addendum to the EHSM, and this document.

3.1 Specific Safety Concerns or Requirements

All samples must be treated as if they are hazardous. The analyst must protect himself/herself from exposure to the sample matrix. Many of the samples that are tested for residue/solids may contain hazardous chemical compounds or biological organisms. The analyst must wear protective clothing (lab coat or apron), eye protection (glasses or face shield), and disposable gloves when handling these samples.

The toxicity or carcinogenicity of chemicals used in this method has not been precisely defined; each chemical should be treated as a potential health hazard, and exposure to these chemicals should be minimized.

Potassium cyanide and sodium cyanide will give off hydrogen cyanide (HCN) gas if combined with strong acids. Inhalation of CN gas can cause irritation, dizziness, nausea, unconsciousness and potentially death.

Pyridine is flammable and will cause severe irritation to the respiratory tract. It can cause dizziness, headaches, nausea, and shortness of breath. Vapors can cause irritation of the eyes. Contact with the eyes can cause severe irritation, possible corneal burns and eye damage.

Sulfuric acid is a strong oxidizer and is a corrosive. It will react violently when combined with organic compounds, possibly producing fire. Inhalation can cause irritation of the nose, throat, mucus membranes, and upper respiratory tract. Contact with the eyes can cause blurred vision, redness, pain, and even blindness.

3.2 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS. MSDS can be found in electronic format on Oasis on the EH&S webpage.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Barium Chloride	Poison Irritant	0.5 mg/m^3 TWA	May be fatal if swallowed. Harmful if inhaled. Avoid contact with eyes, skin, and clothing. Avoid breathing dust. Keep container closed and when in use adequate ventilation.
Chloramine-T	Contact Irritant	None	Harmful if swallowed or inhaled. Causes redness, irritation and pain to skin, eyes, and respiratory tract. Can cause allergic reactions. Symptoms of inhalation can include burning sensation, coughing, wheezing, laryngitis, shortness of breath, sore throat, bronchitis, pneumonitis, and pulmonary edema.
Ferric Nitrate	Oxidizer	None	Causes irritation to the respiratory tract. Causes irritation, redness, and pain to the skin and eyes.
Mercuric Thiocyanate	Poison	0.1 mg/m3 Ceiling (Mercury Compounds)	Extremely Toxic. Causes irritation to the respiratory tract. **May produce Hydrogen Cyanide gas if combined with strong acids.** 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.
Phenol	Corrosive	5 ppm-TWA	Breathing vapor, dust or mist results in digestive disturbances. Will irritate and possibly burn respiratory tract. Rapidly absorbed through the skin with systemic poisoning effects to follow. Discoloration and severe burns may occur, but may be disguised by a loss in pain sensation. Eye burns with redness, pain, blurred vision may occur. May cause severe damage and blindness.
Pyridine	Flammable Irritant	5 ppm-TWA	Inhalation causes severe irritation to the respiratory tract. Symptoms of overexposure include headache, dizziness, nausea, and shortness of breath. Causes severe irritation possibly burns, to the skin. Symptoms include redness and severe pain. Absorption through the skin may occur, resulting in toxic effects similar to inhalation. May act as a photosensitizer. Vapors cause eye irritation. Splashes cause severe irritation, possible corneal burns and eye damage.
Potassium Cyanide, Sodium Cyanide	Poison Corrosive	5 mg/m^3 TWA as CN	**This material will form Hydrogen Cyanide (HCN) gas when combined with strong acids. Breathing HCN gas may result in death.** Corrosive to the respiratory tract. May cause headache, weakness, dizziness, labored breathing nausea and vomiting, which can be followed by weak and irregular heartbeat, unconsciousness, convulsions, coma and death. Solutions are corrosive to the skin and eyes, and may cause deep ulcers, which heal slowly. May be absorbed through the skin, with symptoms similar to those noted for inhalation. Symptoms may include redness, pain, blurred vision, and eye damage.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Sodium Hydroxide	Corrosive Poison	2 ppm, 5 mg/m^3	This material will cause burns if comes into contact with the skin or eyes. Inhalation of Sodium Hydroxide dust will cause irritation of the nasal and respiratory system.
Sulfuric Acid	Corrosive Oxidizer Dehydrator Poison Carcinogen	1 mg/m3-TWA	Inhalation produces damaging effects on the mucous membranes and upper respiratory tract. Symptoms may include irritation of the nose and throat, and labored breathing. Symptoms of redness, pain, and severe burn can occur. Contact can cause blurred vision, redness, pain and severe tissue burns. Can cause blindness.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

4.0 INTERFERENCES

4.1 Ferrous Iron

The reaction is of ferrous iron with 1,10-phenanthroline is free from common interferences. Long storage times or exposure of samples to light must be avoided. Protect from light and analyze as soon as possible.

4.2 Chloride

Highly colored samples can positively interfere. Turbid samples should be filtered.

4.3 Sulfate

Silica above 500mg/L will interfere.

Color and turbidity due to the sample matrix can cause positive interferences that must be accounted for by the use sample blanks analyzed with no reagent. The absorbance or turbidity of the blank is subtracted from the absorbance or turbidity of the treated sample, and the concentration is determined from the corrected response.

4.4 Ortho-Phosphate

Blue colored components may interfere. High concentrations of arsenic may interfere.

4.5 Ammonia

Extreme sample pH values may interfere. Proper sample preservation and adjustment just prior to analysis should alleviate this problem. Turbidity and color in samples must be filtered and/or diluted prior to analysis. Residual chlorine should be removed at the time of sampling by the addition of sodium thiosulfate.

Samples that are tested against a compliance limit must be distilled, or a comparison of distilled and undistilled aliquots must be on file to demonstrate that the sample is free from interferences and that the analysis of the undistilled sample yields comparable results. Comparable results means that the results for the distilled and undistilled samples agree within 10% (percent difference less than 10%) using the distilled aliquot result as the reference.

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4.6 Nitrate

Build up of suspended matter in the reduction column will restrict the sample flow. Since nitrate-nitrogen is found in a soluble state, the sample may be pre-filtered through a glass fiber filter or a 0.45um membrane filter. Highly turbid samples may be pretreated with zinc sulfate before filtration to remove the bulk of particulate matter present in the sample.

Low results might be obtained for samples that contain high concentrations of iron, copper or other metals. EDTA is added to the samples to eliminate this interference.

Samples that contain large concentrations of oil and grease will coat the surface of the cadmium. This interference is eliminated by pre-extracting the sample with an organic solvent.

4.7 Nitrite

There are very few known interferences at concentrations less than 1,000 times that of the nitrite; however, the presence of strong oxidants or reductants in the samples will readily affect the nitrite concentrations. High alkalinity (>600mg/L) will give low results due to a shift in pH.

4.8 Total Phosphorus

High iron concentrations can cause precipitation of, and subsequent loss of, phosphorus. Arsenate is determined similarly to phosphorus and should be considered when present in concentrations higher than phosphorus.

Sample turbidity must be removed by filtration after digestion. Sample color that absorbs in the photometric range used for analysis will also interfere.

4.9 Total Kjeldahl Nitrogen

High nitrate concentrations (10x or more than the TKN level) result in low TKN values. If interference is suspected, samples should be diluted and reanalyzed.

4.10 Hexavalent Chromium

Iron in concentrations of greater than 1mg/L may produce a yellow color, but this is usually not significant when measured at 540nm. Hexavalent molybdenum and mercury salts react to form colored complexes with the color reagent, but the intensities of the colors produced are much lower than for chromium at the specified pH. Mercury and molybdenum concentrations of up to 200mg/L can be tolerated. Vanadium interferes more strongly, but concentrations of up to 10 times that of chromium can be tolerated.

All samples should be adjusted to pH 1.5-2.5 prior to analysis with nitric acid to minimize interference and to allow proper color development.

After acidification, soil digestates should be purged with helium to eliminate excess carbon dioxide gas. Bubble formation in the cuvettes can interfere with readings.

The recovery of soluble forms of hexavalent chromium may be low where the sample matrix contains substances that are readily oxidized by hexavalent chromium (a reducing matrix_. The measurement pH and oxidation-reduction potential may provide information about the ability of hexavalent chromium to persist in the sample.

5.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

5.1 Ferrous Iron

Samples are routinely collected in 250-mL plastic bottles with no preservative. Samples are iced at the time of collection and stored at 4°C (less than 6°C but not frozen) until the time of analysis. Protect from light and analyze the samples as soon as possible.

If the sample(s) cannot be analyzed within 24 hours of collection, filter the samples through a 0.45um filter and adjust the pH to <2 with concentrated HCl. Store the samples at 4°C (less than 6°C but not frozen) and protect from light. Analyze as soon as possible.

5.2 Chloride

Aqueous and soil/sediment samples are routinely collected in 100-mL pre-cleaned plastic containers with no preservative. Holding time is 28 days.

5.3 Sulfate

Aqueous and soil/sediment samples are routinely collected in 120-mL plastic containers with no chemical preservative. The samples are iced at the time of collection and maintained at 4°C (less than 6°C with no frozen samples) until the time of analysis. Samples should be analyzed as soon as possible with a maximum holding time of 28 days.

5.4 Ortho-Phosphate

Aqueous samples are routinely collected in 120-mL plastic bottles and are unpreserved. The samples are iced at the time of collection and stored at 4°C (less than 6°C with no frozen samples) until the time of analysis. The holding time for orthophosphate in liquids is 48 hours.

Soil samples are routinely collected in plastic jars. The samples are iced at the time of collection and stored at 4°C (less than 6C with no frozen sample) until the time of preparation and analysis. The hold time for soil samples is 28 days from collection.

5.5 Ammonia

Aqueous samples are routinely collected in plastic 250-mL containers. The samples are acidified to pH <2 with sulfuric acid, iced at the time of collection, and maintained at 4°C (less than 6°C with no frozen samples) until the time of preparation and analysis. Holding time is 28 days.

NOTE: The pH of each liquid sample must be checked and documented in the lab. If the pH is >2, add additional sulfuric acid until the pH is <=2 and initiate an anomaly report. The volume of sulfuric acid must not exceed 1% of the volume of the container or sample; e.g., no more than 2.5mL of sulfuric acid per 250-mL container or sample.

Residual chlorine should be removed at the time of sampling by the addition of sodium thiosulfate.

Soil and solids samples are routinely collected in plastic containers. The samples are iced at the time of collection and maintained at 4°C (less than 6°C with no frozen samples) until the time of preparation and analysis. The holding time is 28 days.

5.6 Nitrate

Analysis should be made as soon as possible. If analysis can be made within 48 hours, the sample should be preserved by refrigeration at 4°C. When samples must be stored for more than 48 hours, they should be preserved with sulfuric acid (2mL H2SO4 per liter) and refrigerated.

5.7 Nitrite

Analysis should be made within 48 hours on a sample preserved by refrigeration at 4°C.

5.8 Total Phosphorus / Total Kjeldahl Nitrogen

Samples should be collected in plastic or glass bottles and must be preserved with H2SO4 to a pH<2 and cooled to 4°C at the time of collection. Samples should be analyzed as soon as possible after collection. If storage is required, preserved samples are maintained at 4°C and may be held for up to 28 days.

5.9 Hexavalent Chromium

Aqueous samples are routinely collected in 100-mL or 250-mL plastic containers with no preservative. Samples are stored at 4°C (less than 6°C with no frozen samples) and analyzed within 24 hours of collection.

Soil samples should be stored in plastic or glass sealed containers, field-moist, at 4°C (less than 4°C with no frozen samples) until analysis. When stored as above, Cr^{+6} has shown to be quantitatively stable for at least 30 days from the time of collection. The alkaline digest has also been shown to be stable for up to 168 hours after the digestion.

6.0 APPARATUS AND MATERIALS

Konelabs Model 21 Analyzer

7.0 REAGENTS

The preparation of reagents must be documented in accordance with SOP SA-AN-041: *Reagent and Standard Materials Procedures.* See the appropriate SOP Appendix for the reagents used in each analysis.

8.0 STANDARDS

The preparation of the calibration standards must be tracked in accordance with SOP SA-AN-041: *Reagent and Standard Materials Procedures*. See the appropriate SOP Appendix for the standards used in each analysis.

9.0 SAMPLE PREPARATION

ANALYTE	AQUEOUS PREP	SOIL PREP
Ferrous iron	Filter	NA
Chloride	Filter	Soil leachate
Sulfate	Filter	Soil leachate
Ortho-Phosphate	Filter	Soil leachate
Ammonia	Filter*	Soil leachate
Nitrate	Filter	Soil leachate
Nitrite	Filter	Soil leachate
TKN	SOP SA-GE-020	SOP SA-GE-020
Total Phosphorus	SOP SA-GE-020	SOP SA-GE-020
Hexavalent Chromium	Filter	SOP SA-ME-021

*Ammonia samples may be distilled upon request.
See SOP Appendix for the LCS and MS spiking levels.

Basic solid leaching procedure:

Weigh out approximately 5g (5-5.5g) into a 100-mL screw-cap plastic bottle. Record the weight to the nearest 0.1g. Add 100mL of reagent water to each sample container and place on the extractor.

Prepare a method blank by adding 100mL of reagent water to an extraction bottle. Assume a sample weight of 5.0g. Place on extractor.

Prepare a LCS by adding the appropriate volume of spiking solution to 100mL of reagent water in an extraction bottle. Assume a sample weight of 5.0g. Place on extractor.

Weigh two additional 5g aliquots of one of the samples for the MS and MSD. Add the appropriate volume of the spiking solution to the sample. Add 100mL of reagent water to each sample container and place on the extractor.

Rotate for 2 hours for every parameter, except ammonia.

Remove the containers from the extractor and allow the leachates to settle. Filter the extract using a syringe filter with a 0.450-um pore size filter and analyze the extracts as liquid samples.

Ammonia solid leaching procedure:

Weigh out approximately 20g (20-20.4g) into a 100-mL screw-cap plastic bottle. Record the weight to the nearest 0.1g. Add 100mL of 2M potassium chloride to each sample container and place on the extractor.

Prepare a method blank by adding 100mL of reagent water to an extraction bottle. Assume a sample weight of 20g. Place on extractor.

Prepare a LCS by adding the appropriate volume of spiking solution to 100mL of reagent water in an extraction bottle. Assume a sample weight of 20g. Place on extractor.

Weigh two additional 20g aliquots of one of the samples for the MS and MSD. Add the appropriate volume of the spiking solution to the sample. Add 100mL of reagent water to each sample container and place on the extractor.

Rotate for 30 minutes for ammonia.

Remove the containers from the extractor and allow the leachates to settle. Filter the extract using a syringe filter with a 0.450-um pore size filter and analyze the extracts as liquid samples.

Ammonia distillation procedure:
Set controller to 120°C and turn on the heater block to warm up (40 min)

Determine the number of collector tubes needed and align with "M" end in the up position by placing them into a collector tube rack.

Label and fill each collector tube with 1mL of trapping solution (0.016M sulfuric acid). At the end of the distillation, once diluted to 6mL final volume, the final concentration will be 0.003M sulfuric acid.

Place the same number of sample tubes into the sample tube rack.

Label each tube and place 6mL of sample into each sample tube.

Add 1mL of 0.55M borate buffer to each tube.

Immediately push the "D" end of the collector tube over the open end of the sample tube to start the seal.

Place the tube assembly in the press by putting the sample tube through the hole in the base.

Before pressing, place one hand at the breakaway point located on the collector tube to stabilize the assembly.

Apply pressure to the assembly with a smooth motion until the stop ring on the sample tube hits the D end of the collector tube.

Once complete, put on heat resistant gloves and add the tube assemblies to the pre-heated block by placing the sample tube and D end of the collector tube into the block.

Set the timer for 30 minutes.

Once time is up remove the assemblies from the block one at a time and immediately remove the sample tube from the collector tube and invert so the D side is facing up. Note: this should be done within 4 seconds to prevent "suck-back" of the sample.

Once all of the tubes have been removed and disassembled, Parafilm the M end of the tube and allow the tubes to cool for 10 minutes.

Hold the tube horizontally and roll the tube with your fingers to gather the droplets that are stuck to the walls. The tube can be flicked with your finger as well.

With the D end still up, grasp the tube and break it at the breakaway point by pulling the D end towards your body and twisting the tube. Discard the D end of the tube.

Dilute the distillate up to the 6mL mark with distilled water and cap the tube until ready for analysis.

10.0 ANALYTICAL PROCEDURES

See the appropriate SOP Appendix for the analytical procedure.

11.0 DATA ANALYSIS AND CALCULATIONS

See the appropriate SOP Appendix for the analytical procedure.

12.0 QUALITY CONTROL AND DATA ASSESSMENT

QC data must be evaluated against the precision and accuracy criteria set forth in the Method Limit Groups in LIMS and SOP SA-QA-17: *Evaluation of Batch QC Data*. These requirements are summarized in the SOP summaries in the appendices for each analyte.

12.1 Analytical batch

SOP SA-QA-17 provides guidance for establishing and evaluating QC items to be included in an analytical batch. The minimum requirements are a method blank and a lab control standard (LCS) for each batch of twenty or fewer samples. Matrix spikes are analyzed as specified in the analytical method. These requirements are summarized in the SOP summaries in the appendices for each analyte.

12.2 Corrective Action for Out-of-Control Data

When the quality control parameters do not meet the criteria set forth in this SOP, corrective action must be taken in accordance with SOP SA-QA-05: *Preventive and Corrective Action Procedures*. SA-QA-05 provides contingencies for out-of-control data and gives guidance for exceptionally permitting departures from approved policies and procedures.

13.0 METHOD PERFORMANCE

The Reporting Limits (RL), the Method Detection Limits (MDL), and accuracy and precision limits associated with these methods are given in the Method Limit Groups in LIMS.

13.1 Initial and Continuing Demonstration of Capability

Initial and continuing demonstration of capability must be performed in accordance with SOP SA-QA-06*: Training Procedures.*

13.2 Method Detection Limit

The method detection limit must be determined for each analyte in accordance with SOP SA-QA-07: *Determination and Verification of Detection and Reporting Limits.*

14.0 PREVENTIVE MAINTENANCE AND TROUBLESHOOTING

Refer to the manufacturer's guides for trouble-shooting items.

15.0 WASTE MANAGEMENT AND POLLUTION PREVENTION

All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment.

15.1 Waste Streams Produced by the Method

The following waste streams are produced when this method is carried out.

- Excess samples, reagents, and standards must be disposed in accordance with the TestAmerica Savannah Addendum to the Environmental Health and Safety Manual.
- Non-hazardous acidic and alkaline wastewater and samples must be neutralized before disposal into the sewer system.
- Excess aqueous samples – Dispose according to characterization on the sample disposal sheets. Neutralize non-hazardous samples before disposal into drain/sewer. Transfer hazardous samples (identified on disposal sheets) to the waste department for disposal.

- Excess soil and solid samples – Dispose according to characterization on sample disposal sheets. Transfer non-hazardous samples to TCLP container for characterization in hazardous waste department. Transfer hazardous samples (identified on disposal sheets) to waste department for disposal.

- Waste containing pyridine must be isolated and transferred to the waste department for disposal.

16.0 REFERENCES

TestAmerica Savannah's *Quality Assurance Manual (QAM),* current revision

Standard Methods for the Examination of Water and Wastewater; Eighteenth Edition; American Public Health Association: Washington, DC, 1989.

Water Analysis Handbook; HACH Company, 1985

Methods for Chemical Analysis of Water and Wastes; U.S. EPA Office of Research and Development: Cincinnati, OH, March, 1983.

Test Methods for Evaluating Solid Waste, Third Edition; U.S. EPA Office of Solid Waste and Emergency Response: Washington, DC, November 1986.

17.0 TABLES, DIAGRAMS, AND VALIDATION DATA

The appendixes to this SOP contain QC summary tables and application notes relevant to this procedure.

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APPENDIX A- FERROUS IRON SOP SUMMARY AND APPLICATION NOTES

QC ITEM	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
Initial Calibration	Initially and when verification standards fail to meet acceptance criteria	Minimum three point curve with correlation coefficient >=0.995 and acceptable ICV	-reevaluate curve -re-prep and re-analyze one or more standards -prepare new stock, calibration standards, and re-analyze
Initial Calibration Verification(ICV)-independent source	After initial calibration	+/-10% of true value	-reevaluate calibration curve -re-prep and re-analyze ICV standard -re-prep ICV stock -re-calibrate
Continuing Calibration Verification (CCV)	After every 10 absorbance readings	+/-20% of true value	-reevaluate calibration curve -re-prep and re-analyze CCV standard -re-prep CCV stock -re-calibrate
Initial/Continuing Calibration Blank (ICB/CCB)	After verification standards	< RL in MLG	-reevaluate calibration curve -re-prep and re-analyze ICB/CCB standard -locate source of contamination and re-prep and re-analyze ICB/CCB
Lab control sample (LCS)	The ICV and CCV are prepared from a source independent of the calibration standards and is reported as the LCS.		
Matrix spike/Matrix spike duplicate (MS/MSD)	Per batch of 20 or fewer samples (1 MS and 1 MSD per 20 samples)	Recovery within LQM control limits	-report recoveries and flag results as having matrix interference -spike all samples associated with the MS/MSD and evaluate for matrix interferences -dilute the samples to minimize the matrix interferences, re-spike and reanalyze -analyze samples by MSA
Demonstration of Capability (DOC)	Initially per analyst, and then annually thereafter	SOP SA-QA-06	SOP SA-QA-06
Method Detection Limit (MDL)	Annually	SOP SA-QA-07	SOP SA-QA-07

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Ferrous Iron

Application Notes:

For the measurement of Ferrous iron in reducing conditions of ground and acid surface drainage. This analysis is based on SM3500-FeD.

Principle:

Ferrous iron is determined by reaction with phenanthroline in acid solution producing an orange color with a maximum absorbance of 510nm.

Stock Solutions:

Standard Ferrous iron solution:

Dissolve 0.7022g $Fe(NH_4)_2(SO_4)_2.6H_2O$ to 50mL reagent water. Acidify to pH <2 with 1:1 HCl and dilute to 100mL in volumetric glassware.

Working Solutions:

Ammonium acetate buffer

Dissolve 250g $NH_4C_2H_3O_2$ in 150 mL reagent water. Add 700 mL conc. (glacial) acetic acid. *Because NH4C2H3O2 contains a significant amount of iron, new reference standards must be made with each buffer preparation.*

Color Reagent

Dissolve 100 mg $C_{12}H_8N_2.H_2O$ (1,10-phenanthroline monohydrate) in 100 mL reagent water. Add 2 drops of conc. HCl.

Standard solutions

Prepare solutions in the required analytical range. e.g., 0, 0.050, 0.010, 0.020, 0.50, 1.0.

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APPENDIX B- CHLORIDE SOP SUMMARY AND APPLICATION NOTES

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Initial Calibration -minimum 5 points with lowest point @ RL; include zero as point	Before sample analysis, every six months, and when continuing calibration verification fail.	Regression curve with correlation coefficient >=0.995; initial calibration verification within +/-10% of expected value	-Evaluate curve and check calculations -Reanalyze standard(s) -Remake and reanalyze standard(s) -Inspect instrument for proper operation
Initial Calibration Verification (ICV) – independent source	After initial calibration	+/-10% of expected value	-Check calculation -Reanalyze ICV/CCV -Remake and reanalyze ICV/CCV -Recalibrate -Inspect instrument for proper operation
Calibration Verification (CCV)-mid range standard	After every 10 sample measurements	+/-10% of expected value	-Check calculation -Reanalyze ICV/CCV -Remake and reanalyze ICV/CCV -Recalibrate -Inspect instrument for proper operation
Initial Calibration Blank (ICB) and Continuing Calibration Blank (CCB)	After ICV and CCV	< RL in MLG	-Check calculations -Reanalyze ICB/CCB -Determine source of contamination and reanalyze samples if necessary
Method Blank -for analyses that require no preparation, the ICB/CCB are reported as the method blank	Per batch of 20 or fewer samples	< RL in MLG	-Check calculations -Reanalyze method blank -Determine source/cause of contamination and reanalyze associated samples if necessary
LCS	Per batch of 20 or fewer samples	+/- 15% of the expected value	-Check calculations -Reanalyze -SA-QA-17 Decision Matrix
MS/MSD	At a frequency of 5% of samples (one MS and one MSD per twenty samples)	LQM Appendices	-Check calculations -Reanalyze -SA-QA-17 Decision Matrix
Demonstration of Capability (DOC)	Initially per analyst, and then annually thereafter	-linearity criteria -recovery of QCS within 90-110% -determination of MDL	-Reanalyze IDOC
MDL Study	Annually	-SOP SA-QA-07	-evaluate according to SA-QA-07

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Chloride

Application Notes:

For the measurement of Chloride in potable, surface, saline and waste waters.

Principle:

Chloride reacts with mercuric thiocyanate forming a mercuric chloride complex. Released thiocyanate reacts with iron (III) forming a red ferric thiocyanate complex the color produced, measured at 480nm, is proportional to the chloride concentration.

Stock Solutions:

Mercuric Thiocyanate:
Thiocyanate [$Hg(SCN)_2$] dissolved in 1000ml Methanol. Mix and filter as necessary.

Ferric Nitrate:
Dissolve 202g Ferric Nitrate [$Fe(NO_3)_3.9H_2O$] in 800 ml distilled water. Carefully add 44.4ml of concentrated Nitric Acid. Dilute to 1000ml with distilled water. Store in an amber bottle.

Standard Chloride 1000mg/L:
Dissolve 1.6484g dried Sodium Chloride [NaCl] in 1000 ml of distilled water

Working Solutions:

Color Reagent
Dissolve 75ml of stock Mercuric Thiocyanate and 75ml of stock Ferric Nitrate to 500ml with distilled water.

Standard Chloride solutions
Prepare standard solutions in the required analytical range. e.g. 0, 10, 20, 40, 60, 80, 100 mg/l.

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APPENDIX C- SULFATE SOP SUMMARY AND APPLICATION NOTES

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Initial Calibration -minimum 5 points with lowest point @ RL; include zero as point	Before sample analysis, every six months, and when continuing calibration verification fail.	Regression curve with correlation coefficient >=0.995; initial calibration verification within +/-10% of expected value	-Evaluate curve and check calculations -Reanalyze standard(s) -Remake and reanalyze standard(s) -Inspect instrument for proper operation
Initial Calibration Verification (ICV) – independent source	After initial calibration	+/-10% of expected value	-Check calculation -Reanalyze ICV/CCV -Remake and reanalyze ICV/CCV -Recalibrate -Inspect instrument for proper operation
Calibration Verification (CCV)-mid range standard	After every 3-4 sample measurements	+/-10% of expected value	-Check calculation -Reanalyze ICV/CCV -Remake and reanalyze ICV/CCV -Recalibrate -Inspect instrument for proper operation
Initial Calibration Blank (ICB) and Continuing Calibration Blank (CCB)	After ICV and CCV	< RL in MLG	-Check calculations -Reanalyze ICB/CCB -Determine source of contamination and reanalyze samples if necessary
Method Blank -for analyses that require no preparation, the ICB/CCB are reported as the method blank	Per batch of 20 or fewer samples	< RL in MLG	-Check calculations -Reanalyze method blank -Determine source/cause of contamination and reanalyze associated samples if necessary
LCS	Per batch of 20 or fewer samples	+/- 25% of the expected value	-Check calculations -Reanalyze -SA-QA-17 Decision Matrix
MS/MSD	At a frequency of 5% of samples (one MS and one MSD per twenty samples)	Criteria in MLG	-Check calculations -Reanalyze -SA-QA-17 Decision Matrix
Demonstration of Capability (DOC)	Initially per analyst, and then annually thereafter	-linearity criteria -recovery of QCS within 90-110% -determination of MDL	-Reanalyze IDOC
MDL Study	Annually	-SOP SA-QA-07	-evaluate according to SA-QA-07

Sulfate

Application Notes:

For the measurement of Sulfate in potable, surface, and wastewaters.

Principle:

Sulfate ion is precipitated in a strongly acid medium with Barium Chloride. The resulting turbidity is measured photometrically at 405nm and compared with appropriate standard solutions.

Note: EPA 375.4 states to measure turbidities every 30 seconds for 1-5 minutes after the addition of barium chloride, with the maximum turbidity reading used for determining the sulfate concentration. The Konelab instrument performs parallel analyses of each sample, one for turbidity measurements and one for sulfate determination. The turbidity measurements are made 200 seconds after the color reagent is added and every 28 seconds after that for 240 seconds. The turbidity absorbances are presented on the raw data to indicate if the sample is settling out but do not affect how the sulfate result is determined. The sulfate determination is made 300 seconds after the color reagent is added. Although the Konelab instrument will allow multiple sulfate readings to be made, the instrument cannot select the result associated with the highest turbidity; rather, it will perform an average of the results obtained. This instrument configuration cannot be changed to use the sulfate reading taken from the highest turbidity replicate, and is therefore different from that described in the reference method. This is an allowable method modification as defined by EPA.

Stock Solutions:

Stock Sulfate Standard – 1000 mg/L as SO_4:
Dissolve 1.479g anhydrous Na_2SO_4 in distilled water and dilute to 1000mL.

Working Solutions:

Precipitating solution
Dissolve 10.0g of Barium Chloride [BaCl2] 20g Sodium Chloride [NaCl] and 0.5g Gelatin in 300mL of distilled water (requires prolonged stirring to dissolve.
Carefully add 5mL of conc. Hydrochloric acid and dilute to 1000mL with distilled water.

Standard Sulfate solutions
Prepare standard solutions in the required analytical range. e.g. 0, 5, 10, 15, 20, 25, 30, 35 mg/L.

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APPENDIX D- ortho-PHOSPHATE SOP SUMMARY AND APPLICATION NOTES

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Initial Calibration -minimum 5 points with lowest point @ RL; include zero as point	Before sample analysis, every six months, and when continuing calibration verification fail.	Regression curve with correlation coefficient >=0.995; initial calibration verification within +/-10% of expected value	-Evaluate curve and check calculations -Reanalyze standard(s) -Remake and reanalyze standard(s) -Inspect instrument for proper operation
Initial Calibration Verification (ICV) – independent source	After initial calibration	+/-10% of expected value	-Check calculation -Reanalyze ICV/CCV -Remake and reanalyze ICV/CCV -Recalibrate -Inspect instrument for proper operation
Continuing Calibration Verification (CCV)-mid range standard	After every 10 sample measurements	+/-10% of expected value	-Check calculation -Reanalyze ICV/CCV -Remake and reanalyze ICV/CCV -Recalibrate -Inspect instrument for proper operation
Initial Calibration Blank (ICB) and Continuing Calibration Blank (CCB)	After ICV and CCV	< RL in MLG	-Check calculations -Reanalyze ICB/CCB -Determine source of contamination and reanalyze samples if necessary
Method Blank -for analyses that require no preparation, the ICB/CCB are reported as the method blank	Per batch of 20 or fewer samples	< RL in MLG	-Check calculations -Reanalyze method blank -Determine source/cause of contamination and reanalyze associated samples if necessary
LCS	Per batch of 20 or fewer samples	+/- 10% of the expected value	-Check calculations -Reanalyze -SA-QA-17 Decision Matrix
MS/MSD	At a frequency of 5% of samples (one MS and one MSD per twenty samples)	MLG Appendices	-Check calculations -Reanalyze -SA-QA-17 Decision Matrix
Demonstration of Capability (DOC)	Initially per analyst, and then annually thereafter	-linearity criteria -recovery of QCS within 90-110% -determination of MDL	-Reanalyze IDOC
MDL Study	Annually	- SOP SA-QA-07	-evaluate according to SA-QA-07

O-Phosphate

Application Notes:

For the measurement of orthophosphate in potable, surface, saline and, wastewaters.

Principle:

Orthophosphate reacts with ammonium molybdate and antimony potassium tartrate under acidic conditions to form a complex which, when reduced with ascorbic acid, produces an intense blue color which is measured photometrically at 880nm (or at 660nm). The absorbance is proportionate to the amount of orthophosphate in the sample.

Stock Solutions:

Antimony potassium tartrate solution
Dissolve 0.3g $K(SbO)C_4H_4O_6.½\ H_2O$ in 50mL distilled water and dilute to 100mL. Store in a dark bottle and refrigerate.

Ammonium molybdate
Dissolve 4.0g $(NH_4)_6Mo_7O_{24}.4H_2O$ in 100mL of distilled. Store in a plastic container.

Dilute Sulphuric acid
Very slowly add 140ml of conc H_2SO_4 to 1000mL of distilled water. Cool.

Ascorbic Acid solution
Dissolve 1.76g ascorbic acid in 100mL of distilled water. This solution is stable for 5 days when refrigerated.

Standard Phosphate Solution – 1000mg/L as P
Dissolve 4.3936g anhydrous K_2PO_4 in 1000ml of distilled water

Working Solutions:

Reagent 1
Add 75mL of stock Ammonium Molybdate to 250mL of dilute Sulphuric acid. Ad 25mL of stock Antimony Potassium Tartrate to this mixture.

Reagent 2
Stock Ascorbic acid solution.

Mix 28mL of Reagent 1 with 12mL of Reagent 2 – prepare daily.

Standard solutions
Prepare standard solutions in the required analytical range. E.g. 0, 0.2, 0.4, 0.8, 1.2, 1.6, 2.0mg/L.

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APPENDIX E- AMMONIA SOP SUMMARY AND APPLICATION NOTES

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Initial Calibration -minimum 5 points with lowest point @ RL	Before sample analysis, every six months, and when continuing calibration verification fail.	Regression curve with correlation coefficient >=0.995; initial calibration verification within +/-10% of expected value	-Evaluate curve and check calculations -Reanalyze standard(s) -Remake and reanalyze standard(s) -Inspect instrument for proper operation
Initial Calibration Verification (ICV) – independent source	After initial calibration	+/-10% of expected value	-Check calculation -Reanalyze ICV/CCV -Remake and reanalyze ICV/CCV -Recalibrate -Inspect instrument for proper operation
Continuing Calibration Verification (CCV) -mid-range point from the calibration curve	After every 10 sample measurements	+/-10% of expected value	-Check calculation -Reanalyze ICV/CCV -Remake and reanalyze ICV/CCV -Recalibrate -Inspect instrument for proper operation
Initial Calibration Blank (ICB) and Continuing Calibration Blank (CCB)	After ICV and CCV	< RL in MLG	-Check calculations -Reanalyze ICB/CCB -Determine source of contamination and reanalyze samples if necessary
Reporting limit (RL) Standard-lowest standard in initial calibration curve	Daily-optional Required for Fla. DEP samples	Detected	-reanalyze -remake and reanalyze -recalibrate
Method Blank -for analyses that require no preparation, the ICB/CCB are reported as the method blank	Per batch	< RL in MLG	-Check calculations -Reanalyze method blank -Determine source/cause of contamination and reanalyze associated samples if necessary
LCS	Per batch	Criteria in MLG	-Check calculations -Reanalyze -SA-QA-17 Decision Matrix
MS/MSD	Per batch	Criteria in MLG	-Check calculations -SA-QA-17 Decision Matrix
Demonstration of Capability (DOC)	Initially per analyst, and then annually thereafter	-linearity criteria -recovery of QCS within 90-110% -determination of MDL	-Reanalyze samples
MDL Study	Annually	-SOP SA-QA-07	-reanalyze samples

Ammonia-N

Application Notes:

For the measurement of Ammonia in potable, surface, saline and, waste waters.

Principle:

Ammonia reacts with hypochlorite and alkaline phenol in the presence of sodium nitroprusside to form indophenol blue. The color produced is measured at 660nm.

Working Solutions:

Sodium Phenate solution
Dissolve 93mL liquid (≥89%) phenol in 500mL ammonia free water, slowly add 32g NaOH and mix thoroughly and dilute to 1000mL with ammonia free water. **CAUTION**: Wear gloves and eye protection when preparing this solution.

Sodium Hypochlorite solution
Dilute 250mL of 5.25% NaOCl with 250mL ammonia free water.

EDTA buffer
Dissolve 50g of disodium ethylenediamine tetraacetate and 5.5g of NaOH in 1000mL of ammonia free water.

Sodium Nitroprusside solution
Dissolve 0.5g of $Na_2(NO)FE(CN)_5.2H_2O$ in 1000mL of ammonia free water.

Standard Ammonia solutions
Dissolve 3.819g of dried Ammonium chloride [NH_4Cl] in 2000mL of ammonia free water.

Prepare standard solutions in the required analytical range. E.g. 0, 0.2, 0.4, 0.8, 1.2, 1.6, 2.0mg/L.

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APPENDIX F - NITRATE/NITRITE SOP SUMMARY AND APPLICATION NOTES

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Initial Calibration -minimum 5 points with lowest point @ RL; include zero as point	Before sample analysis, every six months, and when continuing calibration verification fail.	Regression curve with correlation coefficient >=0.995; initial calibration verification within +/-10% of expected value	-Evaluate curve and check calculations -Reanalyze standard(s) -Remake and reanalyze standard(s) -Inspect instrument for proper operation
Initial Calibration Verification (ICV) – independent source	After initial calibration	+/-10% of expected value	-Check calculation -Reanalyze ICV/CCV -Remake and reanalyze ICV/CCV -Recalibrate -Inspect instrument for proper operation
Calibration Verification (CCV)-mid range standard	After every 10 sample measurements	+/-10% of expected value	-Check calculation -Reanalyze ICV/CCV -Remake and reanalyze ICV/CCV -Recalibrate -Inspect instrument for proper operation
Initial Calibration Blank (ICB) and Continuing Calibration Blank (CCB)	After ICV and CCV	< RL in MLG	-Check calculations -Reanalyze ICB/CCB -Determine source of contamination and reanalyze samples if necessary
Method Blank -for analyses that require no preparation, the ICB/CCB are reported as the method blank	Per batch of 20 or fewer samples	< RL in MLG	-Check calculations -Reanalyze method blank -Determine source/cause of contamination and reanalyze associated samples if necessary
LCS	Per batch of 20 or fewer samples	+/- 20% of the expected value	-Check calculations -Reanalyze -SA-QA-17 Decision Matrix
MS/MSD	At a frequency of 5% of samples (one MS and one MSD per twenty samples)	Criteria MLG	-Check calculations -Reanalyze -SA-QA-17 Decision Matrix
Demonstration of Capability (DOC)	Initially per analyst, and then annually thereafter	-linearity criteria -recovery of QCS within 80-120% -determination of MDL	-Reanalyze IDOC
MDL Study	Annually	-SOP SA-QA-07	-evaluate according to SA-QA-07

Nitrate / Nitrite

Application Notes:

This method is applicable to the determination of nitrite singly, or nitrite and nitrate combined in drinking water, surface and saline waters, and domestic and industrial wastes. The applicable range of this method is 0.050 to 2.0mg/L nitrate-nitrite nitrogen. The range may be extended with simple dilution.

Principle:

Afiltered sample is passed through a column containing granulated copper-cadmium to reduce nitrate to nitrite. The nitrite (that originally present plus reduced nitrate) is determined by diazotizing with sulfanilamide and coupling with N-(1-naphthyl)-ethylenediamine dihydrochloride to form a highly colored azo dye, which is measured spectrophotometrically. Separate, rather than combine, nitrate-nitrite values are readily obtained by carrying out the procedure first with, and then without, the Cu-Cd reduction step.

Stock Solutions:

Nitrate Standards (1000mg/L NO3-N):
Dissolve 0.6068g $NaNO_3$ in reagent water and dilute to 100mL. For the second source standard, dissolve 0.7218g KNO_3 in reagent water and dilute to 100mL.

Nitrite Standards (1000mg/L NO2-N):
Dissolve 0.4926g $NaNO_2$ in reagent water and dilute to 100mL. For the second source standard, dissolve 0.6076g KNO_2 in reagent water and dilute to 100mL.

Working Solutions:

Ammonium Chloride-EDTA
Dissolve 13g of ammonium chloride and 1.7g of disodium ehtylenediamine tetraacetate in 900mL of reagent water. Adjust pH to 8.5 with concentrated ammonium hydroxide and dilute to 1000mL.

Color Reagent
Dissolve 10g sulfanilamide and 1.0g N-(1-napthyl) ethylene diamine dihydrochloride in a mixture of 100mL concentrated phosphoric acid and 800mL DI water and dilute to 1000mL.

Standards:

Prepare a series of standards by diluting suitable volumes of stock solution to 100mL with reagent water.

APPENDIX G - TOTAL PHOSPHORUS SOP SUMMARY AND APPLICATION NOTES

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Initial Calibration -minimum 5 points with lowest point @ RL; include zero as point	Before sample analysis, every six months, and when continuing calibration verification fail.	Regression curve with correlation coefficient >=0.995; initial calibration verification within +/-10% of expected value	-Evaluate curve and check calculations -Reanalyze standard(s) -Remake and reanalyze standard(s) -Inspect instrument for proper operation
Initial Calibration Verification (ICV) – independent source	After initial calibration	+/-10% of expected value	-Check calculation -Reanalyze ICV/CCV -Remake and reanalyze ICV/CCV -Recalibrate -Inspect instrument for proper operation
Calibration Verification (CCV)-mid range standard	After every 10 sample measurements	+/-10% of expected value	-Check calculation -Reanalyze ICV/CCV -Remake and reanalyze ICV/CCV -Recalibrate -Inspect instrument for proper operation
Initial Calibration Blank (ICB) and Continuing Calibration Blank (CCB)	After ICV and CCV	< RL in MLG	-Check calculations -Reanalyze ICB/CCB -Determine source of contamination and reanalyze samples if necessary
Method Blank -for analyses that require no preparation, the ICB/CCB are reported as the method blank	Per batch of 20 or fewer samples	< RL in MLG	-Check calculations -Reanalyze method blank -Determine source/cause of contamination and reanalyze associated samples if necessary
LCS	Per batch of 20 or fewer samples	+/-40% of the expected value	-Check calculations -Reanalyze -SA-QA-17 Decision Matrix
MS/MSD	At a frequency of 5% of samples (one MS and one MSD per twenty samples)	Criteria in MLG	-Check calculations -Reanalyze -SA-QA-17 Decision Matrix
Demonstration of Capability (DOC)	Initially per analyst, and then annually thereafter	-linearity criteria -recovery of QCS within 60-140% -determination of MDL	-Reanalyze IDOC
MDL Study	Annually	-SOP SA-QA-07	-evaluate according to SA-QA-07

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TOTAL PHOSPHORUS

Application Notes:

This procedure is applicable to the measurement of total phosphorus in potable, surface waters, and domestic and industrial wastes, which are first digested by the Kjeldahl digestion method. This analysis is based on EPA Region 9 SOP #596 and EPA method 365.4.

Principle:

Total phosphorus is converted to orthophosphate ion in the digestion and the colorimetric analysis is based on the reactions that are specific for the orthophosphate ion.

Stock Solutions:

Antimony Potassium Tartrate:
Dissolve 3.0g C8H4K2O12Sb2.H2O in 800mL reagent water and dilute to 1L in volumetric glassware. Store in a dark bottle and refrigerate.

Ammonium Molybdate:
Dissolve 40.0g (NH4)6Mo7O24.4H2O in 800mL reagent water and dilute to 1L in volumetric glassware. Store in a plastic container.

Phosphate Standard:
Dissolve 4.3936g anhydrous K2PO4 in 800mL reagent water and dilute to 1L in volumetric glassware.

Working Solutions:

Color Reagent
Add 35mL concentrated sulfuric acid, 213mL stock ammonium molybdate solution, and 72mL stock antimony potassium tartrate solution to 500mL reagent water. Dilute to 1L in volumetric glassware and mix.

Ascorbic Acid
Dissolve 6.0g C6H8O6 in 50mL reagent water and dilute to 100mL in volumetric glassware. Prepare fresh daily.

Sodium Chloride/Sodium Hydroxide
Dissolve 160g NaCl and 20g NaOH in 600mL reagent water. Let cool and dilute to 1L in volumetric glassware.

Standards:

Prepare a series of standards (e.g. 0.10, 0.30, 0.50, 1.0, 3.0, 5.0) by diluting suitable volumes of stock standard to 1L. Standards must be digested with each digest of samples before analysis.

Diluent:

In a 1-L volumetric, add 40mL concentrated sulfuric acid to 500mL DI water. Bring to volume with DI water and mix.

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APPENDIX H - TKN SOP SUMMARY AND APPLICATION NOTES

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Initial Calibration -minimum 5 points with lowest point @ RL; include zero as point	Before sample analysis, every six months, and when continuing calibration verification fail.	Regression curve with correlation coefficient >=0.995; initial calibration verification within +/-10% of expected value	-Evaluate curve and check calculations -Reanalyze standard(s) -Remake and reanalyze standard(s) -Inspect instrument for proper operation
Initial Calibration Verification (ICV) – independent source	After initial calibration	+/-10% of expected value	-Check calculation -Reanalyze ICV/CCV -Remake and reanalyze ICV/CCV -Recalibrate -Inspect instrument for proper operation
Calibration Verification (CCV)-mid range standard	After every 10 sample measurements	+/-10% of expected value	-Check calculation -Reanalyze ICV/CCV -Remake and reanalyze ICV/CCV -Recalibrate -Inspect instrument for proper operation
Initial Calibration Blank (ICB) and Continuing Calibration Blank (CCB)	After ICV and CCV	< RL in MLG	-Check calculations -Reanalyze ICB/CCB -Determine source of contamination and reanalyze samples if necessary
Method Blank -for analyses that require no preparation, the ICB/CCB are reported as the method blank	Per batch of 20 or fewer samples	< RL in MLG	-Check calculations -Reanalyze method blank -Determine source/cause of contamination and reanalyze associated samples if necessary
LCS	Per batch of 20 or fewer samples	+/-25% of the expected value	-Check calculations -Reanalyze -SA-QA-17 Decision Matrix
MS/MSD	At a frequency of 5% of samples (one MS and one MSD per twenty samples)	Criteria in MLG	-Check calculations -Reanalyze -SA-QA-17 Decision Matrix
Demonstration of Capability (DOC)	Initially per analyst, and then annually thereafter	-linearity criteria -recovery of QCS within 75-125% -determination of MDL	-Reanalyze IDOC
MDL Study	Annually	-SOP SA-QA-07	-evaluate according to SA-QA-07

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TOTAL KJELDAHL NITROGEN

Application Notes:

This method covers the determination of total Kjeldahl nitrogen in drinking, ground, and surface waters as well as domestic and industrial wastes. The procedure converts nitrogen components of biological origin such as amino acids, proteins, and peptides into ammonia, but does not convert the nitrogenous compound of some industrial wastes such as amines, nitro compounds, hydrazones, oximes, and semicarbonous and some refractory tertiary amines. The applicable range is 0.20 to 5.0mg/L TKN. The range may be extended with sample dilution.

Principle:

Total Kjeldahl nitrogen is the sum of free-ammonia and organic nitrogen compounds which are converted to ammonium sulfate under the conditions of digestion. The sample is heated in the presence of sulfuric acid. The resulting residue is cooled, diluted, and analyzed for ammonia. This digested sample may also be used for phosphorus determination.

Working Solutions:

0.80N NaOH:
Mix 32g NaOH and 900mL reagent water in a 1-L volumetric until dissolved. Dilute to volume with reagent water.

Buffer:
Add 35.0g Na2HPO4.7H2O to 500mL reagent water in a 1-L volumetric. Mix and add 20.0g disodium EDTA. Mix and add 50.0g NaOH. Mix until dissolved and bring to volume with reagent water.

Color Reagent:
Add 15.00g sodium salicylate and 0.100g sodium mitroprusside to 50mL reagent water in a 100-mL volumetric. Mix until dissolved and bring to volume with DI water. Make weekly or more often if necessary.

Hypochlorite:
Add 6.4mL commercial 5.25% sodium hypochlorite to a 100-mL volumetric. Bring to volume with DI water and mix well. Prepare fresh daily.

Diluent:
Add 40mL concentrated sulfuric acid to 500mL DI water in a 1-L volumetric. Bring to volume with DI water and mix.

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APPENDIX I- HEXAVALAENT CHROMIUM SOP SUMMARY AND APPLICATION NOTES

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Initial Calibration -minimum 5 points with lowest point @ RL; include zero as point	Before sample analysis, every six months, and when continuing calibration verification fail.	Regression curve with correlation coefficient >=0.995; initial calibration verification within +/-10% of expected value	-Evaluate curve and check calculations -Reanalyze standard(s) -Remake and reanalyze standard(s) -Inspect instrument for proper operation
Initial Calibration Verification (ICV) – independent source	After initial calibration	+/-10% of expected value	-Check calculation -Reanalyze ICV/CCV -Remake and reanalyze ICV/CCV -Recalibrate -Inspect instrument for proper operation
Calibration Verification (CCV)-mid range standard	After every 10 sample measurements	+/-10% of expected value	-Check calculation -Reanalyze ICV/CCV -Remake and reanalyze ICV/CCV -Recalibrate -Inspect instrument for proper operation
Initial Calibration Blank (ICB) and Continuing Calibration Blank (CCB)	After ICV and CCV	< RL in MLG	-Check calculations -Reanalyze ICB/CCB -Determine source of contamination and reanalyze samples if necessary
Method Blank -for analyses that require no preparation, the ICB/CCB are reported as the method blank	Per batch of 20 or fewer samples	< RL MLG	-Check calculations -Reanalyze method blank -Determine source/cause of contamination and reanalyze associated samples if necessary
LCS	Per batch of 20 or fewer samples	+/- 15% of the expected value for aqueous samples +/- 20% of the expected value for soils	-Check calculations -Reanalyze -SA-QA-17 Decision Matrix
MS/MSD	At a frequency of 5% of samples (one MS and one MSD per twenty samples)	+/- 15% of the expected value for aqueous samples +/- 20% of the expected value for soils	-Check calculations -Reanalyze -Spike or post spike all associated samples. Dilute samples as needed until MS recovery is satisfactory. Raise detection limits according to dilution. -SA-QA-17 Decision Matrix
Demonstration of Capability (DOC)	Initially per analyst, and then annually thereafter	-linearity criteria -recovery of QCS within 90-110% -determination of MDL	-Reanalyze IDOC
MDL Study	Annually	-SOP SA-QA-07	-evaluate according to SA-QA-07

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HEXAVALENT CHROMIUM

Application Notes:

For the determination of hexavalent chromium in water, wastewater, and soil extracts and digestates.

Principle:

The sample is acidified to a pH 1.5-2.5 and a color reagent is added (1,5-diphenyl carbazide). The intensity of the purple color, measured at 540nm, is proportional to the concentration of hexavalent chromium present in the sample.

Note: The laboratory uses an pH range of 1.5-2.5 as specified in SM3500-Cr B (On-line Edition) and EPA 7196A. SM3500-Cr D (20th Edition) states that the optimum pH range is currently being re-evaluated by the Standards Methods committee. Release of the newer On-line version with the 1.5-2.5 criteria indicates this range is appropriate for all methods.

Stock Solutions:

1000ppm Hexavalent Chromium Solution:
Purchased to Aldrich.

50ppm Hexavalent Chromium Second Source Stock:
Purchased from Hach.

Working Solutions:

Color Reagent:
Transfer 0.1250g of 1,5-diphenyl-carbaxide to a clean 25mL flask. Add about 10mL acetone ans swirl to dissolve. Dilute to the 25mL mark and mix well. Keep tightly capped and store in the dark.

50% Nitric Acid for pH Adjustment:
Slowly add 50mL of concentrated nitric acid to 50mL DI water. Mix carefully.

Standards:

Prepare standards in the working range, e.g. 0, 0.002, 0.010, 0.025, 0.050, 0.100, 0.300, and 0.500mg/L.

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(Methods: EPA 300.0, EPA 9056, EPA 9056A, and SM4110B)

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1.0 Scope and Application

This SOP gives the procedures for the determination of anions by ion chromatography. The routine matrices analyzed by this procedure are waters and soils. The routine analytes are bromide, chloride, fluoride, nitrate, nitrite, and sulfate. Nitrate and nitrite have a shorter holding time than the other anions and are calibrated independently. For the purposes of this SOP, the term "other anions" refers to bromide, chloride, fluoride, and sulfate.

Nitrate+nitrite, nitrate as N, and nitrite as N can also be reported using this procedure. Refer to Attachment 6 for additional information and the necessary conversions needed for reporting these analytes.

This SOP is most useful for the determination of dirty or highly colored samples that might be problematic for colorimetric determination, or in some instances where a lower limit of detection is required for certain analytes. This procedure may also be used for determining total halogens for heat of combustion determinations.

The reporting limits (RL), the method detection limits (MDL), and the accuracy and precision criteria associated with this procedure are provided in the LIMS Method Limit Groups (MLGs).

This SOP was written by and for TestAmerica's Savannah laboratory.

2.0 Summary of Method

Ion chromatography is a method for the separation of dissolved ionic species using liquid chromatography techniques, along with eluent suppression and conductivity detection. A potassium hydroxide eluent conducts the sample through the chromatography system, which consists of a guard column, a separator column, a suppressor, and a conductivity detector. The guard column screens out potential interferences, the separator column separates the anions, and the conductivity detector detects the anions from their electrical conductivity in solution.

Samples are filtered through a 0.45um filter prior to analysis to remove particulates that may interfere with column flow; therefore, the data may represent only the dissolved ionic species.

Soils are leached by tumbling 5g of soil with 100mL of reagent water. The soil is allowed to settle, and the leachate is filtered and analyzed for the target analytes.

Bomb combustates for the determination of total halogens are collected in reagent water, filtered, and analyzed for the target analytes. (Note: Currently combustates are analyzed for Cl only. Contact Technical Management if other halogens are requested.)

This SOP is based on the following methods: EPA 300.0, EPA 9056, EPA 9056A, and SM4110B.

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3.0 Definitions

Refer to the Glossary Section of the *Quality Assurance Manual* (QAM) for a complete listing of applicable definitions and acronyms.

4.0 Interferences

4.1 Procedural Interferences

4.1.1 Interferences may be caused by contaminants in solvents, reagents, glassware, and other sample processing apparatus and can make identification and/or quantification of the target analytes difficult.

4.1.2 All sample collection containers are single-use disposable containers which limits the potential for contamination. All non-disposable labware must be scrupulously cleaned in accordance with the posted Labware Cleaning Instructions to ensure it is free from contaminants and does not contribute artifacts.

4.1.3 Instrument and/or method blanks are routinely used to demonstrate all reagents and apparatus are free from interferences under the conditions of the analysis.

4.2 Matrix Interferences

4.2.1 Particles from samples may lodge in the system, causing blockage. Therefore, all samples are automatically filtered prior to analysis using the autosampler vial caps which contain 0.45um filters.

4.2.2 Matrix interferences may be caused by contaminants that are co-extracted from the sample matrix. If matrix interference is encountered, the sample may require dilution or another filtration with a 0.20um filter prior to introduction into the 0.45um filter vial caps. The associated batch QC items must also be filtered if any samples or reagents have undergone filtration.

4.2.3 Interfering contamination may occur when a sample containing low concentrations of analytes is analyzed immediately following a sample containing relatively high concentrations of analytes. As such, samples known to be clean should be analyzed first. To prevent carryover into subsequent samples, analysis of reagent blanks may be needed after the analysis of a sample containing high concentrations of analytes. Dionex NaOH wash solution can also be used to rinse the instrument to alleviate carryover.

4.2.4 Large quantities of ions eluting close to the anion of interest will also result in an interference. Separation can be improved by adjusting the eluent concentration and/or flow rate. Sample dilution can also be used. For example, high levels of organic acids that may interfere with inorganic anion analysis may be present in industrial wastes. Two common species, formate and acetate, elute between fluoride and chloride.

4.2.5 The acetate, formate, and other monovalent organic acid anions elute early in the chromatographic run and can interfere with fluoride and/or cause shifts in the retention time of the other anions.

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5.0 Safety

Employees must abide by the policies and procedures in the TestAmerica Environmental Health and Safety Manual (EHSM), the TestAmerica Savannah Addendum to the EHSM, and this document.

This procedure may involve hazardous materials, 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 to follow appropriate safety, waste disposal, and health practices under the assumption that all samples and reagents are potentially hazardous.

The analyst must protect himself/herself from exposure to the sample matrix. Many of the samples that are tested may contain hazardous chemical compounds or biological organisms. The analyst must, at a minimum, wear protective clothing (lab coat), eye protection (safety glasses or face shield), disposable latex or nitrile gloves, and closed-toe, nonabsorbent shoes when handling samples.

5.1 Specific Safety Concerns or Requirements

The toxicity or carcinogenicity of chemicals used in this method has not been precisely defined. Each chemical should be treated as a potential health hazard, and exposure to these chemicals should be minimized.

Potassium hydroxide is a severe corrosive. Contact with the skin can cause irritation or severe burns and scarring. Contact with the eyes can cause irritation, burns, permanent vision impairment or even blindness.

Exercise caution when using syringes with attached filter assemblies. Application of excessive force has, upon occasion, caused a filter disc to burst during the process.

5.2 Primary Materials Used

The following is a list of the materials used in this procedure, which have a serious or significant hazard rating, and a summary of the primary hazards listed in their MSDS.

NOTE: This list does not include all materials used in the procedure. A complete list of materials used in this procedure can be found in the Reagents and Standards Section and the Equipment and Supplies Section of this SOP

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 found using the "MSDS" link on the Oasis homepage, on the EH&S webpage on Oasis, and on the QA Navigator.

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Material	Hazards	Exposure Limit[1]	Signs and Symptoms of Exposure
Potassium Hydroxide	Corrosive	2mg/m^3 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]Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

6.1 Equipment and Instrumentation

Top-loading Balance – Verify in accordance with SOP SA-AN-100: *Laboratory Support Equipment (Verification and Use)*

Ion Chromatography System – Dionex ICS2000 (RFIC)

6.2 Analytical Data System / Software / Hardware

Chromeleon software is used on a Windows-based PC to schedule and acquire data. Target (UNIX and/or Windows) software is used on a Windows-based PC to store, reduce/evaluate, and output the data to the laboratory's LIMS system (i.e., TALS). Target software has the capability of processing data by recognizing a GC peak within any given retention time window. The software allows for calculation of response factors and or construction of a linear regression calibration curve, calculation of response factor statistics (mean and standard deviation), and calculation of concentrations of analytes using either the calibration curve or the response factors.

6.3 Lab Supplies

Guard Column(s) – Dionex AG18

Separator Column(s) – Dionex AS18

Suppressor – ASRS (Auto Self-regenerating Suppressor) 4mm

Volumetric Containers – various sizes; Class A, where applicable. Verify in accordance with SOP SA-AN-100: *Laboratory Support Equipment (Verification and Use)*

Mechanical Pipettes – various sizes. Verify in accordance with SOP SA-AN-100: *Laboratory Support Equipment (Verification and Use)*

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Syringe Filters – Nalgene, 0.20um pore size

Autosampler Vials – with 0.45um filter caps

DI Leach Tumbler

Detergent – FL-70, used for washing non-disposable labware.

6.4 Sample Collection Containers

All sample collection containers are single-use disposable containers which limits the potential for contamination.

The routine sample collection containers supplied by the laboratory are:

Water Samples:
125mL plastic oblong bottles – purchased with Certificate of Analysis attesting to purity.

Soil Samples:
8oz plastic soil jars – purchased with Certificate of Analysis attesting to purity.

7.0 Reagents and Standards

The standards and reagents listed are those in use at the time this SOP was updated. Other standard and reagent stocks, vendors, and concentrations may be used provided they are fully documented and in compliance with method requirements.

7.1 Expiration Dates

Expiration dates (time from initial use or receipt to final use) for standard and reagent materials must be set according to the guidance in this SOP. Note: These are maximum expiration dates and are not to be considered an absolute guarantee of standard or reagent quality. Sound judgment must be used when deciding whether to use a standard or reagent. If there is doubt about the quality of a standard or reagent material, a new material must be obtained or the standard or reagent material verified. Data quality must not be compromised to extend a standard's life – i.e., when in doubt, throw it out.

The expiration date of any standard or reagent must not exceed the expiration date of the standard or reagent that was used to prepare it; that is, the "children may not outlive the parents".

Note: As indicated in the Method Modification Section of this SOP, the default standard expiration dates used by the laboratory have been extended beyond those specified in the reference methods based on the results of a stability study. SC DHEC does not recognize this method modification and requires the method-defined holding times as listed in the SC DHEC Project Requirement Summary (PRS).

7.2 Disposal Information

Unless otherwise specified, all standards and reagents should be disposed of via the drain / sewer system.

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7.3 Reagents

Reagents must be prepared and documented in accordance with SOP SA-AN-041: *Reagent and Standard Materials Procedures.*

Blank Matrix – glass beads. Used for the preparation of soil QC samples.

Laboratory Reagent Water – ASTM Type II.

Dionex Elu Gen Cartridge – Potassium Hydroxide

7.4 Standards

Standards must be prepared and documented in accordance with SOP SA-AN-041: *Reagent and Standard Materials Procedures.* Certificates of analysis or purity must be received with all purchased standards, and scanned and filed in the Data Archival Folder on the G-drive.

First Source Other Anions Mix Source Standard – Inorganic Ventures
Concentration: 200mg/L fluoride; 1000mg/L chloride, bromide, sulfate)
Storage: Refrigerated at 0-6°C
Expiration:
Unopened: Manufacturer's expiration date
Opened: 1 year from date opened

Nitrite Source Standard (1000mg/L NO_2 or 304mg/L as N) – Inorganic Ventures
Storage: Refrigerated at 0-6°C
Expiration:
Unopened: Manufacturer's expiration date
Opened: 6 months from date opened

Nitrate Source Standard (1000mg/L NO_3 or 226mg/L as N) – Inorganic Ventures
Storage: Refrigerated at 0-6°C
Expiration:
Unopened: Manufacturer's expiration date
Opened: 6 months from date opened

Second Source Other Anions Mix Source Standard – Environmental Express
Concentration: 200mg/L fluoride; 1000mg/L chloride, bromide, sulfate
Storage: Refrigerated at 0-6°C
Expiration:
Unopened: Manufacturer's expiration date
Opened: 1 year from date opened

Second Source Nitrite Source Standard - 1000mg/L NO_2 or 304mg/L as N; Environmental Express
Storage: Refrigerated at 0-6°C
Expiration:
Unopened: Manufacturer's expiration date

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Opened: 6 months from date opened

Second Source Nitrate Source Standard - 1000mg/L NO_3 or 226mg/L as N; Environmental Express
Storage: Refrigerated at 0-6°C
Expiration:
Unopened: Manufacturer's expiration date
Opened: 6 months from date opened

Calibration Standards

The calibration standards are prepared as follows:

Nitrate and Nitrite (as N) Calibration Curve:

HICAL (4.0mg/L) – Add 13.16mL of Nitrite Source Standard and 17.70mL of Nitrate Source Standard to a 1L class A volumetric falsk. Bring to volume with Reagent Water.

Calibration Level	Parent Standard	Volume Added (mL)	Final Volume (mL)	Final Concentration[1] (mg/L)
1	CAL-5	25	500	0.05
2	CAL-5	10	100	0.10
3	CAL-5	25	100	0.25
4	CAL-7	25	100	0.50
5	CAL-7	250	500	1.0
6	CAL-7	75	100	1.5
7	HICAL	500	1000	2.0

[1]Concentrations listed are given in terms of the "as N" analytes.
Solvent: Reagent Water
Storage: Refrigerated at 0-6°C
Expiration: 1 month from preparation date

Other Anions Calibration Curve:

Calibration Level	Parent Standard	Volume Added	Final Volume (mL)	Final Concentration (mg/L)
1	Other Anions Mix Source Standard	500uL	500	0.20 (F) 1.0 (Br, Cl, SO4)
2	Other Anions Mix Source Standard	200uL	100	0.40 (F) 2.0 (Br, Cl SO4)
3	Other Anions Mix Source Standard	400uL	100	0.80 (F) 4.0 (Br, Cl SO4)
4	Other Anions Mix Source Standard	5mL	500	2.0 (F) 10 (Br, Cl SO4)
5	Other Anions Mix Source Standard	2mL	100	4.0 (F) 20 (Br, Cl SO4)

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6	Other Anions Mix Source Standard	5mL	100	10 (F) 50 (Br, Cl SO4)

Solvent: Reagent Water
Storage: Refrigerated at 0-6°C
Expiration: 3 months from preparation date
(Note: Bromide is used for NPW analyses only.)

ICV / Water LCS Standards

The ICV and all water LCS are prepared in the same way using the same source standards. A large volume is prepared, and is then ready for analysis as is (i.e., they are "pour and go" standards).

Other Anions ICV/LCS Standard

Parent Standard	Parent Concentration (mg/L)	Volume Added (mL)	Final Volume (mL)	Final Concentration (mg/L)
2nd Source Other Anions Source Standard	200 (F) 1000 (Br, SO4, Cl)	5	500	2 (F) 10 (Br, SO4, Cl)

Solvent: Laboratory Reagent Water
Storage: Refrigerated at 0-6°C
Expiration: 3 months from preparation date

Nitrate and Nitrite ICV/LCS Intermediate Standard (N-NSPK)

Parent Standard	Parent Concentration (mg/L)	Volume Added (mL)	Final Volume (mL)	Final Concentration[1] (mg/L)
2nd Source NO3 Source Standard 2nd Source NO2 Source Standard	1000 (NO3) 1000 (NO2)	22.1 16.4	50	100

[1]Concentrations listed are given in terms of the "as N" analytes.
Solvent: Laboratory Reagent Water
Storage: Refrigerated at 0-6°C
Expiration: 1 month from preparation date

Nitrate and Nitrite ICV/LCS Standard

Parent Standard	Parent Concentration (mg/L)	Volume Added (mL)	Final Volume (mL)	Final Concentration[1] (mg/L)
Nitrate and Nitrite ICV/LCS Intermediate Standard (N-NSPK)	100	5.0	500	1.0

[1]Concentrations listed are given in terms of the "as N" analytes.
Solvent: Laboratory Reagent Water
Storage: Refrigerated at 0-6°C
Expiration: 1 month from preparation date

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8.0 Sample Collection, Preservation, Shipment, and Storage

8.1 Aqueous Samples

Aqueous samples are routinely collected in 125mL oblong plastic containers.

Samples must be iced at the time of collection and maintained at 4°C (less than 6°C but not frozen) until the time of analysis. Samples for nitrate and/or nitrite must be analyzed within 48 hours of collection. Samples for the other anions must be analyzed within 28 days of collection.

8.2 Soil Samples

Soil samples are routinely collected in 8oz plastic soil containers.

Samples must be iced at the time of collection and maintained at 4°C (less than 6°C but not frozen) until the time of preparation. Samples for nitrate and/or nitrite must be prepared within 28 days of collection and analyzed within 48 hours of preparation. Samples for the other anions must be prepared and analyzed within 28 days of collection. Extracts must be stored at 4°C (less than 6°C but not frozen) until the time of analysis.

8.3 Combustate Samples

Once the heat of combustion procedure has been performed (in accordance with SOP GE105), the combustate is transferred to a 125mL plastic container. Combustates must be stored at 4°C (less than 6°C with no frozen samples) until the time of analysis. Combustate samples must be prepared and analyzed within 28 days of collection.

9.0 Quality Control

SOP SA-QA-17: *Evaluation of Batch QC Data* and the SOP Summary in Attachment 1 provide requirements for evaluating QC data.

Batch QC must meet the criteria given in Attachment 3 of this SOP.

9.1 Batch QC

9.1.1 Soil Batch QC

An extraction batch consists of up to 20 environmental samples and the associated QC items extracted (leached) together within a 24 hour period.

The laboratory's default QC items required for each extraction batch are: a method blank, a laboratory control sample (LCS), a matrix spike (MS), and a matrix spike duplicate (MSD). Additionally, for EPA 300.0, another matrix spike (MS) is required.

The routine container supplied for this method is an 8oz container. 5g is required for extraction. It is unlikely that reduced sample initial weights will be necessary to achieve the required batch matrix spike and sample duplicate frequency. If reduced initial weights are required, the minimum extraction weight to be used for the matrix spike samples or sample duplicates is 2.5g.

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Note: Final volumes and spike amounts must be adjusted to compensate for these reduced initial weights.

If there is insufficient sample provided to perform the required MS/MSDs, an NCM must be initiated on all affected samples to denote this situation. If an LCSD has not already been performed, an LCSD should be included in the batch. Insufficient sample is defined as receiving less than a total of 10g.

Note: If an LCS and LCSD are performed, both QC items must be evaluated and reported. Acceptable recoveries (as well as %RPD) for both LCS and LCSD are required.

9.1.2 Water Batch QC

An analytical batch consists of up to 20 environmental samples and the associated QC items analyzed together within a 24 hour period.

The laboratory's default QC items required for each batch are: a method blank, a laboratory control sample (LCS), matrix spike (MS), and an MSD. The following additional QC items are also required:

EPA 300.0 (DW and non-DW): an additional matrix spike
EPA 300.0 (DW only): a low-level LCS
SM4110B: a sample duplicate

Additionally, EPA 300.0 requires an LCSD to be performed quarterly. As there are no preparation steps associated with water samples, the CCV or ICV should satisfy this requirement.

Determination of Sufficient Volume
The routine container supplied for this method is a 125mL container. 5mL is required for analysis. Reduced sample initial volumes may be necessary to achieve the required batch matrix spike and sample duplicate frequency; however, the minimum analysis volume to be used for the matrix spike samples or sample duplicate is 2.5mL.

Note: Spike amounts must be adjusted to compensate for these reduced initial volumes.

If there is insufficient sample volume to perform the required MS/MSDs, the LCS must be performed in duplicate (i.e., LCS/LCSD). An NCM must be initiated on all affected samples to denote this situation. Insufficient sample volume is defined as receiving less than a total of 10mL.

Note: If an LCS and LCSD are performed, both QC items must be evaluated and reported. Acceptable recoveries (as well as %RPD) for both LCS and LCSD are required.

9.2 Instrument QC

9.2.1 Initial Calibration (ICAL)

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The instrument must be calibrated in accordance with SOP SA-QA-16: *Evaluation of Calibration Curves.* This SOP provides requirements for establishing the calibration curve and gives the applicable formulas.

Instrument calibration is performed by analyzing a series of known standards. The calibration curve must consist of a minimum of 3 standards and a blank. The lowest level calibration standard must be at or below the reporting limit, and the remaining standards will define the working range of the analytical system.

The initial calibration standard concentrations currently in use in the laboratory are given in Section 7.3.7. Refer to Section 7.3 for the standard preparation instructions. Other standard concentrations may be used provided they support the reporting limit and are fully documented in accordance with SOP SA-AN-041.

9.2.1.1 ICAL Criteria

The preferred method of calibration and quantitation is a linear regression curve. The regression coefficient (r^2) of the regression curve must be greater than 0.990 for the initial calibration curve to be acceptable.

Note: A minimum of 5 points is required.

Note: The calibration procedures outlined in EPA 9056 and EPA 9056A specify a linear curve to be used and do not incorporate average response factor or quadratic curves, as allowed by the other SW-846 chromatography methods. For this reason, average response factor and quadratic curves are not used for this procedure. As is standard laboratory practice, higher order curves are also prohibited.

LLOQ Verification

EPA 9056A requires verification of the Low-Level of Quantitation (LLOQ). Requant the low point of the calibration curve (or the point equivalent to the RL) as a sample. The concentration must be within 50-150% of the actual concentration to be acceptable.

9.2.2 Second Source Initial Calibration Verification (ICV)

The calibration curve must be verified initially – prior to any sample analyses – in accordance with SOP SA-QA-16 with a standard obtained from a second source.

The ICV must be within +/- 10% of the true value to be acceptable.

The initial calibration verification standard concentration currently in use in the laboratory is equivalent to Level 5 of the ICAL for nitrate and nitrite and to Level 4 of the ICAL for the other anions. Refer to Section 7.3 for the standard preparation instructions. Another standard concentration may be used provided it is mid-level and fully documented in accordance with SOP SA-AN-041.

9.2.3 Initial Calibration Blank (ICB) / Continuing Calibration Blank (CCB)

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The instrument must be shown to be free from contamination by the analysis of calibration blanks. Initial calibration blanks are analyzed immediately ~~after~~ before the ICV. Continuing calibration blanks are analyzed after each CCV.

Initial and continuing calibration blanks must be <MDL to be acceptable.

9.2.4 Continuing Calibration Verification

The initial calibration curve must be verified initially with a mid-level standard for EPA 300.0, EPA 9056, and EPA 9056A, and with a low-level standard (at the reporting limit) for SM4110B. Additionally, a mid-level CCV must be analyzed after every 10 samples for all methods.

The low-level CCV must be within 25% of the true value to be acceptable and the mid-level CCV must be within +/- 10% of the true value to be acceptable.

The mid-level continuing calibration verification standard concentration currently in use in the laboratory is equivalent to Level 5 of the ICAL for nitrate and nitrite and to Level 4 or 5 of the ICAL for the other anions. Refer to Section 7.3 for the standard preparation instructions. Another standard concentration may be used provided it is mid-level and fully documented in accordance with SOP SA-AN-041.

9.2.5 Linear Calibration Range / Linear Dynamic Range

The linear calibration range (LCR), also known as the Linear Dynamic Range (LDR), must be determined initially for SM4110B and EPA 300.0 and verified every 6 months thereafter for EPA 300.0.

The initial demonstration of linearity uses as many standards as necessary to insure the curve is linear. The standards should cover the low and high ends of the calibration range. Each standard must be within +/- 10% of the true value.

The continued demonstration of linearity consists of a minimum of 3 standards and a blank. For the linearity to be verified, the standards must be within +/- 10% of the initial value to be acceptable. If this criterion is not met, the initial demonstration of linearity must be performed.

Note: This study is not meant to extend the linear range of the calibration. If an analyte is measured at a concentration greater than the highest calibration standard, the sample must be analyzed at a dilution to bring the target analyte into the range of the initial calibration.

9.3 Corrective Action for Out-of-Control Data

When the quality control parameters do not meet the criteria set forth in this SOP, corrective action must be taken in accordance with SOP SA-QA-05: *Preventive and Corrective Action Procedures* and the QC Summary Table in Attachment 3. SOP SA-QA-05 provides contingencies for out-of-control data and gives guidance for exceptionally permitting departures from approved policies and procedures. Nonconformance Memos must be initiated to document all instances where QC criteria are not met and all departures from approved policies and procedures.

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10.0 Procedure

10.1 Sample Preparation

10.1.1 Soil Sample Preparation

Soil samples are prepared/extracted using a DI Leach procedure. Soil samples must be homogenized prior to preparation in accordance with SOP SA-QA-15: *Compositing, Homogenization, and Segregation of Samples.*

Remove the samples from the refrigerator and allow them to come to room temperature.

Using a top-loading balance, weigh 4.90-5.10g of the homogenized sample and transfer to a 125mL plastic container. Record the weight of the sample to the nearest 0.01g. Add 100mL of laboratory reagent water to each sample. Tumble the samples at approximately 18rpm for 2 hours +/- 15 minutes. Remove the extracts from the tumbler and allow the sediment to settle. Store extracts at 4°C (less than 6°C but not frozen) until the time of analysis.

When ready to analyze, remove the extracts from the refrigerator and allow them to come to room temperature. Using a disposable syringe, remove a portion of the supernatant liquid and filter through a 0.20um filter. Collect the filtrate in an autosampler tube.

10.1.2 Water Sample Preparation

Remove the samples from the refrigerator and allow them to come to room temperature.

If the sample contains color, turbidity, or particulates, the sample is filtered through a 0.20um filter prior to transferring to the autosampler vial.

Note: If the sample requires filtration through a 0.20um filter, the batch method blank and LCS must also be filtered through a 0.20um filter.

All drinking water samples are initially analyzed straight. To protect the integrity of the instrument, all non-drinking water samples are initially analyzed at a 1:5 dilution.

10.1.3 Combustate Sample Preparation

Combustate samples are prepared in the same manner as water samples, discussed above.

10.2 QC Sample Preparation

10.2.1 Soil QC Sample Preparation

Refer to Section 9.1.1 for the batch QC frequency.

The method blank and the LCS are prepared using glass beads to simulate the sample matrix. The MS and MSD or sample duplicate is prepared using one of the samples in the batch.

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10.2.1.1 Method Blank – Use 5.0g glass beads for the blank matrix and extract as described in Section 10.1.1.

10.2.1.2 Laboratory Control Sample (LCS) and Laboratory Control Sample Duplicate (LCSD) – Use 5.0g glass beads for the blank matrix, add 1.0mL of the applicable 2nd Source Standard to the LCS and LCSD, and extract as described in Section 10.1.1.

To prepare the LCS and LCSD for nitrate and nitrite, use the Nitrate and Nitrite ICV/LCS Standard. To prepare the LCS and LCSD for the other anions, use the 2nd Source Other Anions Source Standard.

10.2.1.3 Matrix Spike (MS) and Matrix Spike Duplicate (MSD) – Add 4.0mL of the applicable 2nd Source Standard to the separate 5.0g aliquots of the sample chosen for the MS and MSD. Extract as described in Section 10.1.1.

To prepare the MS and MSD for nitrate and nitrite, use the use the Nitrate and Nitrite ICV/LCS Standard. To prepare the MS and MSD for the other anions, use the 2nd Source Other Anions Source Standard.

10.2.2 Water QC Sample Preparation

Refer to Section 9.1.2 for the batch QC frequency.

The method blank and the LCS are prepared using reagent water. The MS and MSD or sample duplicate is prepared using one of the samples in the batch.

10.2.2.1 Method Blank – Use 5.0mL laboratory reagent water for the blank matrix. Note: If the associated samples are filtered using a 0.20um filter, the method blank must also be filtered.

10.2.2.2 Laboratory Control Sample (LCS) and Laboratory Control Sample Duplicate (LCSD) – Transfer 5.0mL of the applicable ICV/LCS Standard into an autosampler vial. Note: If the associated samples are filtered using a 0.20um filter, the LCS and LCSD must also be filtered.

10.2.2.3 Low-Level Laboratory Control Sample (LLCS) – Prepare a 1:10 dilution of the applicable ICV/LCS Standard by adding 0.5mL standard into a final volume of 5mL reagent water. (Note: ICAL Level 1 may also be used to satisfy this standard.) This dilution gives the following concentrations: 0.20mg/L (F), 1.0mg/L (Br, SO4, Cl), 0.05 mg/L (NO2 as N) and 0.050 mg/L (NO3 as N). Note: If the associated samples are filtered using a 0.20um filter, the LCS and LCSD must also be filtered.

10.2.2.4 Matrix Spike (MS) / Matrix Spike Duplicate (MSD) – Add 50uL of the applicable Matrix Spike Standard to the separate 5.0mL aliquots of the sample chosen for the MS and MSD.

10.2.3 Combustate QC Sample Preparation

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Combustate batch QC is prepared during the combustion step of the process. Total sulfur is usually analyzed via Konelab along with the total halogen (chloride) performed via IC; therefore, the batch QC is used for both methods. The LCS is spiked at a higher concentration for the total sulfur analysis and must be diluted 1:25 prior to analyzing via IC. Rinsates are usually performed after each injection as well.

10.3 Analysis

10.3.1 Instrument Operating Conditions

The instrument conditions listed in this SOP are provided for guidance purposes. The actual conditions used by the laboratory may be slightly different from those listed here and must be documented in the instrument maintenance log, data system, and/or run log.

Instrument maintenance must be performed in accordance with Attachment 4 of this SOP.

The goal is to have maximum separation between the target compounds in the shortest run time while maintaining sufficient sensitivity to detect the target compounds at the reporting limit and MDL (if required).

Example IC Parameters

Instrument Parameter	Configuration
IC Mode	Gradient / Isocratic
Column	AS18, 4mm
Guard	AG18, 4mm
Sample Loop	25uL
Eluent	KOH EluGen 12-25mM gradient for NO2/NO3 44mM isocratic for the other Anions
SRS Current	112mA
Pressure	≈ 2000-2800psi
Suppressor	4mm ASRS
Conductivity	0.4-2.0uS
Run Time	10-12min

10.3.1.1 Determination of Retention Time Windows

The procedure for the determination of retention time windows is given in SOP SA-QA-08: *Evaluation of Chromatographic Data*. Retention time windows (RTW), i.e., the length of time the instrument will scan for the analyte, must be established initially upon instrument set-up and verified quarterly.

Retention times (RT), i.e., the elution time of the analyte, are verified daily with the analysis of the ICAL or CCV. The retention time for the CCV must fall within the daily retention time window as defined in SOP SA-QA-08.

10.3.2 Initial and Continuing Calibration

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Calibrate the instrument using the standards and criteria described given in Section 9.2.1. Once the calibration has been established and verified with an ICV in accordance with Section 9.2.2, sample analysis may proceed.

Verify the calibration curve with a continuing calibration verification using the standards and criteria described given in Section 9.2.4.

10.3.3 Sample Analysis

Remove the samples or extracts from the refrigerator and allow them to come to room temperature.

The sample or extract must be injected using the same injection volume used for the calibration standards. Samples that are known to be relatively clean should be analyzed first. Samples suspected of containing high concentrations should be analyzed last. Instrument blanks may be analyzed after suspected high concentration samples to allow the detector response to stabilize.

The default procedure is to exclude QC items (method blank and LCS) in determining the maximum number of samples in the clock.

10.3.4 Example Analytical Sequence

An example analytical sequence is listed in Attachment 1.

11.0 Calculations / Data Reduction

11.1 Data Reduction

Data evaluation must be performed in accordance with SA-QA-08: *Evaluation of Chromatographic Data*. This SOP includes specific information regarding the evaluation of chromatographic data, including the requirements for performing manual integrations and the evaluation of retention times.

Data must be evaluated in accordance with SOP SA-QA-02: *Data Generation and Review*.

11.1.1 Target Analyte Identification

The judgment and experience of the analyst and his/her colleagues are important factors in the evaluation of chromatographic data. Inspect each chromatogram to ensure that the peaks are properly identified and that the correct areas have been associated with the corresponding standard peak RT in the data system tabulation.

The evaluation of chromatograms for target analytes must take into account the calibration of the analytical system (initial and continuing calibration response and retention times); whether the peak response falls within the working range of the calibration; and the integration of the peaks. The analyst must also take into account the results from the method blank and laboratory control sample before reporting quantitative

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data. SOP SA-QA-08: *Evaluation of Chromatographic Data* provides additional guidance for the evaluation of chromatographic data.

11.1.2 Manual Integrations

Manual integrations must be documented in accordance with SOP SA-QA-08. Data systems should be adjusted to minimize operator intervention. All chromatographic peaks must be evaluated for overall peak shape and "reasonableness" of integration. Under no circumstances should manual integrations be used to change reasonable data system integrations in order to meet calibration or QC criteria.

11.1.3 Dilutions

If the analyst notices any odor or color and historical data is not available, the analyst shall use their professional judgment for deciding an initial dilution. If the sample requires that more than one anion be reported, the sample shall be diluted so that only the highest target analyte meets or exceeds the midpoint of the calibration curve.

When reporting results from dilutions, appropriate data flags must be used or qualification in a case narrative provided to the client.

For clients who require we provide lower detection limits, a general guide would be to report the dilution detailed above and one additional run at a dilution factor 1/10 of the dilution with the highest target in the upper half of the calibration curve. For example, if samples analyzed at a 1/50 dilution resulted in a target in the upper half of the calibration curve, the sample would be analyzed at a dilution factor of 1/5 to provide lower reporting limits.

11.1.4 Historical Data

Many of the laboratory's clients submit samples for repeat monitoring purposes. LIMS Worksheet Notes and the Historical Data Tracker feature should be used to determine if historical data is available for review.

11.1.5 Chemical Relationships

When available, the following chemical relationships must be evaluated for each sample. If these relationships are not met the Department Manager must be contacted immediately.

- NO3+NO2 > Component analytes
- Refer to Attachment 6 for conversions of nitrate and nitrite to the "as N" analytes

11.1.6 Drinking Water Compliance Evaluation

Public water suppliers (PWS) are governed by EPA-specified Maximum Contaminant Levels (MCL) above which indicates noncompliance. The MCLs associated with this procedure are given in Attachment 5.

11.1.7 Matrix Spike Evaluation

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In the event that an MS/MSD fails high for nitrate and low for nitrite the original sample should be checked for an oxidizing agent. 5mL of sample is poured into a vial and 1 unit of "DPD FREE CHLORINE POWDER POP" is added to the vial. In the event that the sample turns a pink color a nonconformance memo is initiated and the samples are reported.

11.2 Calculations

11.2.1 The calculations associated with batch QC determinations are given in SOP SA-QA-17. Applicable calculations include accuracy (% recovery) and precision (%RPD).

11.2.2 The calculations associated with initial and continuing calibrations and are given in SOP SA-QA-16. Applicable calculations include determination for: calibration factor, standard deviation, relative standard deviation, relative response factor, and relative standard deviation.

11.2.3 The calculation to determine final concentration is given as follows:

$$FinalConcentration = CONC_{Sample} \otimes \frac{F}{I \times dw} \otimes D$$

Where:

$CONC_{Sample}$= Concentration of the sample
F = Final volume/weight
I = Initial volume/weight
D = Dilution factor
dw = % Solids decimal equivalent

Note: This calculation assumes all applicable unit correction factors are applied.

Note: All dry weight corrections are performed automatically in LIMS.

12.0 Method Performance

12.1 Reporting Limit Verification (RLV)

At a minimum, RLVs must be performed initially upon method set-up in accordance with SOP SA-QA-07: *Determination and Verification of Detection and Reporting Limits*.

For analytes and methods certified by DOD ELAP, RLVs must also be performed quarterly thereafter. For all other analytes and methods, RLVs must also be performed annually thereafter. Exceptions may be made for project-specific non-routine analytes.

12.2 Method Detection Limit (MDL) Study

The 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. 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 current MDLs associated with this procedure are given in the Method Limit Group (MLG) in TALS.

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At a minimum, MDL Studies must be performed initially upon method set-up in accordance with SOP SA-QA-07: *Determination and Verification of Detection and Reporting Limits*.

Note: MDL Studies are not required for non-routine analytes provided results are not reported below the RL (i.e., MDL equals RL in TALS).

12.3 Method Detection Limit Verification (MDLV)

At a minimum, MDLVs must be performed initially upon method set-up in accordance with SOP SA-QA-07: *Determination and Verification of Detection and Reporting Limits*.

For analytes and methods certified by DOD ELAP, MDLVs must also be performed quarterly thereafter. For all other analytes and methods, MDLVs must also be performed annually thereafter.

Note: MDLVs are not required for non-routine analytes provided results are not reported below the RL (i.e., MDL equals RL in TALS).

12.4 QC Limit Generation, Control Charting, and Trend Analysis

The control limits for the batch QC items (LCS, MS/MSD) for this procedure are specified in the reference method and cannot be broadened; therefore, the laboratory defaults to the method-defined limits and does not utilize in-house or laboratory-derived limits for the evaluation of batch QC items.

Although the laboratory must default to the method-defined QC limits, control charting is a useful tool and is performed to assess analyte recoveries over time to evaluate trends. Control charting must be performed periodically (at a minimum annually) in accordance with SOP SA-QA-17: *Evaluation of Batch QC Data.*

12.5 Demonstrations of Capability

Initial and continuing demonstration of capability must be performed in accordance with SOP SA-QA-06*: Training Procedures.*

Prior to performing this procedure unsupervised, each new analyst who performs this analysis must demonstrate proficiency per method/analyte combination by successful completion of an initial demonstration of capability.

IDOCs are performed by the analysis of 4 consecutive LCS that meet the method criteria for accuracy and precision. The LCS must be from a second source than that used to prepare the calibration standards. The IDOC must be documented on the IDOC Form shown in SOP SA-QA-06 with documentation routed to the QA Department for filing.

Annual continuing demonstrations of capability (CDOCs) are also required per analyst per method/analyte combination. The CDOC requirement may be met by the consecutive analysis of four LCS all in the same batch, by the analysis of four LCS analyzed in four consecutive batches (in different batches on different days), via acceptable results on a PT study, or analysis of client samples with statistically indistinguishable results when compared to another certified analyst. The CDOC must be documented and routed to the

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QA Department for filing.

12.6 Training Requirements

All training must be performed and documented in accordance with SOP SA-QA-06: *Training Procedures.*

Note: The SOPs listed in the Reference/Cross-Reference Section are applicable to this procedure. All employees performing this procedure must also be trained on these SOPs, and/or have a general understanding of these procedures, as applicable.

13.0 Pollution Control

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (e.g., examining recycling options, ordering chemicals based on quantity needed, preparing reagents based on anticipated usage and reagent stability, etc.). Employees must abide by the policies in Section 13 of the Environmental Health and Safety Manual and the Savannah Addendum to the EHSM.

This procedure has been evaluated for opportunities to minimize the waste generated. Where reasonably feasible, pollution control procedures have been incorporated.

14.0 Waste Management

Waste management practices must be conducted consistent with all applicable federal, state, and local rules and regulations. All waste (i.e., excess reagents, samples, and method process wastes) must be disposed of in accordance with the TestAmerica Savannah Addendum to the EHSM. Waste description rules and land disposal restrictions must be followed.

14.1 Waste Streams Produced by the Method

The following waste streams are produced when this method is carried out:

- Excess aqueous samples – Dispose according to characterization on the sample disposal sheets. Neutralize non-hazardous samples before disposal into drain/sewer. Transfer hazardous samples (identified on disposal sheets) to the waste department for disposal.

- Excess soil and solid samples – Dispose according to characterization on sample disposal sheets. Transfer non-hazardous samples to TCLP container for characterization in hazardous waste department. Transfer hazardous samples (identified on disposal sheets) to waste department for disposal.

15.0 References / Cross-References

- SOP SA-AN-041: *Reagent and Standard Materials Procedures*
- SOP SA-AN-100: *Laboratory Support Equipment (Verification and Use)*
- SOP SA-QA-02: *Data Generation and Review*
- SOP SA-QA-05: *Preventive and Corrective Action Procedures*

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- SOP SA-QA-06: *Training Procedures*
- SOP SA-QA-07: *Determination and Verification of Detection and Reporting Limits (RLs, MDLs, and IDLs)*
- SOP SA-QA-08: *Evaluation of Chromatographic Data*
- SOP SA-QA-15: *Homogenization, Compositing, and Segregation of Samples*
- SOP SA-QA-16: *Evaluation of Calibration Curves*
- SOP SA-QA-17: *Evaluation of Batch QC Data*
- TestAmerica Savannah Quality Assurance Manual
- TestAmerica Environmental Health and Safety Manual
- TestAmerica Savannah Addendum to the Environmental Health and Safety Manual
- Method 300.0 Determination of Inorganic Anions by Ion Chromatography; Revision 2.1, Methods for the Determination of Inorganic Substances in Environmental Samples, August 1993.
- *Test Methods for Evaluating Solid Waste, Third Edition, updates III and IV*; U.S. EPA Office of Solid Waste and Emergency Response: Washington, DC.
    - Method 9056 *Determination of Inorganic Anions by Ion Chromatography*; Revision 0; September 1994. (Update III)
    - Method 9056A *Determination of Inorganic Anions by Ion Chromatography*; Revision 1; February 2007. (Update IV)
- *Standard Methods for the Examination of Water and Wastewater, Online Edition;* American Public Health Association: Washington, DC.
    - SM4020: Quality Assurance/Quality Control
    - SM4110B: *Determination on Anions by Ion Chromatography; Ion Chromatography with Chemical Suppression of Eluent Conductivity*; 2000

16.0 Method Modifications and Clarifications

16.1 Incorporation of Other Matrices

This procedure may be modified to analyze other, non-routine matrices (i.e., waste, wipe, tissue, or filter samples) based on the needs of the client. This must be arranged by the Project Manager at the initiation of the project to ensure thorough client and laboratory understanding of any procedural or QC modifications that must be made to accommodate the matrix. The laboratory will use its routine soil RLs (converted for initial and final volumes, etc.), and soil QC limits to evaluate non-routine matrices. Soil DOCs can be used to satisfy analyst demonstrations of capability for these types of non-routine matrices.

16.2 Other Considerations

16.2.1 For EPA 9056 and EPA 9056A, the laboratory has adopted the soil preparation technique from EPA Method 300.0 with the following modification. For soil extraction, Section 11.7 of EPA Method 300.0 recommends using an amount of laboratory reagent water equal to 10 times the soil weight and mixing with a magnetic stirrer for 10 minutes to perform the extraction. The laboratory performs a DI Leach procedure using laboratory reagent water equal to 20 times the soil weight and tumbling for 2 hours.

16.2.2 EPA 9056 and EPA 9056A do not provide a holding time for fluoride, chloride, bromide, or sulfate. The laboratory has adopted the holding times specified in EPA 300.0. Additionally, the reference methods do not specify a holding time for the soil leaching

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procedure. The laboratory defaults to a 28-day holding time from sample collection to preparation and a 48-hour holding time from sample preparation to analysis for nitrate and nitrite, and a 28-day holding time from sample collection to preparation and analysis for the other anions.

16.2.3 EPA 9056 and EPA 9056A recommend using eluent to dilute samples. The laboratory uses reagent water to perform sample dilutions.

16.2.4 EPA 9056, EPA 9056A, and EPA 300.0 recommend using a carbonate/bicarbonate eluent. Due to advances in instrumentation, the laboratory uses a potassium hydroxide eluent.

16.2.5 EPA 9056 and EPA 9056A requires corrective action be taken for any values detected in the instrument blank and method blank greater than 10% of the reporting limit. The laboratory's RLs are usually within a factor of 3-5 times the MDL, and the laboratory does not report values below its MDL. For these reasons, the laboratory has adopted the criteria that the instrument blank and method blank must be less than the MDL. This requirement is consistent with the requirement found in EPA 300.0.

16.2.6 Section 9.2.4 of EPA Method 300.0 requires a Method Detection Limit (MDL) study to be performed every 6 months or when a new analyst begins. The laboratory performs an MDL study every six months or when a significant change is made to the equipment (ex: new column/guard column type, new instrument, etc.). The laboratory does not use analyst-specific MDLs; therefore, new analysts are required only to perform IDOC as described in Section 12.3.

16.2.7 The calibration procedure outlined in EPA 9056 and EPA 9056A specifies a linear curve to be used and does not incorporate average response factor and/or quadratic curves, as allowed by the other SW-846 chromatography methods. For this reason, average response factor, quadratic curves, and higher order curves are prohibited in this SOP. If needed, and with Department Manager approval, average response factor and quadratic curves may be used for EPA 300.0 and SM4110B.

16.2.8 The standard expiration dates used by the laboratory have been extended beyond those specified in the reference methods. A stability study has been performed and shows the standards are acceptable for use for the timeframes specified in this SOP. Note: SC DHEC does not recognize this method modification and requires the method-defined holding times as listed in the SC DHEC Project Requirement Summary (PRS). (Stock Standards: 1 month, Prepared Standards: 1 day for NO2, and 1 week for all other anions)

16.2.9 The EPA Manual for the Certification of Laboratories Analyzing Drinking Water requires a LFB at the MRL to be performed each day. The laboratory meets this requirement by preparing an LCS at the RL in each EPA 300.0 batch of drinking water samples.

16.2.10 EPA 9056 states that the daily initial CCV must be within 10% to be acceptable and subsequent CCVs must be within 5% to be acceptable. Due to the nature of the sample matrices encountered, the laboratory incorporates the 10% criteria for all CCVs. This criteria is consistent with that defined by EPA 9056A. Additionally, CCV response is verified against the CCV level of the initial calibration curve.

16.2.11 EPA 300.0 requires a Linear Calibration Range to be determined initially and verified every 6 months thereafter. This study is not meant to extend the linear range of the calibration. If an analyte is measured at a concentration greater than the highest calibration standard, the sample must be analyzed at a dilution to bring the target analyte into the range of the initial calibration.

16.2.12 The laboratory's default batch QC items are outlined in Section 9.1. Due to the complexity of the laboratory's work mix (i.e., types of samples, varying state- and client- requirements, etc.) and to aid in scheduling and combining like methods, these default requirements may be more extensive than those specified by the reference methods. For example, the laboratory has adopted the EPA Clean Water Act requirement to perform an MSD per batch for all EPA 300.0 non-drinking water samples. To satisfy client- and/or regulatory expectations for batch precision, an LCSD may be performed when insufficient sample is provided for MS and/or MSD (although not required by some of the methods). Also, to note, matrix spike duplicates are routinely performed for EPA 9056 and EPA 9056A in lieu of sample duplicates (as allowed by the reference methods). If needed, and with Department Manager approval, these additional QC items may be omitted to match those listed within the reference methods. Additionally, where method-specified control limits have not been specified, the laboratory uses default and/or in-house limits.

16.2.13 To aid in scheduling and combining like methods, the laboratory has adopted a tighter CCV frequency for EPA 300.0. That is, EPA 300.0 only requires CCV after every 20 samples; however, the laboratory routinely performs CCV after every 10 samples as required by EPA 9056 and EPA 9056A. If needed, and with Department Manager approval, this frequency may be reduced for EPA 300.0 to match that listed in the reference method.

16.2.14 Per client request, the laboratory has incorporated iodate and iodide into EPA 300.0. Similar procedures and acceptance criteria have been adopted to mirror the other analytes specific in the reference method. A 28-day holding time has been applied.

16.2.15 The SM4110B reference method states to perform an initial calibration daily and whenever CCV is not acceptable. The response of the instrument is very stable; therefore, the laboratory only performs re-calibration upon unacceptable CCV.

16.2.16 The SM4110B reference method states that the MDL should be determined annually but does not mandate a specific procedure to be used. The laboratory performs MDL Studies initially, in accordance with 40CFR Part 136B, and re-establishes the MDL annually or quarterly (as dictated by agency-specific requirements) via an MDLV study.

16.2.17 The reference methods allow the use of 3 calibration points for the initial calibration; however, the laboratory typically performs a minimum of 5 points. 3 points may be used with Technical Manager approval.

17.0 Attachments

The following Tables, Diagrams, and/or Validation Data are included as Attachments:

Attachment 1: SOP Summary

Attachment 2: Sample Collection, Preservation, and Holding Time Table
Attachment 3: QC Summary
Attachment 4: Instrument Maintenance and Troubleshooting
Attachment 5: Maximum Contaminant Level (MCL) Table
Attachment 6: Additional Information Regarding NO3 and NO2 Reporting

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Attachment 1:
SOP Summary

Sample Preparation and Analysis Summary

Samples are analyzed via ion chromatography using eluent suppression and conductivity detection. A potassium hydroxide eluent conducts the sample through the chromatography system, which consists of a guard column, a separator column, a suppressor, and a conductivity detector. The guard column screens out potential interferences, the separator column separates the anions, and the conductivity detector detects the anions from their electrical conductivity in solution.

Soil samples are prepared/leached by tumbling 5g of sample with 100mL reagent water. Prior to analysis, all soil leachates are filtered through a 0.20um filter. Additionally, all water samples and soil leachates are filtered through a 0.45um filter cap prior to analysis to remove particulates that may interfere with column flow. Therefore, the data may represent only the dissolved ionic species.

Bomb combustates for the determination of total halogens are collected in reagent water, filtered, and analyzed for the target analytes.

Example Analytical Sequence

Description	Comments
Blank	Di water
Initial Calibration	
ICB	Method blank
ICV	Second Source (same as LCS)
Samples & Batch QC Items	10 injections, not including LCSD; Includes MS/MSD/DUP
CCV	
CCB	
Samples & Batch QC Items	10 injections including MS/MSD/DUP
CCV	
CCB	
Samples & Batch QC Items	10 injections including MS/MSD/DUP
CCV	
CCB	

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Attachment 2:
Sample Collection, Preservation, and Holding Time Table

Matrix	Routine Sample Container	Default Sample Size	Minimum Sample Size	Chemical Preservation	Thermal Preservation	Dechlorination Agent	Holding Time
Water	125mL plastic	5mL	2.5mL	None	0-6°C	None	F, Cl, Br, SO_4: 28 Days NO2, NO3: 48 Hours
Soil	8oz plastic	5g	2.5g	None	0-6°C	None	F, Cl, Br, SO_4: 28 days[1] NO2, NO3: 28 Days to prep; 48 Hours to analyze
Combustate[2]	125mL plastic	5mL	2.5mL	None	0-6°C	None	28 days[1]

[1] ***Inclusive*** of extraction/combustion time
[2]Combustates are analyzed for Cl only. Contact Technical Management if other halogens are requested.

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Attachment 3:
QC Summary

QC Item	Frequency	Criteria	Corrective Action
Initial Calibration (ICAL) - Minimum 3 standards and a blank	Prior to sample analysis; when CCV fails; at least every 6 months	r^2 >= 0.990	Refer to SOP SA-QA-16
2nd Source Initial Calibration Verification (ICV)	After analysis of initial calibration standards	±10% of true value	Refer to SOP SA-QA-16
Continuing Calibration Verification (CCV)	At the beginning of the analysis, after every 10 samples, and end of the analysis	Mid-Level: ±10% of true value Low-Level: ±25% of true value	Refer to SOP SA-QA-16
Calibration Blank (ICB/CCB): - Can also be used as the method blank.	ICB: Before ICV CCB: After CCV	result < MDL	Refer to SOP SA-QA-17
Method Blank (MB)	1 per batch	result < MDL	Refer to SOP SA-QA-17
Laboratory Control Sample (LCS)	1 per batch	MLG Limits	Refer to SOP SA-QA-17
Laboratory Control Sample Duplicate (LCSD)	1 per batch, when insufficient sample for MS/MSD	MLG Limits	Refer to SOP SA-QA-17
Matrix Spike (MS)	EPA 300.0: 10% of samples performed; i.e., 2 per batch	MLG Limits	Refer to SOP SA-QA-17

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QC Item	Frequency	Criteria	Corrective Action
	Other Methods: 5% of samples performed; i.e., 1 MS per batch		
Matrix Spike Duplicate (MSD)	1 per batch	MLG Limits	Refer to SOP SA-QA-17
Sample Duplicate (SD)	SM 4110B, only: 1 per batch	MLG Limits	Refer to SOP SA-QA-17
Low-Level Laboratory Control Sample (LLCS) – Spiked at the RL	EPA 300.0 DW Only: 1 per batch	The analyte must be detected to be acceptable.	- If the LCS meets criteria, initiate NCM and report data - If the LCS does not meet criteria, reanalyze batch
Initial Demonstration of Capability (IDOC)	Initially, per analyst, per analyte/method/matrix combination	Refer to SOP SA-QA-06	Refer to SOP SA-QA-06 Note: Unsupervised work must not begin until acceptable IDOC is obtained.
Continuing Demonstration of Capability (CDOC)	Annually, per analyst, per analyte/method combination	Refer to SOP SA-QA-06	Refer to SOP SA-QA-06
Reporting Limit Verification (RLV)	Upon method/instrument set-up, per analyte/method/matrix combination. Then quarterly thereafter (for DOD ELAP) or annually thereafter (for non-DOD ELAP)	Refer to SOP SA-QA-07	Refer to SOP SA-QA-07
Method Detection Limit Study (MDL)	Upon method/instrument set-up, per analyte/method/matrix combination and every 6 months thereafter	Refer to SOP SA-QA-07	Refer to SOP SA-QA-07
MDL Verification (MDLV)	Upon method/instrument set-up, per analyte/method/matrix combination.	Refer to SOP SA-QA-07	Refer to SOP SA-QA-07

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QC Item	Frequency	Criteria	Corrective Action
	Then quarterly thereafter (for DOD ELAP) or annually thereafter (for non-DOD ELAP)		
Retention Time Window Study	Annually, with each new column, or if significant method change occurs per SOP SA-QA-08	Define a reasonable window for each analyte or retention time range and apply to each analysis per SOP SA-QA-08	-Evaluate instrument performance, column, and integration system.
Low-Level of Quantitation (LLOQ)	After analysis of initial calibration standards	The concentration must be within 50-150% of the actual concentration to be acceptable.	-Evaluate ion chromatogram and integrations. Check calculations -Reanalyze standards -Remake and reanalyze standard -Recalibrate
Dilution/Fortification	When high concentration of adjacent peak makes identification difficult	Peak in dilution and/or fortified sample within established window.	-Dilute or fortify (spike) sample at higher dilution factor or concentration.
Linear Calibration Range (LCR) Study - EPA 300.0 and SM4110B Only	Upon method/instrument set-up, and when LCR verification fails acceptance criteria.	Within ±10% of the true value	This determines the range at which the instrument is considered linear. The highest concentration of the calibration curve can not exceed the highest concentration of the LCR study.
Linear Calibration Range (LCR) Verification - EPA 300.0 Only	Every 6 months	Within ±10% of the initial value	Re-perform the LCR study

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Attachment 4:
Instrument Maintenance and Troubleshooting

Instrument Labeling

Each instrument must be labeled with its name or ID (e.g., MSA, ICP-D, etc.). Additionally, non-operational instruments must be isolated from service or marked as being out of service. Each piece of equipment has an "Operational / Not Operational" sticker that is used for this purpose.

Maintenance Log

A maintenance log must be established for each piece of equipment used in the laboratory. All maintenance that is performed on the instrument must be recorded in the log including:

- analyst or technician performing the maintenance
- date the maintenance was performed
- detailed explanation of the reason for the maintenance
- resolution of the problem and return to control
- all service calls from instrument representatives

Preventive Maintenance

LABORATORY EQUIPMENT PREVENTIVE MAINTENANCE SCHEDULE								
EQUIPMENT ITEM	Service Interval							SERVICE LEVEL
	D	W	M	Q	SA	A	AN	
Conductivity	X							Record Daily (Approximate Range = 0.3-2)
Pressure	X							Record Daily (Approximate Range = 2100-3000psi)
Eluent							X	Reagent Free – ICS200 system will alert the user when the cartridge expires
Suppressor	X							Check daily for leaks
Analytical/Guard Column							X	Replace as needed

D=daily; W=Weekly; M=monthly; Q=Quarterly; SA=semi-annually; A=annually; AN=as needed

Troubleshooting

Troubleshooting should be documented as outlined above. If possible, troubleshooting is best performed in a step-wise manner to systematically isolate instrument components. Refer to the instrument manufacturer's guides for specific information and strategies. Enlist assistance from technical and/or department management as needed.

Contingency Plan

Maintenance contracts are carried for most instrumentation and close contact is maintained with service personnel to ensure optimal instrument functioning. An extensive spare parts inventory is maintained for routine repairs, consisting of sample loops, filters, guard columns, columns, regulators, and other common instrumentation components. Since

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instrumentation is standardized throughout the laboratory network, spare parts and components can be readily exchanged among the network.

In general, the laboratory has at least one backup unit for each critical unit. In the event of instrument failure, portions of the sample load may be diverted to duplicate instrumentation, the analytical technique switched to an alternate approved technique (such as manual colorimetric determination as opposed to automated colorimetric determination), or samples shipped to another properly certified or approved TestAmerica location.

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Attachment 5:
Maximum Contaminant Level (MCL) Table

Primary Drinking Water Regulations	
Contaminant	**MCL (mg/L)**
Fluoride	4.0
Nitrate (as N)	10
Nitrite (as N)	1

Secondary Drinking Water Regulations	
Contaminant	**MCL (mg/L)**
Chloride	250
Fluoride	2.0
Sulfate	250

Note: Fluoride is part of the Primary Drinking Water Regulations; however, it is also included on the list of Secondary Drinking Water Regulations with a lower MCL.

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Attachment 6:
Additional Information Regarding NO3 and NO2 Reporting

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Reporting the *as N* Analytes:
For Drinking Water analyses, the regulated analytes are Nitrate and Nitrite reported *as Nitrogen*. To accomplish this, the laboratory analyzes nitrate and nitrite and uses the following conversion factor based on the molecular weight of the ion versus the molecular weight of nitrogen:

100mg/L nitrate as NO3 = 22.6mg/L nitrate as N
(i.e., NO3 x 0.226 = NO3 as N)

100mg/L nitrite as NO2 = 30.4mg/L nitrite as N
(i.e., NO2 x 0.304 = NO2 as N)

Target processing methods have been set up to perform this conversion automatically.

Reporting the *plus* Analytes
Some clients may request nitrate and nitrite to be reported together as nitrate+nitrite. To accomplish this, the laboratory analyzes nitrate and nitrite separately and sums the final result. EPA 300.0 indicates sulfuric acid should be used as the preservative when analyzing for nitrate+nitrite; however, since the laboratory is not performing a different chemistry to obtain the *plus* result, the same bottle types and holding times used for "regular" nitrate and nitrite are applied.

18.0 Revision History

Summary of Changes from Previous Revision:

- Minor editorial, grammatical, and formatting changes made. Boilerplate text added.
- Included standard and reagent disposal information, Section 7.2
- Revised NO2/NO3 ICAL standard preparation, Section 7.4
- Revised IC parameters to match current set-up, Section 10.3.1
- Included information for additional sample evaluation when the MS/MSD fails high for nitrate and low for nitrite,Section 11.1.7

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ANIONS BY ION CHROMATOGRAPHY

(Methods: EPA 300.0, EPA 9056, EPA 9056A, and SM4110B)

Approvals (Signature/Date):

Ammie L. Martin — February 21, 2012
Ammie Martin — Date
Quality Assurance Specialist, Designee for:
Andrea Teal, Quality Assurance Manager / Interim Environmental Health and Safety Coordinator

December 27, 2011
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1.0 Scope and Application

This SOP gives the procedures for the determination of anions by ion chromatography. The routine matrices analyzed by this procedure are waters and soils. The routine analytes are bromide, chloride, fluoride, nitrate, nitrite, and sulfate. Nitrate and nitrite have a shorter holding time than the other anions and are calibrated independently. For the purposes of this SOP, the term "other anions" refers to bromide, chloride, fluoride, and sulfate.

Nitrate+nitrite, nitrate as N, and nitrite as N can also be reported using this procedure. Refer to Attachment 6 for additional information and the necessary conversions needed for reporting these analytes.

This SOP is most useful for the determination of dirty or highly colored samples that might be problematic for colorimetric determination, or in some instances where a lower limit of detection is required for certain analytes. This procedure may also be used for determining total halogens for heat of combustion determinations.

The reporting limits (RL), the method detection limits (MDL), and the accuracy and precision criteria associated with this procedure are provided in the LIMS Method Limit Groups (MLGs).

This SOP was written by and for TestAmerica's Savannah laboratory.

2.0 Summary of Method

Ion chromatography is a method for the separation of dissolved ionic species using liquid chromatography techniques, along with eluent suppression and conductivity detection. A potassium hydroxide eluent conducts the sample through the chromatography system, which consists of a guard column, a separator column, a suppressor, and a conductivity detector. The guard column screens out potential interferences, the separator column separates the anions, and the conductivity detector detects the anions from their electrical conductivity in solution.

Samples are filtered through a 0.45um filter prior to analysis to remove particulates that may interfere with column flow; therefore, the data may represent only the dissolved ionic species.

Soils are leached by tumbling 5g of soil with 100mL of reagent water. The soil is allowed to settle, and the leachate is filtered and analyzed for the target analytes.

Bomb combustates for the determination of total halogens are collected in reagent water, filtered, and analyzed for the target analytes. (Note: Currently combustates are analyzed for Cl only. Contact Technical Management if other halogens are requested.)

This SOP is based on the following methods: EPA 300.0, EPA 9056, EPA 9056A, and SM4110B.

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3.0 Definitions

Refer to the Glossary Section of the *Quality Assurance Manual* (QAM) for a complete listing of applicable definitions and acronyms.

4.0 Interferences

4.1 Procedural Interferences

4.1.1 Interferences may be caused by contaminants in solvents, reagents, glassware, and other sample processing apparatus and can make identification and/or quantification of the target analytes difficult.

4.1.2 All sample collection containers are single-use disposable containers which limits the potential for contamination. All non-disposable labware must be scrupulously cleaned in accordance with the posted Labware Cleaning Instructions to ensure it is free from contaminants and does not contribute artifacts.

4.1.3 Instrument and/or method blanks are routinely used to demonstrate all reagents and apparatus are free from interferences under the conditions of the analysis.

4.2 Matrix Interferences

4.2.1 Particles from samples may lodge in the system, causing blockage. Therefore, all samples are automatically filtered prior to analysis using the autosampler vial caps which contain 0.45um filters.

4.2.2 Matrix interferences may be caused by contaminants that are co-extracted from the sample matrix. If matrix interference is encountered, the sample may require dilution or another filtration with a 0.20um filter prior to introduction into the 0.45um filter vial caps. The associated batch QC items must also be filtered if any samples or reagents have undergone filtration.

4.2.3 Interfering contamination may occur when a sample containing low concentrations of analytes is analyzed immediately following a sample containing relatively high concentrations of analytes. As such, samples known to be clean should be analyzed first. To prevent carryover into subsequent samples, analysis of reagent blanks may be needed after the analysis of a sample containing high concentrations of analytes. Dionex NaOH wash solution can also be used to rinse the instrument to alleviate carryover.

4.2.4 Large quantities of ions eluting close to the anion of interest will also result in an interference. Separation can be improved by adjusting the eluent concentration and/or flow rate. Sample dilution can also be used. For example, high levels of organic acids that may interfere with inorganic anion analysis may be present in industrial wastes. Two common species, formate and acetate, elute between fluoride and chloride.

4.2.5 The acetate, formate, and other monovalent organic acid anions elute early in the chromatographic run and can interfere with fluoride and/or cause shifts in the retention time of the other anions.

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5.0 Safety

Employees must abide by the policies and procedures in the TestAmerica Environmental Health and Safety Manual (EHSM), the TestAmerica Savannah Addendum to the EHSM, and this document.

This procedure may involve hazardous materials, 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 to follow appropriate safety, waste disposal, and health practices under the assumption that all samples and reagents are potentially hazardous.

The analyst must protect himself/herself from exposure to the sample matrix. Many of the samples that are tested may contain hazardous chemical compounds or biological organisms. The analyst must, at a minimum, wear protective clothing (lab coat), eye protection (safety glasses or face shield), disposable latex or nitrile gloves, and closed-toe, nonabsorbent shoes when handling samples.

5.1 Specific Safety Concerns or Requirements

The toxicity or carcinogenicity of chemicals used in this method has not been precisely defined. Each chemical should be treated as a potential health hazard, and exposure to these chemicals should be minimized.

Potassium hydroxide is a severe corrosive. Contact with the skin can cause irritation or severe burns and scarring. Contact with the eyes can cause irritation, burns, permanent vision impairment or even blindness.

Exercise caution when using syringes with attached filter assemblies. Application of excessive force has, upon occasion, caused a filter disc to burst during the process.

5.2 Primary Materials Used

The following is a list of the materials used in this procedure, which have a serious or significant hazard rating, and a summary of the primary hazards listed in their MSDS.

NOTE: This list does not include all materials used in the procedure. A complete list of materials used in this procedure can be found in the Reagents and Standards Section and the Equipment and Supplies Section of this SOP

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 found using the "MSDS" link on the Oasis homepage, on the EH&S webpage on Oasis, and on the QA Navigator.

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Material	Hazards	Exposure Limit[1]	Signs and Symptoms of Exposure
Potassium Hydroxide	Corrosive	2mg/m^3 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]Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

6.1 Equipment and Instrumentation

Top-loading Balance – Verify in accordance with SOP SA-AN-100: *Laboratory Support Equipment (Verification and Use)*

Ion Chromatography System – Dionex ICS2000 (RFIC)

6.2 Analytical Data System / Software / Hardware

Chromeleon software is used on a Windows-based PC to schedule and acquire data. Target (UNIX and/or Windows) software is used on a Windows-based PC to store, reduce/evaluate, and output the data to the laboratory's LIMS system (i.e., TALS). Target software has the capability of processing data by recognizing a GC peak within any given retention time window. The software allows for calculation of response factors and or construction of a linear regression calibration curve, calculation of response factor statistics (mean and standard deviation), and calculation of concentrations of analytes using either the calibration curve or the response factors.

6.3 Lab Supplies

Guard Column(s) – Dionex AG18

Separator Column(s) – Dionex AS18

Suppressor – ASRS (Auto Self-regenerating Suppressor) 4mm

Volumetric Containers – various sizes; Class A, where applicable. Verify in accordance with SOP SA-AN-100: *Laboratory Support Equipment (Verification and Use)*

Mechanical Pipettes – various sizes. Verify in accordance with SOP SA-AN-100: *Laboratory Support Equipment (Verification and Use)*

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Syringe Filters – Nalgene, 0.20um pore size

Autosampler Vials – with 0.45um filter caps

DI Leach Tumbler

Detergent – FL-70, used for washing non-disposable labware.

6.4 Sample Collection Containers

All sample collection containers are single-use disposable containers which limits the potential for contamination.

The routine sample collection containers supplied by the laboratory are:

Water Samples:
125mL plastic oblong bottles – purchased with Certificate of Analysis attesting to purity.

Soil Samples:
8oz plastic soil jars – purchased with Certificate of Analysis attesting to purity.

7.0 Reagents and Standards

The standards and reagents listed are those in use at the time this SOP was updated. Other standard and reagent stocks, vendors, and concentrations may be used provided they are fully documented and in compliance with method requirements.

7.1 Expiration Dates

Expiration dates (time from initial use or receipt to final use) for standard and reagent materials must be set according to the guidance in this SOP. Note: These are maximum expiration dates and are not to be considered an absolute guarantee of standard or reagent quality. Sound judgment must be used when deciding whether to use a standard or reagent. If there is doubt about the quality of a standard or reagent material, a new material must be obtained or the standard or reagent material verified. Data quality must not be compromised to extend a standard's life – i.e., when in doubt, throw it out.

The expiration date of any standard or reagent must not exceed the expiration date of the standard or reagent that was used to prepare it; that is, the "children may not outlive the parents".

Note: As indicated in the Method Modification Section of this SOP, the default standard expiration dates used by the laboratory have been extended beyond those specified in the reference methods based on the results of a stability study. SC DHEC does not recognize this method modification and requires the method-defined holding times as listed in the SC DHEC Project Requirement Summary (PRS).

7.2 Disposal Information

Unless otherwise specified, all standards and reagents should be disposed of via the drain / sewer system.

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7.3 Reagents

Reagents must be prepared and documented in accordance with SOP SA-AN-041: *Reagent and Standard Materials Procedures.*

Blank Matrix – glass beads. Used for the preparation of soil QC samples.

Laboratory Reagent Water – ASTM Type II.

Dionex Elu Gen Cartridge – Potassium Hydroxide

7.4 Standards

Standards must be prepared and documented in accordance with SOP SA-AN-041*: Reagent and Standard Materials Procedures.* Certificates of analysis or purity must be received with all purchased standards, and scanned and filed in the Data Archival Folder on the G-drive.

First Source Other Anions Mix Source Standard – Inorganic Ventures
Concentration: 200mg/L fluoride; 1000mg/L chloride, bromide, sulfate)
Storage: Refrigerated at 0-6°C
Expiration:
Unopened: Manufacturer's expiration date
Opened: 1 year from date opened

Nitrite Source Standard (1000mg/L NO_2 or 304mg/L as N) – Inorganic Ventures
Storage: Refrigerated at 0-6°C
Expiration:
Unopened: Manufacturer's expiration date
Opened: 6 months from date opened

Nitrate Source Standard (1000mg/L NO_3 or 226mg/L as N) – Inorganic Ventures
Storage: Refrigerated at 0-6°C
Expiration:
Unopened: Manufacturer's expiration date
Opened: 6 months from date opened

Second Source Other Anions Mix Source Standard – Environmental Express
Concentration: 200mg/L fluoride; 1000mg/L chloride, bromide, sulfate
Storage: Refrigerated at 0-6°C
Expiration:
Unopened: Manufacturer's expiration date
Opened: 1 year from date opened

Second Source Nitrite Source Standard - 1000mg/L NO_2 or 304mg/L as N; Environmental Express
Storage: Refrigerated at 0-6°C
Expiration:
Unopened: Manufacturer's expiration date

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Opened: 6 months from date opened

Second Source Nitrate Source Standard - 1000mg/L NO_3 or 226mg/L as N; Environmental Express
Storage: Refrigerated at 0-6°C
Expiration:
Unopened: Manufacturer's expiration date
Opened: 6 months from date opened

Calibration Standards

The calibration standards are prepared as follows:

Nitrate and Nitrite (as N) Calibration Curve:

HICAL (4.0mg/L) – Add 13.16mL of Nitrite Source Standard and 17.70mL of Nitrate Source Standard to a 1L class A volumetric falsk. Bring to volume with Reagent Water.

Calibration Level	Parent Standard	Volume Added (mL)	Final Volume (mL)	Final Concentration[1] (mg/L)
1	CAL-5	25	500	0.05
2	CAL-5	10	100	0.10
3	CAL-5	25	100	0.25
4	CAL-7	25	100	0.50
5	CAL-7	250	500	1.0
6	CAL-7	75	100	1.5
7	HICAL	500	1000	2.0

[1]Concentrations listed are given in terms of the "as N" analytes.
Solvent: Reagent Water
Storage: Refrigerated at 0-6°C
Expiration: 1 month from preparation date

Other Anions Calibration Curve:

Calibration Level	Parent Standard	Volume Added	Final Volume (mL)	Final Concentration (mg/L)
1	Other Anions Mix Source Standard	500uL	500	0.20 (F) 1.0 (Br, Cl, SO4)
2	Other Anions Mix Source Standard	200uL	100	0.40 (F) 2.0 (Br, Cl SO4)
3	Other Anions Mix Source Standard	400uL	100	0.80 (F) 4.0 (Br, Cl SO4)
4	Other Anions Mix Source Standard	5mL	500	2.0 (F) 10 (Br, Cl SO4)
5	Other Anions Mix Source Standard	2mL	100	4.0 (F) 20 (Br, Cl SO4)

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6	Other Anions Mix Source Standard	5mL	100	10 (F) 50 (Br, Cl SO4)

Solvent: Reagent Water
Storage: Refrigerated at 0-6°C
Expiration: 3 months from preparation date
(Note: Bromide is used for NPW analyses only.)

ICV / Water LCS Standards

The ICV and all water LCS are prepared in the same way using the same source standards. A large volume is prepared, and is then ready for analysis as is (i.e., they are "pour and go" standards).

Other Anions ICV/LCS Standard

Parent Standard	Parent Concentration (mg/L)	Volume Added (mL)	Final Volume (mL)	Final Concentration (mg/L)
2nd Source Other Anions Source Standard	200 (F) 1000 (Br, SO4, Cl)	5	500	2 (F) 10 (Br, SO4, Cl)

Solvent: Laboratory Reagent Water
Storage: Refrigerated at 0-6°C
Expiration: 3 months from preparation date

Nitrate and Nitrite ICV/LCS Intermediate Standard (N-NSPK)

Parent Standard	Parent Concentration (mg/L)	Volume Added (mL)	Final Volume (mL)	Final Concentration[1] (mg/L)
2nd Source NO3 Source Standard 2nd Source NO2 Source Standard	1000 (NO3) 1000 (NO2)	22.1 16.4	50	100

[1]Concentrations listed are given in terms of the "as N" analytes.
Solvent: Laboratory Reagent Water
Storage: Refrigerated at 0-6°C
Expiration: 1 month from preparation date

Nitrate and Nitrite ICV/LCS Standard

Parent Standard	Parent Concentration (mg/L)	Volume Added (mL)	Final Volume (mL)	Final Concentration[1] (mg/L)
Nitrate and Nitrite ICV/LCS Intermediate Standard (N-NSPK)	100	5.0	500	1.0

[1]Concentrations listed are given in terms of the "as N" analytes.
Solvent: Laboratory Reagent Water
Storage: Refrigerated at 0-6°C
Expiration: 1 month from preparation date

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8.0 Sample Collection, Preservation, Shipment, and Storage

8.1 Aqueous Samples

Aqueous samples are routinely collected in 125mL oblong plastic containers.

Samples must be iced at the time of collection and maintained at 4°C (less than 6°C but not frozen) until the time of analysis. Samples for nitrate and/or nitrite must be analyzed within 48 hours of collection. Samples for the other anions must be analyzed within 28 days of collection.

8.2 Soil Samples

Soil samples are routinely collected in 8oz plastic soil containers.

Samples must be iced at the time of collection and maintained at 4°C (less than 6°C but not frozen) until the time of preparation. Samples for nitrate and/or nitrite must be prepared within 28 days of collection and analyzed within 48 hours of preparation. Samples for the other anions must be prepared and analyzed within 28 days of collection. Extracts must be stored at 4°C (less than 6°C but not frozen) until the time of analysis.

8.3 Combustate Samples

Once the heat of combustion procedure has been performed (in accordance with SOP GE105), the combustate is transferred to a 125mL plastic container. Combustates must be stored at 4°C (less than 6°C with no frozen samples) until the time of analysis. Combustate samples must be prepared and analyzed within 28 days of collection.

9.0 Quality Control

SOP SA-QA-17: *Evaluation of Batch QC Data* and the SOP Summary in Attachment 1 provide requirements for evaluating QC data.

Batch QC must meet the criteria given in Attachment 3 of this SOP.

9.1 Batch QC

9.1.1 Soil Batch QC

An extraction batch consists of up to 20 environmental samples and the associated QC items extracted (leached) together within a 24 hour period.

The laboratory's default QC items required for each extraction batch are: a method blank, a laboratory control sample (LCS), a matrix spike (MS), and a matrix spike duplicate (MSD). Additionally, for EPA 300.0, another matrix spike (MS) is required.

The routine container supplied for this method is an 8oz container. 5g is required for extraction. It is unlikely that reduced sample initial weights will be necessary to achieve the required batch matrix spike and sample duplicate frequency. If reduced initial weights are required, the minimum extraction weight to be used for the matrix spike samples or sample duplicates is 2.5g.

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Note: Final volumes and spike amounts must be adjusted to compensate for these reduced initial weights.

If there is insufficient sample provided to perform the required MS/MSDs, an NCM must be initiated on all affected samples to denote this situation. If an LCSD has not already been performed, an LCSD should be included in the batch. Insufficient sample is defined as receiving less than a total of 10g.

Note: If an LCS and LCSD are performed, both QC items must be evaluated and reported. Acceptable recoveries (as well as %RPD) for both LCS and LCSD are required.

9.1.2 Water Batch QC

An analytical batch consists of up to 20 environmental samples and the associated QC items analyzed together within a 24 hour period.

The laboratory's default QC items required for each batch are: a method blank, a laboratory control sample (LCS), matrix spike (MS), and an MSD. The following additional QC items are also required:

EPA 300.0 (DW and non-DW): an additional matrix spike
EPA 300.0 (DW only): a low-level LCS
SM4110B: a sample duplicate

Additionally, EPA 300.0 requires an LCSD to be performed quarterly. As there are no preparation steps associated with water samples, the CCV or ICV should satisfy this requirement.

Determination of Sufficient Volume
The routine container supplied for this method is a 125mL container. 5mL is required for analysis. Reduced sample initial volumes may be necessary to achieve the required batch matrix spike and sample duplicate frequency; however, the minimum analysis volume to be used for the matrix spike samples or sample duplicate is 2.5mL.

Note: Spike amounts must be adjusted to compensate for these reduced initial volumes.

If there is insufficient sample volume to perform the required MS/MSDs, the LCS must be performed in duplicate (i.e., LCS/LCSD). An NCM must be initiated on all affected samples to denote this situation. Insufficient sample volume is defined as receiving less than a total of 10mL.

Note: If an LCS and LCSD are performed, both QC items must be evaluated and reported. Acceptable recoveries (as well as %RPD) for both LCS and LCSD are required.

9.2 Instrument QC

9.2.1 Initial Calibration (ICAL)

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The instrument must be calibrated in accordance with SOP SA-QA-16: *Evaluation of Calibration Curves.* This SOP provides requirements for establishing the calibration curve and gives the applicable formulas.

Instrument calibration is performed by analyzing a series of known standards. The calibration curve must consist of a minimum of 3 standards and a blank. The lowest level calibration standard must be at or below the reporting limit, and the remaining standards will define the working range of the analytical system.

The initial calibration standard concentrations currently in use in the laboratory are given in Section 7.3.7. Refer to Section 7.3 for the standard preparation instructions. Other standard concentrations may be used provided they support the reporting limit and are fully documented in accordance with SOP SA-AN-041.

9.2.1.1 ICAL Criteria

The preferred method of calibration and quantitation is a linear regression curve. The regression coefficient (r^2) of the regression curve must be greater than 0.990 for the initial calibration curve to be acceptable.

Note: A minimum of 5 points is required.

Note: The calibration procedures outlined in EPA 9056 and EPA 9056A specify a linear curve to be used and do not incorporate average response factor or quadratic curves, as allowed by the other SW-846 chromatography methods. For this reason, average response factor and quadratic curves are not used for this procedure. As is standard laboratory practice, higher order curves are also prohibited.

LLOQ Verification

EPA 9056A requires verification of the Low-Level of Quantitation (LLOQ). Requant the low point of the calibration curve (or the point equivalent to the RL) as a sample. The concentration must be within 50-150% of the actual concentration to be acceptable.

9.2.2 Second Source Initial Calibration Verification (ICV)

The calibration curve must be verified initially – prior to any sample analyses – in accordance with SOP SA-QA-16 with a standard obtained from a second source.

The ICV must be within +/- 10% of the true value to be acceptable.

The initial calibration verification standard concentration currently in use in the laboratory is equivalent to Level 5 of the ICAL for nitrate and nitrite and to Level 4 of the ICAL for the other anions. Refer to Section 7.3 for the standard preparation instructions. Another standard concentration may be used provided it is mid-level and fully documented in accordance with SOP SA-AN-041.

9.2.3 Initial Calibration Blank (ICB) / Continuing Calibration Blank (CCB)

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The instrument must be shown to be free from contamination by the analysis of calibration blanks. Initial calibration blanks are analyzed immediately ~~after~~ before the ICV. Continuing calibration blanks are analyzed after each CCV.

Initial and continuing calibration blanks must be <MDL to be acceptable.

9.2.4 Continuing Calibration Verification

The initial calibration curve must be verified initially with a mid-level standard for EPA 300.0, EPA 9056, and EPA 9056A, and with a low-level standard (at the reporting limit) for SM4110B. Additionally, a mid-level CCV must be analyzed after every 10 samples for all methods.

The low-level CCV must be within 25% of the true value to be acceptable and the mid-level CCV must be within +/- 10% of the true value to be acceptable.

The mid-level continuing calibration verification standard concentration currently in use in the laboratory is equivalent to Level 5 of the ICAL for nitrate and nitrite and to Level 4 or 5 of the ICAL for the other anions. Refer to Section 7.3 for the standard preparation instructions. Another standard concentration may be used provided it is mid-level and fully documented in accordance with SOP SA-AN-041.

9.2.5 Linear Calibration Range / Linear Dynamic Range

The linear calibration range (LCR), also known as the Linear Dynamic Range (LDR), must be determined initially for SM4110B and EPA 300.0 and verified every 6 months thereafter for EPA 300.0.

The initial demonstration of linearity uses as many standards as necessary to insure the curve is linear. The standards should cover the low and high ends of the calibration range. Each standard must be within +/- 10% of the true value.

The continued demonstration of linearity consists of a minimum of 3 standards and a blank. For the linearity to be verified, the standards must be within +/- 10% of the initial value to be acceptable. If this criterion is not met, the initial demonstration of linearity must be performed.

Note: This study is not meant to extend the linear range of the calibration. If an analyte is measured at a concentration greater than the highest calibration standard, the sample must be analyzed at a dilution to bring the target analyte into the range of the initial calibration.

9.3 Corrective Action for Out-of-Control Data

When the quality control parameters do not meet the criteria set forth in this SOP, corrective action must be taken in accordance with SOP SA-QA-05: *Preventive and Corrective Action Procedures* and the QC Summary Table in Attachment 3. SOP SA-QA-05 provides contingencies for out-of-control data and gives guidance for exceptionally permitting departures from approved policies and procedures. Nonconformance Memos must be initiated to document all instances where QC criteria are not met and all departures from approved policies and procedures.

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10.0 Procedure

10.1 Sample Preparation

10.1.1 Soil Sample Preparation

Soil samples are prepared/extracted using a DI Leach procedure. Soil samples must be homogenized prior to preparation in accordance with SOP SA-QA-15: *Compositing, Homogenization, and Segregation of Samples*.

Remove the samples from the refrigerator and allow them to come to room temperature.

Using a top-loading balance, weigh 4.90-5.10g of the homogenized sample and transfer to a 125mL plastic container. Record the weight of the sample to the nearest 0.01g. Add 100mL of laboratory reagent water to each sample. Tumble the samples at approximately 18rpm for 2 hours +/- 15 minutes. Remove the extracts from the tumbler and allow the sediment to settle. Store extracts at 4°C (less than 6°C but not frozen) until the time of analysis.

When ready to analyze, remove the extracts from the refrigerator and allow them to come to room temperature. Using a disposable syringe, remove a portion of the supernatant liquid and filter through a 0.20um filter. Collect the filtrate in an autosampler tube.

10.1.2 Water Sample Preparation

Remove the samples from the refrigerator and allow them to come to room temperature.

If the sample contains color, turbidity, or particulates, the sample is filtered through a 0.20um filter prior to transferring to the autosampler vial.

> Note: If the sample requires filtration through a 0.20um filter, the batch method blank and LCS must also be filtered through a 0.20um filter.

All drinking water samples are initially analyzed straight. To protect the integrity of the instrument, all non-drinking water samples are initially analyzed at a 1:5 dilution.

10.1.3 Combustate Sample Preparation

Combustate samples are prepared in the same manner as water samples, discussed above.

10.2 QC Sample Preparation

10.2.1 Soil QC Sample Preparation

Refer to Section 9.1.1 for the batch QC frequency.

The method blank and the LCS are prepared using glass beads to simulate the sample matrix. The MS and MSD or sample duplicate is prepared using one of the samples in the batch.

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10.2.1.1 Method Blank – Use 5.0g glass beads for the blank matrix and extract as described in Section 10.1.1.

10.2.1.2 Laboratory Control Sample (LCS) and Laboratory Control Sample Duplicate (LCSD) – Use 5.0g glass beads for the blank matrix, add 1.0mL of the applicable 2nd Source Standard to the LCS and LCSD, and extract as described in Section 10.1.1.

To prepare the LCS and LCSD for nitrate and nitrite, use the Nitrate and Nitrite ICV/LCS Standard. To prepare the LCS and LCSD for the other anions, use the 2nd Source Other Anions Source Standard.

10.2.1.3 Matrix Spike (MS) and Matrix Spike Duplicate (MSD) – Add 4.0mL of the applicable 2nd Source Standard to the separate 5.0g aliquots of the sample chosen for the MS and MSD. Extract as described in Section 10.1.1.

To prepare the MS and MSD for nitrate and nitrite, use the use the Nitrate and Nitrite ICV/LCS Standard. To prepare the MS and MSD for the other anions, use the 2nd Source Other Anions Source Standard.

10.2.2 Water QC Sample Preparation

Refer to Section 9.1.2 for the batch QC frequency.

The method blank and the LCS are prepared using reagent water. The MS and MSD or sample duplicate is prepared using one of the samples in the batch.

10.2.2.1 Method Blank – Use 5.0mL laboratory reagent water for the blank matrix. Note: If the associated samples are filtered using a 0.20um filter, the method blank must also be filtered.

10.2.2.2 Laboratory Control Sample (LCS) and Laboratory Control Sample Duplicate (LCSD) – Transfer 5.0mL of the applicable ICV/LCS Standard into an autosampler vial. Note: If the associated samples are filtered using a 0.20um filter, the LCS and LCSD must also be filtered.

10.2.2.3 Low-Level Laboratory Control Sample (LLCS) – Prepare a 1:10 dilution of the applicable ICV/LCS Standard by adding 0.5mL standard into a final volume of 5mL reagent water. (Note: ICAL Level 1 may also be used to satisfy this standard.) This dilution gives the following concentrations: 0.20mg/L (F), 1.0mg/L (Br, SO4, Cl), 0.05 mg/L (NO2 as N) and 0.050 mg/L (NO3 as N). Note: If the associated samples are filtered using a 0.20um filter, the LCS and LCSD must also be filtered.

10.2.2.4 Matrix Spike (MS) / Matrix Spike Duplicate (MSD) – Add 50uL of the applicable Matrix Spike Standard to the separate 5.0mL aliquots of the sample chosen for the MS and MSD.

10.2.3 Combustate QC Sample Preparation

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Combustate batch QC is prepared during the combustion step of the process. Total sulfur is usually analyzed via Konelab along with the total halogen (chloride) performed via IC; therefore, the batch QC is used for both methods. The LCS is spiked at a higher concentration for the total sulfur analysis and must be diluted 1:25 prior to analyzing via IC. Rinsates are usually performed after each injection as well.

10.3 Analysis

10.3.1 Instrument Operating Conditions

The instrument conditions listed in this SOP are provided for guidance purposes. The actual conditions used by the laboratory may be slightly different from those listed here and must be documented in the instrument maintenance log, data system, and/or run log.

Instrument maintenance must be performed in accordance with Attachment 4 of this SOP.

The goal is to have maximum separation between the target compounds in the shortest run time while maintaining sufficient sensitivity to detect the target compounds at the reporting limit and MDL (if required).

Example IC Parameters

Instrument Parameter	Configuration
IC Mode	Gradient / Isocratic
Column	AS18, 4mm
Guard	AG18, 4mm
Sample Loop	25uL
Eluent	KOH EluGen 12-25mM gradient for NO2/NO3 44mM isocratic for the other Anions
SRS Current	112mA
Pressure	≈ 2000-2800psi
Suppressor	4mm ASRS
Conductivity	0.4-2.0uS
Run Time	10-12min

10.3.1.1 Determination of Retention Time Windows

The procedure for the determination of retention time windows is given in SOP SA-QA-08: *Evaluation of Chromatographic Data*. Retention time windows (RTW), i.e., the length of time the instrument will scan for the analyte, must be established initially upon instrument set-up and verified quarterly.

Retention times (RT), i.e., the elution time of the analyte, are verified daily with the analysis of the ICAL or CCV. The retention time for the CCV must fall within the daily retention time window as defined in SOP SA-QA-08.

10.3.2 Initial and Continuing Calibration

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Calibrate the instrument using the standards and criteria described given in Section 9.2.1. Once the calibration has been established and verified with an ICV in accordance with Section 9.2.2, sample analysis may proceed.

Verify the calibration curve with a continuing calibration verification using the standards and criteria described given in Section 9.2.4.

10.3.3 Sample Analysis

Remove the samples or extracts from the refrigerator and allow them to come to room temperature.

The sample or extract must be injected using the same injection volume used for the calibration standards. Samples that are known to be relatively clean should be analyzed first. Samples suspected of containing high concentrations should be analyzed last. Instrument blanks may be analyzed after suspected high concentration samples to allow the detector response to stabilize.

The default procedure is to exclude QC items (method blank and LCS) in determining the maximum number of samples in the clock.

10.3.4 Example Analytical Sequence

An example analytical sequence is listed in Attachment 1.

11.0 Calculations / Data Reduction

11.1 Data Reduction

Data evaluation must be performed in accordance with SA-QA-08: *Evaluation of Chromatographic Data*. This SOP includes specific information regarding the evaluation of chromatographic data, including the requirements for performing manual integrations and the evaluation of retention times.

Data must be evaluated in accordance with SOP SA-QA-02: *Data Generation and Review*.

11.1.1 Target Analyte Identification

The judgment and experience of the analyst and his/her colleagues are important factors in the evaluation of chromatographic data. Inspect each chromatogram to ensure that the peaks are properly identified and that the correct areas have been associated with the corresponding standard peak RT in the data system tabulation.

The evaluation of chromatograms for target analytes must take into account the calibration of the analytical system (initial and continuing calibration response and retention times); whether the peak response falls within the working range of the calibration; and the integration of the peaks. The analyst must also take into account the results from the method blank and laboratory control sample before reporting quantitative

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data. SOP SA-QA-08: *Evaluation of Chromatographic Data* provides additional guidance for the evaluation of chromatographic data.

11.1.2 Manual Integrations

Manual integrations must be documented in accordance with SOP SA-QA-08. Data systems should be adjusted to minimize operator intervention. All chromatographic peaks must be evaluated for overall peak shape and "reasonableness" of integration. Under no circumstances should manual integrations be used to change reasonable data system integrations in order to meet calibration or QC criteria.

11.1.3 Dilutions

If the analyst notices any odor or color and historical data is not available, the analyst shall use their professional judgment for deciding an initial dilution. If the sample requires that more than one anion be reported, the sample shall be diluted so that only the highest target analyte meets or exceeds the midpoint of the calibration curve.

When reporting results from dilutions, appropriate data flags must be used or qualification in a case narrative provided to the client.

For clients who require we provide lower detection limits, a general guide would be to report the dilution detailed above and one additional run at a dilution factor 1/10 of the dilution with the highest target in the upper half of the calibration curve. For example, if samples analyzed at a 1/50 dilution resulted in a target in the upper half of the calibration curve, the sample would be analyzed at a dilution factor of 1/5 to provide lower reporting limits.

11.1.4 Historical Data

Many of the laboratory's clients submit samples for repeat monitoring purposes. LIMS Worksheet Notes and the Historical Data Tracker feature should be used to determine if historical data is available for review.

11.1.5 Chemical Relationships

When available, the following chemical relationships must be evaluated for each sample. If these relationships are not met the Department Manager must be contacted immediately.

- NO3+NO2 > Component analytes
- Refer to Attachment 6 for conversions of nitrate and nitrite to the "as N" analytes

11.1.6 Drinking Water Compliance Evaluation

Public water suppliers (PWS) are governed by EPA-specified Maximum Contaminant Levels (MCL) above which indicates noncompliance. The MCLs associated with this procedure are given in Attachment 5.

11.1.7 Matrix Spike Evaluation

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In the event that an MS/MSD fails high for nitrate and low for nitrite the original sample should be checked for an oxidizing agent. 5mL of sample is poured into a vial and 1 unit of "DPD FREE CHLORINE POWDER POP" is added to the vial. In the event that the sample turns a pink color a nonconformance memo is initiated and the samples are reported.

11.2 Calculations

11.2.1 The calculations associated with batch QC determinations are given in SOP SA-QA-17. Applicable calculations include accuracy (% recovery) and precision (%RPD).

11.2.2 The calculations associated with initial and continuing calibrations and are given in SOP SA-QA-16. Applicable calculations include determination for: calibration factor, standard deviation, relative standard deviation, relative response factor, and relative standard deviation.

11.2.3 The calculation to determine final concentration is given as follows:

$$FinalConcentration = CONC_{Sample} \otimes \frac{F}{I \times dw} \otimes D$$

Where:

$CONC_{Sample}$= Concentration of the sample
F = Final volume/weight
I = Initial volume/weight
D = Dilution factor
dw = % Solids decimal equivalent

Note: This calculation assumes all applicable unit correction factors are applied.

Note: All dry weight corrections are performed automatically in LIMS.

12.0 Method Performance

12.1 Reporting Limit Verification (RLV)

At a minimum, RLVs must be performed initially upon method set-up in accordance with SOP SA-QA-07: *Determination and Verification of Detection and Reporting Limits*.

For analytes and methods certified by DOD ELAP, RLVs must also be performed quarterly thereafter. For all other analytes and methods, RLVs must also be performed annually thereafter. Exceptions may be made for project-specific non-routine analytes.

12.2 Method Detection Limit (MDL) Study

The 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. 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 current MDLs associated with this procedure are given in the Method Limit Group (MLG) in TALS.

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At a minimum, MDL Studies must be performed initially upon method set-up in accordance with SOP SA-QA-07: *Determination and Verification of Detection and Reporting Limits*.

Note: MDL Studies are not required for non-routine analytes provided results are not reported below the RL (i.e., MDL equals RL in TALS).

12.3 Method Detection Limit Verification (MDLV)

At a minimum, MDLVs must be performed initially upon method set-up in accordance with SOP SA-QA-07: *Determination and Verification of Detection and Reporting Limits*.

For analytes and methods certified by DOD ELAP, MDLVs must also be performed quarterly thereafter. For all other analytes and methods, MDLVs must also be performed annually thereafter.

Note: MDLVs are not required for non-routine analytes provided results are not reported below the RL (i.e., MDL equals RL in TALS).

12.4 QC Limit Generation, Control Charting, and Trend Analysis

The control limits for the batch QC items (LCS, MS/MSD) for this procedure are specified in the reference method and cannot be broadened; therefore, the laboratory defaults to the method-defined limits and does not utilize in-house or laboratory-derived limits for the evaluation of batch QC items.

Although the laboratory must default to the method-defined QC limits, control charting is a useful tool and is performed to assess analyte recoveries over time to evaluate trends. Control charting must be performed periodically (at a minimum annually) in accordance with SOP SA-QA-17: *Evaluation of Batch QC Data.*

12.5 Demonstrations of Capability

Initial and continuing demonstration of capability must be performed in accordance with SOP SA-QA-06*: Training Procedures.*

Prior to performing this procedure unsupervised, each new analyst who performs this analysis must demonstrate proficiency per method/analyte combination by successful completion of an initial demonstration of capability.

IDOCs are performed by the analysis of 4 consecutive LCS that meet the method criteria for accuracy and precision. The LCS must be from a second source than that used to prepare the calibration standards. The IDOC must be documented on the IDOC Form shown in SOP SA-QA-06 with documentation routed to the QA Department for filing.

Annual continuing demonstrations of capability (CDOCs) are also required per analyst per method/analyte combination. The CDOC requirement may be met by the consecutive analysis of four LCS all in the same batch, by the analysis of four LCS analyzed in four consecutive batches (in different batches on different days), via acceptable results on a PT study, or analysis of client samples with statistically indistinguishable results when compared to another certified analyst. The CDOC must be documented and routed to the

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QA Department for filing.

12.6 Training Requirements

All training must be performed and documented in accordance with SOP SA-QA-06: *Training Procedures*.

Note: The SOPs listed in the Reference/Cross-Reference Section are applicable to this procedure. All employees performing this procedure must also be trained on these SOPs, and/or have a general understanding of these procedures, as applicable.

13.0 Pollution Control

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (e.g., examining recycling options, ordering chemicals based on quantity needed, preparing reagents based on anticipated usage and reagent stability, etc.). Employees must abide by the policies in Section 13 of the Environmental Health and Safety Manual and the Savannah Addendum to the EHSM.

This procedure has been evaluated for opportunities to minimize the waste generated. Where reasonably feasible, pollution control procedures have been incorporated.

14.0 Waste Management

Waste management practices must be conducted consistent with all applicable federal, state, and local rules and regulations. All waste (i.e., excess reagents, samples, and method process wastes) must be disposed of in accordance with the TestAmerica Savannah Addendum to the EHSM. Waste description rules and land disposal restrictions must be followed.

14.1 Waste Streams Produced by the Method

The following waste streams are produced when this method is carried out:

- Excess aqueous samples – Dispose according to characterization on the sample disposal sheets. Neutralize non-hazardous samples before disposal into drain/sewer. Transfer hazardous samples (identified on disposal sheets) to the waste department for disposal.

- Excess soil and solid samples – Dispose according to characterization on sample disposal sheets. Transfer non-hazardous samples to TCLP container for characterization in hazardous waste department. Transfer hazardous samples (identified on disposal sheets) to waste department for disposal.

15.0 References / Cross-References

- SOP SA-AN-041: *Reagent and Standard Materials Procedures*
- SOP SA-AN-100: *Laboratory Support Equipment (Verification and Use)*
- SOP SA-QA-02: *Data Generation and Review*
- SOP SA-QA-05: *Preventive and Corrective Action Procedures*

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- SOP SA-QA-06*: Training Procedures*
- SOP SA-QA-07: *Determination and Verification of Detection and Reporting Limits (RLs, MDLs, and IDLs)*
- SOP SA-QA-08: *Evaluation of Chromatographic Data*
- SOP SA-QA-15: *Homogenization, Compositing, and Segregation of Samples*
- SOP SA-QA-16: *Evaluation of Calibration Curves*
- SOP SA-QA-17: *Evaluation of Batch QC Data*
- TestAmerica Savannah Quality Assurance Manual
- TestAmerica Environmental Health and Safety Manual
- TestAmerica Savannah Addendum to the Environmental Health and Safety Manual
- Method 300.0 Determination of Inorganic Anions by Ion Chromatography; Revision 2.1, Methods for the Determination of Inorganic Substances in Environmental Samples, August 1993.
- *Test Methods for Evaluating Solid Waste, Third Edition, updates III and IV*; U.S. EPA Office of Solid Waste and Emergency Response: Washington, DC.
    - Method 9056 *Determination of Inorganic Anions by Ion Chromatography*; Revision 0; September 1994. (Update III)
    - Method 9056A *Determination of Inorganic Anions by Ion Chromatography*; Revision 1; February 2007. (Update IV)
- *Standard Methods for the Examination of Water and Wastewater, Online Edition;* American Public Health Association: Washington, DC.
    - SM4020: Quality Assurance/Quality Control
    - SM4110B: *Determination on Anions by Ion Chromatography; Ion Chromatography with Chemical Suppression of Eluent Conductivity*; 2000

16.0 Method Modifications and Clarifications

16.1 Incorporation of Other Matrices

This procedure may be modified to analyze other, non-routine matrices (i.e., waste, wipe, tissue, or filter samples) based on the needs of the client. This must be arranged by the Project Manager at the initiation of the project to ensure thorough client and laboratory understanding of any procedural or QC modifications that must be made to accommodate the matrix. The laboratory will use its routine soil RLs (converted for initial and final volumes, etc.), and soil QC limits to evaluate non-routine matrices. Soil DOCs can be used to satisfy analyst demonstrations of capability for these types of non-routine matrices.

16.2 Other Considerations

16.2.1 For EPA 9056 and EPA 9056A, the laboratory has adopted the soil preparation technique from EPA Method 300.0 with the following modification. For soil extraction, Section 11.7 of EPA Method 300.0 recommends using an amount of laboratory reagent water equal to 10 times the soil weight and mixing with a magnetic stirrer for 10 minutes to perform the extraction. The laboratory performs a DI Leach procedure using laboratory reagent water equal to 20 times the soil weight and tumbling for 2 hours.

16.2.2 EPA 9056 and EPA 9056A do not provide a holding time for fluoride, chloride, bromide, or sulfate. The laboratory has adopted the holding times specified in EPA 300.0. Additionally, the reference methods do not specify a holding time for the soil leaching

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procedure. The laboratory defaults to a 28-day holding time from sample collection to preparation and a 48-hour holding time from sample preparation to analysis for nitrate and nitrite, and a 28-day holding time from sample collection to preparation and analysis for the other anions.

16.2.3 EPA 9056 and EPA 9056A recommend using eluent to dilute samples. The laboratory uses reagent water to perform sample dilutions.

16.2.4 EPA 9056, EPA 9056A, and EPA 300.0 recommend using a carbonate/bicarbonate eluent. Due to advances in instrumentation, the laboratory uses a potassium hydroxide eluent.

16.2.5 EPA 9056 and EPA 9056A requires corrective action be taken for any values detected in the instrument blank and method blank greater than 10% of the reporting limit. The laboratory's RLs are usually within a factor of 3-5 times the MDL, and the laboratory does not report values below its MDL. For these reasons, the laboratory has adopted the criteria that the instrument blank and method blank must be less than the MDL. This requirement is consistent with the requirement found in EPA 300.0.

16.2.6 Section 9.2.4 of EPA Method 300.0 requires a Method Detection Limit (MDL) study to be performed every 6 months or when a new analyst begins. The laboratory performs an MDL study every six months or when a significant change is made to the equipment (ex: new column/guard column type, new instrument, etc.). The laboratory does not use analyst-specific MDLs; therefore, new analysts are required only to perform IDOC as described in Section 12.3.

16.2.7 The calibration procedure outlined in EPA 9056 and EPA 9056A specifies a linear curve to be used and does not incorporate average response factor and/or quadratic curves, as allowed by the other SW-846 chromatography methods. For this reason, average response factor, quadratic curves, and higher order curves are prohibited in this SOP. If needed, and with Department Manager approval, average response factor and quadratic curves may be used for EPA 300.0 and SM4110B.

16.2.8 The standard expiration dates used by the laboratory have been extended beyond those specified in the reference methods. A stability study has been performed and shows the standards are acceptable for use for the timeframes specified in this SOP. Note: SC DHEC does not recognize this method modification and requires the method-defined holding times as listed in the SC DHEC Project Requirement Summary (PRS). (Stock Standards: 1 month, Prepared Standards: 1 day for NO2, and 1 week for all other anions)

16.2.9 The EPA Manual for the Certification of Laboratories Analyzing Drinking Water requires a LFB at the MRL to be performed each day. The laboratory meets this requirement by preparing an LCS at the RL in each EPA 300.0 batch of drinking water samples.

16.2.10 EPA 9056 states that the daily initial CCV must be within 10% to be acceptable and subsequent CCVs must be within 5% to be acceptable. Due to the nature of the sample matrices encountered, the laboratory incorporates the 10% criteria for all CCVs. This criteria is consistent with that defined by EPA 9056A. Additionally, CCV response is verified against the CCV level of the initial calibration curve.

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16.2.11 EPA 300.0 requires a Linear Calibration Range to be determined initially and verified every 6 months thereafter. This study is not meant to extend the linear range of the calibration. If an analyte is measured at a concentration greater than the highest calibration standard, the sample must be analyzed at a dilution to bring the target analyte into the range of the initial calibration.

16.2.12 The laboratory's default batch QC items are outlined in Section 9.1. Due to the complexity of the laboratory's work mix (i.e., types of samples, varying state- and client- requirements, etc.) and to aid in scheduling and combining like methods, these default requirements may be more extensive than those specified by the reference methods. For example, the laboratory has adopted the EPA Clean Water Act requirement to perform an MSD per batch for all EPA 300.0 non-drinking water samples. To satisfy client- and/or regulatory expectations for batch precision, an LCSD may be performed when insufficient sample is provided for MS and/or MSD (although not required by some of the methods). Also, to note, matrix spike duplicates are routinely performed for EPA 9056 and EPA 9056A in lieu of sample duplicates (as allowed by the reference methods). If needed, and with Department Manager approval, these additional QC items may be omitted to match those listed within the reference methods. Additionally, where method-specified control limits have not been specified, the laboratory uses default and/or in-house limits.

16.2.13 To aid in scheduling and combining like methods, the laboratory has adopted a tighter CCV frequency for EPA 300.0. That is, EPA 300.0 only requires CCV after every 20 samples; however, the laboratory routinely performs CCV after every 10 samples as required by EPA 9056 and EPA 9056A. If needed, and with Department Manager approval, this frequency may be reduced for EPA 300.0 to match that listed in the reference method.

16.2.14 Per client request, the laboratory has incorporated iodate and iodide into EPA 300.0. Similar procedures and acceptance criteria have been adopted to mirror the other analytes specific in the reference method. A 28-day holding time has been applied.

16.2.15 The SM4110B reference method states to perform an initial calibration daily and whenever CCV is not acceptable. The response of the instrument is very stable; therefore, the laboratory only performs re-calibration upon unacceptable CCV.

16.2.16 The SM4110B reference method states that the MDL should be determined annually but does not mandate a specific procedure to be used. The laboratory performs MDL Studies initially, in accordance with 40CFR Part 136B, and re-establishes the MDL annually or quarterly (as dictated by agency-specific requirements) via an MDLV study.

16.2.17 The reference methods allow the use of 3 calibration points for the initial calibration; however, the laboratory typically performs a minimum of 5 points. 3 points may be used with Technical Manager approval.

17.0 Attachments

The following Tables, Diagrams, and/or Validation Data are included as Attachments:

Attachment 1: SOP Summary

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Attachment 2: Sample Collection, Preservation, and Holding Time Table
Attachment 3: QC Summary
Attachment 4: Instrument Maintenance and Troubleshooting
Attachment 5: Maximum Contaminant Level (MCL) Table
Attachment 6: Additional Information Regarding NO3 and NO2 Reporting

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Attachment 1:
SOP Summary

Sample Preparation and Analysis Summary

Samples are analyzed via ion chromatography using eluent suppression and conductivity detection. A potassium hydroxide eluent conducts the sample through the chromatography system, which consists of a guard column, a separator column, a suppressor, and a conductivity detector. The guard column screens out potential interferences, the separator column separates the anions, and the conductivity detector detects the anions from their electrical conductivity in solution.

Soil samples are prepared/leached by tumbling 5g of sample with 100mL reagent water. Prior to analysis, all soil leachates are filtered through a 0.20um filter. Additionally, all water samples and soil leachates are filtered through a 0.45um filter cap prior to analysis to remove particulates that may interfere with column flow. Therefore, the data may represent only the dissolved ionic species.

Bomb combustates for the determination of total halogens are collected in reagent water, filtered, and analyzed for the target analytes.

Example Analytical Sequence

Description	Comments
Blank	Di water
Initial Calibration	
ICB	Method blank
ICV	Second Source (same as LCS)
Samples & Batch QC Items	10 injections, not including LCSD; Includes MS/MSD/DUP
CCV	
CCB	
Samples & Batch QC Items	10 injections including MS/MSD/DUP
CCV	
CCB	
Samples & Batch QC Items	10 injections including MS/MSD/DUP
CCV	
CCB	

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Attachment 2:
Sample Collection, Preservation, and Holding Time Table

Matrix	Routine Sample Container	Default Sample Size	Minimum Sample Size	Chemical Preservation	Thermal Preservation	Dechlorination Agent	Holding Time
Water	125mL plastic	5mL	2.5mL	None	0-6°C	None	F, Cl, Br, SO_4: 28 Days NO2, NO3: 48 Hours
Soil	8oz plastic	5g	2.5g	None	0-6°C	None	F, Cl, Br, SO_4: 28 days[1] NO2, NO3: 28 Days to prep; 48 Hours to analyze
Combustate[2]	125mL plastic	5mL	2.5mL	None	0-6°C	None	28 days[1]

[1] ***Inclusive*** of extraction/combustion time
[2]Combustates are analyzed for Cl only. Contact Technical Management if other halogens are requested.

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Attachment 3:
QC Summary

QC Item	Frequency	Criteria	Corrective Action
Initial Calibration (ICAL) - Minimum 3 standards and a blank	Prior to sample analysis; when CCV fails; at least every 6 months	r^2 >= 0.990	Refer to SOP SA-QA-16
2nd Source Initial Calibration Verification (ICV)	After analysis of initial calibration standards	±10% of true value	Refer to SOP SA-QA-16
Continuing Calibration Verification (CCV)	At the beginning of the analysis, after every 10 samples, and end of the analysis	Mid-Level: ±10% of true value Low-Level: ±25% of true value	Refer to SOP SA-QA-16
Calibration Blank (ICB/CCB): - Can also be used as the method blank.	ICB: Before ICV CCB: After CCV	result < MDL	Refer to SOP SA-QA-17
Method Blank (MB)	1 per batch	result < MDL	Refer to SOP SA-QA-17
Laboratory Control Sample (LCS)	1 per batch	MLG Limits	Refer to SOP SA-QA-17
Laboratory Control Sample Duplicate (LCSD)	1 per batch, when insufficient sample for MS/MSD	MLG Limits	Refer to SOP SA-QA-17
Matrix Spike (MS)	EPA 300.0: 10% of samples performed; i.e., 2 per batch	MLG Limits	Refer to SOP SA-QA-17

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QC Item	Frequency	Criteria	Corrective Action
	Other Methods: 5% of samples performed; i.e., 1 MS per batch		
Matrix Spike Duplicate (MSD)	1 per batch	MLG Limits	Refer to SOP SA-QA-17
Sample Duplicate (SD)	SM 4110B, only: 1 per batch	MLG Limits	Refer to SOP SA-QA-17
Low-Level Laboratory Control Sample (LLCS) – Spiked at the RL	EPA 300.0 DW Only: 1 per batch	The analyte must be detected to be acceptable.	- If the LCS meets criteria, initiate NCM and report data - If the LCS does not meet criteria, reanalyze batch
Initial Demonstration of Capability (IDOC)	Initially, per analyst, per analyte/method/matrix combination	Refer to SOP SA-QA-06	Refer to SOP SA-QA-06 Note: Unsupervised work must not begin until acceptable IDOC is obtained.
Continuing Demonstration of Capability (CDOC)	Annually, per analyst, per analyte/method combination	Refer to SOP SA-QA-06	Refer to SOP SA-QA-06
Reporting Limit Verification (RLV)	Upon method/instrument set-up, per analyte/method/matrix combination. Then quarterly thereafter (for DOD ELAP) or annually thereafter (for non-DOD ELAP)	Refer to SOP SA-QA-07	Refer to SOP SA-QA-07
Method Detection Limit Study (MDL)	Upon method/instrument set-up, per analyte/method/matrix combination and every 6 months thereafter	Refer to SOP SA-QA-07	Refer to SOP SA-QA-07
MDL Verification (MDLV)	Upon method/instrument set-up, per analyte/method/matrix combination.	Refer to SOP SA-QA-07	Refer to SOP SA-QA-07

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QC Item	Frequency	Criteria	Corrective Action
	Then quarterly thereafter (for DOD ELAP) or annually thereafter (for non-DOD ELAP)		
Retention Time Window Study	Annually, with each new column, or if significant method change occurs per SOP SA-QA-08	Define a reasonable window for each analyte or retention time range and apply to each analysis per SOP SA-QA-08	-Evaluate instrument performance, column, and integration system.
Low-Level of Quantitation (LLOQ)	After analysis of initial calibration standards	The concentration must be within 50-150% of the actual concentration to be acceptable.	-Evaluate ion chromatogram and integrations. Check calculations -Reanalyze standards -Remake and reanalyze standard -Recalibrate
Dilution/Fortification	When high concentration of adjacent peak makes identification difficult	Peak in dilution and/or fortified sample within established window.	-Dilute or fortify (spike) sample at higher dilution factor or concentration.
Linear Calibration Range (LCR) Study - EPA 300.0 and SM4110B Only	Upon method/instrument set-up, and when LCR verification fails acceptance criteria.	Within ±10% of the true value	This determines the range at which the instrument is considered linear. The highest concentration of the calibration curve can not exceed the highest concentration of the LCR study.
Linear Calibration Range (LCR) Verification - EPA 300.0 Only	Every 6 months	Within ±10% of the initial value	Re-perform the LCR study

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Attachment 4:
Instrument Maintenance and Troubleshooting

Instrument Labeling

Each instrument must be labeled with its name or ID (e.g., MSA, ICP-D, etc.). Additionally, non-operational instruments must be isolated from service or marked as being out of service. Each piece of equipment has an "Operational / Not Operational" sticker that is used for this purpose.

Maintenance Log

A maintenance log must be established for each piece of equipment used in the laboratory. All maintenance that is performed on the instrument must be recorded in the log including:

- analyst or technician performing the maintenance
- date the maintenance was performed
- detailed explanation of the reason for the maintenance
- resolution of the problem and return to control
- all service calls from instrument representatives

Preventive Maintenance

LABORATORY EQUIPMENT PREVENTIVE MAINTENANCE SCHEDULE								
EQUIPMENT ITEM	Service Interval							SERVICE LEVEL
	D	W	M	Q	SA	A	AN	
Conductivity	X							Record Daily (Approximate Range = 0.3-2)
Pressure	X							Record Daily (Approximate Range = 2100-3000psi)
Eluent							X	Reagent Free – ICS200 system will alert the user when the cartridge expires
Suppressor	X							Check daily for leaks
Analytical/Guard Column							X	Replace as needed

D=daily; W=Weekly; M=monthly; Q=Quarterly; SA=semi-annually; A=annually; AN=as needed

Troubleshooting

Troubleshooting should be documented as outlined above. If possible, troubleshooting is best performed in a step-wise manner to systematically isolate instrument components. Refer to the instrument manufacturer's guides for specific information and strategies. Enlist assistance from technical and/or department management as needed.

Contingency Plan

Maintenance contracts are carried for most instrumentation and close contact is maintained with service personnel to ensure optimal instrument functioning. An extensive spare parts inventory is maintained for routine repairs, consisting of sample loops, filters, guard columns, columns, regulators, and other common instrumentation components. Since

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instrumentation is standardized throughout the laboratory network, spare parts and components can be readily exchanged among the network.

In general, the laboratory has at least one backup unit for each critical unit. In the event of instrument failure, portions of the sample load may be diverted to duplicate instrumentation, the analytical technique switched to an alternate approved technique (such as manual colorimetric determination as opposed to automated colorimetric determination), or samples shipped to another properly certified or approved TestAmerica location.

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Attachment 5:
Maximum Contaminant Level (MCL) Table

Primary Drinking Water Regulations	
Contaminant	**MCL (mg/L)**
Fluoride	4.0
Nitrate (as N)	10
Nitrite (as N)	1

Secondary Drinking Water Regulations	
Contaminant	**MCL (mg/L)**
Chloride	250
Fluoride	2.0
Sulfate	250

Note: Fluoride is part of the Primary Drinking Water Regulations; however, it is also included on the list of Secondary Drinking Water Regulations with a lower MCL.

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Attachment 6:
Additional Information Regarding NO3 and NO2 Reporting

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Reporting the *as N* Analytes:

For Drinking Water analyses, the regulated analytes are Nitrate and Nitrite reported *as Nitrogen*. To accomplish this, the laboratory analyzes nitrate and nitrite and uses the following conversion factor based on the molecular weight of the ion versus the molecular weight of nitrogen:

100mg/L nitrate as NO3 = 22.6mg/L nitrate as N
(i.e., NO3 x 0.226 = NO3 as N)

100mg/L nitrite as NO2 = 30.4mg/L nitrite as N
(i.e., NO2 x 0.304 = NO2 as N)

Target processing methods have been set up to perform this conversion automatically.

Reporting the *plus* Analytes

Some clients may request nitrate and nitrite to be reported together as nitrate+nitrite. To accomplish this, the laboratory analyzes nitrate and nitrite separately and sums the final result. EPA 300.0 indicates sulfuric acid should be used as the preservative when analyzing for nitrate+nitrite; however, since the laboratory is not performing a different chemistry to obtain the *plus* result, the same bottle types and holding times used for "regular" nitrate and nitrite are applied.

18.0 Revision History

Summary of Changes from Previous Revision:

- Minor editorial, grammatical, and formatting changes made. Boilerplate text added.
- Included standard and reagent disposal information, Section 7.2
- Revised NO2/NO3 ICAL standard preparation, Section 7.4
- Revised IC parameters to match current set-up, Section 10.3.1
- Included information for additional sample evaluation when the MS/MSD fails high for nitrate and low for nitrite,Section 11.1.7

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OIL & GREASE AND TOTAL PETROLEUM HYDROCARBONS (HEM & SGT-HEM)

(Methods: EPA 1664A and EPA 9071B)

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1.0 Scope and Application

This SOP gives the procedures for the determination of n-hexane extractable material (HEM, also called oil & grease) and the components of n-hexane extractable material that are not adsorbed by silica gel (SGT-HEM, also called total petroleum hydrocarbons or TPH) by gravimetry.

The routine matrices are in water and soil samples. Other matrices may be performed as outlined in Section 16.

The reporting limits (RL), the method detection limits (MDL), and the accuracy and precision criteria associated with this procedure are provided in the LIMS Method Limit Groups (MLGs).

This SOP was written by and for TestAmerica's Savannah laboratory.

2.0 Summary of Method

Aqueous Samples: The preserved contents of the sampling container (nominally 500mL) are transferred to a separatory funnel. The container is rinsed with n-hexane and added to the separatory funnel. The sample is serially extracted three times with n-hexane, and the extract is dried over sodium sulfate. The solvent is then evaporated from the extract, and the dried residue is weighed to determine the HEM content. If SGT-HEM is to be determined, the residue is re-dissolved in n-hexane and treated with silica gel. The solution is filtered to remove the silica gel, the solvent is evaporated and the SGT-HEM is weighed.

Soil Samples: A 30g aliquot of homogenized sample is dried with anhydrous sodium sulfate and serially extracted three times with n-hexane using an ultrasonic extractor. The extract is filtered, and the solvent is evaporated. The dried residue is weighed to determine the HEM content. If SGT-HEM is to be determined, the residue is re-dissolved in n-hexane and is treated with silica gel. The solution is filtered to remove the silica gel, the solvent is evaporated, and the SGT-HEM is weighed.

This SOP is based on the following methods: EPA Method 1664, Revision A (February 1999) and SW-846 Method 9071B.

3.0 Definitions

Refer to the Glossary Section of the *Quality Assurance Manual* (QAM) for a complete listing of applicable definitions and acronyms.

HEM – n-hexane extractable material, also called oil & grease

SGT-HEM – silica gel treated n-hexane extractable material, also called total petroleum hydrocarbons or TPH

4.0 Interferences

4.1 Procedural Interferences

4.1.1 Interferences may be caused by contaminants in solvents, reagents, glassware, and other sample processing apparatus and can make identification and/or quantification of the target analytes difficult.

4.1.2 All sample collection containers are single-use disposable containers which limits the potential for contamination. All non-disposable labware must be scrupulously cleaned in accordance with the posted Labware Cleaning Instructions to ensure it is free from contaminants and does not contribute artifacts.

4.1.3 High purity reagents and solvents are used to help minimize interference problems. Acetone, hexane, and sulfuric acid must be verified prior to use in accordance with the TestAmerica Solvent Lot Testing Program.

4.1.4 Instrument and/or method blanks are routinely used to demonstrate all reagents and apparatus are free from interferences under the conditions of the analysis.

4.1.5 The major interferences with this procedure are artifacts introduced during extraction. Reagent blanks must be analyzed and monitored closely to check for laboratory contamination. Glassware must be thoroughly cleaned. Remove all fingerprints and moisture on the outside of the weighing pan prior to placing in the desiccator.

4.1.6 This procedure is entirely empirical. Duplicate results can be obtained with a high degree of precision only by strict adherence to all details of the process. Solvent removal results in the loss of short-chain hydrocarbons and simple aromatics by volatilization. Significant portions of petroleum distillates from gasoline through No. 2 diesel fuel are lost in this process. Strict adherence to sample drying time is necessary to standardize the gradual loss of weight due to volatilization.

4.1.7 During the cooling of the distillation pan and extracted material, a gradual increase in weight may be observed if a desiccator is not used. Make sure all samples are dried, stored, and weighed in a consistent manner to ensure reproducible results. Sample extracts must be stored in a desiccator for at least 30 minutes, preferably overnight, prior to weighing. Make sure the desiccant is maintained properly.

4.2 Matrix Interferences

4.2.1 Matrix interferences may be caused by contaminants that are co-extracted from the sample matrix.

4.2.2 The presence of moisture in the sample extract can cause a considerable positive bias; therefore, carefully dry all extracts using anhydrous sodium sulfate to exclude water from the final extract. Some sample matrices can increase the amount of water partitioned into the organic extraction fluid. When the extraction solvent from this type of sample is dried

with sodium sulfate, the drying capacity of the sodium sulfate can be exceeded, thus allowing sodium sulfate to dissolve and pass into the tared pan. If this occurs, re-dissolve the extract in hexane, filter, and capture the extract in a clean, tared pan; and continue on with the analysis.

5.0 Safety

Employees must abide by the policies and procedures in the TestAmerica Environmental Health and Safety Manual (EHSM), the TestAmerica Savannah Addendum to the EHSM, and this document.

This procedure may involve hazardous materials, 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 to follow appropriate safety, waste disposal, and health practices under the assumption that all samples and reagents are potentially hazardous.

The analyst must protect himself/herself from exposure to the sample matrix. Many of the samples that are tested may contain hazardous chemical compounds or biological organisms. The analyst must, at a minimum, wear protective clothing (lab coat), eye protection (safety glasses or face shield), disposable nitrile gloves, and closed-toe, nonabsorbent shoes when handling samples.

5.1 Specific Safety Concerns or Requirements

The toxicity or carcinogenicity of chemicals used in this method has not been precisely defined; each chemical should be treated as a potential health hazard, and exposure to these chemicals should be minimized.

Hexane and acetone are flammable solvents. They can cause irritation to the respiratory tract. Overexposure can cause fatigue, lightheadedness, headache, dizziness, and blurred vision.

The evaporation procedure produces vapors that can be flammable if the proper safety procedures are not followed. **IT IS EXPRESSLY FORBIDDEN TO USE HOT PLATES FOR THE EVAPORATION OF FLAMMABLE SOLVENTS.** The Speed Vap III or equivalent will be used. All evaporation must be performed under a fume hood.

Sulfuric acid is a strong oxidizer and is a corrosive. It will react violently when combined with organic compounds, possibly producing fire. Inhalation can cause irritation of the nose, throat, mucus membranes, and upper respiratory tract. Contact with the eyes can cause blurred vision, redness, pain, and even blindness.

Methylene chloride is a carcinogen and an irritant. It causes irritation to the respiratory tract and has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting, and headache. Methylene chloride may be absorbed through the skin and can cause irritation and pain to the skin and eyes.

5.2 Primary Materials Used

The following is a list of the materials used in this procedure, which have a serious or significant hazard rating, and a summary of the primary hazards listed in their MSDS.

NOTE: This list does not include all materials used in the procedure. A complete list of materials used in this procedure can be found in the Reagents and Standards Section and the Equipment and Supplies Section of this SOP

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 found using the "MSDS" link on the Oasis homepage, on the EH&S webpage on Oasis, and on the QA Navigator.

Material	Hazards	Exposure Limit[1]	Signs and Symptoms of Exposure
Acetone	Flammable	1000ppm TWA	Inhalation of vapors irritates the respiratory tract. May cause coughing, dizziness, dullness, and headache.
Hexane	Flammable Irritant	500ppm TWA	Inhalation of vapors irritates the respiratory tract. Overexposure may cause lightheadedness, nausea, headache, and blurred vision. Vapors may cause irritation to the skin and eyes.
Methylene Chloride	Carcinogen Irritant	25ppm TWA 125ppm 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	1mg/m^3 TWA	Inhalation produces damaging effects on the mucous membranes and upper respiratory tract. Symptoms may include irritation of the nose and throat, and labored breathing. Symptoms of redness, pain, and severe burn can occur. Contact can cause blurred vision, redness, pain and severe tissue burns. Can cause blindness.
[1]Exposure limit refers to the OSHA regulatory exposure limit.			
Note: Always add acid to water to prevent violent reactions.			

6.0 Equipment and Supplies

6.1 Equipment and Instrumentation

Analytical Balance – Verify in accordance with SOP SA-AN-100: *Laboratory Support Equipment (Verification and Use)*

Thermometers – Verify in accordance with SOP SA-AN-100: *Laboratory Support Equipment (Verification and Use)*

Desiccator

Oven – for anhydrous sodium sulfate

Horizon Speed Vap III

Ultrasonic disrupter – Tekmar or equivalent with horn-type titanium-tipped sonication probe. The sonicator should be capable of operating in the pulse mode at full power.

Sonabox – The sonicator should be placed in the sonabox to reduce noise. The sonabox must be placed under a fume hood.

Mechanical Shaking Device

Data System – Computer system with an RS236 port to link an analytical balance and the software required to electronically transfer weights into LIMs. Also use Microsoft Excel software to capture raw data on a spreadsheet.

Laboratory timer

6.2 Lab Supplies

Volumetric Containers – various sizes; Class A, where applicable. Verify in accordance with SOP SA-AN-100: *Laboratory Support Equipment (Verification and Use)*

Mechanical Pipettes – various sizes. Verify in accordance with SOP SA-AN-100: *Laboratory Support Equipment (Verification and Use)*

Disposable Graduated Pipettes – various sizes. Verify in accordance with SOP SA-AN-100: *Laboratory Support Equipment (Verification and Use)*

Gas-Tight Syringes – various sizes. Verify in accordance with SOP SA-AN-100: *Laboratory Support Equipment (Verification and Use)*

pH paper

Separatory funnels: 1L and 2L with Teflon stopcock

Filter Paper: #41 Whatman, 12.5cm diameter

Glass Wool

Glass funnel

Weigh pans, disposable – Aluminum

Magnetic stirrer

Uncontrolled Copy

PTFE-coated magnetic stirring bars

Pre-cleaned 500mL soil extraction bottles: Discard the bottles after use.

Stainless steel spatulas

Detergent – FL-70 or equivalent, used for washing non-disposable labware.

Glass stirring rods

6.3 Sample Collection Containers
All sample collection containers are single-use disposable containers which limits the potential for contamination.

The routine sample collection containers supplied by the laboratory are purchased with Certificate of Analysis attesting to purity as listed below:

Water: 500mL amber glass
Solid: 16oz soil jar

7.0 Reagents and Standards

7.1 Expiration Dates
Expiration dates (time from initial use or receipt to final use) for standard and reagent materials must be set according to the guidance in this SOP. Note: These are maximum expiration dates and are not to be considered an absolute guarantee of standard or reagent quality. Sound judgment must be used when deciding whether to use a standard or reagent. If there is doubt about the quality of a standard or reagent material, a new material must be obtained or the standard or reagent material verified. Data quality must not be compromised to extend a standard's life – i.e., when in doubt, throw it out.

The expiration date of any standard or reagent must not exceed the expiration date of the standard or reagent that was used to prepare it; that is, the "children may not outlive the parents".

Unless listed elsewhere in this SOP, the expiration dates given below apply.

7.1.1 The expiration date for unopened standards and reagents is the manufacturer's expiration date.

7.1.2 The expiration date for opened stock reagents is the manufacturer's expiration date or 5 years from the date opened, whichever is sooner.

7.1.3 The expiration date for opened stock standards is the manufacturer's expiration date or 12 months from the date opened, whichever is sooner.

7.1.4 The expiration date for prepared reagents is 6 months from the date prepared or the expiration date of the parent reagent, whichever is sooner.

7.1.5 The expiration date for prepared standards is 3 months from the date prepared or the

expiration date of the parent standard, whichever is sooner.

7.2 Reagents

Reagents must be prepared and documented in accordance with SOP SA-AN-41: *Reagent and Standard Materials Procedures.*

Acetone, hexane, and sulfuric acid must be verified prior to use in accordance with the TestAmerica Solvent Lot Testing Program.

7.2.1 Purchased Reagents

7.2.1.1 Blank Matrix – Ottawa sand. Used for the preparation of soil QC samples.

7.2.1.2 Laboratory Reagent Water – ASTM Type II

7.2.1.3 Sodium sulfate, Na_2SO_4 – anhydrous, granular, purified in an oven at 450°C overnight.
Storage: Room temperature, in sealed glass container
Expiration: 6 months from purification

7.2.1.4 n-Hexane – 85% purity
Storage: Room temperature, in Flammables Cabinet
Expiration: manufacturer's expiration date

7.2.1.5 Sulfuric acid (H_2SO_4) – concentrated, reagent grade
Storage: Room temperature, in Acids Cabinet under a hood
Expiration: manufacturer's expiration date

7.2.1.6 Silica gel: Anhydrous, 75-150 micrometers, Davisil Grade 923. Dry at 110°C for 24 hours minimum, and store in a tightly sealed container.
Storage: Room temperature, in sealed glass container
Expiration: 6 months from purification

7.2.1.7 Desiccant
Storage: Room temperature, in sealed glass container
Expiration: manufacturer's expiration date

7.2.1.8 Acetone – residue grade
Storage: Room temperature, in Flammables Cabinet
Expiration: manufacturer's expiration date

7.2.1.9 Methylene Chloride – residue grade
Storage: Room temperature, in Solvent Cabinet
Expiration: manufacturer's expiration date

7.2.2 Prepared Reagents

7.2.2.1 Sulfuric acid solution (1:1 H_2SO_4) – Carefully add an equal volume of acid into an equal volume of reagent water.
Storage: Room temperature, in Acids Cabinet under a hood
Expiration: 6 months from preparation

7.3 Standards

Standards must be prepared and documented in accordance with SOP SA-AN-41: *Reagent and Standard Materials Procedures.* Certificates of analysis or purity must be received with all purchased standards, and scanned and filed in the Data Archival Folder on the G-drive.

7.3.1 Purchased Standards

7.3.1.1 Hexadecane – 98% minimum purity
Storage: Room temperature, in cabinet under a hood
Expiration: manufacturer's expiration date

7.3.1.2 Stearic acid – 98% minimum purity
Storage: Room temperature, in cabinet under a hood
Expiration: manufacturer's expiration date

7.3.2 Prepared Standards

7.3.2.1 MS/LCS Spiking Solution – contains stearic acid and hexadecane in acetone at a concentration of 2.0mg/mL each.

Quantitatively transfer, in small aliquots, 2.000g ± 0.020g stearic acid and 2.000g ± 0.020g hexadecane to a 100mL volumetric flask containing 10mL acetone. Complete the quantitative transfer of the solution with 50mL of acetone and dilute to volume with acetone. Transfer the solution to a glass container equipped with a Teflon-lined cap. Mark the solvent level on the side of the container. This level should be marked each time the solution is used.

Note: The solution may require warming for complete dissolution of stearic acid.

Immediately prior to use, verify the solvent volume. If solvent has evaporated, dilute to the mark with acetone before proceeding. Warm to re-dissolve all visible precipitate. If doubt exists about the concentration of the spiking solution, verify the concentration by transferring 1.0mL of the solution to a tared flask and evaporating the solvent. The residue should be 40mg±1mg. If not, prepare a new spiking solution.

The table is based on a 500mL extraction volume for aqueous samples and a 30g extraction volume for soil samples, assuming 100% solids.

Matrix	HEM (MS)	HEM (PAR/LCS)	SGT-HEM (MS)	SGT-HEM (LCS)
Water	40mg/L	40mg/L	20mg/L	20mg/L
Soil	1300mg/kg	1300mg/kg	670mg/kg	670mg/kg

Storage: in the dark at room temperature
Expiration: 3 months from preparation

8.0 Sample Collection, Preservation, Shipment, and Storage

8.1 Aqueous Samples

Aqueous samples are routinely collected in 500mL amber glass containers with Teflon-lined lids containing 2mL 1:1 sulfuric acid preservative. The preservative should be sufficient to achieve a sample pH of less than 2. Duplicate samples are collected in the field for QC purposes.

Note: Smaller containers may be used for sample collection if the concentration of HEM or SGT-HEM is known to be greater than 500mg/L.

Samples must be iced at the time of collection and maintained at 4°C (less than 6°C but not frozen) until the time of preparation. Samples must be prepared within 28 days of collection. Extracts are stored in the desiccator until the time of analysis and analyzed as soon as possible after extraction.

NCMs must be initiated for samples collected in improper containers and containing improper or insufficient preservatives. NCMs must be initiated for samples where the sample container is not >90% full.

8.1.1 Preservation Checks

Sample preservation is checked when the samples are extracted. Refer to Section 10.3.2.3.

8.2 Soil Samples

Soil samples are routinely collected in 16oz soil jars.

Samples must be iced at the time of collection and maintained at 4°C (less than 6°C but not frozen) until the time of preparation. Samples must be prepared within 28 days of collection. Extracts are stored in the desiccator until the time of analysis and analyzed as soon as possible after extraction.

9.0 Quality Control

SOP SA-QA-17: *Evaluation of Batch QC Data* and the SOP Summary in Attachment 3 provide requirements for evaluating QC data.

9.1 Batch QC

A preparation batch consists of up to 20 environmental samples and the associated QC items. The routine minimum QC items required for each preparation batch are: a method blank, a laboratory control sample (LCS), a matrix spike (MS), and a matrix spike duplicate (MSD).

If there is insufficient sample to perform the MS/MSD, the LCS must be prepared in duplicate (i.e., LCSD), and an NCM must be initiated to denote this situation.

Note: If an LCS and LCSD are performed, both QC items must be evaluated and reported.

Acceptable recoveries (as well as %RPD) for both LCS and LCSD are required.

- For aqueous samples, the routine container supplied for this method is a 500mL container. 500mL is required for extraction. Insufficient sample is defined as receiving less than a total of 1500mL.
- For soil samples, the routine container supplied for this method is a 16oz container. 30g is required for extraction. Insufficient sample volume is defined as receiving less than a total of 60g.

Batch QC must meet the criteria given in Attachment 3 of this SOP.

9.2 Instrument QC

9.2.1 Initial Calibration (ICAL)

These samples are analyzed using the analytical balance. The balance must be calibrated in accordance with SOP SA-AN-100: *Laboratory Support Equipment (Verification and Use)*.

9.2.2 Quality Control Sample (QCS, Second Source Verification)

A Quality Control Sample (QCS) must be analyzed monthly. The QCS must be prepared using a standard obtained from a second source. This requirement is met with each batch as the LCS is a secondary source.

The QCS must be within the LCS limits in the MLG to be acceptable.

The QCS concentration currently in use in the laboratory is equivalent to the LCS. Refer to Section 7.3.2.1 for the standard preparation instructions. Another standard concentration may be used provided it is fully documented in accordance with SOP SA-AN-41.

9.2.3 Continuing Calibration Verification

In addition to the verifications required by SOP SA-AN-100, the balance calibration must be verified before and after each analytical batch with a 2mg and a 1000mg weight.

The 2mg weight must be within +/-0.2mg to be acceptable.

The 1000mg weight must be within +/-5mg to be acceptable.

9.3 Corrective Action for Out-of-Control Data

When the quality control parameters do not meet the criteria set forth in this SOP, corrective action must be taken in accordance with SOP SA-QA-05: *Preventive and Corrective Action Procedures* and the QC Summary Table in Attachment 3. SOP SA-QA-05 provides contingencies for out-of-control data and gives guidance for exceptionally permitting departures from approved policies and procedures. Nonconformance Memos must be initiated to document all instances where QC criteria are not met and all departures from approved policies and procedures.

10.0 Procedure

10.1 Sample Preparation

The sample preparation steps are an integral part of the analysis and are included in Section 10.3.

10.2 QC Sample Preparation

10.2.1 Aqueous QC Sample Preparation

10.2.1.1 Method Blank – Add 500mL of laboratory reagent water to the separatory funnel designated for the method blank. Prepare as a sample following the procedure outlined in Section 10.3.2.

10.2.1.2 Laboratory Control Sample (LCS) – Add 500mL of laboratory reagent water to the separatory funnel designated for the LCS. Spike the LCS with 0.50mL of the 40mg/mL spiking solution. Prepare as a sample following the procedure outlined in Section 10.3.2.

10.2.1.3 Matrix Spike(s) (i.e., MS/MSD) – Add 500mL of sample to the separatory funnel designated for the MS and MSD. Spike the MS/MSD with 0.50mL of the 40mg/mL spiking solution. Prepare as a sample following the procedure outlined in Section 10.3.2.

10.2.2 Solid QC Sample Preparation

10.2.2.1 Method Blank – Weigh 30g of Ottawa Sand into the extraction vessel designated for the method blank. Prepare as a sample following the procedure outlined in Section 10.3.3.

10.2.2.2 Laboratory Control Sample (LCS) – Weigh 30g of Ottawa Sand into the extraction vessel designated for the LCS. Prior to adding the sodium sulfate to the samples, spike the LCS with 1.0mL of the 40mg/mL spiking solution. Prepare as a sample following the procedure outlined in Section 10.3.3.

10.2.2.3 Matrix Spike(s) (i.e., MS/MSD) – Weigh 2 additional 30g portions of the sample in the batch selected as the MS and MSD into the designated extraction vessels. Prior to adding the sodium sulfate to the samples, spike the MS and MSD with 1.0mL of the 40mg/mL spiking solution. Prepare as a sample following the procedure outlined in Section 10.3.3.

10.3 Sample Analysis

The default procedure is to exclude QC items (method blank, LCS/LCSD, and MS/MSD) in determining the maximum number of samples in the batch.

10.3.1 Preparation of the Weighing Pans

Inspect each desiccator cabinet door and the desiccant in each desiccator prior to beginning the preparation of the weighing pans. The door should seal securely. The

desiccant should be blue. If the desiccant begins to turn pink around the edges, it must be replaced. See Attachment 4 for routine maintenance items.

10.3.1.1 Collect the required number of aluminum pans (one for each sample and QC item) and label each pan.

Note: From this point forward, wear gloves when handling the pans.

10.3.1.2 Rinse each pan with 5-10mL of hexane. Discard the rinses in the flammable waste container. Set the pans on a Kimwipe under the hood to allow the residual solvent to evaporate.

10.3.1.3 Place the pans in the desiccator for at least two hours (overnight is recommended).

10.3.1.4 Check the calibration of the analytical balance and record the results for the 2mg and 1000mg weights. The results of these weights must be within ±10% at 2mg (i.e., 0.0018g-0.0022g) and ±0.5% at 1000mg (i.e., 0.9950g-1.0050g).

10.3.1.5 Remove each pan directly from the desiccator and weigh the pan on the analytical balance. Record the weight of the pan (the "tare weight") in the "HEM Tare Weight" cell.

10.3.1.6 Place the pans back into the desiccator until it is time to determine the residue of the samples and quality control items in Section 10.3.5.

10.3.2 Aqueous Sample Extraction

10.3.2.1 Remove the samples from the refrigerator and let them warm to room temperature.

10.3.2.2 Note the volume of sample in the container. The sample container should be full at the time of receipt. If the sample container is not >90% full, initiate a Nonconformance Memo.

10.3.2.3 Check and record the pH of each sample as follows:

- Mix the sample by inverting the sample several times.
- Dip a stirring rod into the sample and then touch the stirring rod to a piece of narrow range pH paper. Rinse the stirring rod with n-hexane (to ensure no extractable material is lost on the stirring rod) and collect the rinsate in the separatory funnel.
- Note the pH of the sample on the prep batch sheet. The pH of a properly preserved sample should be less than or equal to 2.
- If the pH is >2, carefully add 1:1 sulfuric acid to adjust to pH <=2. Do not sub-divide water samples in the laboratory for oil and grease. Notify the Project Manager via a non-conformance report.

10.3.2.4 Gather and label the glassware and materials listed below as needed to extract the samples:

- 1 2L Teflon separatory funnel for each sample and QC item
- 1 glass funnel for each sample and QC item

- 1 filter paper, folded into a cone, for each sample and QC item
- 5g purified sodium sulfate for each sample and QC item
- 125mL glass flask or 125mL Erlenmeyer flask for each sample and QC item

The minimum QC items for each batch of twenty or fewer samples are listed in Section 9.1.

10.3.2.5 Fold the filter paper twice into a cone shape and place it into a glass funnel. Add approximately 5g of purified sodium sulfate to the cone to serve as a drying column for the extract.

10.3.2.6 Rinse all glassware thoroughly with n-hexane.

10.3.2.7 Mark the sample bottle at the water meniscus for later determination of sample volume.

10.3.2.8 Mix the sample well by inverting the container several times and transfer the entire sample into the separatory funnel.

Note: Do not divide or subsample an oil and grease sample. The entire sample must be used and the bottle rinsed (Section 10.3.2.9) to remove all traces of oils and greases from the container.

10.3.2.9 Add 30mL of n-hexane to each sample bottle, shake the container for 10 seconds, and transfer the solvent to the separatory funnel.

10.3.2.10 Shake each funnel for 10-20 seconds and release the pressure under the hood.

10.3.2.11 Place six separatory funnels on the automatic shaker and shake the samples for approximately five minutes.

10.3.2.12 Remove the separatory funnels from the shaker and place on the rack. Place the next six separatory funnels on the shaker while the water/hexane layers are separating in the first set. Allow the solvent/extract phase (top) to separate from the water phase (bottom) for a minimum of five minutes.

Note: While one set of funnels is shaking, another set should be on the ring stands to allow the extract to separate from water layer. Continue to rotate the samples between the shaking and separating processes.

10.3.2.13 Collect the water phase in the sample container. Drain the solvent phase/extract through the drying column prepared in Section 10.3.2.5, collecting the extract into a labeled hexane-rinsed 125mL beaker or Erlenmeyer flask.

10.3.2.14 If an emulsion forms between the phases and is greater than one-third the volume of the solvent layer, the analyst must employ additional techniques to break up the emulsion. If the emulsion is very bad, perform all three extractions before attempting to break the emulsion. Use one or more of the following procedures to break the emulsion:

1) Stir the extract vigorously with a glass rod or spatula.

2) Drop small quantities of purified sodium sulfate into the emulsion. The sodium sulfate crystals will bind with the emulsion and "pull" the emulsion away from the extract.
3) Prepare a larger filter funnel with more sodium sulfate and a larger piece of filter paper. Pass the emulsion through the filter and collect the extract. Use small aliquots of hexane to rinse the filter.
4) Pour the emulsion into a larger beaker and add sufficient sodium sulfate to bind the emulsion. Break up the hardened sodium sulfate with a glass rod or spatula. Pass the extract through a new glass funnel/filter paper setup and collect the extract.
5) Transfer the extract to a larger beaker and add "dry ice" to the emulsion to freeze it. The "dry ice" will cause the emulsion to freeze and sink to the bottom of the beaker. Pass the extract through a new glass funnel/filter paper setup and collect the extract. If dry ice is not available, cover the beaker with aluminum foil and place the beaker in a freezer for about an hour. The emulsion will freeze and sink to the bottom.

10.3.2.15 Return the water phase to the separatory funnel and repeat the above extraction process two more times (Sections 10.3.2.9 – 10.3.2.13), collecting the three extracts for each sample into the same flask or beaker used for the extract rinse. Rinse the top of the funnel and the filter paper with n-hexane, drain into the labeled receiving flask or beaker and cover the extracts with aluminum foil or cap until the time of analysis. Then proceed to Section 10.3.5.

10.3.3 Soil Sample Extraction

Soil samples must be homogenized prior to preparation in accordance with SOP SA-QA-15: *Compositing, Homogenization, and Segregation of Samples.*

10.3.3.1 Remove the samples from the refrigerator and let them warm to room temperature. Scan or type the samples into the prep batch sheet.

10.3.3.2 Gather the required glassware for the extraction, which includes:

- 1 500mL extraction bottle for each sample and QC item
- 1 glass funnel for each sample and QC item
- 1 filter paper, folded into a cone, for each sample and QC item
- 1 300mL Zymark tube for each sample and QC item
- 1 40mL vial fitted with a Teflon-lined septa for each sample and QC item

The minimum QC items for each batch of twenty or fewer samples are listed in Section 9.1.

10.3.3.3 Fold the filter paper twice into a cone shape and place it into a glass funnel.

10.3.3.4 Rinse all glassware thoroughly with n-hexane.

10.3.3.5 Weigh 30g of each homogenized sample into a pre-cleaned, labeled 500mL extraction bottle. Record the weight for all the samples in this batch.

10.3.3.6 Add granular, purified sodium sulfate to each sample and QC item and stir with a glass rod or stainless steel spatula to form a sandy, free-flowing mixture. The sodium sulfate combines with the water in the sample to "dry" the sample (remove the water). Additional sodium sulfate may be required if the sample is very wet.

Note: The LCS and matrix spike(s) must be spiked prior to the addition of the sodium sulfate.

10.3.3.7 Immediately cover the sample with 70 L of hexane to about 1 inch over the solids. Stir the sample to break up any lumps that may have formed.

10.3.3.8 Place the tip of the sonicator horn in the center of the beaker about ½ inch below the surface of the solvent but above the solid portion.

10.3.3.9 Sonicate for three minutes with the output control knob set at 10, the mode switch set to pulse, and percent duty cycle set at 50%. If the sonication is properly performed, the solids and solvent will vigorously mix each time the sonicator pulses.

10.3.3.10 Pour the extract through the filter/funnel and collect the extract in a pre-cleaned Zymark tube.

10.3.3.11 Repeat Sections 10.3.7 through 10.3.10 two more times with fresh 70mL portions of solvent and collect the extract in the vessel. Cover the Zymark tube with aluminum foil, and leave it under the hood until the concentration step.

Note: Clean the sonicator horn between samples by rinsing with n-hexane.

10.3.3.12 Dispose of the extraction bottle and sample residue by placing in a waste container kept under a hood until removed from the laboratory.

10.3.3.13 Concentrate the extract to approximately 1mL using the Zymark nitrogen blow down apparatus at a constant temperature of 50°C. The Zymark pressure ranges from 4 to 15psi. Zymark tubes with smaller sample volumes are processed with higher pressures (15psi); while, Zymark tubes with larger volumes (those approaching the red "fill" line) are processed with lower pressures (4-5psi).

10.3.3.14 Rinse the Zymark tube with 10mL of hexane and transfer the extract to the weighing pan. Rinse the Zymark tube with two additional 10mL portions of hexane and collect in the weighing pan. Store the extract at room temperature until it is time to evaporate and determine the residue (Sections 10.3.5 and 10.3.6).

10.3.5 Non-Recovery Evaporation Technique (Oil & Grease / HEM Determination)

10.3.5.1 Turn on the SpeedVap III, and set the temperature to 50°C. Allow the instrument to reach this set point before beginning the solvent evaporations.

10.3.5.2 Transfer a portion, approximately 60mL, of the extract to a labeled, tared aluminum pan. Place five pans on each SpeedVap III and close the cover. Increase the flow until the surface of the solvent starts to move but not so high that the solvent is blown out of the pan. Allow the pans to remain in the SpeedVap III until the just a small volume of solvent remains. Open the cover and add the remaining extract to the

appropriate pan. Rinse the flask or beaker with a small volume of hexane and add that to the pan.

10.3.5.3 Remove the sample extract from the Speed Vap III when just a small volume of solvent remains in the pan. Place the pan on a clean surface under the hood that has been covered with a Kimwipe, and allow the pan to cool. Wipe the outside of the pan using a Kimwipe to remove excess moisture and fingerprints.

Note: If the pan remains in the Speed Vap III after all of the solvent has evaporated, oil and grease residue may be lost.

10.3.5.4 Place the pan in the desiccator for a minimum of 2 hours (preferably overnight).

10.3.6 Residue Determination

10.3.6.1 Inspect the residue in the pan for crystals. Crystal formation is an indication that sodium sulfate may have dissolved and passed into the pan. This may happen if the drying capacity of the sodium sulfate is exceeded or if the sample is not adjusted to a low pH. If crystals are observed, re-dissolve the extract in n-hexane, filter into another tared pan, and repeat the solvent evaporation procedure in Section 10.3.5.

10.3.6.2 Check the calibration of the analytical balance and record the results for the 2mg and 1000mg weights on the spreadsheet. The results of these weights must be within ± 10% at 2 mg and ±0.5% at 1000 mg.

Remove each pan directly from the desiccator and weigh the pan on the analytical balance. Record the weight of the pan and residue in the "HEM Weight 1" cell.

Place the pan back into the desiccator for a minimum of one hour and repeat Section 10.3.6.2 for each pan, recording the weight in the "HEM Weight 2" cell.

If the difference between the Hem Weight 1 and HEM Weight 2 is less than 0.5mg (i.e., 0.00050g) OR the percent difference between the HEM Weight 1 and HEM Weight 2 is less than 4.0%, the pan has achieved constant weight and can be used for calculation of the residue.

If the difference between the HEM Weight 1 and HEM Weight 2 is greater than 0.5mg (i.e., 0.00050g) AND the percent difference is greater than 4.0%, the pan has not achieved constant weight. Place the pan back into the desiccator for one hour. Remove the pan from the desiccator and weigh and record the result in the "HEM Weight 3" field. Repeat until the difference between two consecutive weights is less than 0.5mg OR the percent difference between two consecutive residue weights is less than 4.0%.

The following equations are used to determine difference and percent difference:

$$Difference = |W_2 - W_1| \otimes \frac{1000mg}{g}$$

Where:
W_2 = second weight of the pan + residue (g)

W_1 = initial weight of pan + residue (g)

$$PercentDifference = \left| \frac{W_s - W_i}{W_i} \right| \otimes 100$$

Where:
W_s = second weight of residue (g)
W_i = initial weight of residue (g)
And where:
W_s = (second weight of pan + residue) – (Second weight of pan)
W_i = (initial weight of pan + residue) – (initial weight of pan)

10.3.7 TPH / SGT-HEM Determination

The residue is re-dissolved in hexane and silica gel is added to the sample to bind the polar components of the residue (the fats and animal and vegetable oils). The components of the sample that do not bind with the silica gel are primarily due to components from petroleum. The silica gel-treated (SGT) component of the sample is also called the total petroleum hydrocarbons.

10.3.7.1 Working under a hood, add 10mL of hexane to the pan and swirl to re-dissolve the residue.

10.3.7.2 Transfer the hexane/residue to a labeled, hexane-rinsed weigh pan. Rinse the original pan with three additional 10mL aliquots of hexane and combine all of the rinses in the weigh pan.

10.3.7.3 If the HEM residue is less than or equal to 1000mg, then add 3g of anhydrous silica gel to the pan for each 100mg of residue. For example, if the residue is 750mg, add 24g of silica gel. A maximum of 30g of silica gel can be added to the pan for 1000mg of residue.

Oil and Grease Residue (mg)	Weight of Silica Gel to Add
< 100	3g
100-200	6g
200-300	9g
300-400	12g
400-500	15g
500-600	18g
600-700	21g
700-800	24g
800-900	27g
900-1000	30g
>1000g	Section 10.3.7.4

10.3.7.4 If the HEM residue exceeds 1000mg, transfer the extract to a 100mL volumetric flask. Rinse the extract container with 2-3 small portions of n-hexane and add to the volumetric flask. Dilute to 100mL with n-hexane.

Treat an aliquot of the solution equal to 1000mg with 30g of silica gel. The volume of n-hexane containing the residue to treat with 30g of silica gel is calculated as follows:

$$V = \frac{(1000mg)(100mL)}{W}$$

Where:
V = volume of solution containing residue equal to or less than 1000mg (mL)
W = weight of residue (mg) dissolved in 100mL of n-hexane

This step is referred to as the "extract splitting procedure".

Transfer the aliquot (V) to a clean glass vessel. Dilute to approximately 100mL with n-hexane, and add 30g of silica gel.

10.3.7.5 Add a stirring bar to the flask, and stir the solution on a magnetic stirrer for a minimum of 5 minutes. Alternatively, allow the extract to stand for 30 minutes swirling occasionally.

10.3.7.6 Filter the solution through n-hexane moistened filter paper or glass wool into a pre-dried, tared pan. Rinse the silica gel and filter paper (or glass wool) with several small amounts of n-hexane to complete the transfer.

10.3.7.7 Evaporate the solution (Section 10.3.5) and determine the SGT-HEM (Section 10.3.6).

10.3.7.8 Note: If HEM is a non-detect (ND), the TPH/SGT-HEM determination step is not performed. A value of "HEM ND" is entered as the SGT-HEM initial weight (pan) for the sample, and TALS generates a result of 0 for TPH/SGT-HEM. If all samples requesting TPH/SGT-HEM are non-detects for HEM, this step is omitted on the QC (MB, LCS/LCSD) as well. A theoretical value is given (half of the HEM result) for the QC results to provide associated SGT-HEM data.

11.0 Calculations / Data Reduction

11.1 Data Reduction

Data must be evaluated in accordance with SOP SA-QA-02: *Data Generation and Review.*

11.1.1 MS/MSD Evaluation

If the concentration of a target analyte in the un-spiked (native) sample is more than four times the theoretical concentration of the matrix spike, the recovery is not reported and the data is flagged.

11.1.2 Dilutions

Unless otherwise specified by a client QAPP, results from a single analysis are reported as long as the largest target analyte (when multiple analytes are present) is in the upper half if the calibration range. When reporting results from dilutions, appropriate data flags must be used or qualification in a case narrative provided to the client.

For clients who require we provide lower detection limits, a general guide would be to report the dilution detailed above and one additional run at a dilution factor 1/10 of the dilution with the highest target in the upper half of the calibration curve. For example, if samples analyzed at a 1/50 dilution resulted in a target in the upper half of the calibration curve, the sample would be analyzed at a dilution factor of 1/5 to provide lower reporting limits.

11.1.3 Historical Data

Many of the laboratory's clients submit samples for repeat monitoring purposes. Prior to analysis, verify LIMS Worksheet Notes and/or use the Historical Data tracker feature to determine if historical data is available for review.

11.1.4 Chemical Relationships

When available, the following chemical relationships must be evaluated for each sample. If these relationships are not met the Department Manager must be contacted immediately.

- HEM > SGT-HEM
- Oil & Grease > TPH

11.2 Calculations

11.2.1 The calculations associated with batch QC determinations are given in SOP SA-QA-17. Applicable calculations include accuracy (% recovery) and precision (%RPD).

11.2.2 The calculation to determine final concentration is given below:

11.2.2.1 Oil & Grease (HEM) (Aqueous Samples)

$$HEM(mg/L) = \frac{W_h}{V_s}$$

Where:
W_h = weight of residue (mg) (no silica gel treatment)
V_s = volume of sample extracted (L)

11.2.2.2 Oil & Grease (HEM) (Soil Samples)

$$HEM(mg/kg, dw) = \frac{W_h}{W_x \otimes solids}$$

Where:
W_h = weight of residue (mg) (no silica gel treatment)

W_x = Weight of sample extracted (kg)
Solids = decimal equivalent of percent solids (percent solids/100)

11.2.2.3 TPH (SGT-HEM) (Aqueous Samples)

$$SGT-HEM(mg/L)=\frac{W_h}{V_s}$$

Where:
W_h = weight of residue (mg) (with silica gel treatment)
V_s = sample volume extracted (L)

11.2.2.4 TPH (SGT-HEM) (Soil Samples)

$$SGT-HEM(mg/kg,dw)=\frac{W_h}{W_x \otimes solids}$$

Where:
W_h = weight of residue (mg) (with silica gel treatment)
W_x = Weight of sample extracted (kg)
Solids = decimal equivalent of percent solids (percent solids/100)

11.2.2.5 If the "extract splitting procedure" (Section 10.3.7.4) was performed for SGT-HEM, use the following equation to determine the corrected total weight of SGT-HEM (W_c) in the un-split extract. Use this value for W_h in the equations in Section 11.2.3.3 and 11.2.3.4 above.

$$W_c(mg)=\frac{V_t}{V}\otimes W_d(mg)$$

Where:
W_d = residue in aliquot of extract diluted to 100mL (mg)
V_t = final volume of extract (mL) (usually 100mL)
V = volume of aliquot used to determine residue (mL) (Section 10.7.4)

12.0 Method Performance

12.1 Reporting Limit Verification (RLV)

At a minimum, RLVs must be performed initially upon method set-up in accordance with SOP SA-QA-07: *Determination and Verification of Detection and Reporting Limits.*

For analytes and methods certified by DOD ELAP, RLVs must also be performed quarterly thereafter. For analytes and methods certified by NELAC, RLVs must also be performed annually thereafter. Exceptions may be made for project-specific non-routine analytes.

12.2 Method Detection Limit (MDL) Study

The 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. MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix and may not

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be achievable in all environmental matrices. The current MDLs associated with this procedure are given in the Method Limit Group (MLG) in TALS.

At a minimum, MDL Studies must be performed initially upon method set-up in accordance with SOP SA-QA-07: *Determination and Verification of Detection and Reporting Limits.*

Note: The MDL for water samples must be less than or equal to 1.4mg/L. The recommended spike concentration for the determination of the MDL is 2.5mg/L.

12.3 Method Detection Limit Verification (MDLV)

At a minimum, MDLVs must be performed initially upon method set-up in accordance with SOP SA-QA-07: *Determination and Verification of Detection and Reporting Limits.*

For analytes and methods certified by DOD ELAP, MDLVs must also be performed quarterly thereafter. For analytes and methods certified by NELAC, MDLVs must also be performed annually thereafter.

Note: MDLVs are not required for non-routine analytes provided results are not reported below the RL (i.e., MDL equals RL in TALS).

12.4 QC Limit Generation, Control Charting, and Trend Analysis

The control limits for the batch QC items (LCS and MS/MSD) for this procedure are specified in the reference method and cannot be broadened; therefore, the laboratory defaults to the method-defined limits and does not utilize in-house or laboratory-derived limits for the evaluation of batch QC items.

Although the laboratory must default to the method-defined QC limits, control charting is a useful tool and is performed to assess analyte recoveries over time to evaluate trends. Control charting must be performed periodically (at a minimum annually) in accordance with SOP SA-QA-17: *Evaluation of Batch QC Data.*

12.5 Demonstrations of Capability

Initial and continuing demonstration of capability must be performed in accordance with SOP SA-QA-06*: Training Procedures.*

Prior to performing this procedure unsupervised, each new analyst who performs this analysis must demonstrate proficiency per method/analyte combination by successful completion of an initial demonstration of capability. The IDOC is performed by the analysis of 4 consecutive LCSs that meet the method criteria for accuracy and precision listed below. The LCSs must be from a second source than that used to prepare the calibration standards. The IDOC must be documented on the IDOC Form shown in SOP SA-QA-06 with documentation routed to the QA Department for filing.

Parameter	Initial Precision and Recovery	
	Accuracy (Average Recovery)	Precision (Standard Deviation)

Oil & Grease (HEM)	83-101	10
TPH (SGT-HEM)	83-116	13

Annual continuing demonstrations of capability (CDOCs) are also required per analyst per method/analyte combination. The CDOC requirement may be met by the consecutive analysis of four LCS all in the same batch, by the analysis of four LCS analyzed in four consecutive batches (in different batches on different days), via acceptable results on a PT study, or analysis of client samples with statistically indistinguishable results when compared to another certified analyst. The CDOC must be documented and routed to the QA Department for filing.

12.6 Training Requirements

All training must be performed and documented in accordance with SOP SA-QA-06: *Training Procedures*.

Note: The SOPs listed in the Reference/Cross-Reference Section are applicable to this procedure. All employees performing this procedure must also be trained on these SOPs, and/or have a general understanding of these procedures, as applicable.

13.0 Pollution Control

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (e.g., examining recycling options, ordering chemicals based on quantity needed, preparing reagents based on anticipated usage and reagent stability, etc.). Employees must abide by the policies in Section 13 of the Environmental Health and Safety Manual and the Savannah Addendum to the EHSM.

This procedure has been evaluated for opportunities to minimize the waste generated. Where reasonably feasible, pollution control procedures have been incorporated.

14.0 Waste Management

Waste management practices must be conducted consistent with all applicable federal, state, and local rules and regulations. All waste (i.e., excess reagents, samples, and method process wastes) must be disposed of in accordance with Section 9 of the TestAmerica Savannah Addendum to the EHSM. Waste description rules and land disposal restrictions must be followed.

14.1 Waste Streams Produced by the Method

The following waste streams are produced when this method is carried out:

- Excess acidic aqueous samples – Dispose according to characterization on the sample disposal sheets. Neutralize non-hazardous samples before disposal into drain/sewer. Transfer hazardous samples (identified on disposal sheets) to the waste department for disposal.

- Excess soil and solid samples – Dispose according to characterization on sample

disposal sheets. Transfer non-hazardous samples to TCLP container for characterization in hazardous waste department. Transfer hazardous samples (identified on disposal sheets) to waste department for disposal.

- Flammable waste (acetone, and hexane extracts, rinsings, and standards) – Transfer to a satellite container designated for flammable waste and transfer to waste disposal department when the container is full.

- Methylene chloride rinses from silica cleanup – Transfer to a satellite container designated for chlorinated solvent waste and transfer to the waste disposal department when the container is full.

15.0 References / Cross-References

- SOP SA-AN-41: *Reagent and Standard Materials Procedures*
- SOP SA-AN-100: *Laboratory Support Equipment (Verification and Use)*
- SOP SA-QA-02: *Data Generation and Review*
- SOP SA-QA-05: *Preventive and Corrective Action Procedures*
- SOP SA-QA-06: *Training Procedures*
- SOP SA-QA-07: *Determination and Verification of Detection and Reporting Limits*
- SOP SA-QA-08: *Evaluation of Chromatographic Data*
- SA-QA-10: *Validation of New Analytical Capabilities and Instrumentation*
- SOP SA-QA-15: *Homogenization, Compositing, and Segregation of Samples*
- SOP SA-QA-17: *Evaluation of Batch QC Data*
- TestAmerica Savannah Quality Assurance Manual
- TestAmerica Environmental Health and Safety Manual (CW-E-M-001)
- TestAmerica Savannah Addendum to the Environmental Health and Safety Manual
- Method 1664, Revision A: *n-Hexane Extractable Material (HEM; Oil and Grease) and Silica Gel treated n-Hexane Extractable Material (SGT-HEM; Non-Polar Material) by Extraction and Gravimetry;* Document EPA-821-R-98-002, USEPA Office of Water, Washington, DC 20460, February 1999.
- Analytical Method Guidance for EPA Method 1664A Implementation and Use (40 CFR part 136); Document EPA-821-R-00-003, USEPA Office of Water, Washington, DC 20460, February 2000.
- EPA Memorandum: *Modifications to Method 1664A*; January 16, 2009
- Test Methods for Evaluation Solid Waste, Third Edition; U.S. EPA Office of Solid Waste and Emergency Response: Washington, DC; Updates III and IV
  - EPA Method 9071B: *n-Hexane Extractable Material (HEM) for Sludge, Sediment, and Solid Samples*; Revision 2, April 1998

16.0 Method Modifications and Clarifications

16.1 Incorporation of Other Matrices

16.1.1 EPA Method 1664A has been modified to accommodate soil samples. The adaptation of the procedure for solid matrices is based on Method 1664A with the extraction taken from EPA 3550B and EPA 9071B. The sample volume has been reduced from 1L (in the reference method) to 500mL for routine HEM samples. Sonication is used for the extraction of solid samples.

The volume of spiking solution has been adjusted to keep the concentrations of the LCS and MS at the concentrations specified in EPA 1664A. The spiking solution is prepared with a concentration ten-fold higher than recommended in the reference method (EPA 1664A) while maintaining the same total amount of HEM in the LCS and MS/MSD. The method recommends 10mL of a 4.0mg/mL solution; while, the SOP has 0.5mL of a 40mg/mL solution for water samples and 1.0mL of a 40mg/mL solution for soil samples. The total HEM spike is 20mg for water samples and 40mg for soils samples.

Specific procedures for soil samples are outlined in the body of this SOP.

16.1.2 EPA 1664A has been modified to analyze waste samples based on the needs of the client. Waste matrices are non-routine, and the laboratory is not currently NELAC certified for this matrix. The laboratory uses its routine soil RLs (converted for initial and final volumes, etc.) and default QC limits to evaluate waste samples. Soil DOCs can be used to satisfy analyst demonstrations of capability for this type of non-routine matrix. Ottawa sand is used as the blank matrix for wastes unless specifically requested otherwise by the project.

Waste samples are routinely collected in 125mL amber glass containers with PTFE-lined lids. Waste samples must be iced at the time of collection and maintained at 4°C (less than 6°C but not frozen) until the time of preparation. Waste samples must be prepared within 28 days of collection. Extracts are stored in the desiccator until the time of analysis and analyzed as soon as possible after extraction.

Waste samples are diluted in hexane and filtered through a glass funnel fitted with filter paper and a small amount of sodium sulfate. The extract is collected in a tared pan, the solvent evaporated, and the weight of residue determined.

The required glassware for the extraction includes:

- 1 glass funnel for each sample and QC item
- 1 filter paper, folded into a cone, for each sample and QC item
- 1 40mL vial fitted with a Teflon-lined septum for each sample and QC item

Fold the filter paper twice into a cone shape and place it into a glass funnel. Rinse all glassware thoroughly with n-hexane. Weigh 1g of the homogenized sample into a pre-cleaned, labeled 40mL vial. Add 2.0g of sodium sulfate to each sample. Add 25mL of hexane to each vial. Stir the sample to break up any lumps that may have formed. Pour the extract through the filter/funnel and collect the extract in a tared pan. Evaporate solvent according to the procedures outlined in the body of this SOP.

Waste QC Samples are prepared as follows:

- Method Blank – Weigh 1g of Ottawa Sand into the extraction vessel designated for the method blank.
- Laboratory Control Sample – Weigh 1g of Ottawa Sand into the extraction vessel designated for the LCS. Prior to adding the sodium sulfate to the samples, spike the LCS with 1.0mL of the 40mg/mL spiking solution.
- Matrix Spike(s) – Weigh an additional 1g portion of the sample in the batch selected as the MS into the extraction vessel designated for the MS. Prior to adding the sodium sulfate to the samples, spike the MS with 1.0mL of the

40mg/mL spiking solution.

16.2 Other Considerations

16.2.1 A nitrogen blow-down apparatus is used to evaporate the solvent and concentrate the residue.

16.2.2 Aluminum pans are used to contain the final residue.

16.2.3 EPA Method 1664A contains very specific requirements in regards to method validation (Sections 9.1 and 9.2 of the method). The laboratory performs method validations in accordance with SOP SA-QA-10: *Validation of New Analytical Capabilities and Instrumentation* and Section 12.0 of this SOP.

16.2.4 EPA Method 1664A specifies that stream, river, and open water samples are to be collected in 500mL wide mouth glass jars; while, effluents, monitoring wells, and other samples are collected in narrow mouth glass jars. The laboratory supplies 500mL wide mouth containers, unless specifically requested to do otherwise.

16.2.5 The laboratory has incorporated the minimum batch QC items as outlined in Section 9.1. To satisfy common regulatory and/or client requests for precision data and/or to facilitate scheduling and data evaluation, some QC items are routinely performed that differ from those required in the reference methods (i.e., MS/MSD and or LCS/LCSD). Other clarifications are listed below:

EPA Method 1664A specifies a matrix spike to be performed for 5% of samples from a given sampling site. The laboratory performs matrix spikes per 5% of samples analyzed. The matrix spikes are chosen at random unless client-specific matrix spikes are requested.

EPA Method 1664A states that matrix spike duplicates are recommended but not required. EPA Method 9071B states to perform a matrix duplicate per batch. The laboratory's default batch QC include MS and MSD.

EPA Method 1664A refers to an On-going Precision and Recovery (OPR) sample. This sample is equivalent to the laboratory's Laboratory Control Sample (LCS).

16.2.6 If HEM is a non-detect (ND), the TPH/SGT-HEM determination step is not performed. A value of "HEM ND" is entered as the SGT-HEM initial weight (pan) for the sample, and TALS generates a result of 0 for TPH/SGT-HEM. If all samples requesting TPH/SGT-HEM are non-detects for HEM, this step is omitted on the QC (MB, LCS/LCSD) as well. A theoretical value is given (half of the HEM result) for the QC results to provide associated SGT-HEM data.

17.0 Attachments

The following Tables, Diagrams, and/or Validation Data are included as Attachments:

Attachment 1: SOP Summary
Attachment 2: Sample Collection, Preservation, and Holding Time Table
Attachment 3: QC Summary

Attachment 4: Instrument Maintenance and Troubleshooting

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Attachment 1:
SOP Summary

Aqueous Samples:
The preserved contents of the sampling container (nominally 500mL) are transferred to a separatory funnel. The container is rinsed with n-hexane and added to the separatory funnel. The sample is serially extracted three times with n-hexane, and the extract is dried over sodium sulfate. The solvent is then evaporated from the extract, and the dried residue is weighed to determine the HEM content. If SGT-HEM is to be determined, the residue is re-dissolved in n-hexane and treated with silica gel. The solution is filtered to remove the silica gel, the solvent is evaporated and the SGT-HEM is weighed.

Soil Samples:
A 30g aliquot of homogenized sample is dried with anhydrous sodium sulfate and serially extracted three times with n-hexane using an ultrasonic extractor. The extract is filtered, and the solvent is evaporated. The dried residue is weighed to determine the HEM content. If SGT-HEM is to be determined, the residue is re-dissolved in n-hexane and is treated with silica gel. The solution is filtered to remove the silica gel, the solvent is evaporated, and the SGT-HEM is weighed.

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Attachment 2:
Sample Collection, Preservation, and Holding Time Table

Matrix	Routine Sample Container	Routine Sample Size	Minimum Sample Size	Dechlorination Agent	Chemical Preservation	Thermal Preservation	Holding Time[2]
Water	500mL amber glass with Teflon lined lid	500mL	500mL	Not Applicable	1:1 H_2SO_4 to pH<2	4°C[1]	28 days
Soil	16oz soil jar	30g	30g	Not Applicable	Not Applicable	4°C[1]	28 days

[1]Samples are collected on ice and maintained at <6°C with no frozen samples.
[2]Inclusive of preparation and analysis.

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Attachment 3:
QC Summary

QC Item	Frequency	Criteria	Corrective Action
Analytical Balance Calibration	Daily	In accordance with SOP SA-AN-100	Recalibrate balance
Quality Control Standard (QCS) - Second Source	Monthly	Within LCS limits in the MLG	Recalibrate balance
Continuing Calibration Verification (CCV)	At the beginning and end of the analysis (2mg weight and 1000mg weight)	2mg weight: within +/-0.2mg 1000mg weight: within +/-5mg	Terminate the analysis, fix the problem and reanalyze the previous samples.
Batch Definition	Up to 20 field samples, extracted together within 24-hr period	result <1/2 RL	Evaluate according to SOP SA-QA-17
Method Blank	One per batch	result <1/2 RL	Evaluate according to SOP SA-QA-17
Laboratory Control Sample (LCS)	One per batch	Within limits listed in the MLG	Evaluate according to SOP SA-QA-17
Laboratory Control Sample Duplicate (LCSD)	One per batch, when insufficient sample is available for MS/MSD	Within limits listed in the MLG	Evaluate according to SOP SA-QA-17
Matrix Spike (MS)	One per batch	Within limits listed in the MLG	Evaluate according to SOP SA-QA-17
Matrix Spike Duplicate (MSD)	One per batch	Within limits listed in the MLG	Evaluate according to SOP SA-QA-17
Initial Demonstration of Capability (IDOC)	Initially, per analyst, per analyte/method/matrix combination	Within the method specified DOC accuracy and precision criteria	Refer to SOP SA-QA-06 Note: Unsupervised work must not begin until acceptable IDOC is obtained.

QC Item	Frequency	Criteria	Corrective Action
Continuing Demonstration of Capability (CDOC)	Annually, per analyst, per analyte/method/matrix combination	Within the method specified DOC accuracy and precision criteria	Refer to SOP SA-QA-06
Reporting Limit Verification (RLV)	Upon method/instrument set-up, per analyte/method/matrix combination. Then quarterly thereafter (for DOD ELAP) or annually thereafter (for NELAC)	Refer to SOP SA-QA-07	Refer to SOP SA-QA-07
Method Detection Limit Study (MDL)	Upon method/instrument set-up, per analyte/method/matrix combination	Refer to SOP SA-QA-07 Water MDL must be <1.4mg/L	Refer to SOP SA-QA-07
MDL Verification (MDLV)	Upon method/instrument set-up, per analyte/method/matrix combination. Then quarterly thereafter (for DOD ELAP) or annually thereafter (for NELAC)	Refer to SOP SA-QA-07	Refer to SOP SA-QA-07

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Attachment 4:
Instrument Maintenance and Troubleshooting

Instrument Labeling
Each instrument must be labeled with its name or ID (e.g., MSA, ICP-D, etc.). Additionally, non-operational instruments must be isolated from service or marked as being out of service. Each piece of equipment has an "Operational / Not Operational" sticker that is used for this purpose.

Maintenance Log
A maintenance log must be established for each piece of equipment used in the laboratory. All maintenance that is performed on the instrument must be recorded in the log including:

- analyst or technician performing the maintenance
- date the maintenance was performed
- detailed explanation of the reason for the maintenance
- resolution of the problem and return to control
- all service calls from instrument representatives

Preventive Maintenance
Refer to the instrument manufacturer's guides for trouble-shooting items.

EQUIPMENT ITEM	Service Interval							SERVICE LEVEL
	D	W	M	Q	SA	A	AN	
Zymark Extract Concentrator Service Schedule								
Bath		X						Change water, scrub bath, and dust outside weekly.
Temperature Verification		X						Verify bath temperature and adjust weekly.
Sensor Diagnostic Test		X						Check each position and adjust weekly.

D = daily; W = Weekly; M = monthly; Q = Quarterly; SA = semi-annually; A = annually; AN = as needed

Troubleshooting
Troubleshooting should be documented as outlined above. If possible, troubleshooting is best performed in a step-wise manner to systematically isolate instrument components. Refer to the instrument manufacturer's guides for specific information and strategies. Enlist assistance from technical and/or department management as needed.

Contingency Plan
Maintenance contracts are carried for most instrumentation and close contact is maintained with service personnel to ensure optimal instrument functioning. An extensive spare parts inventory is maintained for routine repairs. Since instrumentation is standardized throughout the laboratory network, spare parts and components can be readily exchanged among the network.

In general, the laboratory has at least one backup unit for each critical unit. In the event of instrument failure, portions of the sample load may be diverted to duplicate instrumentation, the analytical technique switched to an alternate approved technique (such as manual colorimetric determination as opposed to automated colorimetric determination), or samples shipped to another properly certified or approved TestAmerica location.

Sonicator "Tune" Procedures

A tune check must be performed on the sonic dismembrator each day it is used. This tune is to ensure that the sonic dismembrator is operating within the manufacturer's specifications and that the instrument is delivering the minimum wattage output specified by the procedure. Full maintenance is to be performed as needed by a shift supervisor. All checks and adjustments to the sonicator must be documented in the maintenance log.

Unplug the top horn connection on the rear panel of the instrument. Check to ensure that the bottom horn connection is as tight as possible. Turn the pulser switch off. Turn the output to 10. Check to ensure that the horn being tuned is free of obstruction.

Carefully push the tune switch in. At the same time, adjust the tuning knob so that the output meter reads as low as possible. The output meter must read below 20%. If the instrument will not tune to below 20%, a shift supervisor must be informed immediately.

NOTE: The tuning switch must not be held in for longer than 10 seconds. Holding the switch in for more than 10 seconds may cause the horn being tuned to overheat.

Instrument Weekly Maintenance

Turn the instrument off. Disconnect both horns from the back of the instruments. Place both horns on a clean padded surface.

Remove the 1" tip from the horn. Inspect the tip for any pitting and any irregularities. The tip is to be replaced with a new tip if any pitting or irregularities are found. Clean the tip and its threads with a paper towel and methylene chloride.

Check to ensure that the connection with the horn is tight and free of any foreign matter. Carefully reinsert the 1" tip into the horn and tighten the tip. Care must be taken to ensure that the tip is as tight as possible, re-inspect the tip for any irregularities after tightening.

Perform the above procedure on the second horn.

After maintenance is complete, re-connect the bottom horn and re-tune the instrument. If the horn will not re-tune, see the Department Manager. Any horn that cannot be re-tuned will be replaced. If the instrument will not tune, a service technician will be called. The instrument will not be used until all tuning criteria are met.

Desiccator Maintenance

The following checks must be performed daily:

- Desiccant is active
- Hygrometer is in the low humidity zone
- Door is making an air tight seal

The desiccator door must remain closed and seated whenever possible.

When the desiccant turns from blue to light purple, discard desiccant and refill pan with fresh desiccant.

The desiccant must be disposed in accordance with the EHSM.

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18.0 Revision History

Summary of Changes from Previous Revision:

- Removed acetone rinse for glassware. Currently, only a hexane rinse is employed. (2011 Internal Audit Finding) Section 10.3.1.2
- Revised reagent water type from ASTM Type I instead of ASTM Type II. (Note: This is a typographical change and does not reflect a procedural change.) Section 7.2.1.2
- Removed procedure for lot testing silica gel per lot. Method blanks performed with each batch provide evidence of continued acceptability of reagents and materials. (2011 Internal Audit Finding) Section 7.2.1.6.1 and Section 7.2.1.6.2
- Revised sample volume determination procedure to incorporate a gravimetric procedure (via use of the balance) as opposed to volumetric (via marking the meniscus on the side of the bottle). Section 10.3.2.9
- Added section on procedure for TPH/SGT-HEM when HEM is non-detect. Section 10.3.7 and Section 16.2.6

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SOLID / RESIDUE DETERMINATIONS

(Methods: Various)

Approvals (Signature/Date):

Andrea Teal — October 23, 2012
Andrea Teal — Date
Quality Assurance Manager

K. Wayne Robbins — August 15, 2012
Wayne Robbins — Date
Technical Director / EHS Coordinator

Carol M Webb — June 29, 2012
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1.0 Scope and Application

This SOP contains the procedures for the determination of various residue analytes in water, soil, and waste samples.

The following analytes, matrices, methods, and technologies are included in this SOP:

Analyte	Matrix	Reference Method	Technology
Total Dissolved Solids	Water	EPA 160.1 and SM2540C	Gravimetric
Total Suspended Solids	Water	EPA 160.2 and SM2540D	Gravimetric
Total Solids	Water	EPA 160.3 and SM 2540B	Gravimetric
Volatile Dissolved Solids	Water and Waste (oil)	EPA 160.4 and SM2540E	Gravimetric
Volatile Suspended Solids	Water and Waste (oil)	EPA 160.4 and SM2540E	Gravimetric
Volatile Solids	Water and Waste (oil)	EPA 160.4 and SM2540E	Gravimetric
Fixed Solids	Water and Waste (oil)	EPA 160.4	Gravimetric
Ash Content	Water and Waste (oil)	SM 2540E	Gravimetric
Settleable Solids	Water	EPA 160.5 and SM2540F	Volumetric
Percent Moisture Fixed Solids Total Solids Total Volatile Solids Ash Content	Soil and Waste (oil)	SM2540G	Gravimetric
Percent Moisture Percent Solids (Used for Dry Weight Corrections)	Soil	EPA 3500B EPA 3500C EPA 3550B EPA 3550C	Gravimetric

The reporting limits (RL) and accuracy and precision criteria associated with this procedure are provided in the LIMS Method Limit Groups (MLGs).

This SOP was written by and for TestAmerica's Savannah laboratory.

2.0 Summary of Method

2.1 Total Solids (TS) – also called Total Residue

A well-mixed sample is transferred to a tared evaporating dish, and evaporated at 103°C-105°C. The residue that remains in the dish after the liquid has been evaporated is the total residue (or total solids) of the sample. The total solids are reported as milligrams of residue per liter of sample (mg/L).

The total solids is an approximate measure of the sum of the total suspended solids (TSS) and total dissolved solids (TDS). The total residue may not be equal to the sum of the TSS and TDS since the TDS is determined at 180°C and the total residue is determined at 103°C-105°C.

Note: The procedure used by the laboratory for the determination of percent solids/moisture as it pertains to dry weight determination is performed in a similar manner

as the method procedure described above but the method-specified QC steps are not required. Refer to the applicable % Solids/% Moisture Work Instruction (in Attachment 15) for information on this procedure.

2.2 Volatile Solids (VS) and Ash Content/Fixed Solids

After the total solids has been determined, the residue is heated to 550 ± 50°C to determine the volatile solids. The portion of the residue that is lost during the 550°C heating step is defined as the volatile solids.

The residue remaining after heating to 550°C is the ash content or the fixed solids.

2.3 Total Suspended Solids (TSS) – also called Non-filterable Solids/Residue

A well-mixed sample is filtered through a tared glass fiber filter. The residue trapped on the filter pad is dried at 103°C-105°C. The weight of the residue (mg) divided by the volume of the sample filtered (L) is the total suspended solids. The total suspended solids are reported as milligrams of residue per liter of sample (mg/L).

Note: The TSS analysis is also referred to as "non-filterable residue" because the portion of the sample that is trapped on the filter pad is measured.

2.4 Volatile Suspended Solids (VSS)

The volatile suspended solids are determined by placing the filter containing the TSS residue into a muffle furnace at 550 ± 50°C. The portion of the residue that is volatilized at 550°C is the volatile suspended solid.

2.5 Total Dissolved Solids (TDS) – also called Filterable Solids/Residue

A well-mixed sample is filtered through a tared glass fiber filter. The filtrate (the liquid portion passing through the filter) is transferred to a tared evaporating dish and the liquid is evaporated at 103°C-105°C. The oven is then heated to 180°C± 2°C. The residue that remains after the sample has been heated at 180°C is the total dissolved solids portion of the sample. The total dissolved solids are reported as milligrams of residue per liter of sample (mg/L).

Note: The TDS analysis is also referred to as "filterable residue" because the dissolved solids in the portion of the sample that passes through the filter pad is measured.

2.6 Volatile Dissolved Solids (VDS)

The volatile dissolved solids are determined by placing the remaining TDS residue in a muffle furnace at 550°C ± 50°C. The weight that is lost after the sample has been heated at 550°C is the volatile dissolved solids portion of the sample.

2.7 Settleable Solids – also called Settable Matter

A well-mixed sample is transferred to an Imhoff cone and measured volumetrically.

2.8 This SOP is based on the methods listed in Section 1.

3.0 Definitions

Refer to the Glossary Section of the *Quality Assurance Manual* (QAM) for a complete listing of applicable definitions and acronyms.

4.0 Interferences

4.1 Procedural Interferences

4.1.1 Interferences may be caused by contaminants in solvents, reagents, glassware, and other sample processing apparatus and can make quantification of the target analytes difficult.

4.1.2 All sample collection containers are single-use disposable containers which limits the potential for contamination. All non-disposable labware must be scrupulously cleaned in accordance with the posted Labware Cleaning Instructions to ensure it is free from contaminants and does not contribute artifacts.

4.1.3 High purity reagents and solvents are used to help minimize interference problems.

4.1.4 Instrument and/or method blanks are routinely used to demonstrate all reagents and apparatus are free from interferences under the conditions of the analysis.

4.1.5 Since the basis of the gravimetric procedure is the difference in the weight of the crucible containing the residue and the tare weight of the crucible, care must be taken to ensure that the crucible is not treated in such a manner to add or lose weight. Oils from the hand can be added to the dish by touching the dish with "ungloved" hands.

4.2 Matrix Interferences

4.2.1 For some settleable solids samples a separation of settleable and floating materials will occur. In these instances, the volume attributed to the floating materials is not included in the determination of the final result.

4.2.2 Highly mineralized water containing calcium, magnesium, chloride, or sulfate will require prolonged drying. A crust may form in some samples, trapping water underneath.

4.2.3 An excellent discussion on some of the problems that may be encountered when determining solids is found in Section 2540 of the *Standard Methods for the Examination of Water and Wastewater*, 20th Edition.

5.0 Safety

Employees must abide by the policies and procedures in the TestAmerica Environmental Health and Safety Manual (EHSM), the TestAmerica Savannah Addendum to the EHSM, and this document.

This procedure may involve hazardous materials, 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 to follow appropriate safety, waste disposal, and health practices under the assumption that all samples and reagents are potentially hazardous.

The analyst must protect himself/herself from exposure to the sample matrix. Many of the samples that are tested may contain hazardous chemical compounds or biological organisms. The analyst must, at a minimum, wear protective clothing (lab coat), eye protection (safety glasses or face shield), disposable nitrile or latex gloves, and closed-toe, nonabsorbent shoes when handling samples.

5.1 Specific Safety Concerns or Requirements

All samples must be treated as if they are hazardous. The analyst must protect himself/herself from exposure to the sample matrix. Many of the samples that are tested for residue/solids may contain hazardous chemical compounds or biological organisms. The analyst must wear protective clothing (lab coat or apron), eye protection (glasses or face shield), and disposable gloves when handling these samples.

The toxicity or carcinogenicity of chemicals used in this method has not been precisely defined. Each chemical should be treated as a potential health hazard, and exposure to these chemicals should be minimized.

The analyst should handle samples that have been dried at 180°C with caution. This temperature can cause skin burns and damage to unprotected countertops. Gloves and tongs suitable for handling samples that have been placed in a muffle furnace must be used by the analyst to prevent the severe burns that can occur by contact with samples or containers at or near 550°C. The analyst is cautioned to make sure the surface that sample containers are placed on after removal from the oven or muffle furnace is capable of handling the heat without damage.

5.2 Primary Materials Used

The following is a list of the materials used in this procedure, which have a serious or significant hazard rating, and a summary of the primary hazards listed in their MSDS.

Note: This list does not include all materials used in the procedure. A complete list of materials used in this procedure can be found in the Reagents and Standards Section and the Equipment and Supplies Section of this SOP

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 found using the "MSDS" link on the Oasis homepage, on the EH&S webpage on Oasis, and on the QA Navigator.

Material	Hazards	Exposure Limit[1]	Signs and Symptoms of Exposure
Methanol	Flammable Poison Irritant	200ppm 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.			
[2]Always add acid to water to prevent violent reactions.			

6.0 Equipment and Supplies

6.1 Equipment and Instrumentation

Analytical Balance – Verify in accordance with SOP SA-AN-100: *Laboratory Support Equipment (Verification and Use)*

Top-loading Balance – Verify in accordance with SOP SA-AN-100: *Laboratory Support Equipment (Verification and Use)*

Vacuum Pump – with appropriate tubing and water trap

Drying Oven – capable of maintaining a constant temperature of 103-105°C for SM2540 and 180°C ± 2°C

Muffle Furnace – capable of maintaining a constant temperature of 550 +/- 50°C. Note: The temperature correction factors assigned by the external calibration technician during the annual calibration should be used to adjust the muffle furnace thermometer accordingly.

Thermometers – Verify in accordance with SOP SA-AN-100: *Laboratory Support Equipment (Verification and Use)*

6.2 Lab Supplies

Volumetric Containers – various sizes; Class A, where applicable. Verify in accordance with SOP SA-AN-100: *Laboratory Support Equipment (Verification and Use)*

Filtration Apparatus – consisting of a 47mm filter magnetic funnel and a 1000mL filtrate receiving flask with a sidearm. The filter funnel should extend into the receiving flask to ensure that no filtrate is suctioned into the water trap. (This is especially important if TDS is to be determined on the filtrate.) A rubber or Neoprene stopper is used to attach the filter support to the receiving flask.

Gelman Filter – Type A/E glass fiber filter discs; 47-mm

Porcelain evaporating dishes - appropriate volume capacity. The evaporating dishes must be clearly marked so that samples transferred to the dishes can be clearly identified.

Imhoff Cone – verified in accordance with SOP SA-AN-100: *Laboratory Support Equipment (Verification and Use)*

Imhoff Cone Holder

Forceps – to handle the filter if TSS is being determined in conjunction with TDS

Aluminum Pans

Desiccator

Spatula or other utensil for transferring sample

Glass Rods

Nochromix – used to clean evaporating dishes and crucibles

Detergent – Liquinox, used to wash non-disposable labware

6.3 Sample Collection Containers

All sample collection containers are single-use disposable containers which limits the potential for contamination.

The routine samples containers supplied by the laboratory for these tests are listed below:

500mL Plastic containers – used for all aqueous analyses except Settable Residue
1000mL Plastic containers – used for Settable Residue
8oz plastic soil containers – used for SM2540G.

All containers must be purchased with Certificate of Analysis attesting to purity.

7.0 Reagents and Standards

7.1 Expiration Dates

Expiration dates (time from initial use or receipt to final use) for standard and reagent materials must be set according to the guidance in this SOP. Note: These are maximum expiration dates and are not to be considered an absolute guarantee of standard or reagent quality. Sound judgment must be used when deciding whether to use a standard or reagent. If there is doubt about the quality of a standard or reagent material, a new material must be obtained or the standard or reagent material verified. Data quality must not be compromised to extend a standard's life – i.e., when in doubt, throw it out.

The expiration date of any standard or reagent must not exceed the expiration date of the standard or reagent that was used to prepare it; that is, the "children may not outlive the parents".

7.2 Reagents

Reagents must be prepared and documented in accordance with SOP SA-AN-041: *Reagent and Standard Materials Procedures.*

7.2.1 Laboratory Reagent Water – ASTM Type I

7.2.2 Desiccant – Drierite, blue to light purple color change
Storage: Store in a tightly closed container in a cool, dry location. Store away from incompatible materials.
Expiration: Unopened: Manufacturer's expiration date; Opened: 1year from date opened

7.2.3 Blank Matrix – Ottawa sand or equivalent. Used for the preparation of soil QC samples.
Storage: Room temperature
Expiration: Unopened: Manufacturer's expiration date; Opened: 1year from date opened

7.2.4 Methanol – Purge and Trap grade
Storage: Flammable Cabinet
Expiration: Unopened: Manufacturer's expiration date; Opened: 1year from date opened

7.3 Standards

Standards must be prepared and documented in accordance with SOP SA-AN-041: *Reagent and Standard Materials Procedures.* Certificates of analysis or purity must be received with all purchased standards, and scanned and filed in the Data Archival Folder on the G-drive.

7.3.1 TDS/TS Check Standard – used as the LCS; QCI Solids Standard. This standard must be stored in the refrigerator.
Storage: Room Temperature
Expiration: Unopened: Manufacturer's expiration date; Opened: 1year from date opened

A certificate of analysis must be kept on file and the standard tracked according to SOP SA-AN-041.

7.3.2 TSS Microcrystalline cellulose ("Sigmacell") – purchased from Sigma
Storage: Room Temperature
Expiration: Unopened: Manufacturer's expiration date; Opened: 5 years from date opened

7.3.3 TSS Check Standard (100mg/L) – used as the LCS

Weigh 0.1000g of Sigmacell into a clean, dry 1L beaker. Add approximately 150mL of laboratory reagent water to the beaker and stir the water/Sigmacell mixture for one hour at the highest setting obtainable without splashing. Transfer the suspension to a 1L volumetric flask, rinsing the beaker several times with laboratory reagent water, and adding the rinses to the volumetric flask. Make sure that all of the suspension is transferred to the flask. (Do not add the stir bar to the flask). Dilute to the 1L mark with laboratory reagent water and mix the water/Sigmacell suspension well. Transfer the 100mg/L TSS standard to a plastic container large enough to provide some headspace for mixing the suspension prior to taking an aliquot from the container.
Storage: Room Temperature

Expiration: Prepare weekly

8.0 Sample Collection, Preservation, Shipment, and Storage

8.1 Aqueous Samples

Aqueous samples are routinely collected in the following containers:

EPA Method 160.5 – 1L plastic containers

EPA Methods 160.1, 160.2, 160.3, 160.4, Std Methods 2540B, 2540C, 2540D, 2540E, and 2540F – 500mL plastic containers

Samples must be iced at the time of collection and maintained at 4°C (less than 6°C but not frozen) until the time of analysis.

Refer to Attachment 2 for holding time information.

8.2 Soil Samples

Soil samples for SM2540G are routinely collected in 8oz plastic soil containers.

Percent solids samples used for the determination of dry weight are generally subsampled from the Metals or General Department sample portion. If the samples are for volatiles analyses only, then the soil samples are subsampled from the volatiles portion after the Volatiles Department has completed preparation of the sample.

Samples must be iced at the time of collection and maintained at 4°C (less than 6°C but not frozen) until the time of analysis.

Refer to Attachment 2 for holding time information.

8.3 Waste (Oil) Samples

Waste (oil) samples for Standard Methods 2540E and 2540G are routinely collected in 4oz amber glass soil containers.

Samples must be iced at the time of collection and maintained at 4°C (less than 6°C but not frozen) until the time of analysis.

Refer to Attachment 2 for holding time information.

9.0 Quality Control

SOP SA-QA-17: *Evaluation of Batch QC Data* and the SOP Summary in Attachment 3 provide requirements for evaluating QC data.

9.1 Batch QC

An analytical batch consists of up to 20 environment samples and the associated quality control (QC) items.

The default QC items performed for each analytical batch for this procedure are: a method blank and two separate sample duplicates; i.e., a minimum of 1 sample duplicate per 10 samples, equivalent to a 10 % frequency. The following methods also incorporate a laboratory control sample (LCS): EPA 160.1 and SM2540C, EPA 160.2 and SM2540D, and EPA 160.3 and SM2540B, EPA 160.4 and SM2540E.

If there is insufficient sample to perform the sample duplicates, an NCM must be initiated. Note: Do not use field QC samples such as the equipment blank or field blank to perform the sample duplicates.

Batch QC must meet the criteria given in Attachment 3 of this SOP.

9.2 Corrective Action for Out-of-Control Data

When the quality control parameters do not meet the criteria set forth in this SOP, corrective action must be taken in accordance with SOP SA-QA-05: *Preventive and Corrective Action Procedures* and the QC Summary Table in Attachment 3.

SOP SA-QA-05 provides contingencies for out-of-control data and gives guidance for exceptionally permitting departures from approved policies and procedures.

Nonconformance Memos must be initiated to document all instances where QC criteria are not met and all departures from approved policies and procedures.

10.0 Procedure

10.1 Field Sample Preparation

See Attachments 5 – 15 for details on field sample preparation.

10.2 QC Sample Preparation

See Attachments 5 – 15 for details on QC sample preparation.

10.3 Analysis

See Attachments 5 – 15 for details on sample analysis.

11.0 Calculations / Data Reduction

11.1 Data Reduction

Data must be evaluated in accordance with SOP SA-QA-02: *Data Generation and Review.*

11.1.1 These tests are empirical tests; therefore, there are no data reduction steps specific to these procedures.

11.1.2 Historical Data

Many of the laboratory's clients submit samples for repeat monitoring purposes. Prior to analysis, verify LIMS Worksheet Notes and/or use the Historical Data Tracker feature to determine if historical data is available for review.

11.1.3 Chemical Relationships

When available, the following chemical relationships must be evaluated for each sample. If these relationships are not met the Department Manager must be contacted immediately.

- Total Solids (TS) ≥ TDS or TSS
- TDS ≥ individual anions

11.2 Calculations

11.2.1 The calculations associated with batch QC determinations are given in SOP SA-QA-17. Applicable calculations include accuracy (% recovery) and precision (%RPD).

11.2.2 The calculations associated with each analyte/method are contained in Attachments 5 through 15.

12.0 Method Performance

12.1 Reporting Limit Verification (RLV)

At a minimum, RLVs must be performed initially upon method set-up in accordance with SOP SA-QA-07: *Determination and Verification of Detection and Reporting Limits*. The reporting limit is based on the lowest discernable value achievable by the labware used in this procedure.

For analytes and methods certified by DOD ELAP, RLVs must also be performed quarterly thereafter. For all other analytes and methods, RLVs must also be performed annually thereafter. Exceptions may be made for project-specific non-routine analytes.

12.2 Method Detection Limit Study (MDL)

Method detection limits as generated in accordance with 40CFR Part 136 Appendix B are not applicable to this procedure. The reporting limit is based on the lowest discernable value achievable by the labware used in this procedure.

12.3 Method Detection Limit Verification (MDLV)

MDL verifications as outlined in SOP SA-QA-07: *Determination and Verification of Detection and Reporting Limits* are not applicable to this procedure. The reporting limit is based on the lowest discernable value achievable by the labware used in this procedure.

12.4 QC Limit Generation, Control Charting, and Trend Analysis

The control limits for the batch QC items (i.e., LCS/LCSD and SD) for this procedure are not specified by the reference methods; therefore, the laboratory defaults to in-house and/or laboratory-derived limits for the evaluation of batch QC items.

Although the laboratory must default to the method-defined QC limits, control charting is a useful tool and is performed to assess analyte recoveries over time to evaluate trends. Control charting must be performed periodically (at a minimum annually) in accordance with SOP SA-QA-17: *Evaluation of Batch QC Data.*

12.5 Demonstrations of Capability

Initial and continuing demonstration of capability must be performed in accordance with SOP SA-QA-06*: Training Procedures.*

Prior to performing this procedure unsupervised, each new analyst who performs this analysis must demonstrate proficiency per method by successful completion of an initial demonstration of capability.

The IDOC is performed by the analysis of 4 consecutive LCSs that meet the method criteria for accuracy and precision, or, when LCS are not available, by the analysis of client samples with statistically indistinguishable results when compared to another certified analyst. The IDOC must be documented and routed to the QA Department for filing.

Annual continuing demonstrations of capability (CDOCs) are also required per analyst per method/analyte combination. The CDOC requirement may be met by via the consecutive analysis of four LCS (where available) all in the same batch, by the analysis of four LCS analyzed in four consecutive batches (in different batches on different days), acceptable results on a PT study or by the analysis of client samples with statistically indistinguishable results when compared to another certified analyst. The CDOC must be documented and routed to the QA Department for filing.

12.6 Training Requirements

All training must be performed and documented in accordance with SOP SA-QA-06: *Training Procedures.*

Note: The SOPs listed in the Reference/Cross-Reference Section are applicable to this procedure. All employees performing this procedure must also be trained on these SOPs, and/or have a general understanding of these procedures, as applicable.

13.0 Pollution Control

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (e.g., examining recycling options, ordering chemicals based on quantity needed, preparing reagents based on anticipated usage and reagent stability, etc.). Employees must abide by the policies in Section 13 of the Environmental Health and Safety Manual and the Savannah Addendum to the EHSM.

This procedure has been evaluated for opportunities to minimize the waste generated. Where reasonably feasible, pollution control procedures have been incorporated.

14.0 Waste Management

Waste management practices must be conducted consistent with all applicable federal, state, and local rules and regulations. All waste (i.e., excess reagents, samples, and method process wastes) must be disposed of in accordance with Section 9 of the TestAmerica Savannah Addendum to the EHSM. Waste description rules and land disposal restrictions must be followed.

14.1 Waste Streams Produced by the Method

The following waste streams are produced when this method is carried out:

- Non-hazardous acidic and alkaline wastewater and samples – Neutralize before disposal into drain/sewer system
- Excess aqueous samples – Dispose according to characterization on the sample disposal sheets. Neutralize non-hazardous samples before disposal into drain/sewer. Transfer hazardous samples (identified on disposal sheets) to the waste department for disposal.
- Acidic rinsings – Neutralize before disposal into drain/sewer system
- Excess soil and solid samples – Dispose according to characterization on sample disposal sheets. Transfer non-hazardous samples to TCLP container for characterization in hazardous waste department. Transfer hazardous samples (identified on disposal sheets) to the waste department for disposal.

15.0 References / Cross-References

- SOP SA-AN-041: *Reagent and Standard Materials Procedures*
- SOP SA-AN-100: *Laboratory Support Equipment (Verification and Use)*
- SOP SA-BA-080: *Specific Conductance*
- SOP SA-QA-02: *Data Generation and Review*
- SOP SA-QA-05: *Preventive and Corrective Action Procedures*
- SOP SA-QA-06: *Training Procedures*
- SOP SA-QA-15: *Homogenization, Compositing, and Segregation of Samples*
- SOP SA-QA-17: *Evaluation of Batch QC Data*
- TestAmerica Savannah Quality Assurance Manual
- TestAmerica Environmental Health and Safety Manual
- TestAmerica Savannah Addendum to the Environmental Health and Safety Manual
- *Methods for Chemical Analysis of Water and Waste*; U.S. EPA Office of Research and Development: Cincinnati, OH, March, 1983.
- *Standard Methods for the Examination of Water and Wastewater*, Online Edition; American Public Health Association: Washington, D.C.
- *Test Methods for Evaluating Solids Waste, Third Edition (Updates III and IV)*, SW-846; USEPA Office of Solids Waste and Emergency response, Washington, D.C.

16.0 Method Modifications and Clarifications

16.1 EPA 160.5 and SM2540F do not specify batch QC requirements. To comply with NELAC and/or state-specific batch QC requirements, the laboratory performs a method blank and a sample duplicate with each batch.

16.2 SM2450F gives volumetric and gravimetric procedures for the determination of settleable matter. The laboratory only performs the volumetric procedure.

16.3 The gravimetric analytical methods call for drying samples for a minimum of 1 hour. The laboratory's standard procedure is to dry samples for a minimum of 4 hours. The filters are placed back in the oven for an additional hour or overnight to evaluate if a constant weight has been achieved.

16.4 Although samples comprised of a waste matrix are not addressed in the Standards Methods reference methods, the laboratory has adapted these methods to accommodate waste samples.

16.5 With the exception of the Settleable Matter, the EPA reference methods do not specify holding times for these analyses; rather, these reference methods state to perform the test "as soon as possible". The Standard Methods reference methods state to perform the test "as soon as possible" preferably within 24 hours, but no longer than 7 days. As such, the laboratory has adopted a 7-day holding time for all these analyses.

16.6 The laboratory's procedures for Percent Solids/Percent Moisture used to determine dry weight correction are taken from EPA 3500B, EPA 3500C, EPA 3550B, and EPA 3550C. These procedures are performed in a similar manner as the EPA 160.3 and SM2540B and SM2540G method procedures; however, those method-specified QC steps are not required.

17.0 Attachments

The following Tables, Diagrams, and/or Validation Data are included as Attachments:

Attachment 1: SOP Summary
Attachment 2: Sample Collection, Preservation, and Holding Time
Attachment 3: QC Summary
Attachment 4: Table Preventative Maintenance and Troubleshooting
Attachment 5: Total Solids (TS) Procedures – EPA 160.3 and SM2540B
Attachment 6: Volatile Solids (VS), Ash Content, and Fixed Solids Procedures – EPA 160.4 and SM2540E
Attachment 7: Total Suspended Solids (TSS) Procedures – EPA 160.2 and SM2540D
Attachment 8: Volatile Suspended Solids (VSS) Procedures – EPA 160.4 and SM2540E
Attachment 9: Total Dissolved Solids (TDS) Procedures – EPA 160.1 and SM2540C
Attachment 10: Volatile Dissolved Solids (VDS) Procedures – EPA 160.4 and SM2540E
Attachment 11: Settleable Solids Procedures – EPA 160.5 and SM2540F

Attachment 12: Percent Moisture and Total Solids Procedures – SM2540G
Attachment 13: Volatile Solids, Ash Content, and Fixed Solids Procedures – SM2540G
Attachment 14: Constant Weight Determination Procedures
Attachment 15: Percent Moisture and Percent Solids (for Dry Weight Correction)

Attachment 1:
SOP Summary

Analyte	Matrix	Reference Method	Technology
Total Dissolved Solids	Water	EPA 160.1 and SM2540C	Gravimetric
Total Suspended Solids	Water	EPA 160.2 and SM2540D	Gravimetric
Total Solids	Water	EPA 160.3 and SM 2540B	Gravimetric
Volatile Dissolved Solids	Water and Waste (oil)	EPA 160.4 and SM2540E	Gravimetric
Volatile Suspended Solids	Water and Waste (oil)	EPA 160.4 and SM2540E	Gravimetric
Volatile Solids	Water and Waste (oil)	EPA 160.4 and SM2540E	Gravimetric
Fixed Solids	Water and Waste (oil)	EPA 160.4	Gravimetric
Ash Content	Water and Waste (oil)	SM 2540E	Gravimetric
Settleable Solids	Water	EPA 160.5 and SM2540F	Volumetric
Percent Moisture Fixed Solids Total Solids Total Volatile Solids Ash Content	Soil and Waste (oil)	SM2540G	Gravimetric
Percent Moisture Percent Solids (Used for Dry Weight Corrections)	Soil	EPA 3500B EPA 3500C EPA 3550B EPA 3550C	Gravimetric

Sample Preparation and Analysis Summary

Total Solids (TS) – also called Total Residue
A well-mixed sample is transferred to a tared evaporating dish, and evaporated at 103°C-105°C. The residue that remains in the dish after the liquid has been evaporated is the total residue (or total solids) of the sample. The total solids are reported as milligrams of residue per liter of sample (mg/L).

The total solids is an approximate measure of the sum of the total suspended solids (TSS) and total dissolved solids (TDS). The total residue may not be equal to the sum of the TSS and TDS since the TDS is determined at 180°C and the total residue is determined at 103°C-105°C.

Note: The procedure used by the laboratory for the determination of percent solids/moisture as it pertains to dry weight determination is performed in a similar manner as the method procedure described above but the method-specified QC steps are not required.

Volatile Solids (VS) and Ash Content/Fixed Solids
After the total solids has been determined, the residue is heated to 550 ± 50°C to determine the volatile solids. The portion of the residue that is lost during the 550°C heating step is defined as the volatile solids.

The residue remaining after heating to 550°C is the ash content or the fixed solids.

Total Suspended Solids (TSS) – also called Non-filterable Solids/Residue
A well-mixed sample is filtered through a tared glass fiber filter. The residue trapped on the filter pad is dried at 103°C-105°C. The weight of the residue (mg) divided by the volume of the sample filtered (L) is the total suspended solids. The total suspended solids are reported as milligrams of residue per liter of sample (mg/L).

Note: The TSS analysis is also referred to as "non-filterable residue" because the portion of the sample that is trapped on the filter pad is measured.

Volatile Suspended Solids (VSS)
The volatile suspended solids are determined by placing the filter containing the TSS residue into a muffle furnace at 550 ± 50°C. The portion of the residue that is volatilized at 550°C is the volatile suspended solid.

Total Dissolved Solids (TDS) – also called Filterable Solids/Residue
A well-mixed sample is filtered through a tared glass fiber filter. The filtrate (the liquid portion passing through the filter) is transferred to a tared evaporating dish and the liquid is evaporated at 103°C-105°C. The oven is then heated to 180°C± 2°C. The residue that remains after the sample has been heated at 180°C is the total dissolved solids portion of the sample. The total dissolved solids are reported as milligrams of residue per liter of sample (mg/L).

Note: The TDS analysis is also referred to as "filterable residue" because the dissolved solids in the portion of the sample that passes through the filter pad is measured.

Volatile Dissolved Solids (VDS)
The volatile dissolved solids are determined by placing the remaining TDS residue in a muffle furnace at 550°C ± 50°C. The weight that is lost after the sample has been heated at 550°C is the volatile dissolved solids portion of the sample.

Settleable Solids – also called Settable Matter
A well-mixed sample is transferred to an Imhoff cone and measured volumetrically.

Attachment 2:
Sample Collection, Preservation, and Holding Time Table

Matrix	Method	Routine Sample Container	Routine Sample Size	Minimum Sample Size	Chemical Preservation	Dechlorination Agent	Thermal Preservation	Holding Time[1]
Water	EPA 160.1 EPA 160.2 EPA 160.3 EPA 160.4 SM2540B SM2540C SM2540D SM2540E	1L plastic	500mL	500mL	Not Applicable	Not Applicable	4°C[2]	7 days
Water	EPA 160.5 SM2540F	1L plastic	1L	1L	Not Applicable	Not Applicable	4°C[2]	48 hours
Soil	SM2540G	8oz plastic soil jar	10g	10g	Not Applicable	Not Applicable	4°C[2]	7 days
Waste (oil)	SM2540E SM2540G	4oz amber glass soil jar	10g	10g	Not Applicable	Not Applicable	4°C[2]	7 days

[1]From collection to analysis
[2]Less than 6°C, with no frozen samples

Attachment 3:
QC Summary

Method	QC Item	Frequency	Criteria	Corrective Action
EPA 160.1 & SM 2540C (Total Dissolved Solids)	Method Blank (MB)	One per batch	<RL listed in the MLG	Re-analyze batch
	Laboratory Control Sample (LCS)	One per batch	Within %Recovery listed in the MLG	Re-analyze batch
	Laboratory Control Sample Duplicate (LCSD)	One per batch	Within %RPD listed in the MLG	Re-analyze batch
	Sample Duplicate (SD)	One per batch, or one for every 10 samples analyzed, whichever is greater	Within %RPD listed in the MLG	Flag associated data
	Initial Demonstration of Capability (IDOC)	Initially, per analyst per method	Within %RPD listed in the MLG	Re-perform DOC; Unsupervised work cannot proceed until successful IDOC is obtained.
	Continuing Demonstration of Capability (CDOC)	Annually, per analyst per method	Within %RPD listed in the MLG	Re-perform DOC

Method	QC Item	Frequency	Criteria	Corrective Action
EPA 160.2 & SM 2540D (Total Suspended Solids)	Method Blank (MB)	One per batch	<RL listed in the MLG	Re-analyze batch
	Laboratory Control Sample (LCS)	One per batch	Within %Recovery listed in the MLG	Re-analyze batch
	Laboratory Control Sample Duplicate (LCSD)	One per batch	Within %RPD listed in the MLG	Re-analyze batch
	Sample Duplicate (SD)	One per batch, or one for every 10 samples analyzed, whichever is greater	Within %RPD listed in the MLG	Flag associated data
	Initial Demonstration of Capability (IDOC)	Initially, per analyst per method	Within %RPD listed in the MLG	Re-perform DOC; Unsupervised work cannot proceed until successful IDOC is obtained.
	Continuing Demonstration of Capability (CDOC)	Annually, per analyst per method	Within %RPD listed in the MLG	Re-perform DOC

Method	QC Item	Frequency	Criteria	Corrective Action
EPA 160.3 & SM 2540B (Total Solids)	Method Blank (MB)	One per batch	<RL listed in the MLG	Re-analyze batch
	Laboratory Control Sample (LCS)	One per batch	Within %Recovery listed in the MLG	Re-analyze batch
	Laboratory Control Sample Duplicate (LCSD)	One per batch	Within %RPD listed in the MLG	Re-analyze batch
	Sample Duplicate (SD)	One per batch, or one for every 10 samples analyzed, whichever is greater	Within %RPD listed in the MLG	Flag associated data
	Initial Demonstration of Capability (IDOC)	Initially, per analyst per method	Within %RPD listed in the MLG	Re-perform DOC; Unsupervised work cannot proceed until successful IDOC is obtained.
	Continuing Demonstration of Capability (CDOC)	Annually, per analyst per method	Within %RPD listed in the MLG	Re-perform DOC

Method	QC Item	Frequency	Criteria	Corrective Action
EPA 160.4 & SM 2540E (Volatile Dissolved Solids, Volatile Suspended Solids, Volatile Solids, Fixed Solids, and Ash Content)	Method Blank (MB)	One per batch	<RL listed in the MLG	Re-analyze batch
	Laboratory Control Sample (LCS)	One per batch	Within %Recovery listed in the MLG	Re-analyze batch
	Laboratory Control Sample Duplicate (LCSD)	One per batch, when insufficient sample for SD	Within %RPD listed in the MLG	Re-analyze batch
	Sample Duplicate (SD)	One per batch, or one for every 10 samples analyzed, whichever is greater	Within %RPD listed in the MLG	Flag associated data
	Initial Demonstration of Capability (IDOC)	Initially, per analyst per method	Within %RPD listed in the MLG	Re-perform DOC; Unsupervised work cannot proceed until successful IDOC is obtained.
	Continuing Demonstration of Capability (CDOC)	Annually, per analyst per method	Within %RPD listed in the MLG	Re-perform DOC

Method	QC Item	Frequency	Criteria	Corrective Action
EPA 160.5 & SM 2540F (Settleable Solids)	Method Blank (MB)	One per batch	<RL listed in the MLG	Re-analyze batch
	Sample Duplicate (SD)	One per batch, or one for every 10 samples analyzed, whichever is greater	Within %RPD listed in the MLG	Flag associated data
	Initial Demonstration of Capability (IDOC)	Initially, per analyst per method	Within %RPD listed in the MLG	Re-perform DOC; Unsupervised work cannot proceed until successful IDOC is obtained.
	Continuing Demonstration of Capability (CDOC)	Annually, per analyst per method	Within %RPD listed in the MLG	Re-perform DOC

Method	QC Item	Frequency	Criteria	Corrective Action
SM 2540G (Percent Moisture, Fixed Solids, Total Solids, Total Volatile Solids, Ash Content)	Method Blank* (MB)	One per batch	<RL listed in the MLG	Re-analyze batch
	Sample Duplicate (SD)	One per batch, or one for every 10 samples analyzed, whichever is greater	Within %RPD listed in the MLG	Flag associated data
	Initial Demonstration of Capability (IDOC)	Initially, per analyst per method	Within %RPD listed in the MLG	Re-perform DOC; Unsupervised work cannot proceed until successful IDOC is obtained.
	Continuing Demonstration of Capability (CDOC)	Annually, per analyst per method	Within %RPD listed in the MLG	Re-perform DOC

***Note: Method Blank not performed for Total Solids, Fixed Solids, or % Moisture.**

Attachment 4:
Preventative Maintenance and Troubleshooting

Instrument Labeling

Each instrument must be labeled with its name or ID (e.g., MSA, ICP-D, etc.). Additionally, non-operational instruments must be isolated from service or marked as being out of service. Each piece of equipment has an "Operational / Not Operational" sticker that is used for this purpose.

Maintenance Log

A maintenance log must be established for each piece of equipment used in the laboratory. All maintenance that is performed on the instrument must be recorded in the log including:

- analyst or technician performing the maintenance
- date the maintenance was performed
- detailed explanation of the reason for the maintenance
- resolution of the problem and return to control
- all service calls from instrument representatives

Desiccator Checks, Maintenance, and Use

The following checks must be performed daily:

- Desiccant is active.
- Hygrometer is in the low humidity zone.
- Door is making an air-tight seal.

The desiccator door must remain closed and seated whenever possible. When the desiccant turns from blue to light purple, discard desiccant and refill pan with fresh desiccant. The desiccant must be disposed in accordance with the Waste Disposal Section of the TestAmerica Savannah Addendum to the Environmental Health and Safety Manual.

Attachment 5:

Total Solids (TS) Procedures
EPA 160.3 and SM2540B

Preparation of the Evaporating Dishes

Thoroughly clean the 100mL and 200mL evaporating dishes that will be required for the determination of TS. It may be necessary to place Nochromix in the dish for a short time to remove dissolved solids from a previous analysis.

Rinse the evaporating dishes thoroughly with reagent water and allow them to dry. The evaporating dishes should be placed in the oven at 180°C to facilitate the drying.

After the evaporating dish has remained in the oven at 180°C for a minimum of two hours, remove the dishes from the oven and place in the desiccator. Do not close the door of the desiccator completely until the dishes have cooled. After the dishes have cooled, close the door of the desiccator. The evaporating dishes may be stored in the desiccator until ready for use.

Note: If the evaporating dishes are placed into the desiccator while hot and the door is sealed, a vacuum may form as the dishes cool. If this occurs, the door of the desiccator may be difficult to open.

Determination of Total Solids

Verify that the analytical balance is working within the proper parameters in accordance with SOP SA-AN-100: *Laboratory Support Equipment (Verification and Use).*

Remove the evaporating dish from the desiccator and weigh it on the analytical balance. Record the evaporating dish ID and the weight of the dish to the nearest 0.0001g in the LIMS AD batch.

Note: Remember never to touch dishes with bare hands as this can cause oils to deposit and affect the weight of the dish.

Mix the sample well before taking an aliquot for analysis. Transfer the aliquot of the well-mixed sample to a graduated cylinder. Record the volume of sample in the LIMS AD batch.

Note: The sample aliquot to be used in the determination of total residue may be estimated from the specific conductance as obtained using the procedures outlined in SOP SA-GE-193: *Measurement of Analytes using the PC Titrate Auto-Analyzer.* If the conductance is greater than 200umho/cm refer to the chart in Attachment 9 for appropriate sample volume. If the conductance is less than 200umho/cm, use 100mL of sample. If the concentration of the suspended particulates of the sample is significant, a smaller aliquot should be used.

Carefully add the sample to the tared evaporating dish (up to 100mL). A few milliliters of reagent water may be used to help transfer any particulates adhering to the side of the graduated cylinder to the evaporating dish. Place the dish containing the sample into the

drying oven maintained at 103°C-105°C to evaporate the liquid portion of the sample. This will generally require overnight in the oven.

After the liquid has evaporated, remove the evaporation dishes from the oven, allow them to cool slightly, and place them in the desiccator. Remember not to place the evaporating dishes on a surface that can cause dirt or other foreign objects to adhere to the dishes. After the dishes are cool, place them in the desiccator.

Allow the evaporating dishes to remain in the desiccator until completely cool.

Weigh the evaporating dish on the analytical balance and record the weight of the dish and the residue.

The total solids is calculated using the following equation:

$$TS, mg/L = \frac{(A - B)}{V} \otimes 1000 mg/g$$

Where
A = weight of sample residue and evaporating dish (g)
B = weight of evaporating dish (g)
V = volume of sample used to determine the TS (L)

QC Sample Preparation and Analysis

For each batch of 20 or fewer samples prepare a minimum of one method blank, one laboratory control sample, and two separate sample duplicates.

For the method blank, follow the procedures outlined above and use 100mL of reagent water to serve as the blank matrix.

For the laboratory control sample, follow the procedures outlined above and use 100mL of the TDS/TS Check Standard.

For the sample duplicate sample, follow the procedures outlined above and prepare and analyze one sample in duplicate per batch or 1 sample in duplicate for every 10 samples analyzed, whichever is greater.

Attachment 6:

Volatile Solids (VS), Ash Content, and Fixed Solids Procedures EPA 160.4 and SM2540E

Preparation of the Evaporating Dishes for Volatile Solids

Thoroughly clean the 100mL and 200mL evaporating dishes that will be required for the determination of TS. It may be necessary to place Nochromix in the dish for a short time to remove dissolved solids from a previous analysis.

Rinse the evaporating dishes thoroughly with reagent water and allow them to dry. The evaporating dishes should be placed in the oven at 180°C to facilitate the drying.

After the evaporating dish has remained in the oven at 180°C for a minimum of two hours, remove the dishes from the oven and place them into the muffle furnace. Turn on the muffle furnace, and elevate the temperature 100°C per hour until it reaches 550°C ± 50°C. After one hour at 550°C, remove the dishes from the muffle furnace, allow them to cool for a few minutes, and place them in the desiccator. Do not close the door of the desiccator completely until the dishes have cooled. After the dishes have cooled, close the door of the desiccator. The evaporating dishes may be stored in the desiccator until ready for use.

Note: If the evaporating dishes are placed into the desiccator while hot and the door is sealed, a vacuum may form as the dishes cool. If this occurs, the door of the desiccator may be difficult to open.

Determination of Volatile Solids, Ash Content, and Fixed Solids

Once the Total Solids has been determined, place the evaporating dish containing the sample residue into the muffle furnace. Turn on the muffle furnace, and elevate the temperature 100°C per hour until it reaches 550°C ± 50°C.

After one hour at 550°C, remove the evaporation dishes from the muffle furnace, allow them to cool for a few minutes, and place them in the desiccator. Remember not to place the evaporating dishes on a surface that can cause dirt, or other foreign objects, to adhere to the dishes or on a surface that will melt when hot dishes are placed on it.

Allow the evaporating dishes to remain in the desiccator until completely cool.

Note: Remember never to touch dishes with bare hands as this can cause oils to deposit and affect the weight of the dish.

Verify that the analytical balance is working within the proper parameters in accordance with SOP SA-AN-100: *Laboratory Support Equipment (Verification and Use)*.

Weigh the evaporating dish on the analytical balance and record the weight of the dish and the residue to the nearest 0.0001g on the bench sheet.

The Volatile Solids is calculated:

$$VS, mg/L = \frac{(R_{105} - R_{550})}{V} \otimes 1000 mg/g$$

where
V = volume of sample analyzed
R_{550} = (weight of residue and dish at 550°C)g - (weight of dish)g
R_{105} = (weight of residue and dish at 105°C)g - (weight of dish)g

The Fixed Solids is calculated:

$$mg_fixed_solids/L = \frac{(R_{550})}{V} \otimes 1000 mg/g$$

where
V = volume of sample analyzed
R_{550} = (weight of residue and dish at 550°C)g - (weight of dish)g

The ash content is calculated:

$$Ash_Content, \% = \frac{R_{550}}{R_{105}} \otimes 100$$

where
R_{550} = (weight of residue and dish at 550°C)g - (weight of dish)g
R_{105} = (weight of residue and dish at 105°C)g - (weight of dish)g

QC Sample Preparation and Analysis

For each batch of 20 or fewer samples prepare a minimum of one method blank, one LCS, and two separate sample duplicates.

For the method blank, follow the procedures outlined above and use 100mL of reagent water to serve as the blank matrix.

For the sample duplicate sample, follow the procedures outlined above and prepare and analyze one sample in duplicate per batch or 1 sample in duplicate for every 10 samples analyzed, whichever is greater.

Attachment 7:

Total Suspended Solids (TSS) Procedures
EPA 160.2 and SM2540D

Preparation of the Filters

Filters may be purchased from an approved vendor that are pre-cleaned and pre-weighed for the intended use. If pre-weighed filters are not used, the following procedures must be followed prior to use of the filters for TSS:

Assemble the filtration apparatus consisting of a 47mm filter magnetic funnel and a 1000mL filtrate receiving flask with a sidearm. The filter funnel should extend into the receiving flask to ensure that no filtrate is suctioned into the water trap. (This is especially important if TDS is to be determined on the filtrate.) A rubber or Neoprene stopper is used to attach the filter support to the receiving flask.

Remove the filter from the box with forceps and place it on the filter support. The rough side of the filter must face up. Attach the reservoir funnel and clamp the filter in place.

Add about 25mL of reagent water to the reservoir funnel. Turn on the vacuum pump and pull the water through the funnel. Add two additional 25mL aliquots of reagent water, allowing each to pass through the filter. After the third aliquot, leave the pump on for about 1 minute after the water has gone through the filter to allow any water trapped in the filter to move through.

Turn off the pump and release the vacuum. Carefully remove the filter disc from the filter support and place the filter in an aluminum pan. The filter should be placed on the edge of the aluminum pan so that the filter has minimum contact with the pan. After ten minutes or so in the oven, the filter may be placed in the bottom of the pan. (If a wet filter is placed in contact with the bottom of the pan, the filter may stick to the pan.)

Dry the filter at 103°C-105°C for a minimum of one hour.

Remove the filter from the oven and place in a petri dish or other appropriate container. Several filters may be placed on top of each other. Store the filters in the desiccator until ready for use.

Determination of Total Suspended Solids

Calibrate and verify that the analytical balance is working within the proper parameters in accordance with SOP SA-AN-100: *Laboratory Support Equipment (Verification and Use).*

Place a numbered aluminum pan on the analytical balance. Tare the aluminum pan.

Using the forceps, remove a filter from the desiccator and place the filter in the tared aluminum pan on the balance. Record the weight of the filter in the run log. If the filters are preweighed, check several filters per lot to verify accuracy of weight. Otherwise, record the weight from the filter's pan.

The filter may be stored in the aluminum pan at ambient conditions for a short time. If the time between the determination of the tare weight of the filter and the use of the filter is

extended, the filter should be stored in the desiccator or other covered container to prevent dust and other materials from adhering to the filter.

Assemble the filtration apparatus consisting of a 47mm filter magnetic funnel and a 1000mL filtrate receiving flask with a sidearm. The filter funnel should extend into the receiving flask to ensure that no filtrate is suctioned into the water trap. (This is especially important if TDS is to be determined on the filtrate.) A rubber or Neoprene stopper is used to attach the filter support to the receiving flask.

Using forceps, transfer a filter from the numbered aluminum pan to the filter support with the rough side up. Clamp the filter into place and add a small aliquot (~5mL) of reagent water to the filtering apparatus to seat the filter.

Mix the sample well before taking an aliquot for analysis. The sample size can range from 1mL to 200mL, depending on how much suspended material is in the sample. Transfer an aliquot of the well-mixed sample to a graduated cylinder. Record the sample identification, pan number, and volume of sample in the LIMS AD batch.

Note: Wide bore pipettes can also be used to transfer the aliquot from the sample container to the filtration device. Narrow bore pipettes must not be used.

Note: The volume of sample analyzed should produce a residue of 100mg with a target range between 1.0mg and 200mg. If the mass of residue falls below this range, use more sample.

Turn on the pump and carefully pour the entire sample aliquot into the filter funnel. Use a small aliquot of reagent water to rinse the entire sample out of the graduated cylinder and onto the filter. Use three 10mL portions of reagent water to rinse down the sides of the filter funnel to transfer all of the particulates on to the filter.

Allow the pump to remain on for about a minute after all of the particulates have been transferred to the filter to remove the water trapped on the filter.

Turn off the pump and release the vacuum. Carefully remove the filter disc from the filter support with the forceps and place the filter in an aluminum pan. The filter should be placed on the edge of the aluminum pan so that the filter has minimum contact with the pan. After ten minutes or so in the oven, the filter may be placed in the bottom of the pan (if a wet filter is placed in contact with the bottom of the pan, the filter may stick to the pan).

Dry the filter at 103°C-105°C for at least one hour, preferably overnight. Record the temperature of the oven and the time that the samples are placed into the oven in the LIMS AD batch.

Remove the filters and pans from the oven and place them into the desiccator.

Place an aluminum pan onto the analytical balance and tare. Transfer the filter to the tared aluminum pan. Record the weight of the filter and residue in the LIMS AD batch.

The total suspended solids is calculated using the following equation:

$$TSS, mg/L = \frac{(A - B)}{V} \otimes 1000 mg/g$$

where
A = weight of pan, sample residue, and filter (g)
B = weight of pan and filter (g)
V = volume of sample used to determine the TSS (L)

QC Sample Preparation and Analysis

For each batch of 20 or fewer samples prepare a minimum of one method blank, one laboratory control sample, and two separate sample duplicates.

For the method blank, follow the procedures outlined above and use 200mL of reagent water to serve as the blank matrix.

For the laboratory control sample, follow the procedures outlined above and use 200mL of the TSS Check Standard.

For the sample duplicate sample, follow the procedures outlined above and prepare and analyze one sample in duplicate per batch or 1 sample in duplicate for every 10 samples analyzed, whichever is greater.

Attachment 8:

Volatile Suspended Solids (VSS) Procedures
EPA 160.4 and SM2540E

Preparation of the Filters

Filters may be purchased from an approved vendor that are pre-cleaned and pre-weighed for the intended use. If pre-weighed filters are not used, the following procedures must be followed prior to use of the filters for VSS:

Note that these filters will be used to determine the Total Suspended Solids and the Volatile Suspended Solids.

Assemble the filtration apparatus consisting of a 47mm filter magnetic funnel and a 1000mL filtrate receiving flask with a sidearm. The filter funnel should extend into the receiving flask to ensure that no filtrate is suctioned into the water trap. (This is especially important if TDS is to be determined on the filtrate.) A rubber or Neoprene stopper is used to attach the filter support to the receiving flask.

Remove the filter from the box with forceps and place it on the filter support. The rough side of the filter must face up. Attach the reservoir funnel and clamp the filter in place.

Add about 30mL of reagent water to the reservoir funnel. Turn on the vacuum pump and pull the water through the funnel. Leave the pump on for a few moments after the water has gone through the filter to allow any water trapped in the filter to move through.

Turn off the pump and release the vacuum. Carefully remove the filter disc from the filter support and place the filter in an aluminum pan. The filter should be placed on the edge of the aluminum pan so that the filter has minimum contact with the pan. After ten minutes or so in the oven, the filter may be placed in the bottom of the pan. (If a wet filter is placed in contact with the bottom of the pan, the filter may stick to the pan.)

Dry the filter at 103°C-105°C for a minimum of one hour.

Remove the pans containing the filters from the oven and place them into the muffle furnace. Turn on the muffle furnace, and elevate the temperature 100°C per hour until it reaches 550°C ± 50°C.

After one hour at 550°C ± 50°C, remove the aluminum pans containing the filters from the muffle furnace oven and place the filters in a petri dish or other appropriate container. Several filters may be placed on top of each other. Store the filters in the desiccator until ready for use.

Determination of Volatile Suspended Solids

Volatile suspended solids are the difference in the residue at 105°C (TSS) and the residue that remains after the sample is volatilized at 550°C. The filter used for VSS must be prepared as described above, unless pre-weighed filters are purchased. The associated TSS method blank must be taken through the ignition procedure described in this section. If the sample was analyzed in duplicate for TSS, both replicates must be taken though the ignition procedure.

After the TSS has been determined, carefully transfer the numbered aluminum pan, filter, and residue into the muffle furnace. If there is quite a bit of organic material in the TSS residue, potentially harmful gases may evolve as the sample ignites. The muffle furnace should be maintained under a hood or in a well ventilated area.

Note: Long handled tongs and proper gloves must be used when working with the muffle furnace.

Turn on the muffle furnace, and elevate the temperature 100°C per hour until it reaches 550°C ± 50°C. Allow the sample to remain in the muffle furnace for at least one hour at 550 ± 50°C.

After one hour, remove the pans and filters from the furnace and place them on a non-reactive surface to cool slightly. Transfer the pans and filters to the desiccator and allow them to cool to ambient temperature.

Note: Be careful when placing the pans on any surface when they are taken out of the muffle furnace. Even though the heat will quickly dissipate, the pans/filters will be very hot and should not be placed on a surface that will melt or ignite.

Place the numbered aluminum pan on the analytical balance and tare. Place the filter containing the residue into the tared aluminum pan. Record the weight of the filter and residue on the run log. Calculate the volatile suspended solids.

Note: Caution must be taken when handling a filter that contains residue. The analyst must be careful not to lose any of the residue. If an obvious loss occurs, contact the department manager and repeat the analysis.

The volatile suspended solids is calculated as follows:

$$VSS(mg/L) = = \frac{(R_{105} - R_{550})}{V} \otimes 1000mg/g$$

Where
R_{105} = residue at 105°C (g)
R_{550} = residue at 550°C (g)

QC Sample Preparation and Analysis

For each batch of 20 or fewer samples prepare a minimum of one method blank, one LCS, and two separate sample duplicates.

For the method blank, follow the procedures outlined above and use 200mL of reagent water to serve as the blank matrix.

For the sample duplicate sample, follow the procedures outlined above and prepare and analyze one sample in duplicate per batch or 1 sample in duplicate for every 10 samples analyzed, whichever is greater.

Attachment 9:

Total Dissolved Solids (TDS) Procedures
EPA 160.1 and SM2540C

Preparation of the Evaporating Dishes

Thoroughly clean the 100mL and 200mL evaporating dishes that will be required for the determination of TDS. It may be necessary to place Nochromix in the dish for a short time to remove dissolved solids from a previous analysis.

Rinse the evaporating dishes thoroughly with reagent water and allow them to dry. The evaporating dishes should be placed in the oven at 180°C to facilitate the drying.

After the evaporating dish has remained in the oven at 180°C for a minimum of two hours, remove the dishes from the oven and place in the desiccator. Do not close the door of the desiccator completely until the dishes have cooled. After the dishes have cooled, close the door of the desiccator. The evaporating dishes may be stored in the desiccator until ready for use.

NOTE: If the evaporating dishes are placed into the desiccator while hot and the door is sealed, a vacuum may form as the dishes cool. If this occurs, the door of the desiccator may be difficult to open.

Determination of Total Dissolved Solids

Calibrate and verify that the analytical balance is working within the proper parameters in accordance with SOP SA-AN-100: *Laboratory Support Equipment (Verification and Use)*.

Note: If TSS is to be determined along with the TDS, the filter used in the filtration step should be prepared in accordance with Attachment 7.

Measure the weight of the pre-labeled, pre-cleaned porcelain dishes to the nearest 0.1mg on an analytical balance.

Measure the conductivity of the unfiltered samples in accordance with SOP SA-BA-80: *Specific Conductance*. If the specific conductance of the sample is less than 200umho/cm, filter 100mL of the sample. If the specific conductance is greater than 200umho/cm, filter and analyze an aliquot of sample that will give a TDS residue of less than or equal to 200mg. Suggested volumes are listed below. Record the specific conductivity in the LIMS AD batch.

Conductivity (umho/cm)	Sample Volume (mL)
0-200	100
201-2000	50
2001-4000	25
4001-6000	10
>6000	5

Assemble the filtration apparatus.

Place a filter pad in the filter support with the rough side up. If determining TSS on the same sample, transfer a tared filter from the numbered aluminum pan to the filter support with the rough side up. Clamp the filter into place.

Mix the sample well before taking an aliquot for analysis. Transfer the aliquot, determined by specific conductance above, of the well mixed sample to a graduated cylinder. Do not use a narrow bore pipette to measure the aliquot if TSS is also being determined because a pipette may strain some of the particulates out of the sample aliquot. Record the sample identification, dish number, and volume of sample in the LIMS AD batch.

Note: The volume needed for the TSS and the volume of sample needed for the TDS may be different. A sample with a low conductivity but containing a high concentration of suspended solids may require several filters to obtain the needed volume of sample for TDS. In this case, the TSS and TDS should not be analyzed from the same sample aliquot. The sample aliquot must be selected to give a TDS residue of less than or equal to 200mg.

Turn on the pump and carefully pour the entire sample aliquot into the filter funnel. Use a small aliquot of reagent water to rinse the entire sample out of the graduated cylinder and on to the filter. Use another small portion of reagent water to rinse down the sides of the filter funnel to transfer all of the particulates onto the filter.

Allow the pump to remain on for a few moments after all of the particulates have been transferred to the filter to remove the water trapped on the filter.

Turn off the pump and release the vacuum. If TSS is being determined concurrently, carefully remove the filter disc from the filter support with the forceps and place the filter in an aluminum pan. Transfer the filtrate from the receiving flask to a properly labeled container.

Note: The entire filtrate must be used for the determination of TDS; that is, the sample filtrate and the reagent water used to rinse the graduated cylinder and the receiving flask are included in the aliquot for TDS. Since the reagent water contains very low concentrations of dissolved solids, the addition of small quantities of reagent water will have no net effect on the concentration of TDS in the sample.

Remove the evaporating dish from the desiccator and weigh it on the analytical balance. Record the evaporating dish ID and the weight of the dish to the nearest 0.0001g in the LIMS AD batch.

Carefully add the filtered sample to the appropriate tared evaporating dish and place it in the drying oven maintained at 103°C-105°C to evaporate the liquid portion of the sample. This will generally require overnight in the oven. After the sample has evaporated, increase the temperature to 180°C ± 2°C and heat for a minimum of 2 hours.

Remove the evaporation dishes from the oven, allow them to cool slightly, and place them in the desiccator. Remember not to place the evaporating dishes on a surface that can cause dirt or other foreign objects to adhere to the dishes.

Allow the evaporating dishes to remain in the desiccator until completely cool.

Weigh the evaporating dish on the analytical balance and record the weight of the dish and the residue.

The total dissolved solids is calculated using the following equation:

$$TDS, mg/L = \frac{A - B}{V} \otimes 1000 mg/g$$

where
A = weight of sample residue and evaporating dish (g)
B = weight of evaporating dish (g)
V = volume of sample analyzed (L)

QC Sample Preparation and Analysis

For each batch of 20 or fewer samples prepare a minimum of one method blank, one laboratory control sample, and two separate sample duplicates.

For the method blank, follow the procedures outlined above and use 100mL of reagent water to serve as the blank matrix.

For the laboratory control sample, follow the procedures outlined above and use 100mL of the TS/TDS Check Standard.

For the sample duplicate sample, follow the procedures outlined above and prepare and analyze one sample in duplicate per batch or 1 sample in duplicate for every 10 samples analyzed, whichever is greater.

Attachment 10:

Volatile Dissolved Solids (VDS) Procedures
EPA 160.4 and SM2540E

Preparation of Evaporating Dishes

Note that the same evaporating dishes will be used to determine the TDS and the VDS.

Thoroughly clean the 100mL and 200mL evaporating dishes that will be required for the determination of VDS. It may be necessary to place Nochromix in the dish for a short time to remove dissolved solids from a previous analysis.

Rinse the evaporating dishes thoroughly with reagent water and allow them to dry. The dishes should be placed in the oven at 180°C to facilitate the drying.

Remove the evaporating dishes from the oven and place them in the muffle furnace. Turn on the muffle furnace, and elevate the temperature 100°C per hour until it reaches 550°C ± 50°C.

After the evaporating dishes have remained in the muffle furnace at 550°C ± 50°C for a minimum of one hour, remove the dishes and place them in the desiccator. Do not seal the door of the desiccator completely until the dishes have cooled. After the evaporating dishes have cooled, close the door of the desiccator. The evaporating dishes may be stored in the desiccator until ready for use.

Note: If the evaporating dishes are placed into the desiccator while hot and the top or door is sealed, a vacuum may form as the dishes cool. The top or door may be difficult to open if this occurs. This can occur very readily after the evaporating dishes have been removed from the muffle furnace.

Determination of Volatile Dissolved Solids

Determine the Total Dissolved Solids as described in Attachment 9. Use evaporating dishes that have been prepared as above.

Place the evaporating dishes in the muffle furnace. Turn on the muffle furnace, and elevate the temperature 100°C per hour until it reaches 550°C ± 50°C.

After the evaporating dishes have remained in the muffle furnace at 550°C ± 50°C for a minimum of one hour, remove the evaporating dishes from the muffle furnace and place them on a non-reactive surface to cool. Caution: the dishes will be extremely hot when removed. Do not place them on a surface that will melt or ignite

Note: One option is to turn the muffle furnace off and let then dishes cool in the furnace.

After the dishes have cooled, place them in desiccator for at least one hour or overnight.

Remove the dishes from the desiccator and weigh the dishes to constant weight using the procedure outlined in Attachment 14.

The volatile dissolved solids is calculated:

$$Volatile\ dissolved\ solids,\ mg/L = \frac{R_{180} - R_{550}}{V} \otimes 1000 mg/g$$

Where:
R_{180} = (weight of sample residue + evaporating dish @ 180°C)g - (evaporating dish weight)g = residue at 180°C (g)
R_{550} = (weight of residue + evaporating dish @ 550°C)g - (weight of evaporating dish)g = residue at 550°C (g)
V = volume of sample analyzed

QC Sample Preparation and Analysis

For each batch of 20 or fewer samples prepare a minimum of one method blank and two separate sample duplicates.

For the method blank, follow the procedures outlined above and use 100mL of reagent water to serve as the blank matrix.

For the sample duplicate sample, follow the procedures outlined above and prepare and analyze one sample in duplicate per batch or one sample in duplicate for every 10 samples analyzed, whichever is greater.

Attachment 11:

Settleable Solids Procedures
EPA 160.5 and SM2540F

Sample Preparation and Analysis

Remove the samples from the refrigerator and allow them to come to room temperature.

Rinse the Imhoff cone well with reagent water. Wash with detergent, if needed.

Place the Imhoff cone in the holder and pour 1L of sample into the cone.

Allow the sample to settle for 45 minutes. Stir the sample gently with a glass rod to free matter stuck to the sides of the Imhoff cone.

Allow the sample to settle an additional 15 minutes. Read the volume of settleable matter and report the results in mL/L. If less than 0.1mL of material has settled in the bottom, report the result as <0.1mL/L.

For some samples a separation of settleable and floating materials will occur. In these instances, the volume attributed to the floating materials is not included in the determination of the final result.

QC Sample Preparation and Analysis

For each batch of 20 or fewer samples prepare a minimum of one method blank and two separate sample duplicates.

For the method blank, follow the procedures outlined above and use 1L of reagent water to serve as the blank matrix.

For the sample duplicate sample, follow the procedures outlined above and prepare and analyze one sample in duplicate per batch or 1 sample in duplicate for every 10 samples analyzed, whichever is greater.

Attachment 12:

Percent Moisture and Total Solids Procedures SM2540G

Preparation of the Evaporating Dishes or Crucibles

Thoroughly clean the evaporating dishes or crucibles that will be required for the determination of total solids. It may be necessary to place Nochromix in the dish for a short time to remove dissolved solids from a previous analysis.

Rinse the evaporating dishes or crucibles thoroughly with reagent water and allow them to dry. The dishes should be placed in the oven at 105°C to facilitate the drying.

After the evaporating dish has remained in the oven at 105°C for a minimum of two hours, remove the dishes from the oven and place in the desiccator. Do not close the door of the desiccator completely until the dishes have cooled. After the dishes have cooled, close the door of the desiccator. The evaporating dishes and crucibles may be stored in the desiccator until ready for use.

NOTE: If the dishes are placed into the desiccator while hot and the door is sealed, a vacuum may form as the dishes cool. If this occurs, the door of the desiccator may be difficult to open.

Determination of Total Solids (or Percent Moisture)

Calibrate and verify that the top loading balance is working within the proper parameters in accordance with SOP SA-AN-100: *Laboratory Support Equipment (Verification and Use)*.

Label the drying dish with an identification number.

Measure the weight of the pre-labeled, pre-cleaned dishes to the nearest 0.1mg on an analytical balance.

Thoroughly mix the sample with a stainless steel spatula or glass rod. It is important to obtain a homogeneous mixture prior to sub-sampling so that the sub-sample will accurately reflect the composition of the sample. Leaves, rocks, and other foreign materials should not be included in the sub-sample.

NOTE: If there is any doubt as to how to treat a given sample, contact the department manager to determine the proper course of action. SOP SA-QA-15: *Homogenization, Compositing, and Segregation of Samples* provides guidance for homogenizing samples.

Add approximately a 25-50g aliquot of the well-mixed sample to the dish. Record the weight to the nearest 0.1mg.

Place the sample in the drying oven, maintained at 103°C -105° for at least 12 hours (overnight).

Remove the evaporation dishes from the oven, allow them to cool slightly, and place them in the desiccator. Remember not to place the evaporating dishes on a surface that can cause dirt or other foreign objects to adhere to the dishes.

Allow the evaporating dishes to remain in the desiccator until completely cool.

Weigh the evaporating dish on the analytical balance and record the weight of the dish and the residue.

The percent solids is calculated using the following equation:

$$percent_total_solids = \frac{A - B}{W} x\,100$$

Where:
A = weight of sample residue and evaporating dish (g)
B = weight of evaporating dish (g)
W = weight of sample used to determine the total solids (g)
i.e., W = weight $_{wetsample + dish}$ – weight$_{dish}$

The percent moisture is calculated using the following equation:

$$percent_moisture = \frac{W - (A - B)}{W} x\,100$$

Where:
A = weight of sample residue and evaporating dish (g)
B = weight of evaporating dish (g)
W = weight of sample used to determine the total solids (g)
i.e., W = weight $_{wetsample + dish}$ – weight$_{dish}$

QC Sample Preparation and Analysis

For each batch of 20 or fewer samples prepare a minimum of one method blank and two separate sample duplicates.

For the method blank, follow the procedures outlined above and use 10g of Ottawa Sand that has been prepared for metals analysis to serve as the blank matrix.

For the sample duplicate sample, follow the procedures outlined above and prepare and analyze one sample in duplicate per batch or 1 sample in duplicate for every 10 samples analyzed, whichever is greater.

Attachment 13:

Volatile Solids, Ash Content, and Fixed Solids Procedures SM2540G

Preparation of the Evaporating Dishes and Crucibles

Note that the same evaporating dishes will be used to determine the % moisture and total solids.

Thoroughly clean the evaporating dishes or crucibles that will be required for the determination of ash content and fixed solids. It may be necessary to place Nochromix in the dish for a short time to remove dissolved solids from a previous analysis.

Rinse the evaporating dishes thoroughly with reagent water and allow them to dry. The dishes should be placed in the oven at 105°C to facilitate the drying.

Remove the evaporating dishes from the oven and place them in the muffle furnace. Turn on the muffle furnace, and elevate the temperature 100°C per hour until it reaches 550°C ± 50°C.

After the evaporating dishes have remained in the muffle furnace at 550°C ± 50°C for a minimum of one hour, remove the dishes and place them in the desiccator. Do not seal the door of the desiccator completely until the dishes have cooled. After the evaporating dishes have cooled, close the door of the desiccator. The evaporating dishes may be stored in the desiccator until ready for use.

Note: If the evaporating dishes are placed into the desiccator while hot and the top or door is sealed, a vacuum may form as the dishes cool. The top or door may be difficult to open if this occurs. This can occur very readily after the evaporating dishes have been removed from the muffle furnace.

Determination of Volatile Solids, Ash Content, and Fixed Solids

Determine Percent Moisture, Total Solids (2540G) as described in Attachment 13. Use evaporating dishes that have been prepared as above.

Place the evaporating dishes in the muffle furnace. Turn on the muffle furnace, and elevate the temperature 100°C per hour until it reaches 550°C ± 50°C.

After the evaporating dishes have remained in the muffle furnace at 550°C ± 50°C for a minimum of one hour, remove the evaporating dishes from the muffle furnace and place them on a non-reactive surface to cool. Caution: the dishes will be extremely hot when removed. Do not place them on a surface that will melt or ignite

Note: One option is to turn the muffle furnace off and let then dishes cool in the furnace.

After the dishes have cooled, place them in desiccator for at least one hour or overnight.

Remove the dishes from the desiccator and weigh the dishes to constant weight using the procedure outlined in Attachment 14.

The Ash Content is calculated using the following equation:

$$percent_ash = \frac{A}{B} x\ 100$$

Where:
A = weight of sample residue after heating to 550°C (g)
B = weight of sample residue after heating to 105°C (g)

The Ash content can also be reported as % Fixed Solids

The Volatile Solids is calculated using the following equation:

$$percent_volatile_solids = \frac{B - A}{B} x\ 100$$

Where:
A = weight of sample residue after heating to 550°C (g)
B = weight of sample residue after heating to 105°C (g)

QC Sample Preparation and Analysis

For each batch of 20 or fewer samples prepare a minimum of one method blank, one LCS, and two separate sample duplicates.

For the method blank, follow the procedures outlined above and use 10g of Ottawa Sand that has been prepared for metals analysis to serve as the blank matrix.

For the sample duplicate sample, follow the procedures outlined above and prepare and analyze one sample in duplicate per batch or 1 sample in duplicate for every 10 samples analyzed, whichever is greater.

Attachment 14:

Constant Weight Determination Procedures

For all residue determinations except percent solids/moisture, the constant weight must be achieved. To verify constant weight has been achieved, replicate weighings of each sample must occur. Each sample must be placed back into the oven at the appropriate temperature and re-weighed after a minimum of 1 hour.

If the percent difference between the initial and subsequent weighing is less than 4% or the difference is less than 0.5mg from the previous weighing, the sample has been dried to a constant weight, and no further drying time is needed.

If the percent difference is greater than 4%, or if the difference is greater than 0.5mg, the sample must be dried and re-weighed until the "constant weight" criteria are achieved.

NOTE: The "percent difference" and "difference" limits are separate criteria. Only one has to be met. The difference criteria will apply to low concentration samples and the percent difference criteria to higher concentration samples.

Attachment 15

Percent Solids/Moisture Procedures (Used for Dry Weight Correction) EPA 3500B, EPA 3500C, EPA 3550B, and EPA 3550C

Determination of Percent Solids (or Percent Moisture)

Calibrate and verify that the top loading balance is working within the proper parameters in accordance with SOP SA-AN-100: *Laboratory Support Equipment (Verification and Use).*

Label an aluminum pan with an identification number. Create a new Moisture Batch in Analyst Desktop in LIMS and enter the LIMS IDs of the samples to be tested. Weigh the pan on the top loading balance. Record the weight (to the nearest 0.01g) of the aluminum pan in the column labeled "DishWeight" in the row of the corresponding sample. Do NOT TARE the balance after weighing the dish.

Thoroughly mix the sample with a stainless steel spatula or glass rod. It is important to obtain a homogeneous mixture prior to sub-sampling so that the sub-sample will accurately reflect the composition of the sample. Leaves, rocks, and other foreign materials should not be included in the sub-sample.

Note: If there is any doubt as to how to treat a given sample, contact the department manager to determine the proper course of action. SOP SA-QA-15: Homogenization, *Compositing, and Segregation of Samples* provides guidance for homogenizing samples.

Add approximately a 10g aliquot of the well-mixed sample to the un-tared aluminum pan. The amount can vary by as much as +/- 50%. Record the weight to the nearest 0.01g in the column labeled "SampleMassWet."

Place the sample in the drying oven, maintained at 105°C +/- 5°C, for a minimum of 4 hours for % solids used for dw determination.

Remove the aluminum pan from the oven and allow to cool. Remember not to place the aluminum pan on a surface that can cause dirt or other foreign objects to adhere to the pan. Ensure that the surface that the pan is placed on can handle the temperature of the aluminum pan without damage.

Weigh the aluminum pan containing the sample residue on the top-loading balance and record the weight to the nearest 0.01g in the column labeled "SampleMassDry."

The LIMS program automatically calculates the results for Percent Solids according to the equations listed below.

Use the following equation to calculate the Percent Solids:

$$Percent\ Solids = \frac{A - B}{C - D} x\ 100$$

Where:
A = weight of dry sample and aluminum pan
B = weight of aluminum pan
C = weight of wet sample and aluminum pan
D = weight of aluminum pan

Use the following equation to express the percent solids as a decimal equivalent ("Solids") for calculating sample results on a "dry weight basis":

$$\text{Solids} = \frac{\text{Percent Solids}}{100},$$

Or

$$Solids = \frac{A - B}{C - D}$$

Where:
A = weight of dry sample and aluminum pan
B = weight of aluminum pan
C = weight of wet sample and aluminum pan
D = weight of aluminum pan

Use the following equation to express the percent solids as percent moisture:

$$\text{Percent Moisture} = 100 - \text{Percent Solids}$$

Revision History

Summary of Changes from Previous Revision:

- Minor editorial changes and clarifications made.
- Added sections on % Solids/% Moisture procedures used for dry weight correction and the applicable attachment.
- Added requirement to place samples in muffle furnace prior to turning on the oven. Then gradually increase the temperature 100C every 10 minutes until the temperature reaches 550C. Attachment 6, Attachment 8, Attachment 10, and Attachment 13
- Revised reagent storage requirements for TDS/TS check standard to allow storage at room temperature. Section 7.3.1
- Added information on laboratory's % Solids/% Moisture (used for dry weight calculations) to Method Modifications and Clarifications section. Section 16.6

Measurement of Analytes using the PC Titrate Auto-Analyzer

(Methods: Various)

Approvals (Signature/Date):

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1.0 Scope and Application

This SOP contains the procedures for the determination of various analytes using the PC Titrate Auto-analyzer. The following analytes may be determined:

Analyte	Routine Matrix	Reference Method
Acidity (as Calcium Carbonate)	Water	EPA 305.1 SM 2310B
Alkalinity[1]	Water	EPA 310.1 SM 2320B
Conductivity[2]	Water	EPA 120.1 EPA 9050A SM 2510B
pH	Water	EPA 150.1 EPA 9040B EPA 9040C SM 4500H$^+$ B
Salinity	Water	SM 2520B

[1]In addition to total alkalinity, the analyzer can calculate the following carbonate species: phenolphthalein alkalinity, bicarbonate alkalinity, carbonate alkalinity, hydroxide alkalinity, and free carbon dioxide. The calculations for CO_2 and forms of alkalinity are taken from Standard Methods 4500-CO_2 D.

[2]In addition to conductivity, the analyzer can calculate resistivity.

This procedure is also used, in conjunction with SOP SA-AN-100: *Laboratory Support Equipment (Verification and Use)* to determine the conductivity of the deionized water generated and used by the lab.

A complete target analyte list, the reporting limits (RL), the method detection limits (MDL), and the accuracy and precision criteria associated with this procedure are provided in the LIMS Method Limit Groups (MLGs).

This SOP was written by and for TestAmerica's Savannah laboratory.

2.0 Summary of Method

An appropriate volume of sample or sample extract is introduced into the PC Titrate Auto-analyzer. Analyses for Acidity, Alkalinity, Conductivity, pH, and Salinity may be performed using manual methodologies as long as all of the QA/QC requirements of this document are met.

2.1 Acidity Determination

The pH of the sample aliquot is determined. The sample is titrated to a pH of 8.2 using a commercially prepared 0.1N NaOH solution. Titration to pH 8.2 reflects the bicarbonate equivalence point. The 8.2 endpoint is chosen because many waters contain naturally occurring bicarbonate salts. Determination of acidity by titrating to this endpoint makes acidity independent of any bicarbonate salts that may have been present in the original

sample. This bicarbonate-independent acidity is often termed the CO_2-acidity, since the dissolved CO_2 and other gases are measured.

Samples which are known or suspected to contain ferrous or ferric iron may be treated with hydrogen peroxide prior to titration, to ensure a true mineral acidity is obtained. The pH of the sample aliquot is determined. Commercially prepared 0.02N H_2SO_4 is added in to achieve a pH of 4 or less. An aliquot of H_2O_2 is added to the solution, which is boiled for two minutes, then cooled to room temperature. The sample is then titrated using standardized NaOH to a pH of 8.2.

2.2 Alkalinity Determination
Total alkalinity, as calcium carbonate, is determined in water samples by titrating the sample to an endpoint of pH 4.5 with standardized acid. Phenolphthalein alkalinity is determined by titrating to an endpoint of 8.3. The other carbonate species that can be reported with this procedure are calculated using the results of the sample's total alkalinity and pH.

2.3 Conductivity Determination
The conductivity of a sample is determined at a temperature that is as close to 25°C as possible using a self-contained conductivity meter. The instrument adjusts the conductivity automatically based on the cell constant and sample temperature. The resistivity is calculated using the sample conductivity.

2.4 pH Determination
The pH of liquid samples is determined potentiometrically using a pH electrode meter with temperature compensation.

2.5 Salinity Determination
The salinity of a sample is determined as a function of conductivity once the instrument has been calibrated for salinity using a 0.5078N NaCl standard to determine the practical salinity value.

2.7 This SOP is based on the methods listed in Section 1.

3.0 Definitions

Refer to the Glossary Section of the *Quality Assurance Manual* (QAM) for a complete listing of applicable definitions and acronyms.

4.0 Interferences

4.1 Procedural Interferences

4.1.1 Interferences may be caused by contaminants in solvents, reagents, glassware, and other sample processing apparatus and can make quantification of the target analytes difficult.

4.1.2 All sample collection containers are single-use disposable containers which limits the potential for contamination. All non-disposable labware must be scrupulously cleaned in accordance with the posted Labware Cleaning Instructions to ensure it is free from contaminants and does not contribute artifacts.

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4.1.3 High purity reagents and solvents are used to help minimize interference problems. Sulfuric acid must be verified prior to use in accordance with the TestAmerica Solvent Lot Testing Program.

4.1.4 Instrument and/or method blanks are routinely used to demonstrate all reagents and apparatus are free from interferences under the conditions of the analysis.

4.1.5 Conductivity Procedural Interferences

4.1.5.1 Platinum electrodes can degrade and operate erratically. If this happens, as evidenced by anomalous results or flaking off of the platinum black coating, the electrode should be replaced.

4.1.5.2 The conductivity cell can become coated with oil or dirt. It is essential that the cell be thoroughly rinsed and, if necessary, cleaned well with a soft brush and detergent between samples. Alternatively, the cell can be cleaned with 1:1 isopropanol and 10N HCl.

4.1.5.3 The use of conductivity to determine salinity can produce false negatives for non-ionic solutions. The use of density to determine salinity is less precise; however, it works for all dissolved solutions.

4.2 Matrix Interferences

4.2.1 Matrix interferences may be caused by contaminants that are co-extracted from the sample matrix. The sample may require cleanup or dilution prior to analysis to reduce or eliminate the interferences.

4.2.2 Interfering contamination may occur when a sample containing low concentrations of analytes is analyzed immediately following a sample containing relatively high concentrations of analytes. As such, samples known to be clean should be analyzed first. To prevent carryover into subsequent samples, analysis of reagent blanks may be needed after the analysis of a sample containing high concentrations of analytes.

4.2.3 Acidity-specific Matrix Interferences

4.2.3.1 Reduced iron, aluminum, and other reduced polyvalent cations will not be included in the measured acidity unless they are first oxidized with the peroxide treatment.

4.2.3.2 Samples containing suspended matter may cause erratic pH readings. Allow sufficient time for the pH to stabilize between titrant additions.

4.2.3.3 Dissolved gases such as CO_2, hydrogen sulfide, and ammonia, which contribute to acidity, may be lost or gained during sampling, storage, and titration. Avoid shaking the sample container vigorously. Avoid sample exposure to the atmosphere prior to titration.

4.2.4 Alkalinity-specific Matrix Interferences

4.2.4.1 Extremely turbid, oily, and soap-containing samples will affect the analysis by coating the pH electrode.

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4.2.4.2 Shaking an alkalinity sample will introduce CO_2 into the sample causing elevated alkalinity readings.

4.2.5 Conductivity- and Salinity-specific Matrix Interferences

Exposure to the atmosphere will cause an upward drift in samples with low conductivity due to the adsorption of carbon dioxide. Samples with an oily matrix may coat the platinum plates, causing sluggish readings.

4.2.6 pH-specific Matrix Interferences

4.2.6.1 To prevent cross-contamination, thorough rinsing of the electrode is required between readings.

4.2.6.2 Temperature effects are minimized by the use of a pH meter that automatically compensates for temperature. The temperature of the samples must be at or near the temperature of the buffer solutions (i.e., room temperature). This automatic temperature compensation device must be verified quarterly in accordance with SOP SA-AN-100.

5.0 Safety

Employees must abide by the policies and procedures in the TestAmerica Environmental Health and Safety Manual (EHSM), the TestAmerica Savannah Addendum to the EHSM, and this document.

This procedure may involve hazardous materials, 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 to follow appropriate safety, waste disposal, and health practices under the assumption that all samples and reagents are potentially hazardous.

The analyst must protect himself/herself from exposure to the sample matrix. Many of the samples that are tested may contain hazardous chemical compounds or biological organisms. The analyst must, at a minimum, wear protective clothing (lab coat), eye protection (safety glasses or face shield), disposable nitrile gloves, and closed-toe, nonabsorbent shoes when handling samples.

5.1 Specific Safety Concerns or Requirements

5.1.1 Acidity-Specific Safety Concerns or Requirements

Hydrogen peroxide is a dangerous oxidizer and an extreme corrosive. The vapors are corrosive and irritating to the respiratory tract. Contact to the skin can cause redness, pain and severe burns. Contact with the eyes can cause permanent tissue destruction. Contact with organic material can cause fires.

Sodium hydroxide is a severe corrosive. Contact with the skin can cause irritation or severe burns and scarring. Contact with the eyes can cause irritation, burns, permanent vision impairment or even blindness.

5.1.2 Alkalinity-Specific Safety Concerns or Requirements

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Samples that contain high concentrations of carbonates or organic matter, or samples that are at elevated pH can react violently when acids are added. Acids must be added to samples under a hood to avoid splash/splatter hazards and/or possibly toxic vapors that will be given off when the samples are acidified.

Sulfuric acid is a strong oxidizer and is a corrosive. It will react violently when combined with organic compounds, possibly producing fire. Inhalation can cause irritation of the nose, throat, mucus membranes, and upper respiratory tract. Contact with the eyes can cause blurred vision, redness, pain, and even blindness.

5.1.3 pH-Specific Safety Concerns or Requirements

Proper care must be used when handling the reagents. The buffers used in this procedure contain reagents that can be poisonous.

5.2 Primary Materials Used

The following is a list of the materials used in this procedure, which have a serious or significant hazard rating, and a summary of the primary hazards listed in their MSDS.

Note: This list does not include all materials used in the procedure. A complete list of materials used in this procedure can be found in the Reagents and Standards Section and the Equipment and Supplies Section of this SOP

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 found using the "MSDS" link on the Oasis homepage, on the EH&S webpage on Oasis, and on the QA Navigator.

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Material	Hazards	Exposure Limit[1]	Signs and Symptoms of Exposure
Sulfuric Acid	Corrosive Oxidizer Dehydrator Poison Carcinogen	1 mg/m^3 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.
Hydrogen Peroxide	Oxidizer Corrosive	1 ppm TWA	**Danger! Strong oxidizer**. Contact with other material may cause a fire. Corrosive. Light sensitive. May be harmful if swallowed. May cause central nervous system effects. Eye contact may result in permanent eye damage. May cause severe respiratory tract irritation with possible burns. Causes eye and skin irritation and possible burns. May cause severe digestive tract irritation with possible burns. Vapors are corrosive and irritating to the respiratory tract. Vapors are very corrosive and irritating to the eyes and skin.
Sodium Hydroxide	Corrosive	2 mg/m^3 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]Exposure limit refers to the OSHA regulatory exposure limit.			
Note: Always add acid to water to prevent violent reactions.			

6.0 Equipment and Supplies

6.1 Equipment and Instrumentation

PC-Titrate Plus Auto Titration System – Version 3, Hinterland Software

pH Meter with an automatic temperature adjustment probe. Calibrate each day of use in accordance with attachment 5.

Conductivity meter with 1.18K conductivity cell or equivalent, an automatic temperature adjustment probe.

Thermometers – Verify in accordance with SOP SA-AN-100: *Laboratory Support Equipment (Verification and Use).*

Analytical Balance – Verify in accordance with SOP SA-AN-100: *Laboratory Support Equipment (Verification and Use).*

Top-loading Balance – Verify in accordance with SOP SA-AN-100: *Laboratory Support Equipment (Verification and Use).*

Data System: PC-Titrate Hinterland software is used to acquire, store, reduce, and output data. This software has the capability of storing calibration curves, and monitoring QC parameters with control charting.

6.2 Lab Supplies

Volumetric Containers – various sizes; Class A, where applicable. Verify in accordance with SOP SA-AN-100: *Laboratory Support Equipment (Verification and Use).*

Mechanical Pipettes – various sizes. Verify in accordance with SOP SA-AN-100: *Laboratory Support Equipment (Verification and Use).*

Disposable Graduated Pipettes – various sizes. Verify in accordance with SOP SA-AN-100: *Support Equipment (Verification and Use).*

Disposable Transfer Pipettes – various sizes

Medicine cups – 30mL, disposable

Detergent – used for washing non-disposable labware.

Centrifuge vials – 50mL from Mansci (P/N: PC-100-429/1000)

Magnetic Stirrer

Stir Bars – Teflon-coated

Beakers – 150mL glass

Volumetric flasks – 1000mL

Ferrous/Ferric Iron test strips – Ferrous/Ferric Iron test strips provide a quick and easy way to determine if iron is present in a sample.

Iron test strips should be checked upon receipt to make sure that they are functioning properly.

-Examine the test strip. If the paper is discolored or looks worn, it may be defective.
-Place a piece of the test strip on a watch glass or other suitable surface and add a few drops of a certified iron solution onto the paper.
-Compare the color of the paper to the reference colors. If the colors match, the paper can be used. If not, acquire new paper.

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Dip a clean disposable Pasteur pipette into the sample and touch the pipet to the iron test strip. Compare the color to the reference. Note: to ensure cross-contamination does not occur, use a new pipette for each sample.

Note: Do not dip the iron test strip into a sample.

6.3 Sample Collection Containers

All sample collection containers are single-use disposable containers which limits the potential for contamination.

The routine sample collection containers supplied by the laboratory are:

Aqueous samples:
125mL or 250mL plastic containers

Soil and waste samples:
8oz plastic or 4oz amber glass containers

7.0 Reagents and Standards

7.1 Expiration Dates

Expiration dates (time from initial use or receipt to final use) for standard and reagent materials must be set according to the guidance in this SOP. Note: These are maximum expiration dates and are not to be considered an absolute guarantee of standard or reagent quality. Sound judgment must be used when deciding whether to use a standard or reagent. If there is doubt about the quality of a standard or reagent material, a new material must be obtained or the standard or reagent material verified. Data quality must not be compromised to extend a standard's life – i.e., when in doubt, throw it out.

The expiration date of any standard or reagent must not exceed the expiration date of the standard or reagent that was used to prepare it; that is, the "children may not outlive the parents".

7.2 Reagents

Reagents must be prepared and documented in accordance with SOP SA-AN-041: *Reagent and Standard Materials Procedures.*

Sulfuric acid must be verified prior to use in accordance with the TestAmerica Solvent Lot Testing Program.

Blank Matrix – Ottawa Sand. Used for the preparation of soil QC samples.

Laboratory Reagent Water – ASTM Type II.

7.2.1 Acidity-Specific Reagents

7.2.1.1 Laboratory Reagent Water – ASTM Type II reagent water used for the preparation of reagents and the standardization of NaOH must be boiled for a minimum of 15

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minutes then cooled to room temperature. The pH and the conductivity must be taken and recorded in the acidity analysis log.

**Note: Reagent water must be prepared fresh daily. Reagent water must have a pH $\geq$ 6 and the conductivity must be <2.0umhos/cm.

7.2.1.2 Hydrogen peroxide, 30% (H_2O_2) - commercially prepared
Storage: Refrigerated at 4°C in a dark, well-ventilated area separated from combustible substances, reducing agents, strong bases and organics. Do not store on wooden shelves or floors.
Expiration: Manufacturer's expiration date

7.2.1.3 Sulfuric acid, 0.02N (H_2SO_4) – commercially prepared
Storage: Room temperature, in acid cabinet
Expiration: Manufacturer's expiration date

7.2.1.4 Sodium hydroxide 0.1N (NaOH) Solution – commercially prepared (Fisher SS276-4) or equivalent. Caution: Sodium hydroxide is caustic and will destroy unprotected clothing and cause chemical burns on unprotected skin and eyes.
Storage: Room temperature away from incompatibles
Expiration: Manufacturer's expiration date

7.2.2 Alkalinity-Specific Purchased Reagents

7.2.2.1 Commercially prepared 0.02N Sulfuric Acid Solution (VWR EM-SX1243-F-3 or equivalent).
Storage: Room temperature, in acid cabinet
Expiration: Manufacturer's expiration date or 2 years from the date opened

7.2.3 Conductivity-Specific Reagents
There are no items for this section.

7.2.4 pH-Specific Reagents
There are no items for this section.

7.2.5 Salinity-Specific Reagents
There are no items for this section.

7.3 Standards

Standards must be prepared and documented in accordance with SOP SA-AN-041: *Reagent and Standard Materials Procedures.* Certificates of analysis or purity must be received with all purchased standards, and scanned and filed in the Data Archival Folder on the Public Drive or attached to the TALS batch.

7.3.1 Acidity-Specific Standards

7.3.1.1 Sulfuric acid (H_2SO_4), 0.02N (1000mg/L acidity) - commercially prepared, used to prepare the laboratory control standards. (Alkalinity Titrant)
Storage: Room temperature, in acid cabinet

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Expiration: Manufacture's expiration date

7.3.2 Alkalinity-Specific Standards

7.3.2.1 Refer to pH information listed in Section 7.3.5, for information on initial calibration and second source calibration verification standards.

7.3.2.2 Laboratory Control Standard (LCS) – Mineral Standard (QCI). The concentration of this standard varies with each lot.
Storage: reagent cabinet
Expiration: Manufacturer's expiration date or 2 years from the date opened, whichever is sooner.

7.3.3 Conductivity-Specific Standards

7.3.3.1 Potassium Chloride (KCl) – reagent grade
Storage: Room temperature
Expiration: Manufacturer's expiration date

7.3.3.2 Second Source Calibration Verification Standard (1,000umhos/cm) – purchased from YSI
Storage: Room temperature
Expiration: Manufacturer's expiration date

7.3.3.3 Linear Calibration Verification Standards
- 1413uS NCLabs C-37L or equivalent
- 6668uS NCLabs C-37P or equivalent
- 12900uS NCLabs C-37Q or equivalent
- 47600uS Fisher 7225-32 or equivalent

7.3.3.4 Daily Calibration Standard – 1413uS Ricca cat no.2236.52.32 or equivalent

7.3.3.5 Reporting Limit Standard (5.0mhos/cm) – Fill a 250mL volumetric flask with approximately 75mL of reagent water. Carefully add 1.25mL of the second source calibration verification standard (1,000umhos/cm). Dilute to volume with reagent water.
Storage: Room temperature
Expiration: One month from preparation date

7.3.4 pH-Specific Standards

7.3.4.1 Calibration standards: Certified pH buffers to bracket pH range of samples. Suggested buffers are 2.0, 4.0, 7.0, 10.0, and 12.0. These buffer solutions are also used for continuing calibration verifications (CCV).
Storage: solvent cabinet
Expiration: manufacturer's expiration date or 2 years from the date opened, whichever is sooner.

7.3.4.2 Initial Calibration Verification (ICV): certified solution from ERA or other vendor. The check standard must be purchased from a separate source than that used to purchase the calibration standards.

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Storage: solvent cabinet
Expiration: manufacturer's expiration date or 2 years from the date opened, whichever is sooner.

7.3.5 Salinity-Specific Standards

0.5078N NaCl – Fisher 7225-32 or equivalent
Storage: Reagent Cabinet BOD lab
Expiration: Manufacturer's expiration date

Laboratory Control Standard – 29.64g of the 0.5078N NaCl solution, diluted to 1L.

8.0 Sample Collection, Preservation, Shipment, and Storage

Refer to Attachment 2 for a summary of the sample collection, preservation, shipment, storage, and holding time requirements associated with these procedures.

9.0 Quality Control

SOP SA-QA-17: *Evaluation of Batch QC Data* and the SOP Summary in Attachment 3 provide requirements for evaluating QC data.

9.1 Batch QC

9.1.1 Acidity Batch QC

Acidity batches need to be bracketed with lab control samples (LCS). Start batch with an LCS and end batch with an LCSD.

9.1.1.1 Aqueous

An analytical batch consists of up to 20 environment samples and the associated quality control items. The minimum QC items required for each analytical batch are: a method blank, a laboratory control sample (LCS), a laboratory control sample duplicate (LCSD at the end of the analysis), and a sample duplicate to be performed on a minimum of 10% of samples or one per batch – whichever is greater.

This frequency equates to the following:

- For a batch of 10 or fewer samples, the minimum QC items are a method blank, an LCS, LCSD (at the end of the analysis), and 1 sample duplicate.
- For a batch of 11-20 samples, the minimum QC items are a method blank, an LCS, LCSD (at the end of the analysis), 1 sample duplicate (from sample 1-10), and another sample duplicate (from sample 11-20).

9.1.2 Alkalinity Batch QC

Alkalinity batches need to be bracketed with lab control samples (LCS). Start batch with an LCS and end batch with an LCSD.

9.1.2.1 Aqueous

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An analytical batch consists of up to 20 environment samples and the associated quality control items. The minimum QC items required for each analytical batch are: a method blank, a laboratory control sample (LCS), a laboratory control sample duplicate (LCSD at the end of the analysis), and a sample duplicate to be performed on a minimum of 10% of samples or one per batch – whichever is greater.

This frequency equates to the following:

- For a batch of 10 or fewer samples, the minimum QC items are a method blank, an LCS, LCSD (at the end of the analysis), and 1 sample duplicate.
- For a batch of 11-20 samples, the minimum QC items are a method blank, an LCS, LCSD (at the end of the analysis), 1 sample duplicate (from sample 1-10), and another sample duplicate (from sample 11-20).

Note: The EPA Manual for the Certification of Laboratories Analyzing Drinking Water requires a LFB at the MRL to be performed each day. Therefore, if analyzing drinking water samples for alkalinity, an LCS at the RL must also be included in the required batch QC.

9.1.3 Conductivity Batch QC

9.1.3.1 Aqueous

An analytical batch consists of up to 20 environment samples and the associated quality control items. The minimum QC items required for each analytical batch are: a method blank, a laboratory control sample (LCS), and a sample duplicate to be performed on a minimum of 10% of samples or one per batch – whichever is greater.

This frequency equates to the following:

- For a batch of 10 or fewer samples, the minimum QC items are a method blank, an LCS, and 1 sample duplicate.
- For a batch of 11-20 samples, the minimum QC items are a method blank, an LCS, 1 sample duplicate (from sample 1-10), and another sample duplicate (from sample 11-20).

If there is insufficient sample volume to perform the required sample duplicate(s), the LCS must be prepared in duplicate (i.e., LCS/LCSD). An NCM must be initiated on all affected samples to denote this situation. Insufficient sample volume is defined as receiving less than a total of 60mL.

Note: If an LCS and LCSD are performed, both QC items must be evaluated and reported. Acceptable recoveries (as well as %RPD) for both LCS and LCSD are required.

Note: The EPA Manual for the Certification of Laboratories Analyzing Drinking Water requires a LFB at the MRL to be performed each day. Therefore, if analyzing drinking water samples for conductivity, an LCS at the RL must also be included in the required batch QC.

Batch QC must meet the criteria given in Attachment 3 of this SOP.

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9.1.4 pH Batch QC

9.1.4.1 Aqueous

The minimum QC items required for each pH batch are: a laboratory control sample (LCS), and two sample duplicates (1 for every 10 samples).

If there is insufficient sample volume to perform the required sample duplicate(s), the LCS must be prepared in duplicate (i.e., LCS/LCSD). An NCM must be initiated on all affected samples to denote this situation. Insufficient sample volume is defined as receiving less than a total of 60mL.

Note: If an LCS and LCSD are performed, both QC items must be evaluated and reported. Acceptable recoveries (as well as %RPD) for both LCS and LCSD are required.

Note: The EPA Manual for the Certification of Laboratories Analyzing Drinking Water requires a LFB at the MRL to be performed each day. Therefore, if analyzing drinking water samples for pH, an LCS at the RL must also be included in the required batch QC.

9.1.5 Salinity Batch QC

9.1.5.1 Aqueous

An analytical batch consists of up to 20 environment samples and the associated quality control items. The minimum QC items required for each analytical batch are: a method blank, a laboratory control sample (LCS), and a sample duplicate to be performed on a minimum of 10% of samples or one per batch – whichever is greater.

This frequency equates to the following:

- For a batch of 10 or fewer samples, the minimum QC items are a method blank, an LCS, and 1 sample duplicate.
- For a batch of 11-20 samples, the minimum QC items are a method blank, an LCS, 1 sample duplicate (from sample 1-10), and another sample duplicate (from sample 11-20).

If there is insufficient sample volume to perform the required sample duplicate(s), the LCS must be prepared in duplicate (i.e., LCS/LCSD). An NCM must be initiated on all affected samples to denote this situation. Insufficient sample volume is defined as receiving less than a total of 60mL.

Note: If an LCS and LCSD are performed, both QC items must be evaluated and reported. Acceptable recoveries (as well as %RPD) for both LCS and LCSD are required.

9.2 Instrument QC

9.2.1 Initial Calibration (ICAL)

The instrument must be calibrated in accordance with SOP SA-QA-16: *Evaluation of Calibration Curves*. This SOP provides requirements for establishing the calibration curve and gives the applicable formulas.

The initial calibration standard concentrations currently in use in the laboratory are as follows:

pH (used for Alkalinity and Acidity as well)

Standard Level	Concentration (mg/L)
1	4
2	7
3	10

Conductivity Linear calibration verification (annual)

Standard Level	Concentration (uS)
1	1413
2	6668
3	12900
4	47600

Salinity

Standard Level	Concentration (Practical Salinity value)
1	35

Refer to Section 7.3 for the standard preparation instructions. Other standard concentrations may be used provided they support the reporting limit and are fully documented in accordance with SOP SA-AN-041.

pH (used for Alkalinity and Acidity as well):
For routine samples with pH range of 4-10, calibrate the meter using pH 4.0, 7.0, and 10 buffer solutions. For the curve to be acceptable, each calibration buffer's reading must be within 0.05 pH units from the actual value.

For samples with pH below 4 and above 10, analyze a 2.0 or a 12.0 buffer to check the calibration accuracy at that level. If the pH 2.0 and/or the pH 12.0 are recovered within 0.1 units then the results for the sample may be reported. If the standard is not recovered within acceptable limits then the meter must be recalibrated with 3 buffers that bracket the range of samples to be reported. It is acceptable to group those samples that exceed the pH 4-10 range and reanalyze them on a separate calibration after all other samples are analyzed.

Note: Pour fresh pH buffer solution from the stock bottle each day. Do not reuse over multiple days.

Calibration frequency – Daily

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Note: Samples falling outside the calibrated range of the pH meter can not be reported as quantitative values. For example, if a sample reads 1.5 and the meter is calibrated to pH 2.0 the sample must be reported as <2.0.

Conductivity
The conductivity meter is calibrated with a multi-point standard annually. The meter is calibrated daily with a fresh aliquot of 1413 standard and read back. The acceptance criteria for the read back is the same as the LCS +/- 10%.

Salinity
The meter is calibrated for salinity as needed, when salinity analysis is required.

9.2.2 Second Source Initial Calibration Verification (ICV)

The calibration curve must be verified initially – prior to any sample analyses – in accordance with SOP SA-QA-16 with a standard obtained from a second source.

The pH (Alkalinity/Acidity) ICV must be within 0.1 pH units of the actual value to be acceptable. The initial calibration verification standard concentration currently in use in the laboratory is equivalent to pH 7.0.

The Conductivity ICV (LCS) must be within 90-110% of the actual value to be acceptable.

The Salinity ICV (LCS) must be within 70-130% of the actual value to be acceptable.

Refer to Section 7.3 for the standard preparation instructions. Another standard concentration may be used provided it is mid-level and fully documented in accordance with SOP SA-AN-041.

9.2.3 Initial Calibration Blank (ICB) / Continuing Calibration Blank (CCB)

The instrument must be shown to be free from contamination by the analysis of calibration blanks. Initial calibration blanks are analyzed for fluoride immediately following the initial calibration. Continuing calibration blanks are analyzed for fluoride after each of the continuing calibration verification (CCV) standards.

Initial and continuing calibration blanks must be <RL to be acceptable.

9.2.4 Continuing Calibration Verification

The initial calibration curve for pH and fluoride must be verified prior to sample analysis, after every ten samples, and at the end of the sequence with a mid-level standard.

The CCV for pH must be within 0.1 pH units of the actual value to be acceptable. The continuing calibration verification standard concentration currently in use in the laboratory is equivalent to pH 7.0.

Alkalinity/Acidity requires two separate CCV. First, the pH CCV must be analyzed and be within 0.1 pH units of the actual value to be acceptable. The pH continuing calibration verification standard concentration currently in use in the laboratory is equivalent to pH 7.0. In addition to the pH CCV, an LCS must also be analyzed for Alkalinity and Acidity. The

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LCS must be within the limits defined in the LIMS MLG to be acceptable. The lab accomplishes this by analyzing the LCS at the beginning of the batch, prior to sample analysis, and the LCSD at the end of the batch.

The CCV for conductivity must be within 90-110% of the actual value to be acceptable.

Refer to Section 7.3 for the standard preparation instructions. Another standard concentration may be used provided it is mid-level and fully documented in accordance with SOP SA-AN-041.

9.2.5 Conductivity Reporting Limit Standard

The reporting limit must be verified at the beginning of the analysis with the 5umhos/cm standard.

The reporting limit (RL) standard must be within 50% of the true value to be acceptable.

Refer to Section 7.3 for the standard preparation instructions. Another standard concentration may be used provided it is mid-level and fully documented in accordance with SOP AN41.

Note: Correct the conductivity of the RL standard by subtracting the conductivity of the deionized water from the conductivity of the RL standard before calculating the recovery.

9.3 Corrective Action for Out-of-Control Data

When the quality control parameters do not meet the criteria set forth in this SOP, corrective action must be taken in accordance with SOP SA-QA-05: *Preventive and Corrective Action Procedures* and the QC Summary Table in Attachment 3. SOP SA-QA-05 provides contingencies for out-of-control data and gives guidance for exceptionally permitting departures from approved policies and procedures. Nonconformance Memos must be initiated to document all instances where QC criteria are not met and all departures from approved policies and procedures.

10.0 Procedure

10.1 Sample Preparation

10.1.1 Acidity Sample Preparation

Remove the samples to be analyzed from the storage refrigerator. Allow the samples to equilibrate to room temperature. Test each sample for ferrous or ferric iron and residual chlorine.

If the sample does not test positive for ferrous or ferric iron, proceed with Sample Analysis.

If the sample tests positive for ferrous or ferric iron, hot peroxide pretreatment (Section 10.1.1.1) may be performed, upon client request, to eliminate this interference. Notify

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the project manager via a non-conformance memo if samples test positive.

10.1.1.1 Peroxide Pre-treatment

- Transfer 50mL of the sample to a 150mL beaker.
- Using a calibrated pH meter (calibrated as per Attachment 5), measure the pH of the sample.
- If the pH is greater than 4.0, add 0.02N H_2SO_4 in 5mL increments until the pH is less than or equal to 4.0. Record the adjusted pH and volume of H_2SO_4 used in the acidity analysis log.
- Remove the pH probe from the sample and add 5 drops of 30% H_2O_2 to the sample. Place the sample on a hot plate and heat to boiling. Allow the sample to boil rapidly for 2 – 4 minutes. Cool the sample to room temperature and proceed with Sample Analysis.

  **NOTE -- 30% H_2O_2 IS A VERY DANGEROUS CHEMICAL. ALL SAFETY EQUIPMENT (safety glasses, lab coat, and gloves) MUST BE EMPLOYED WHEN HANDLING THIS CHEMICAL.

10.1.2 Alkalinity Sample Preparation

Remove the samples from the refrigerator and allow them to come to room temperature.

Pour 30-40 mL of sample into a 50mL conical tube.

10.1.3 Conductivity / Salinity Sample Preparation

Remove the samples from the refrigerator and allow them to come to room temperature (approximately 25°C).

Pour 40-45mL of sample into a 50mL conical tube. Note: There must be enough sample volume to fully submerge the conductivity cell or erroneous results will be determined.

Note: If the sample has an organic layer, the organic layer should be separated from the aqueous layer before the conductivity is measured using a separatory funnel. The Project Manager must be notified via an NCM if this occurs.

10.1.4 pH Sample Preparation

Remove the samples from the refrigerator and allow them to come to room temperature.

Pour 30-40mL of sample into a 50mL conical tube.

10.2 QC Sample Preparation

10.2.1 Acidity

The method blank is reagent water, and is treated exactly as a sample. Laboratory

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control standards are comprised of 0.02N H2SO4, which has been commercially prepared. The laboratory control standard is analyzed after the method blank to determine that the titration procedure is in control.

10.2.2 Alkalinity

Prepare the method blank using laboratory reagent water.

Prepare the laboratory control standard using the Mineral Standard (Section 7.3.2.2)

10.2.3 Conductivity / Salinity

Method Blank – Pour 40-45mL of reagent water into a 50mL conical tube.

Laboratory Control Sample – Pour 40-45mL of the 1,000umhos/cm second source standard into a 50mL conical tube.

Sample Duplicate – Prepare a separate aliquot of a sample.

10.2.4 pH

Prepare the laboratory control standard using the second source pH buffer 7.0 (Section 7.3.5.2)

10.3 Analysis

10.3.1 Instrument Operating Conditions

The instrument conditions listed in this SOP are provided for guidance purposes. The actual conditions used by the laboratory may be slightly different from those listed here and must be documented in the instrument maintenance log, data system, and/or run log.

Instrument maintenance must be performed in accordance with Attachment 4 of this SOP.

Instrument Operation:

- From the main menu, select the desired pre-loaded run template.
- The QC that is pre-loaded should not be altered. Scan or key the sample IDs into the run sequence.
- Once the samples are entered into the run sequence, print out a copy of the sequence to reduce errors when loading the auto sampler tray. Use 50L disposable centrifuge tubes and add 40mL of sample. Leave the caps off of the centrifuge tubes.
  Note: Always load a 4.0 buffer at the end of the run, so the probes will not dry out. This buffer is not keyed in as part of the run.
- After the samples are loaded in correct sequence, select the start button on the screen.
- Upon completion of the run, print the results by preparing a report. Refer to the PC-Titrate Manual Ch.2.07.02, and 2.07.03, located in the Data Archival folder on the Public Access drive.
- Once a report is generated, there is the option of exporting the data to the LIMS

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system.

For more information about the instrument refer to the instrument manual located on the Public Access drive: Public_QA:\Data Archival\Laboratory Files\General\Instrument Manuals.

10.3.2 Initial and Continuing Calibration

Calibrate the instrument using the standards and criteria described given in Section 9.2.1. Once the calibration has been established and verified with an ICV in accordance with Section 9.2.2, sample analysis may proceed.

Verify the calibration curve with a continuing calibration verification using the standards and criteria described given in Section 9.2.4.

10.3.3 Sample Analysis

Remove the samples or extracts from the refrigerator and allow them to come to room temperature.

The sample / extract must be analyzed using the same volume used for the calibration standards.

The default procedure is to include QC items (method blank, LCS, MS/MSD, and SD) in determining the maximum number of samples in the clock.

10.3.4 Example Analytical Sequence

An example analytical sequence is provided in Attachment 1.

11.0 Calculations / Data Reduction

11.1 Data Reduction

Data must be evaluated, reviewed, and reported in accordance with SOP SA-QA-02: *Data Generation and Review*.

11.1.1 MS/MSD Evaluation

If the concentration of a target analyte in the un-spiked (native) sample is more than four times the theoretical concentration of the matrix spike, the recovery is not reported and the data is flagged.

11.1.2 Historical Data

Many of the laboratory's clients submit samples for repeat monitoring purposes. Prior to analysis, verify LIMS Worksheet Notes and/or use the Historical Data Tracker feature to determine if historical data is available for review.

11.1.3 Chemical Relationships

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When available, the following chemical relationships must be evaluated for each sample. If these relationships are not met the Department Manager must be contacted immediately.

- Alkalinity is directly proportional to Conductivity
- At pH levels below 10 the majority of total alkalinity will be in the form of bicarbonate alkalinity
- Salinity can be estimated from the chloride concentration: S = 1.80655*Cl

11.1.4 Drinking Water Compliance Evaluation

Public water suppliers (PWS) are governed by EPA-specified Maximum Contaminant Levels (MCL) above which indicates noncompliance. The MCLs associated with this procedure are given in Attachment 6. Initiate a Nonconformance Memo if any sample contains a detection above these levels.

11.2 Calculations

The calculations associated with batch QC determinations are given in SOP SA-QA-17. Applicable calculations include accuracy (% recovery) and precision (%RPD).

The calculations associated with initial and continuing calibrations and are given in SOP SA-QA-16. Applicable calculations include determination for: calibration factor, standard deviation, relative standard deviation, relative response factor, and relative standard deviation.

The calculation to determine final concentration is performed by the PC-Titrate Plus.

11.2.1 pH

11.2.1.1 The calculation to determine the percent recovery is given as follows:

$$\%Rec = \frac{H}{H_T} x\ 100\ \%$$

Where:

H = hydrogen ion from pH measurement
H_T = theoretical hydrogen ion concentration

11.2.1.2 The calculation to determine the percent RPD is given as follows:

$$\%RPD = \left| \frac{H_1 - H_2}{(H_1 - H_2)/2} \right| x100\%$$

Where:

H_1 = hydrogen ion concentration of replicate 1
H_2 = hydrogen ion concentration of replicate 2

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11.2.2 Alkalinity

11.2.2.1 The alkalinity, as mg/L calcium carbonate, is calculated as follows:

$$Alkalinity(total) = \frac{A \otimes N}{V} \otimes \frac{50mg}{meq} \otimes \frac{1000mL}{L}$$

Where:
A = volume of standard acid (mL) to bring pH to 4.5
N = normality of standard acid (meq/mL)
V = volume of sample titrated (mL)

11.2.2.2 The phenolphthalein alkalinity, as mg/L calcium carbonate, is calculated as follows:

$$Alkalinity(phenolphthalein) = \frac{D \otimes N}{V} \otimes \frac{50mg}{meq} \otimes \frac{1000mL}{L}$$

Where:
D = volume of standard acid (mL) to bring pH to 8.3
N = normality of standard acid (meq/mL)
V = volume of sample titrated (mL)

11.2.2.3 The low level alkalinity (<20mg/L), as mg/L calcium carbonate, is calculated as follows:

$$Alkalinity(low) = \frac{(2B - C) \otimes N}{V} \otimes \frac{50mg}{meq} \otimes \frac{1000mL}{L}$$

Where:
B = volume of titrant to first endpoint (mL)
C = volume of titrant to lower pH by 0.3 units (mL)
N = normality of the sulfuric acid solution (meq/mL)
V = volume of sample titrated (mL)

11.2.2.4 The bicarbonate alkalinity, as mg/L calcium carbonate, is calculated as follows:

$$Bicarbonate = \frac{Alk_T - 5.0 \otimes 10^{(pH-10)}}{1 + 0.94 \otimes 10^{(pH-10)}}$$

Where:
Alk_T = Total Alkalinity

11.2.2.5 The carbonate alkalinity is calculated as follows:

$$Carbonate = 0.94 \otimes AlkBicarb \otimes 10^{(pH-10)}$$

Where:
AlkBicarb = bicarbonate alkalinity

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11.2.2.6 The hydroxide alkalinity is calculated as follows:

$$Hydroxide = 5 \otimes 10^{(pH-10)}$$

11.2.2.7 The free carbon dioxide is calculated as follows:

$$FreeCarbonDioxide = 2 \otimes AlkBicarb \otimes 10^{(6-pH)}$$

Where:
AlkBicarb = bicarbonate alkalinity

11.2.2.8 The total carbon dioxide is calculated as follows:

$$CarbonDioxide = FCO_2 + (0.44 \otimes (2 \otimes AlkBicarb + AlkCarb))$$

Where:
FCO_2 = free carbon dioxide
AlkBicarb = bicarbonate alkalinity
AlkCarb = carbonate alkalinity

11.2.3 pH

11.2.3.1 The calculation to determine final concentration is given as follows:

$$pH = -1(\log[H^+])$$

$$\frac{pH}{-1} = \log[H^+]$$

$$anitlog\left(\frac{pH}{-1}\right) = [H^+]$$

pH is a logarithmic function; that is, an increase or decrease of one pH unit represents a ten-fold change in hydrogen ion concentration. The following table demonstrates the relationship between pH and hydrogen ion concentration:

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pH	Hydrogen Ion Concentration
1.0	0.10 = (1×10^{-1})
2.0	0.010 = (1×10^{-2})
3.0	0.0010 = (1×10^{-3})
4.0	0.00010 = (1×10^{-4})
5.0	0.000010 = (1×10^{-5})
6.0	0.0000010 = (1×10^{-6})
7.0	0.00000010 = (1×10^{-7})
8.0	0.000000010 = (1×10^{-8})
9.0	0.0000000010 = (1×10^{-9})
10.0	0.00000000010 = (1×10^{-10})
11.0	0.000000000010 = (1×10^{-11})
12.0	0.0000000000010 = (1×10^{-12})
13.0	0.00000000000010 = (1×10^{-13})
14.0	0.000000000000010 = (1×10^{-14})

11.2.4 Acidity

11.2.4.1 If H_2O_2 pretreatment is not required, the acidity, as calcium carbonate, is calculated as follows:

$$Acidity(mg/L) = \frac{Vb}{Vs} \otimes Nb \otimes \frac{50mg}{meq} \otimes \frac{1000mL}{L}$$

Where:

Vb = volume of NaOH titrant used (mL)
Vs = volume of sample used (mL)
Nb = normality of the NaOH titrant (meq/mL)

11.2.4.2 If pretreated with H_2O_2 and H_2SO_4 (Section 10.1.1.1.1), the acidity, as calcium carbonate, is calculated as follows:

$$Acidity(mg/L) = \frac{(A \otimes B) - (C \otimes D)}{Vs} \otimes \frac{50mg}{meq} \otimes \frac{1000mL}{L}$$

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Where:

A = volume of NaOH titrant used (mL)
B = normality of the NaOH (meq/mL)
C = volume of H2SO4 used to adjust pH <4.0 (mL)
D = normality of H2SO4 (meq/mL)
Vs = volume of sample used (mL)

If the result is negative, the acidity is reported as less than the RL. A negative acidity indicates that the sample should be tested for alkalinity.

11.2.4.3 Conductivity – The instrument has automatic temperature compensation and the readout can be used directly as μmho/cm.

11.2.4.4 Resistivity – Resistivity is the reciprocal of conductivity.

Resistivity(uohm-cm) = 1/Conductivity(umho/cm)

11.2.4.5 Salinity –The instrument has a direct readout for salinity.

12.0 Method Performance

12.1 Reporting Limit Verification (RLV)

At a minimum, RLVs must be performed initially upon method set-up in accordance with SOP SA-QA-07: *Determination and Verification of Detection and Reporting Limits.*

For analytes and methods certified by DOD ELAP, RLVs must also be performed quarterly thereafter. For all other analytes and methods, RLVs must also be performed annually thereafter. Exceptions may be made for project-specific non-routine analytes.

12.2 Method Detection Limit Study (MDL)

Method detection limits as generated in accordance with 40CFR Part 136 Appendix B are not applicable to this procedure. The reporting limit for these analyses is based on the lowest discernable value achievable by the instrument/labware.

12.3 QC Limit Generation, Control Charting, and Trend Analysis

The control limits for the batch QC items (e.g., LCS, MS/MSD, SD) for this procedure are specified in the reference method and cannot be broadened; therefore, the laboratory defaults to the method-defined limits and does not utilize in-house or laboratory-derived limits for the evaluation of batch QC items.

Although the laboratory must default to the method-defined QC limits, control charting is a useful tool and is performed to assess analyte recoveries over time to evaluate trends. Control charting must be performed periodically (at a minimum annually) in accordance with SOP SA-QA-17: *Evaluation of Batch QC Data.*

12.4 Demonstrations of Capability

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Initial and continuing demonstration of capability must be performed in accordance with SOP SA-QA-06*: Training Procedures.*

Prior to performing this procedure unsupervised, each new analyst who performs this analysis must demonstrate proficiency per method/analyte combination by successful completion of an initial demonstration of capability. The IDOC is performed by the analysis of 4 consecutive LCSs that meet the method criteria for accuracy and precision. The IDOC must be documented and routed to the QA Department for filing.

Annual continuing demonstrations of capability (CDOCs) are also required per analyst per method/analyte combination. The CDOC requirement may be met by the consecutive analysis of four LCS all in the same batch, by the analysis of four LCS analyzed in four consecutive batches (in different batches on different days), via acceptable results on a PT study, or analysis of client samples with statistically indistinguishable results when compared to another certified analyst. The CDOC must be documented and routed to the QA Department for filing.

12.5 Training Requirements

All training must be performed and documented in accordance with SOP SA-QA-06: *Training Procedures.*

Note: The SOPs listed in the Reference/Cross-Reference Section are applicable to this procedure. All employees performing this procedure must also be trained on these SOPs, and/or have a general understanding of these procedures, as applicable.

13.0 Pollution Control

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (e.g., examining recycling options, ordering chemicals based on quantity needed, preparing reagents based on anticipated usage and reagent stability, etc.). Employees must abide by the policies in Section 13 of the Environmental Health and Safety Manual and the Savannah Addendum to the EHSM.

This procedure has been evaluated for opportunities to minimize the waste generated. Where reasonably feasible, pollution control procedures have been incorporated.

14.0 Waste Management

Waste management practices must be conducted consistent with all applicable federal, state, and local rules and regulations. All waste (i.e., excess reagents, samples, and method process wastes) must be disposed of in accordance with Section 9 of the TestAmerica Savannah Addendum to the EHSM. Waste description rules and land disposal restrictions must be followed.

14.1 Waste Streams Produced by the Method

The following waste streams are produced when this method is carried out:

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- Excess samples, reagents, and standards must be disposed in accordance with the TestAmerica Savannah Addendum to the EHSM.

- Excess aqueous samples and acidic sample extracts – Dispose according to characterization on the sample disposal sheets. Neutralize non-hazardous samples before disposal into drain/sewer. Transfer hazardous samples (identified on disposal sheets) to the waste department for disposal.

- Excess soil and solid samples – Dispose according to characterization on sample disposal sheets. Transfer non-hazardous samples to TCLP container for characterization in hazardous waste department. Transfer hazardous samples (identified on disposal sheets) to waste department for disposal.

- Titration residue – Neutralize and dispose into the drain/sewer.

15.0 References / Cross-References

- SOP SA-AN-041: *Reagent and Standard Materials Procedures*
- SOP SA-AN-100: *Laboratory Support Equipment (Verification and Use)*
- SOP SA-QA-02: *Data Generation and Review*
- SOP SA-QA-05: *Preventive and Corrective Action Procedures*
- SOP SA-QA-06: *Training Procedures*
- SOP SA-QA-07: *Determination and Verification of Detection and Reporting Limits*
- SOP SA-QA-15: *Homogenization, Compositing, and Segregation of Samples*
- SOP SA-QA-16: *Evaluation of Calibration Curves*
- SOP SA-QA-17: *Evaluation of Batch QC Data*
- TestAmerica Savannah Quality Assurance Manual
- TestAmerica Environmental Health and Safety Manual (CW-E-M-001)
- TestAmerica Savannah Addendum to the Environmental Health and Safety Manual
- *PC –Titrate Software Manual,* Man-Tech Associates, Guelph, Ontario, Canada, Version 3
- *Methods for Chemical Analysis of Water and Wastes*; U.S. EPA Office of Research and Development: Cincinnati, OH, March, 1983.
    - Method 120.1 Conductance (Specific Conductance, umhos at 25°C); 1982
    - Method 150.1 pH (Electrometric); 1982
    - Method 305.1 Acidity (Titrimetric); 1974
    - Method 310.1 Alkalinity (Titrimetric); 1978
- *Standard Methods for the Examination of Water and Wastewater, Online Edition*; American Public Health Association; Washington, D.C.
    - Method 2310B Acidity, Titration Method; 1997
    - Method 2320B Alkalinity, Titration Method; 1997
    - Method 2510B Conductivity, Laboratory Method; 1997
    - Method 2520B Salinity, Electrical Conductivity Method; 2000
    - Method 4500-H^+ B pH Value, Electrometric Method; 2000
- *Methods for the Evaluating of Solid Waste, Third Edition, SW-846*; USEPA Office of Solids Waste and Emergency Response: Washington, D.C.
    - Method 9040B pH Electrometric Measurement; 1995
    - Method 9040C pH Electrometric Measurement; 2005
    - Method 9050A Revision 1 Specific Conductance; December 1996

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16.0 Method Modifications and Clarifications

16.1 The reference method was written specifically for drinking water and source water samples; however, the laboratory may perform other types of water samples using this procedure.

16.2 This procedure may be modified to analyze other matrices (e.g., wipe samples) based on the needs of the client. This will need to be arranged by the Project Manager at the initiation of the project.

Waste and tissue matrices are non-routine, and the laboratory is not currently NELAC certified for these matrices. The laboratory uses its routine soil RLs and MDLs (converted for initial and final volumes, etc.), and soil QC limits to evaluate waste and tissue samples. Soil DOCs can be used to satisfy analyst demonstrations of capability for these types of non-routine matrices. Ottawa sand is used as the blank matrix for tissue samples unless a "true" tissue matrix is required by the project.

16.4 The EPA Manual for the Certification of Laboratories Analyzing Drinking Water requires a LFB at the MRL to be performed each day. The laboratory meets this requirement by preparing an LCS at the RL in each batch of drinking water samples. The EPA DW Manual does not specify criteria for the low-level LCS; therefore, the laboratory defaults to 50-150%.

16.5 The holding time given in the methods EPA 150.1 and SM 4500H+ B is listed as "immediately". Table II of 40CFR Part 136 defines a 15 minute holding time from collection to analysis for these methods. The 15 minute holding time has been adopted by the laboratory for these methods, and the HF-flag is used to qualify results outside the holding time for these methods.

16.6 The holding time listed in the methods for SW-846 9040B and 9040C is "as soon as possible". Following guidance from MICE (Methods Information Communication Exchange Service), the laboratory has adopted a 24-hour holding time for aqueous samples (SW-846 9040B and 9040C). The H-flag is used to qualify results outside holding time for these methods.

16.7 The sample preparation procedure has been modified from the guidance in EPA Method 305.1 to match Standard Methods 2310B. EPA Method 305.1 specifies that all samples with a pH greater than 4 be treated as ferrous/ferric iron containing samples.

16.8 The holding time for EPA Method 120.1 has been changed from 24 hours to the 28 days as allowed by SW-846 Method 9050A and in accordance with Table II of 40CFR Part 136.

16.9 Method SM2510B does not contain a specific holding time requirement. The laboratory has defaulted to a 28 day holding time as allowed by SW-846 Method 9050A for this test.

17.0 Attachments

The following Tables, Diagrams, and/or Validation Data are included as Attachments:

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Attachment 1: SOP Summary
Attachment 2: Sample Collection, Preservation, and Holding Time Table
Attachment 3: QC Summary
Attachment 4: Instrument Maintenance and Troubleshooting
Attachment 5: pH Meter Calibration and Use
Attachment 6: Maximum Contaminant Level (MCL) Table

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Attachment 1:
SOP Summary

Sample Preparation and Analysis Summary

An appropriate volume of sample or sample extract is introduced into the PC Titrate Auto-analyzer.

Alkalinity Determination

Total alkalinity, as calcium carbonate, is determined in water samples by titrating the sample to an endpoint of pH 4.5 with standardized acid. Phenolphthalein alkalinity is determined by titrating to an endpoint of 8.3. The other carbonate species that can be reported with this procedure are calculated using the results of the sample's total alkalinity and pH.

Analytical Sequence for Alkalinity samples:

Description	Comments
Initial pH Calibration	Batch begins with the analysis of the method blank
ICV - pH	Second Source
Method Blank	
LCS – Alkalinity	0.02N H_2SO_4 std
Samples & Batch QC Items	Up to 8 analyses
CCV	Level 7.0 pH
Samples & Batch QC Items	Up to 10 analyses
CCV	Level 7.0 pH
*LCSD-Alkalinity	0.02N H_2SO_4 std

*The batch must end with a bracketing LCSD

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pH Determination

The pH of liquid samples is determined potentiometrically using a pH electrode meter with temperature compensation.

Analytical Sequence for pH:

Description	Comments
Initial Calibration	Batch begins with the analysis of the ICV standard
ICV	Second Source
Samples & Batch QC Items	Up to 10 analyses, excluding the CCV
CCV	Level 7.0 pH
Samples & Batch QC Items	Up to 10 analyses, excluding the CCV
CCV	Level 7.0 pH

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Acidity Determination

The pH of the sample aliquot is determined. The sample is titrated to a pH of 8.2 using standardized NaOH. Titration to pH 8.2 reflects the bicarbonate equivalence point. The 8.2 endpoint is chosen because many waters contain naturally occurring bicarbonate salts. Determination of acidity by titrating to this endpoint makes acidity independent of any bicarbonate salts that may have been present in the original sample. This bicarbonate-independent acidity is often termed the CO_2-acidity, since the dissolved CO_2 and other gases are measured.

Samples which are known or suspected to contain ferrous or ferric iron may be treated with hydrogen peroxide prior to titration, to ensure a true mineral acidity is obtained. The pH of the sample aliquot is determined. Commercially prepared 0.02N H_2SO_4 is added in to achieve a pH of 4 or less. An aliquot of H_2O_2 is added to the solution, which is boiled for two minutes, then cooled to room temperature. The sample is then titrated using standardized NaOH to a pH of 8.2.

Analytical Sequence for Acidity samples:

Description	Comments
Initial pH Calibration	Batch begins with the analysis of the method blank
ICV - pH	Second Source
Method Blank	
LCS - Acidity	0.02N H_2SO_4 std
Samples & Batch QC Items	Up to 10 analyses
CCV	Level 7.0 pH
Samples & Batch QC Items	Up to 10 analyses
CCV	Level 7.0 pH
*LCSD-Acidity	0.02N H_2SO_4 std

*The batch must end with a bracketing LCSD

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Conductivity and Salinity Determination

To measure conductivity of a sample approximately 45mL of the sample is poured into the sample analysis container. The conductivity probe is submerged in the sample and the signal is allowed to stabilize. It is very important that sufficient sample is poured up so that the conductivity plates on the probe are completely covered by the sample. If they are not completely covered then erroneous results will be obtained.

To measure salinity of a sample approximately 45mL of the sample is poured into the sample analysis container. The conductivity probe is submerged in the sample and the signal is allowed to stabilize. It is very important that sufficient sample is poured up so that the conductivity plates on the probe are completely covered by the sample. If they are not completely covered then erroneous results will be obtained.

Analytical Sequence for Conductivity and Salinity samples:

Description	Comments
1413 Conductivity Cal Check	Batch begins with the analysis of the method blank
Salinity Cal Std	
DI rinse	
Conductivity RL Std	5.0 ppm Std
Method Blank	
LCS – Cond	
LCS – Salinity	
Samples & Batch QC Items	Up to 6 analyses
CCV - Cond	Same standard as LCS may be used
CCB	
Samples & Batch QC Items	Up to 10 analyses
CCV - Cond	Same standard as LCS may be used
CCB	
*LCSD - Salinity	

*The sequence continues for up to 20 field samples and must be bracketed with a CCV/CCB for conductivity and the LCSD for salinity.

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Attachment 2:
Sample Collection, Preservation, and Holding Time Table

Listed below are the holding times and preservation requirements:

Matrix	Analyte	Sample Container	Minimum Sample Size	Chemical Preservation	Thermal Preservation	Dechlorination Agent	Holding Time[2]
Water	Alkalinity	250mL plastic	125mL	None	4°C[1]	None	14 days
Water	Acidity	250mL plastic	125mL	None	4°C[1]	None	14 days
Water	Conductivity	125mL plastic	125mL	None	4°C[1]	None	28 days
Water	pH	125mL plastic	50mL	None	4°C[1]	None	15 minutes
Water	pH	125mL plastic	50mL	None	4°C[1]	None	24 hours

[1] Less than 6°C with no frozen samples.
[2] Inclusive of preparation and analysis.

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Attachment 3:
QC Summary

Acidity, Alkalinity, and pH

QC Item	Frequency	Criteria	Corrective Action
Initial Calibration (ICAL) Multi-point: Min 3 standards	pH and Alkalinity – Daily	pH, Acidity, and Alkalinity: each calibration buffer's reading must be within 0.05 pH units from the actual value.	Recalibrate
2nd Source Initial Calibration Verification (ICV)	After each ICAL	pH, Acidity, and Alkalinity: within 0.1 pH units	Recalibrate
Continuing Calibration Verification (CCV)	At the beginning and end of the analysis, and every 10 samples	pH, Acidity, and Alkalinity: within 0.1 pH units	Terminate the analysis, correct problem, and reanalyze the previous 10 samples.
Batch Definition	No more than 20 field samples prepared together w/in 24-hour time period	Not Applicable	Not Applicable
Method Blank (MB)	One per batch	<RL	Re-extract (if necessary) and reanalyze batch
Laboratory Control Sample (LCS)	One per batch	MLG %R	Re-extract (if necessary) and reanalyze batch
Laboratory Control Sample Duplicate (LCSD)	Acidity and Alkalinity Only: One per batch All Other Analytes: One per batch when insufficient sample	MLG %R & %RPD	Reanalyze batch

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QC Item	Frequency	Criteria	Corrective Action
	provided for SD/MS/MSD		
Sample Duplicate (SD)	pH, Acidity, and Alkalinity: One per 10% of samples (i.e., 1 SD for a batch of <10 samples; 2 SD for a batch >10 samples)	MLG %RPD	Flag and report data
Initial Demonstration of Capability (IDOC)	Initially, per analyst, per method/analyte/matrix combination	Within the method specified accuracy and precision criteria	Re-perform IDOC (Unsupervised performance may not occur until successful IDOC is obtained.)
Continuing Demonstration of Capability (CDOC)	Annually, per analyst, per method/analyte/matrix combination	Within the method specified accuracy and precision criteria	Re-perform CDOC. Re-training, as applicable.
Reporting Limit Verification	Upon method/instrument set-up, per analyte/method/matrix combination. Then quarterly thereafter (for DOD ELAP) or annually thereafter (for non-DOD ELAP)	Refer to SOP SA-QA-07	Refer to SOP SA-QA-07

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Conductivity / Salinity

QC Item	Frequency	Criteria	Corrective Action
Initial Calibration (ICAL)	Annually: Minimum 4 standards and 1 blank Daily: Single point (1413umhos/cm) Daily: Single point (35 Practical Salinity Value)	Each std +/-10% of TV 90-110% 90-110%	Recalibrate
2nd Source Initial Calibration Verification (ICV)	After each ICAL	Within ±10%	Recalibrate
Reporting Limit Verification (RLV)	At the beginning of an analysis run	Within ±50%	Recalibrate
Continuing Calibration Verification (CCV)	At the beginning and end of the analysis, and every 10 samples	Within ±10%	Terminate the analysis, correct problem, and reanalyze the previous 10 samples.
Initial and Continuing Calibration Blank (ICB/CCB)	After ICV and every CCV	<RL	Terminate the analysis, correct problem and reanalyze the previous 10 samples
Batch Definition	No more than 20 field samples prepared together w/in 24-hour time period	Not Applicable	Not Applicable
Method Blank (MB)	One per batch	<RL	Re-extract (if necessary) and reanalyze batch
Laboratory Control Sample (LCS)	One per batch	MLG	Re-extract (if necessary) and reanalyze batch

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Laboratory Control Sample Duplicate (LCSD)	One per batch when insufficient sample provided for SD	MLG	Reanalyze batch
Sample Duplicate (SD)	One per 10% of samples (i.e., 1 SD for a batch of <10 samples; 2 SD for a batch >10 samples)	MLG	Flag and report data
Initial Demonstration of Capability (IDOC)	Initially, per analyst, per method/analyte/matrix combination	Within the method specified accuracy and precision criteria	Re-perform IDOC (Unsupervised performance may not occur until successful IDOC is obtained.)
Continuing Demonstration of Capability (CDOC)	Annually, per analyst, per method/analyte/matrix combination	Within the method specified accuracy and precision criteria	Re-perform CDOC. Re-training, as applicable.

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Attachment 4:
Instrument Maintenance and Troubleshooting

Instrument Labeling
Each instrument must be labeled with its name or ID (e.g., MSA, ICP-D, etc.). Additionally, non-operational instruments must be isolated from service or marked as being out of service. Each piece of equipment has an "Operational / Not Operational" sticker that is used for this purpose.

Maintenance Log
A maintenance log must be established for each piece of equipment used in the laboratory. All maintenance that is performed on the instrument must be recorded in the log including:

- analyst or technician performing the maintenance
- date the maintenance was performed
- detailed explanation of the reason for the maintenance
- resolution of the problem and return to control
- all service calls from instrument representatives

Preventive Maintenance
Refer to the instrument manufacturer's guides for trouble-shooting items.

Care should be taken to maintain the hotplate(s) so that the temperatures can be regulated efficiently.

Contingency Plan
Maintenance contracts are carried for most instrumentation and close contact is maintained with service personnel to ensure optimal instrument functioning. An extensive spare parts inventory is maintained for routine repairs, consisting of tubing and other common instrumentation components. Since instrumentation is standardized throughout the laboratory network, spare parts and components can be readily exchanged among the network.

In general, the laboratory has at least one backup unit for each critical unit. In the event of instrument failure, portions of the sample load may be diverted to duplicate instrumentation, the analytical technique switched to an alternate approved technique (such as manual colorimetric determination as opposed to automated colorimetric determination), or samples shipped to another properly certified or approved TestAmerica location.

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Attachment 5:
pH Meter Calibration and Use

pH Meter Calibration

Instrument calibration is performed by analyzing a series of known standards. The calibration standards define the working range of the analytical system. The pH meter must be calibrated with 3 buffers on each day of use, pH 4 to pH 10.

Make sure that the pH meter has warmed up and that pH is the selected mode of operation. The lot numbers of the pH buffers must be recorded on the pH data sheet. Calibrate the pH meter according to the manufacturer's instructions.

Calibrate the meter using pH 4.0, 7.0, and 10 buffer solutions.

Note: The electrode should be immersed in the buffer but should not come in contact with the Teflon-coated stir bar. Allow the solution to stir slowly while the measurement is being made.

Tabulate the concentrations and corresponding responses for each analyte. Compare the meter's reading of each buffer's pH to the actual value. For the curve to be acceptable, each calibration buffer's reading must be within 0.05 pH units from the actual value.

> Note: Pour fresh pH buffer solution from the stock bottle each day. Do not reuse over multiple days.

Sample Analysis

Remove the samples or extracts from the refrigerator and allow them to come to room temperature.

Place an aliquot of the well-mixed sample into a beaker on the magnetic stir plate. Make sure that the electrode is immersed in the sample but does not contact the Teflon-coated stir bar.

Allow the sample to slowly stir while the pH measurement is being made. Record the pH to the nearest 0.01 pH unit after the reading has stabilized.

After all measurements have been made, return the electrode to the 7 buffer solution or electrode storage solution and set the meter to "standby." The meter should be left "on" when not in use.

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Attachment 6:
Maximum Contaminant Level (MCL) Table

Secondary Drinking Water Regulations	
Contaminant	**MCL**
pH	6.5 - 8.5 SU

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18.0 Revision History

Summary of Changes from Previous Revision:

- Minor editorial, grammatical, and/or formatting changes made.
- Removed reference to fluoride by EPA 340.2 and SM 4500^{-} C. These methods are no longer performed by the laboratory. (Soil matrices, dilution procedures, and MDLs were specific to these methods, so reference to these items has been removed as well.)
- Removed reference to G-drive and replaced with Public_QA drive. Section 7.3 and Section 10.3.1
- Added note that COAs can be attached to the TALS AD batch. Section 7.4

UNCONTROLLED

TOTAL HARDNESS as $CaCO_3$ by TITRIMETRIC EDTA

(Methods: EPA 130.2 & SM2340C)

Approvals (Signature/Date):

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1.0 Scope and Application

This SOP gives the procedures for the determination of hardness by titration.

The routine matrix performed by the laboratory is a water matrix. With Technical Management approval, this procedure can be modified to incorporate other matrices. Soil samples are non-routine, and, therefore, are not included in this SOP.

The reporting limits (RL) and the accuracy and precision criteria associated with this procedure are provided in the LIMS Method Limit Groups (MLGs).

This SOP was written by and for TestAmerica's Savannah laboratory.

2.0 Summary of Method

Calcium and magnesium ions present in a sample are chelated into a soluble complex when a solution of disodium ethylenediamine tetraacetic acid (Na_2EDTA) is added. Eriochrome Black T, which turns a red-wine color in the presence of calcium and magnesium ions, is used as the indicator of the titration endpoint. When all of the calcium and magnesium ions have been complexed, and there is an excess of cations (predominantly Na and Mg), the Eriochrome Black T turns blue signaling the endpoint of the titration.

This SOP is based on the following methods: EPA Method 130.2 and Standard Methods 2340C.

3.0 Definitions

Refer to the Glossary Section of the *Quality Assurance Manual* (QAM) for a complete listing of applicable definitions and acronyms.

Chelation – a chemical reaction that results in a bond being formed between a metal ion and an organic molecule. The resulting complex is called a chelate and contains the metal ion firmly bound with the organic molecule to form a ring structure.

Titration – a method of analysis that determines the precise endpoint of a reaction and the precise quantity of reactant in the titration flask. A burette is routinely used to deliver the second reactant to the flask. An indicator is used to detect the endpoint of the reaction.

4.0 Interferences

4.1 Procedural Interferences

4.1.1 Interferences may be caused by contaminants in solvents, reagents, glassware, and other sample processing apparatus and can make identification and/or quantification of the target analytes difficult.

4.1.2 All sample collection containers are single-use disposable containers which limits the potential for contamination. All non-disposable labware must be scrupulously cleaned in accordance with the posted Labware Cleaning Instructions to ensure it is free from contaminants and does not contribute artifacts.

4.1.3 High purity reagents and solvents are used to help minimize interference problems. Hydrochloric acid must be verified prior to use in accordance with the TestAmerica Solvent Lot Testing Program.

4.1.4 Reagent and/or method blanks are routinely used to demonstrate all reagents and apparatus are free from interferences under the conditions of the analysis.

4.2 Matrix Interferences

4.2.1 Matrix interferences may be caused by contaminants that are co-extracted from the sample matrix.

4.2.2 Interfering contamination may occur when a sample containing low concentrations of analytes is analyzed immediately following a sample containing relatively high concentrations of analytes. As such, samples known to be clean should be analyzed first. To prevent carryover into subsequent samples, analysis of reagent blanks may be needed after the analysis of a sample containing high concentrations of analytes.

4.2.3 Large concentrations of metal ions can interfere by fading the indicator, or making the endpoint indistinct. Inhibitors can be used to decrease the effects of metal ions on the titration. Refer to EPA Method 130.2 or Standard Methods 2340C for determining which inhibitors apply to the sample matrix being titrated.

4.2.4 Suspended matter may interfere with the endpoint. Eliminate this interference by evaporating the 25mL of the sample to dryness in a 550°C muffle oven until all organic matter has been oxidized. Dissolve the residue remaining in 20mL of 1N HCl. Neutralize to pH 7 with 1N NaOH. Dilute to 25mL with reagent water. Cool to room temperature and titrate.

5.0 Safety

Employees must abide by the policies and procedures in the TestAmerica Environmental Health and Safety Manual (EHSM), the TestAmerica Savannah Addendum to the EHSM, and this document.

This procedure may involve hazardous materials, 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 to follow appropriate safety, waste disposal, and health practices under the assumption that all samples and reagents are potentially hazardous.

The analyst must protect himself/herself from exposure to the sample matrix. Many of the samples that are tested may contain hazardous chemical compounds or biological organisms. The analyst must, at a minimum, wear protective clothing (lab coat), eye protection (safety glasses or face shield), disposable latex or nitrile gloves, and closed-toe, nonabsorbent shoes when handling samples.

5.1 Specific Safety Concerns or Requirements

All samples must be treated as if they are hazardous. The analyst must protect himself/herself from exposure to the sample matrix. Many of the samples that are tested may contain hazardous chemical compounds or biological organisms. The analyst must wear protective clothing (lab coat), eye protection (glasses or face shield), and disposable latex or nitrile gloves when handling samples.

The toxicity or carcinogenicity of chemicals used in this method has not been precisely defined. Each chemical must be treated as a potential health hazard, and exposure to these chemicals must be minimized.

Caution must be taken when handling ammonium hydroxide (NH_4OH). NH_4OH gives off strong, suffocating odors, which will burn the nose and throat. Handling of this reagent must be done under a properly functioning fume hood. Protective equipment must be utilized at all times.

Hydrochloric acid is extremely hazardous as an oxidizer, a corrosive, a poison, and is reactive. Inhalation of the vapors can cause coughing; choking; irritation of the nose, throat, and respiratory tract; breathing difficulties; and lead to pneumonia and pulmonary edema. Contact with the skin can cause severe burns, redness, and pain. Acid vapors are irritating and can cause damage to the eyes. Contact with the eyes can cause permanent damage.

5.2 Primary Materials Used

The following is a list of the materials used in this procedure, which have a serious or significant hazard rating, and a summary of the primary hazards listed in their MSDS.

Note: This list does not include all materials used in the procedure. A complete list of materials used in this procedure can be found in the Reagents and Standards Section and the Equipment and Supplies Section of this SOP

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 found using the "MSDS" link on the Oasis homepage, on the EH&S webpage on Oasis, and on the QA Navigator.

Material	Hazards	Exposure Limit[1]	Signs and Symptoms of Exposure
Ammonium Hydroxide	Corrosive Poison	50ppm TWA	Vapors and mists cause irritation to the respiratory tract. Causes irritation and burns to the skin and eyes.
Hydrochloric Acid	Corrosive Poison	5ppm 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]Exposure limit refers to the OSHA regulatory exposure limit.			
Note: Always add acid to water to prevent violent reactions.			

6.0 Equipment and Supplies

6.1 Equipment and Instrumentation

pH Meter – Calibrate each day of use in accordance with Attachment 5.

Analytical Balance – Verify in accordance with SOP SA-AN-100: *Laboratory Support Equipment (Verification and Use)*

Thermometers – Verify in accordance with SOP SA-AN-100: *Laboratory Support Equipment (Verification and Use)*

Burette – with 0.02mL increments. Verify in accordance with SOP SA-AN-100: *Laboratory Support Equipment (Verification and Use)*

6.2 Lab Supplies

Volumetric Containers – various sizes; Class A, where applicable. Verify in accordance with SOP SA-AN-100: *Laboratory Support Equipment (Verification and Use)*

Disposable Graduated Pipettes – various sizes. Verify in accordance with SOP SA-AN-100: *Laboratory Support Equipment (Verification and Use)*

pH paper

Magnetic Stir Plate and Stir Bars

Hot Plate

Dessicator and Dessicant

Detergent – Alconox or equivalent, used for washing non-disposable labware.

6.3 Sample Collection Containers

All sample collection containers are single-use disposable containers which limits the potential for contamination.

The routine sample collection containers supplied by the laboratory are:

500mL plastic – purchased with Certificate of Analysis attesting to purity.

7.0 Reagents and Standards

7.1 Expiration Dates

Expiration dates (time from initial use or receipt to final use) for standard and reagent materials must be set according to the guidance in this SOP. Note: These are maximum expiration dates and are not to be considered an absolute guarantee of standard or reagent quality. Sound judgment must be used when deciding whether to use a standard or reagent. If there is doubt about the quality of a standard or reagent material, a new material must be obtained or the standard or reagent material verified. Data quality must not be compromised to extend a standard's life – i.e., when in doubt, throw it out.

The expiration date of any standard or reagent must not exceed the expiration date of the standard or reagent that was used to prepare it; that is, the "children may not outlive the parents".

7.2 Reagents

Reagents must be prepared and documented in accordance with SOP SA-AN-41: *Reagent and Standard Materials Procedures.*

Hydrochloric acid must be verified prior to use in accordance with the TestAmerica Solvent Lot Testing Program.

7.2.1 Purchased Reagents

7.2.1.1 Laboratory Reagent Water – ASTM Type II

7.2.1.2 Disodium ethylenediamine tetraacetic acid dihydrate (Na_2EDTA) – reagent grade.
LIMS Name: EDTA
Storage: Store in tightly closed container at room temperature, away for incompatible substances
Expiration:
Unopened: Manufacturer's expiration or 5 years
Opened: Manufacturer's expiration or 5 years

7.2.1.3 Calcium Standard (10,000mg/L) – obtained from the Metals Department
LIMS Name: Caspx10000
Storage: GE Walk-in Refrigerator at <4°C
Unopened: Manufacturer's expiration or 5 years
Opened: Manufacturer's expiration or 5 years

7. 2.1.5 Ammonium Chloride (NH_4Cl) – reagent grade.

LIMS Name: NH4Clsalt
Storage: Store in a tightly closed container, in a cool, dry ventilated area away from sources of heat, moisture, and incompatibles.
Expiration:
Unopened: Manufacturer's expiration or 5 years
Opened: Manufacturer's expiration or 5 years

7. 2.1.6 Ammonium Hydroxide (NH_4OH) – reagent grade.
LIMS Name: NH4OH
Storage: Store in a tightly closed container, in a cool, dry ventilated area. Protect from heat, direct sunlight, and incompatibles.
Expiration:
Unopened: Manufacturer's expiration or 5 years
Opened: Manufacturer's expiration or 5 years

7. 2.1.7 Hydrochloric Acid (HCl) – reagent grade. Stable under ordinary conditions of use and storage.
LIMS Name: HCl
Storage: Store in a cool, dry, ventilated storage area with acid resistant floors and good drainage. Store away from sunlight, heat, water, and incompatible materials.
Expiration:
Unopened: Manufacturer's expiration or 5 years
Opened: Manufacturer's expiration or 5 years

7. 2.1.8 Nitric acid (HNO_3): reagent grade. Stable under ordinary conditions of use and storage.
LIMS Name:HNO3Conc
Storage: Store in a cool, dry, ventilated storage area with acid resistant floors and good drainage. Store away from sunlight, heat, water, and incompatible materials.
Expiration:
Unopened: Manufacturer's expiration date
Opened: 5 years from date of opening

7. 2.1.9 Buffer Solution – Buffer Solution Hardness 1 (pH 10.1 $\pm$ 0.1), purchased from HACH.
LIMS Name: HardBuff
Storage: Store in a tightly closed plastic container. Protect from light.
Expiration:
Unopened: Manufacturer's expiration date
Opened: Manufacturer's expiration date or within 1 year of opening

7.2.1.10 Eriochrome Black T indicator solution – commercially prepared.
LIMS Name:BlkTSol
Storage: room temperature
Expiration:
Unopened: Manufacturer's expiration date
Opened: Manufacturer's expiration date or within 1 year of opening

7. 2.1.11 Methyl Red Indicator – commercially prepared. Store at room temperature.
LIMS Name: MethylRed
Storage: Store in a tightly closed container away from heat and sources of ignition. Store in a cool, dry, well ventilated area, away from incompatibles.

Expiration:
Unopened: Manufacturer's expiration date
Opened: Manufacturer's expiration date or within 1 year of opening

7. 2.1.12 Calcium Carbonate ($CaCO_3$) – reagent grade. Dried at 105°C for at least two hours.
LIMS Name:CaCO3
Storage: room temperature in a desiccator
Expiration:
Unopened: Manufacturer's expiration date
Opened: Manufacturer's expiration date or within 5 year of opening

7.2.2 Prepared Reagents

7. 2.2.1 Hydrochloric Acid (1:1) – Under a ventilation hood, place 250mL reagent water in a glass beaker. Add a stir bar and place on a magnetic stirplate. While stirring, SLOWLY add 250mL of concentrated HCl. After the solution cools, transfer to a glass or plastic storage container.
LIMS Name: 1:1HCl
Storage: Store in a cool, dry, ventilated storage area with acid resistant floors and good drainage. Store away from sunlight, heat, water, and incompatible materials.
Expiration: 1 year from preparation date

CAUTION: Heat is generated as the acid mixes with the water. Always add acid to water. This solution is acidic and will cause skin burns, immediate eye damage, and destroy unprotected clothing.

7. 2.2.2 Nitric Acid (1:1) – Under a ventilation hood, place 250mL reagent water in a glass beaker. Add a stir bar and place on a magnetic stirplate. While stirring, SLOWLY add 250mL of concentrated HNO_3. After the solution cools, transfer to a glass or plastic storage container.
LIMS Name: 1:1HNO3
Storage: Store in a cool, dry, ventilated storage area with acid resistant floors and good drainage. Store away from sunlight, heat, water, and incompatible materials.
Expiration: 1 year from preparation date

CAUTION: Heat is generated as the acid mixes with the water. Always add acid to water. This solution is acidic and will cause skin burns, immediate eye damage, and destroy unprotected clothing.

7. 2.2.3 Ammonium Hydroxide Solution (3N) – Add 210mL of NH_4OH to a 1L volumetric flask containing approximately 500mL of reagent water. Dilute to volume with reagent water. Note: Ammonium hydroxide gives off strong, suffocating fumes. Perform this step under a hood.
LIMS Name: NH4OH(3N)
Storage: GE Walk-in Refrigerator at 4°C
Expiration: 3 months from preparation date

7. 2.2.4 Ammonium Hydroxide solution (1N) – Add 70mL of NH_4OH to a 1L volumetric flask containing approximately 500mL of reagent water. Dilute to volume with reagent water.
LIMS Name: NH4OH(1N)

Storage: GE Walk-in Refrigerator at 4°C
Expiration: 3 months from preparation date

7. 2.2.5 Standard Calcium Solution (0.02N) – Place 1.000g $CaCO_3$ in a 500mL flask. Dropwise, add 1:1 HCl solution until all of the $CaCO_3$ has dissolved. Add 200mL reagent water. Boil 5 minutes to expel the CO_2. Cool. Add a few drops of methyl red indicator and adjust to an intermediate orange color by adding 3N NH_4OH or 1:1HCl as required. Transfer to a 1L volumetric flask and dilute to volume with reagent water.
LIMS Name: CaCO3
Storage: GE Walk-in Refrigerator at 4°C
Expiration: 3 months from preparation date

7. 2.2.6 Standard EDTA titrant (0.02N) – Dissolve 3.723g Na_2EDTA in a 1L volumetric flask containing approximately 800mL of reagent water. Dilute to volume with reagent water. Standardize as needed (at least weekly) against a calcium carbonate solution.
LIMS Name: EDTA(Hard)
Storage: GE Walk-in Refrigerator at 4°C
Expiration: 3 months from preparation date

7. 2.2.6.1 Standardization Procedure

7. 2.2.6.1.1 Place 10mL 0.02N Standard Calcium Solution in an Erlenmeyer flask containing about 50mL of reagent water. Add 1mL of the Buffer Solution. Add 1-2 drops of Eriochrome Black T. Titrate slowly, while stirring, until the last reddish tinge disappears. When approaching the endpoint, titrate dropwise in 3-5 second intervals, until a blue color appears. Total titration time should be less than 5 minutes from the time the buffer was added.

7. 2.2.6.1.2 Calculate the normality (N) of the EDTA titrant as follows:

$$N \quad of \quad EDTA = \frac{0.20}{ML\ EDTA}$$

7. 2.2.6.1.3 Repeat this process two more times and average the results to obtain the normality of the EDTA.

7.3 Standards

Standards must be prepared and documented in accordance with SOP SA-AN-41: *Reagent and Standard Materials Procedures.* Certificates of analysis or purity must be received with all purchased standards, and scanned and filed in the Data Archival Folder on the G-drive.

7.3.1 Calcium Carbonate Lab Control Standard (400mg $CaCO_3$/L) – Spike 1.0mL of the 10,000 mg/L Calcium Standard to 10mL of reagent water. Analyze once per batch of twenty or fewer samples. This standard is made and analyzed once per analysis batch. The preparation is noted in the LIMS analysis batch.

7.3.2 pH Meter Calibration Standards – Certified pH buffers to bracket pH range of samples. Suggested buffers are 2.0, 4.0, 7.0, 10.0, and 12.0. These buffer solutions are also used for continuing calibration verifications (CCV).
LIMS Name: 2.0Buff, 4.0Buff, 7.0Buff, 10Buff, 12Buff
Storage: solvent cabinet
Expiration: Manufacturer's expiration date or 2 years from the date opened

8.0 Sample Collection, Preservation, Shipment, and Storage

8.1 Aqueous Samples

The reference methods are inconsistent or unclear on collection, preservation, and holding time requirements. EPA 130.2 states to preserve samples to pH<2 with nitric acid and to ice after collection, and does not list a holding time requirement. SM2340C does not list a preservation or holding time requirement. The Methods Update Rule (MUR) states to preserve samples to pH<2 with nitric acid and specifies a holding time of 6 months.

The laboratory defaults to the following:
Aqueous samples are routinely collected in 500mL plastic containers containing 3.0mL 1:1 nitric acid preservative. The preservative should be sufficient to achieve a sample pH of less than 2.

Samples must be iced at the time of collection and maintained at 4°C (less than 6°C but not frozen) until the time of analysis. Samples must be analyzed within 6 months of collection.

NCMs must be initiated for samples collected in improper containers and containing improper or insufficient preservatives.

8.1.1 Preservation Checks – pH Verification

For each sample,
- Place a piece of pH paper in a disposable medicine cup.
- Pour a few drops of sample into the medicine cup and note the color change of the pH paper.
- If the pH is not less than 2, initiate a Nonconformance Memo. Adjust the sample pH to less than 2 using 1:1 nitric acid.

> Note: If samples are received unpreserved, they are acidified with 1:1 nitric acid to pH <2, and must held for 24 hours (after acidification) prior to analysis - in the same manner Metals samples are treated.

Note: To avoid cross-contamination, use a separate medicine cup and piece of pH paper per sample. Do not dip the pH paper into the sample container. The pH paper dye may bleed into the sample and affect sample results.

9.0 Quality Control

SOP SA-QA-17: *Evaluation of Batch QC Data* and the SOP Summary in Attachment 3 provide requirements for evaluating QC data.

9.1 Batch QC

An analysis batch consists of up to 20 environmental samples and the associated QC items analyzed together within a 24 hour period.

The minimum QC items required for each analysis batch are: a method blank, a laboratory control sample (LCS), and a sample duplicate to be performed on a minimum of 10% of samples or one per batch – whichever is greater.

This frequency equates to the following:

- For a batch of 10 or fewer samples, the minimum QC items are a method blank, an LCS, and 1 sample duplicate.
- For a batch of 11-20 samples, the minimum QC items are a method blank, an LCS, 1 sample duplicate (from sample 1-10), and another sample duplicate (from sample 11-20).

The routine container supplied for this method is a 500mL container. 25mL is required for analysis.

If there is insufficient sample volume to perform the required sample duplicates(s), the LCS must be prepared in duplicate (LCS/LCSD). An NCM must be initiated on all affected samples to denote this situation. Insufficient sample volume is defined as receiving less than a total of 50mL.

Batch QC must meet the criteria given in Attachment 3 of this SOP.

Note: If an LCS and LCSD are performed, both QC items must be evaluated and reported. Acceptable recoveries (as well as %RPD) for both LCS and LCSD are required.

9.2 Instrument QC

Not applicable to this procedure.

9.3 Corrective Action for Out-of-Control Data

When the quality control parameters do not meet the criteria set forth in this SOP, corrective action must be taken in accordance with SOP SA-QA-05: *Preventive and Corrective Action Procedures* and the QC Summary Table in Attachment 3. SOP SA-QA-05 provides contingencies for out-of-control data and gives guidance for exceptionally permitting departures from approved policies and procedures. Nonconformance Memos must be initiated to document all instances where QC criteria are not met and all departures from approved policies and procedures.

10.0 Procedure

10.1 Sample Preparation and Analysis

Remove the samples from the refrigerator and allow them to come to room temperature.

Note: Pretreatment of the sample is not required.

Place 25mL of sample in a beaker. Dilute to approximately 50mL with reagent water. Add a magnetic stir bar and place on a stir plate.

Add 1mL of buffer solution. Check the pH (Attachment 5). Add additional buffer solution, when necessary, to obtain a pH of 9.9-10.1.

Add 1 to 2 drops of Eriochrome Black T.

Titrate slowly with standardized EDTA titrant until the reddish tinge begins to disappear. Continue to titrate 1 drop every 3-5 seconds until the solution turns blue. Total titration time should be less than 5 minutes from the time the buffer was added.

10.2 QC Sample Preparation

10.2.1 Method Blank – Use 25mL of reagent water and prepare as a sample following the procedure outlined in Section 10.1.

10.2.2 Laboratory Control Sample – Use 10mL of Calcium Carbonate Lab Control Standard and prepare as a sample following the procedure outlined in Section 10.1.

10.2.3 Sample Duplicate – Prepare the chosen sample in duplicate.

11.0 Calculations / Data Reduction

11.1 Data Reduction

Data must be evaluated in accordance with SOP SA-QA-02: *Data Generation and Review.*

11.1.1 Historical Data

Many of the laboratory's clients submit samples for repeat monitoring purposes. Prior to analysis, verify LIMS Worksheet Notes and/or use the TALS Historical Data Tracker Feature to determine if historical data is available for review.

11.2 Calculations

11.2.1 The calculations associated with batch QC determinations are given in SOP SA-QA-17. Applicable calculations include accuracy (% recovery) and precision (%RPD).

11.2.2 The calculation to determine final concentration is given as follows:

$$Hardness, mg/L\ as\ CaCO_3 = \frac{A \otimes N \otimes 50000}{mL\ sample}$$

Where:

A = volume of EDTA titrant (mL)
N = standardized normality of EDTA
50000 = correction factor for equivalent weight
i.e.,

$$N \otimes 50000 = (meq/mL) \otimes ((50 mg CaCO_3/meq) \otimes (1000 mL/L) = mg CaCO_3/L$$

12.0 Method Performance

12.1 Reporting Limit Verification (RLV)

At a minimum, RLVs must be performed initially upon method set-up in accordance with SOP SA-QA-07: *Determination and Verification of Detection and Reporting Limits.*

For analytes and methods certified by DOD ELAP, RLVs must also be performed quarterly thereafter. For all other analytes and methods, RLVs must also be performed annually thereafter. Exceptions may be made for project-specific non-routine analytes.

12.2 Method Detection Limit Study (MDL)

Method detection limits as generated in accordance with 40CFR Part 136 Appendix B are not applicable to this procedure. The reporting limit is based on the lowest discernable value achievable by the instrument/labware.

12.3 Method Detection Limit Verification (MDLV)

MDL verifications as outlined in SOP SA-QA-07: *Determination and Verification of Detection and Reporting Limits* are not applicable to this procedure.

12.4 QC Limit Generation, Control Charting, and Trend Analysis

The control limits for the batch QC items (LCS and SD) for this procedure are specified in the reference method and cannot be broadened; therefore, the laboratory defaults to the method-defined limits and does not utilize in-house or laboratory-derived limits for the evaluation of batch QC items.

Although the laboratory must default to the method-defined QC limits, control charting is a useful tool and is performed to assess analyte recoveries over time to evaluate trends. Control charting must be performed periodically (at a minimum annually) in accordance with SOP SA-QA-17: *Evaluation of Batch QC Data.*

12.5 Demonstrations of Capability

Initial and continuing demonstration of capability must be performed in accordance with SOP SA-QA-06*: Training Procedures.*

Prior to performing this procedure unsupervised, each new analyst who performs this analysis must demonstrate proficiency per method/analyte combination by successful completion of an initial demonstration of capability. The IDOC is performed by the analysis of 4 consecutive LCSs that meet the method criteria for accuracy and precision or by obtaining acceptable single blind PT or QC results. The IDOC must be documented and routed to the QA Department for filing.

Annual continuing demonstrations of capability (CDOCs) are also required per analyst per method/analyte combination. The CDOC requirement may be met by the consecutive analysis of four LCS all in the same batch, by the analysis of four LCS analyzed in four consecutive batches (in different batches on different days), or via acceptable results on a single blind PT or QC study. The CDOC must be documented and routed to the QA Department for filing.

12.6 Training Requirements

All training must be performed and documented in accordance with SOP SA-QA-06: *Training Procedures*.

Note: The SOPs listed in the Reference/Cross-Reference Section are applicable to this procedure. All employees performing this procedure must also be trained on these SOPs, and/or have a general understanding of these procedures, as applicable.

13.0 Pollution Control

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (e.g., examining recycling options, ordering chemicals based on quantity needed, preparing reagents based on anticipated usage and reagent stability, etc.). Employees must abide by the policies in Section 13 of the Environmental Health and Safety Manual and the Savannah Addendum to the EHSM.

This procedure has been evaluated for opportunities to minimize the waste generated. Where reasonably feasible, pollution control procedures have been incorporated.

14.0 Waste Management

Waste management practices must be conducted consistent with all applicable federal, state, and local rules and regulations. All waste (i.e., excess reagents, samples, and method process wastes) must be disposed of in accordance with Section 9 of the TestAmerica Savannah Addendum to the EHSM. Waste description rules and land disposal restrictions must be followed.

14.1 Waste Streams Produced by the Method

The following waste streams are produced when this method is carried out:

- Excess aqueous samples – Dispose according to characterization on the sample disposal sheets. Neutralize non-hazardous samples before disposal into drain/sewer. Transfer hazardous samples (identified on disposal sheets) to the waste department for disposal.

15.0 References / Cross-References

- SOP SA-AN-041: *Reagent and Standard Materials Procedures*
- SOP SA-AN-100: *Laboratory Support Equipment (Verification and Use)*
- SOP SA-QA-02: *Data Generation and Review*
- SOP SA-QA-05: *Preventive and Corrective Action Procedures*
- SOP SA-QA-06: *Training Procedures*
- SOP SA-QA-07: *Determination and Verification of Detection and Reporting Limits*
- SOP SA-QA-17: *Evaluation of Batch QC Data*
- TestAmerica Savannah Quality Assurance Manual
- TestAmerica Environmental Health and Safety Manual (CW-E-M-001)
- TestAmerica Savannah Addendum to the Environmental Health and Safety Manual
- *Methods for the Chemical Analysis of Water and Wastes;* U.S. EPA Office of Research and Development, Cincinnati, OH, March, 1983.
  - EPA 130.2: *Hardness, Total (mg/L as $CaCO_3$) (Titrimetric, EDTA)*; Editorial Edition 1978, 1982
- *Standard Methods for the Examination of Water and Wastewater, Online Edition;* American Public Health Association: Washington, DC.
  - SM 2020: Quality Assurance/Quality Control; 2001
  - SM 2340C: *Hardness: EDTA Titrimetric Method*; 1997

16.0 Method Modifications and Clarifications

16.1 The reference method was written specifically for drinking water and source water samples; however, the laboratory may perform other types of water samples using this procedure. With Technical Management approval, this procedure can be modified to incorporate other matrices. Soil samples are non-routine, and, therefore, are not included in this SOP.

16.2 EPA Method 130.2 and Standard Methods 2340C require pretreatment of wastewaters and highly polluted waters using the digestion procedures specified in the Atomic Adsorption methods. The laboratory processes all samples without pretreatment.

16.3 The reference methods are inconsistent or unclear on collection, preservation, and holding time requirements. EPA 130.2 states to preserve samples to pH<2 with nitric acid and to ice after collection, and does not list a holding time requirement. SM2340C does not list a preservation or holding time requirement. The Methods Update Rule states to preserve samples to pH<2 with nitric acid and specifies a holding time of 6 months. The laboratory defaults to the requirements outlined in Section 8.1.

16.4 EPA 130.2 does not specify QC criteria or frequency; therefore, the laboratory defaults to that specified in SM2020 for both EPA 130.2 and SM2340C.

17.0 Attachments

The following Tables, Diagrams, and/or Validation Data are included as Attachments:

Attachment 1: SOP Summary
Attachment 2: Sample Collection, Preservation, and Holding Time Table
Attachment 3: QC Summary
Attachment 4: Instrument Maintenance and Troubleshooting
Attachment 5: pH Meter Calibration and Use

Attachment 1:
SOP Summary

Sample Preparation and Analysis Summary

Calcium and magnesium ions present in a sample are chelated into a soluble complex when a solution of disodium ethylenediamine tetraacetic acid (Na_2EDTA) is added. Eriochrome Black T, which turns a red-wine color in the presence of calcium and magnesium ions, is used as the indicator of the titration endpoint. When all of the calcium and magnesium ions have been complexed, and there is an excess of cations (predominantly Na and Mg), the Eriochrome Black T turns blue signaling the endpoint of the titration.

Attachment 2:
Sample Collection, Preservation, and Holding Time Table

Matrix	Routine Sample Container	Routine Sample Size	Minimum Sample Size	Chemical Preservation	Thermal Preservation	Dechlorination Agent	Holding Time
Water	500mL plastic	250mL	25mL	3mL 1:1 HNO_3	4°C[1]	None	6 months

[1]0-6°C, with no frozen samples

Attachment 3:
QC Summary

QC Item	Frequency	Criteria	Corrective Action
Batch Definition	Up to 20 field samples, analyzed together w/in 24-hr timeframe	Not Applicable	Not Applicable
Method Blank (MB)	One per batch	<RL	Refer to SOP SA-QA-17
Laboratory Control Sample (LCS)	One per batch	Within limits listed in the MLG: 75-125%R	Refer to SOP SA-QA-17
Laboratory Control Sample Duplicate (LCSD)	One per batch, when insufficient sample is provided for SD	Within limits listed in the MLG: 75-125%R <30% RSD	Refer to SOP SA-QA-17
Sample Duplicate (SD)	10% of samples analyzed; i.e., 2 separate sample duplicates per batch of twenty samples	Within limits listed in the MLG: <30% RSD	Refer to SOP SA-QA-17
Reporting Limit Verification (RLV)	Upon method set-up. Then quarterly thereafter (for DOD ELAP) or annually thereafter (for non-DOD ELAP)	Qualitatively identified	Refer to SOP SA-QA-07
Initial Demonstration of Capability (IDOC)	Initially, per analyst	Within limits listed in the MLG: 75-125%R <30% RSD	Refer to SOP SA-QA-06 (Unsupervised work may not begin until successful IDOC is obtained.)
Continuing Demonstration of Capability (CDOC)	Annually, per analyst	Within limits listed in the MLG: 75-125%R <30% RSD	Refer to SOP SA-QA-06

Attachment 4:
Instrument Maintenance and Troubleshooting

Note: This procedure is a titrimetric procedure, and, with the exception of the pH meter, does not involve any other instrumentation. The items listed below are specific to the pH meter.

Instrument Labeling

Each instrument must be labeled with its name or ID (e.g., MSA, ICP-D, etc.). Additionally, non-operational instruments must be isolated from service or marked as being out of service. Each piece of equipment has an "Operational / Not Operational" sticker that is used for this purpose.

Maintenance Log

A maintenance log must be established for each piece of equipment used in the laboratory. All maintenance that is performed on the instrument must be recorded in the log including:

- analyst or technician performing the maintenance
- date the maintenance was performed
- detailed explanation of the reason for the maintenance
- resolution of the problem and return to control
- all service calls from instrument representatives

Preventive Maintenance

Refer to the instrument manufacturer's guides for trouble-shooting items.

pH METER
PREVENTIVE MAINTENANCE SCHEDULE

EQUIPMENT / ITEM	Service Interval							SERVICE LEVEL
	D	W	M	Q	SA	A	AN	
Probe	X							Check fill level daily and clean probe as necessary

D = daily; W = Weekly; M = monthly; Q = Quarterly; SA = semi-annually; A = annually; AN = as needed

Desiccator Maintenance

The following checks must be performed daily:

- Desiccant is active
- Hygrometer is in the low humidity zone
- Door is making an air tight seal

The desiccator door must remain closed and seated whenever possible. When the desiccant turns from blue to light purple, discard desiccant, in accordance with the TestAmerica Savannah Addendum to the EHSM, and refill pan with fresh desiccant.

Troubleshooting

Troubleshooting should be documented as outlined above. If possible, troubleshooting is best performed in a step-wise manner to systematically isolate instrument components. Refer to the instrument manufacturer's guides for specific information and strategies. Enlist assistance from technical and/or department management as needed.

Contingency Plan

In general, the laboratory has at least one backup unit for each critical unit. In the event of instrument failure, portions of the sample load may be diverted to duplicate instrumentation, the analytical technique switched to an alternate approved technique (such as manual colorimetric determination as opposed to automated colorimetric determination), or samples shipped to another properly certified or approved TestAmerica location.

Spare pH meter probes are available as are other pH meters for use as backups.

Attachment 5:
pH Meter Calibration and Use

pH Meter Calibration

- Instrument calibration is performed by analyzing a series of known standards. The calibration standards define the working range of the analytical system. The pH meter must be calibrated with 3 buffers on each day of use, pH 7 to pH 12.

- Make sure that the pH meter has warmed up and that pH is the selected mode of operation. The lot numbers of the pH buffers must be recorded on the pH data sheet. Calibrate the pH meter according to the manufacturer's instructions.

- Since the pH range to be measured is 9.9 – 10.0, calibrate the meter using pH 7.0, 10.0, and 12.0 buffer solutions.

  Note: The electrode should be immersed in the buffer but should not come in contact with the Teflon-coated stir bar. Allow the solution to stir slowly while the measurement is being made.

- Tabulate the concentrations and corresponding responses for each analyte. Compare the meter's reading of each buffer's pH to the actual value. For the curve to be acceptable, each calibration buffer's reading must be within 0.05 pH units from the actual value.

  Note: Pour fresh pH buffer solution from the stock bottle each day. Do not reuse over multiple days.

Sample Analysis

- Remove the samples or extracts from the refrigerator and allow them to come to room temperature.

- Place an aliquot of the well-mixed sample into a beaker on the magnetic stir plate. Make sure that the electrode is immersed in the sample but does not contact the Teflon-coated stir bar.

- Allow the sample to slowly stir while the pH measurement is being made. Record the pH to the nearest 0.01 pH unit after the reading has stabilized.

- After all measurements have been made, return the electrode to the 7 buffer solution or electrode storage solution and set the meter to "standby." The meter should be left "on" when not in use.

18.0 Revision History

Summary of Changes from Previous Revision:

- Minor editorial edits and clarifications made.
- Added requirement to hold samples requiring acidification upon receipt for 24 hours (after acidification) prior to analysis. Section 8.1
- Added note that if an LCS and LCSD are performed, both QC items must be evaluated and reported. Acceptable recoveries (as well as %RPD) for both LCS and LCSD are required. Section 9.1
- Revised amount of Calcium Carbonate LCS Standard used to prepare LCS. Section 10.2.2
- Removed requirement that IDOC must be from a second source and IDOC must be documented using the IDOC Form. These items are no longer mandatory. Section 12.5
- Clarified requirements and frequency for RLVs, MDL Studies, and MDLVs to be consistent with SOP SA-QA-07 and to include the quarterly frequency as defined by DOD. Section 12.1 - 12.3 and Attachment 3
- Added section on troubleshooting. Attachment 4

Review Summary:
This SOP was further reviewed on 10/27/12 during the annual review cycle, and no edits were found to be necessary. An alpha-character has been appended (i.e, SA-GE-201, Rev. 3A) to denote this review was performed.

CARBON CONTENT IN WATER: Total Carbon (TC), Total Organic Carbon (TOC), and Total Inorganic Carbon (TIC)

(Methods: EPA 9060, EPA 9060A, SM5310B, & EPA 415.1)

Approvals (Signature/Date):

Ammie L. Martin February 21, 2012
Ammie Martin Date
Quality Assurance Specialist, Designee for:
Andrea Teal, Quality Assurance Manager / Interim Environmental Health and Safety Coordinator

December 27, 2011
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1.0 Scope and Application

This SOP gives the procedures for the determination of total carbon (TC), total inorganic carbon (TIC), and total organic carbon (TOC) in water samples by combustion.

Total carbon is comprised of total organic carbon and total inorganic carbon.

- If TOC is requested, the sample is acidified upon collection and analyzed.
- If TC is requested, an unacidified sample is collected and analyzed.
- If TIC is requested, the sample is collected and analyzed twice (once for the acidified portion and once for the unacidified portion) and the TOC result is subtracted from the TC result to obtain TIC.

Note: The determination of TOC involves purging the sample which eliminates any volatile organic compounds (VOCs) from the sample matrix. For this reason, the TOC reported is actually the non-purgeable TOC. The determination of TC or TIC involves analyzing the sample without purging the VOCs from the sample matrix.

This procedure can also be used for the determination of dissolved organic carbon (DOC), dissolved inorganic carbon (DIC), and dissolved carbon (DC) by performing a filtration on the sample prior to acidification and analysis.

The reporting limits (RL), the method detection limits (MDL), and the accuracy and precision criteria associated with this procedure are provided in the LIMS Method Limit Groups (MLGs).

This SOP was written by and for TestAmerica's Savannah laboratory.

2.0 Summary of Method

Total organic carbon (TOC) is measured by purging an acid-preserved sample to eliminate the inorganic carbon present in the sample. The acid converts any carbonates present in the sample to CO_2, which is then purged out of the sample. The sample is then combusted at 680°C. The carbon is converted to carbon dioxide and is measured by an infrared (IR) detector.

Total carbon is performed in the same manner as TOC using unpreserved vials and eliminating the purging step. TIC is determined using a calculation by subtracting the TOC result from the TC result.

Dissolved organic carbon (DOC) is performed by filtering an aliquot of unpreserved sample with a 0.45um filter and preserving the filtrate to a pH of <2 with 50% HCl. The filtered sample is analyzed using the same procedures and equipment as the TOC analysis.

The following equation shows the relationship between the various forms of carbon that can be measured with this procedure:

$$TC = TOC + TIC$$

Where:
TC = total carbon
TOC = total organic carbon
TIC = total inorganic carbon

2.3 This SOP is based on the following methods: EPA Method 415.1, Standard Methods 5310B, EPA Method 9060, and EPA Method 9060A.

3.0 Definitions

Refer to the Glossary Section of the *Quality Assurance Manual* (QAM) for a complete listing of applicable definitions and acronyms.

4.0 Interferences

4.1 Procedural Interferences

4.1.1 Interferences may be caused by contaminants in solvents, reagents, glassware, and other sample processing apparatus and can make identification and/or quantification of the target analytes difficult.

4.1.2 All sample collection containers are single-use disposable containers which limits the potential for contamination. All non-disposable labware must be scrupulously cleaned in accordance with the posted Labware Cleaning Instructions to ensure it is free from contaminants and does not contribute artifacts.

4.1.3 High purity reagents and solvents are used to help minimize interference problems. Hydrochloric acid must be verified prior to use in accordance with the TestAmerica Solvent Lot Testing Program.

4.1.4 Instrument and/or method blanks are routinely used to demonstrate all reagents and apparatus are free from interferences under the conditions of the analysis.

4.2 Matrix Interferences

4.2.1 Matrix interferences may be caused by contaminants that are co-extracted from the sample matrix. The sample may require cleanup or dilution prior to analysis to reduce or eliminate the interferences.

4.2.2 Interfering contamination may occur when a sample containing low concentrations of analytes is analyzed immediately following a sample containing relatively high concentrations of analytes. As such, samples known to be clean should be analyzed first. To prevent carryover into subsequent samples, analysis of reagent blanks may be needed after the analysis of a sample containing high concentrations of analytes.

4.2.3 Sample particulates that are larger than the syringe will not be included in the carbon determination. This condition may cause low results if the particulates contain

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measurable levels of carbon. The analyst must be cognizant of samples that may clog the sample syringe, and initiate an NCM, as applicable, to address this situation.

4.2.4 Liquid samples containing oil or that are oily in nature will deactivate the catalyst which may require regeneration or replacement prior to running other samples.

4.2.5 It is very important that the inorganic carbon be removed from the sample prior to the direct determination of the TOC (non-purgeable TOC). Failure to remove the inorganic component of the sample may result in artificially high TOC results.

5.0 Safety

Employees must abide by the policies and procedures in the TestAmerica Environmental Health and Safety Manual (EHSM), the TestAmerica Savannah Addendum to the EHSM, and this document.

This procedure may involve hazardous materials, 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 to follow appropriate safety, waste disposal, and health practices under the assumption that all samples and reagents are potentially hazardous.

The analyst must protect himself/herself from exposure to the sample matrix. Many of the samples that are tested may contain hazardous chemical compounds or biological organisms. The analyst must, at a minimum, wear protective clothing (lab coat), eye protection (safety glasses or face shield), disposable gloves, and closed-toe, nonabsorbent shoes when handling samples.

5.1 Specific Safety Concerns or Requirements

5.1.1 The furnace on the carbon analyzer must be cooled to room temperature before maintenance is performed. The temperature of the analyzer furnace can reach 680°C. The analyst must be careful to avoid touching these very hot surfaces.

5.1.2 Care must be taken when handling hydrochloric acid (HCl) and solutions of hydrochloric acid. The 2N HCl must be handled with caution.

Hydrochloric acid is extremely hazardous as an oxidizer, a corrosive, a poison, and is reactive. Inhalation of the vapors can cause coughing, choking, irritation of the nose, throat, and respiratory tract, breathing difficulties, and lead to pneumonia and pulmonary edema. Contact with the skin can cause severe burns, redness, and pain. Acid vapors are irritating and can cause damage to the eyes. Contact with the eyes can cause permanent damage. The acid should be added slowly to minimize potentially violent reactions that will splatter acid.

5.1.3 The carrier gas MUST remain on while the instrument is in operation. The analyst must ensure that there is sufficient carrier gas available in the tank to complete the run. Failure to have carrier gas running through the instrument while the combustion furnace is at the operating temperature may result in melting of the combustion tubes.

5.2 Primary Materials Used

The following is a list of the materials used in this procedure, which have a serious or significant hazard rating, and a summary of the primary hazards listed in their MSDS.

Note: This list does not include all materials used in the procedure. A complete list of materials used in this procedure can be found in the Reagents and Standards Section and the Equipment and Supplies Section of this SOP

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 found using the "MSDS" link on the Oasis homepage, on the EH&S webpage on Oasis, and on the QA Navigator.

Material	Hazards	Exposure Limit [1]	Signs and Symptoms of Exposure
Hydrochloric Acid	Corrosive Poison	5ppm 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]Exposure limit refers to the OSHA regulatory exposure limit.			
Note: Always add acid to water to prevent violent reactions.			

6.0 Equipment and Supplies

6.1 Equipment and Instrumentation

Shimadzu TOC-VCPN Carbon Analyzer

6.2 Analytical Data System / Software / Hardware

The TOC Control software is used on a Windows-based PC to schedule and acquire data and to output the data to the laboratory's LIMS system (i.e., TALS). This software allows for the construction of a calibration curve and calculation of concentrations of analytes.

6.3 Lab Supplies

Volumetric Containers – various sizes; Class A, where applicable. Verify in accordance with SOP SA-AN-100: *Support Equipment (Verification and Use)*

Disposable Graduated Pipettes – various sizes. Verify in accordance with SOP SA-AN-100: *Support Equipment (Verification and Use)*

Disposable Transfer Pipettes – various sizes

Filters – 0.45um, disposable

Syringes – 10mL, disposable

Autosampler vials – 25mL

pH paper

Detergent – Liquinox, used for washing non-disposable labware.

Ultra pure compressed air, regulator, and appropriate fittings – The compressed air must contain less than 1ppm of carbon dioxide. This gas is used as the carrier and sparge gas for the analysis of aqueous samples.

Stir plate

Stir bars – Teflon

6.4 Sample Collection Containers

All sample collection containers are single-use disposable containers which limits the potential for contamination.

The sample collection containers supplied by the laboratory are purchased with Certificate of Analysis attesting to purity. The routine bottle kits supplied are as follows:

Analyte	Bottle Type	Preservative	Other Equipment
TOC	125mL amber glass	2mL 50% HCl	None
TC	40mL VOA	None	None
TIC	125mL amber glass	2mL 50% HCl	None
	40mL VOA	None	None
DOC	125mL amber glass	2mL 50% HCl	10mL syringe 0.45um filter
DC	40mL VOA	None	10mL syringe 0.45um filter
DIC	125mL amber glass	2mL 50% HCl	10mL syringe 0.45um filter
	40mL VOA	None	10mL syringe 0.45um filter

7.0 Reagents and Standards

7.1 Expiration Dates

Expiration dates (time from initial use or receipt to final use) for standard and reagent materials must be set according to the guidance in this SOP. Note: These are maximum expiration dates and are not to be considered an absolute guarantee of standard or reagent quality. Sound judgment must be used when deciding whether to use a standard

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or reagent. If there is doubt about the quality of a standard or reagent material, a new material must be obtained or the standard or reagent material verified. Data quality must not be compromised to extend a standard's life – i.e., when in doubt, throw it out.

The expiration date of any standard must not exceed the expiration date of the standard that was used to prepare it; that is, the "children may not outlive the parents".

7.2 Reagents

Reagents must be prepared and documented in accordance with SOP SA-AN-041: *Reagent and Standard Materials Traceability.*

Hydrochloric acid must be verified prior to use in accordance with the TestAmerica Solvent Lot Testing Program.

7.2.1 Laboratory Reagent Water – ASTM Type II

7.2.2 Hydrochloric acid (HCl) – concentrated, reagent grade or better. Store in original container at room temperature.
Storage: reagent cabinet
Expiration: manufacturer's expiration date or 2 years from the date opened, whichever is sooner

7.2.3 Hydrochloric acid solution (50% HCl) – Add approximately 400mL of laboratory reagent water to a 2L beaker. Place the beaker on a magnetic stir plate located under a hood. Add a Teflon stir bar to the laboratory reagent water and turn the stir plate on. Slowly add 500mL of concentrated HCl to the beaker in small aliquots. After the entire 500mL of HCl has been added, continue mixing and allow the solution to cool. After the solution has cooled, transfer to a 1L volumetric flask and dilute to volume with laboratory reagent water. This solution must be stored in a glass container at room temperature. ***Caution: Hydrochloric acid is extremely toxic, has a suffocating odor, and can cause severe chemical burns.***
Storage: Acid storage cabinet, away from incompatibles
Expiration: 2 years from the date prepared or the expiration date of the parent reagent, whichever is sooner

7.2.4 2N HCl Solution – Slowly add 42mL of concentrated HCl to approximately 150mL of laboratory reagent water contained in a 250-mL volumetric flask. Dilute to volume with laboratory reagent water. Transfer the reagent to a glass container. This reagent must be prepared under a hood. This solution must be stored in a glass container at room temperature. ***Caution: Hydrochloric acid is extremely toxic, has a suffocating odor, and can cause severe chemical burns.***
Storage: reagent cabinet
Expiration: 2 years from the date prepared or the expiration date of the parent reagent, whichever is sooner

7.3 Standards

Standards must be prepared and documented in accordance with SOP SA-AN-041: *Reagent and Standard Materials Traceability.* Certificates of analysis or purity must be

received with all purchased standards, and scanned and filed in the Data Archival Folder on the G-drive.

7.3.1 Potassium Hydrogen Phthalate (KHP) Salt – Primary source; reagent grade; purchased from EMD Chemicals
Storage: Store in original container at room temperature in a desiccator
Expiration: manufacturer's expiration date or 5 years from the date opened, whichever is sooner

Note: This material must be lightly crushed, to break up lumps, and dried at 120°C prior to preparation of the stock standard.

7.3.2 ICAL/MS/MSD Stock Standard, 2000mg/L – Prepared by adding 4.256g of primary source KHP Salt to 1L reagent water.
Storage: Refrigerated at 4°C
Expiration: 3 months from the date prepared or the expiration date of the parent reagent, whichever is sooner

7.3.2 Calibration Standards
Prepare the calibration standards according to the recipe in the following table. Smaller volumes and different concentrations may be prepared if approved by the department manager.

Calibration Standard Level	ICAL/MS/MSD Stock Standard Volume (mL)	Final Volume (mL)	Calibration Standard Concentration (mg/L)
1	0.0	1000	0.0
2	0.50	1000	1.0
3	2.5	1000	5.0
4	5.0	1000	10
5	12.5	1000	25
6	25	1000	50
7	50	1000	100

Adjust the pH of each standard to less than 2 with 50% HCl. Transfer the calibration standards to amber storage bottles.
Storage: Refrigerated at 4°C
Expiration: 3 months from the date prepared or the expiration date of the parent reagent, whichever is sooner

7.3.3 Initial Calibration Verification (ICV)

Second Source Potassium Hydrogen Phthalate (KHP) Salt – Reagent grade, purchased from Fisher Scientific. Note: The ICV standard must be purchased from a source independent of that used to prepare the calibration standards. Store in original container at room temperature.
Storage: Desiccator
Expiration: manufacturer's expiration date or 5 years from the date opened, whichever is sooner

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Note: This material must be lightly crushed, to break up lumps, and dried at 120°C prior to preparation of the stock standard.

7.3.4 ICV Stock Standard – Transfer 4.256g of the Second Source KHP to a 1L volumetric and dissolve in a small amount of laboratory reagent water. Dilute to volume with laboratory reagent water. The stock standard has a concentration of 2000mg C/L. Transfer the stock standard to an amber storage bottle. Storage: Refrigerated at 4°C
Expiration: 3 months from the date prepared or the expiration date of the parent reagent, whichever is sooner

7.3.5 Laboratory Control Sample (LCS) Solution (20mg/L) – Add 10mL ICV Stock Standard to 1L reagent water. Adjust the pH to less than 2 with 50% HCl. Transfer the stock standard to an amber storage bottle.
Storage: Refrigerated at 4°C
Expiration: 3 months from the date prepared or the expiration date of the parent reagent, whichever is sooner

7.3.6 Sodium Carbonate ($NaCO_3$) – reagent grade. Store in original container at room temperature.
Storage: reagent cabinet
Expiration: manufacturer's expiration date or 5 years from the date opened, whichever is sooner

7.3.7 Sodium Bicarbonate ($NaHCO_3$) – reagent grade. Store in original container at room temperature.
Storage: reagent cabinet
Expiration: manufacturer's expiration date or 5 years from the date opened, whichever is sooner

7.3.8 Carbonate/Bicarbonate TIC Check Solution – Place approximately 800mL of laboratory reagent water in a 1L volumetric flask. Add 1.38g $NaHCO_3$ and 1.77g $NaCO_3$ and mix until all solid is dissolved into solution. Dilute to 1L with laboratory reagent water and adjust the pH to <2 with 50% HCl. Transfer to an amber glass container and store at room temperature.
Storage: reagent cabinet
Expiration: 2 years from the date prepared or the expiration date of the parent reagent, whichever is sooner

8.0 Sample Collection, Preservation, Shipment, and Storage

8.1 Aqueous Samples

All aqueous samples must be iced at the time of collection and maintained at 4°C (less than 6°C but not frozen) until the time of analysis. Samples must be analyzed within 28 days of collection.

NCMs must be initiated for samples collected in improper containers and containing improper or insufficient preservatives. NCMs must be initiated for samples for TC and/or

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TIC analysis that are received containing headspace.

8.1.1 TOC

Aqueous samples for TOC are routinely collected in 125mL amber glass containers containing 2.0mL of 50% hydrochloric acid preservative is added to the container. This preservative should be sufficient to achieve a sample pH of <2.

8.1.2 TC

Aqueous samples for TC are routinely collected in 40mL VOA vials with no preservative. The 40mL VOA must be filled with no headspace to minimize the loss of the volatile organic components.

Note: a 125mL amber glass container may also be used, provided the sample is collected with no headspace.

8.1.3 TIC

Aqueous samples for TIC require 2 sets of bottles to be supplied:
- 1 set as outlined above for TOC
- 1 set as outlined above for TC

8.1.4 DOC

Aqueous samples for DOC should be filtered and preserved in the field, within 2 hours of collection. The samples are filtered using a 0.45um filter, and the filtrate should be collected in a 125mL container containing hydrochloric acid preservative sufficient to achieve a sample pH of <2.

Note: Samples for DOC may be collected in unpreserved containers, filtered in the lab upon receipt, and then preserved with HCl; however, this is not recommended as the 2-hour holding time will not be met. An NCM must be initiated for samples collected and preserved in this manner.

8.1.5 DC

Aqueous samples for DC should be filtered in the field, within 2 hours of collection, using a 0.45um filter. The filtrate should be collected in a 40mL VOA vial with no preservative. The 40mL VOA must be filled with no headspace to minimize the loss of the volatile organic components.

Note: a 125mL amber glass container may also be used, provided the sample is collected with no headspace.

8.1.6 DIC

Aqueous samples for DIC require 2 sets of bottles to be supplied:
- 1 set as outlined above for DOC
- 1 set as outlined above for DC

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8.1.7 Preservation Checks

8.1.7.1 pH Verification

For each preserved sample,
- Place a piece of pH paper in a disposable medicine cup.
- Pour a few drops of sample into the medicine cup and note the color change of the pH paper.
- If the pH is greater than 2, initiate a Nonconformance Memo. Adjust the sample pH to <2 using 50% HCl.

Note: To avoid cross-contamination, use a separate medicine cup and piece of pH paper per sample. Do not dip the pH paper into the sample container. The pH paper dye may bleed into the sample and affect sample results.

9.0 Quality Control

SOP SA-QA-17: *Evaluation of Batch QC Data* and the SOP Summary in Attachment 4 provide requirements for evaluating QC data.

9.1 Batch QC

An analytical batch consists of up to 20 environment samples and the associated quality control items. The minimum QC items required for each analytical batch are: a method blank, a laboratory control sample (LCS), a matrix spike (MS), and a matrix spike duplicate (MSD) or sample duplicate (SD).

If there is insufficient sample to perform the MS or sample duplicate, an NCM must be initiated and the LCS must be prepared in duplicate (i.e., LCSD must be performed).

Note: If an LCS and LCSD are performed, both QC items must be evaluated and reported. Acceptable recoveries (as well as %RPD) for both LCS and LCSD are required.

Batch QC must meet the criteria given in Attachment 4 of this SOP.

9.2 Instrument QC

9.2.1 Initial Calibration (ICAL)

The instrument must be calibrated in accordance with SOP SA-QA-16: *Evaluation of Calibration Curves.* This SOP provides requirements for establishing the calibration curve and gives the applicable formulas.

Instrument calibration is performed by analyzing a series of known standards. The calibration curve contains 3 ranges. Each range consists of a minimum of 4 standards. The lowest level calibration standard must be at or below the reporting limit, and the remaining standards will define the working range of the analytical system.

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The initial calibration standard concentrations currently in use in the laboratory are as follows:

Range	Calibration Level	Calibration Standard Level	Concentration (mg/L)
1	1	1	0
1	2	2	1.0
1	3	3	5.0
1	4	4	10
2	5	2	1.0
2	6	4	10
2	7	5	25
2	8	6	50
3	9	2	1.0
3	10	5	25
3	11	6	50
3	12	7	100

Refer to Section 7.3.2 for the standard preparation instructions. Other standard concentrations may be used provided they support the reporting limit and are fully documented in accordance with SOP SA-AN-041.

The purge is turned off to analyze the initial calibration. Each initial calibration standard is analyzed by the instrument up to 5 times. The instrument chooses the best 3 of the 5 analyses and averages this result to determine the response for the associated concentration.

9.2.1.1 ICAL Criteria

The correlation coefficient (r) of the regression curve must be greater than 0.995 for the initial calibration curve to be acceptable.

Note: The calibration must be performed quarterly (i.e., once every 3 months) at a minimum.

9.2.2 Second Source Initial Calibration Verification (ICV)

The calibration curve must be verified after the initial calibration is established, prior to any sample analyses, in accordance with SOP SA-QA-16 with a standard obtained from a second source.

The initial calibration verification standard concentration currently in use in the laboratory is 400mg/L. Refer to Section 7.3.3 for the standard preparation instructions. Another standard concentration may be used provided it is mid-level and fully documented in accordance with SOP SA-AN-041.

The ICV must be within +/-10% of the true value to be acceptable.

9.2.3 Initial Calibration Blank (ICB) / Continuing Calibration Blank (CCB)

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The instrument must be shown to be free from contamination by the analysis of calibration blanks. Initial calibration blanks are analyzed immediately following the initial calibration. Continuing calibration blanks are analyzed at the beginning and end of each batch.

Initial and continuing calibration blanks must be <1/2RL to be acceptable.

9.2.4 Continuing Calibration Verification

The initial calibration curve must be verified every 10 analyses with a mid-level standard.

The CCV must be within +/-10% of the true value to be acceptable.

The continuing calibration verification standard concentrations currently in use in the laboratory are equivalent to the 5.0, 50, and 200mg/L levels of the ICAL. Refer to Section 7.3.2 for the standard preparation instructions. Another standard concentration may be used provided it is mid-level and fully documented in accordance with SOP SA-AN-041.

9.2.5 Carbonate/Bicarbonate TIC Check Solution

This solution is analyzed once daily as a check of the efficiency of the acidification and sparging to remove inorganic carbon from samples being analyzed for TOC.

Transfer an aliquot of the Carbonate/Bicarbonate TIC Solution to a labeled autosampler vial.

Adjust the pH of the solution to <2 with 50% HCl and analyze.

Evaluate the result. If the TIC Check standard is less than the RL, continue with the sequence. If not, check the pH of the standard to make sure that it is less than 2.0, remake the standard, and reanalyze the standard. If the result is still greater than 2.0, contact the supervisor immediately. The instrument may need to be serviced.

9.3 Corrective Action for Out-of-Control Data

When the quality control parameters do not meet the criteria set forth in this SOP, corrective action must be taken in accordance with SOP SA-QA-05: *Preventive and Corrective Action Procedures* the QC Summary Table in Attachment 4. SOP SA-QA-05 provides contingencies for out-of-control data and gives guidance for exceptionally permitting departures from approved policies and procedures. Nonconformance Memos must be initiated to document all instances where QC criteria are not met and all departures from approved policies and procedures.

10.0 Procedure

10.1 Sample Preparation

Remove the samples from the refrigerator and allow them to come to room temperature.

10.2 QC Sample Preparation

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Prepare the method blank by adding 25mL of laboratory reagent water that has been adjusted to a pH of <2 with 50% HCl to an autosampler vial.

Prepare the LCS by adding 25mL of LCS Solution to an autosampler vial.

Prepare the matrix spikes (MS and MSD) by adding 0.25mL of the 2000mg/L ICAL/MS/MSD Stock Standard Solution to 25mL of sample.

Prepare a sample duplicate.

10.3 Analysis

10.3.1 Instrument Operating Conditions

The instrument conditions listed in this SOP are provided for guidance purposes. The actual conditions used by the laboratory may be slightly different from those listed here and must be documented in the instrument maintenance log, data system, and/or run log.

Instrument maintenance must be performed in accordance with Attachment 2 of this SOP.

Turn the Shimadzu TOC-VCPN on and set the following parameters:

Analyzer Furnace: 680°C
Carrier Gas: 150mL/min (ultrapure compressed air)
Purge Gas: 50mL/min (ultrapure compressed air)
Minimum Number of Replicates: 3
Maximum Number of Replicates: 5

Allow the instrument to equilibrate and the furnace to reach the set point before processing samples.

10.3.2 Initial and Continuing Calibration

Calibrate the instrument using the standards and criteria described given in Section 9.2.1. Once the calibration has been established and verified with an ICV in accordance with Section 9.2.2, sample analysis may proceed.

Verify the calibration curve with a continuing calibration verification using the standards and criteria described given in Section 9.2.4.

10.3.3 Sample Analysis

The sample must be analyzed using the same volume used for the calibration standards. Samples that are known to be relatively clean should be analyzed first. Samples suspected of containing high concentrations should be analyzed last. Instrument blanks may be analyzed after suspected high concentration samples to allow the detector response to stabilize.

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Each sample must be analyzed in quadruplicate for EPA 9060 and EPA 9060A, and in triplicate for SM5310B and EPA 415.1. If a sample contains a concentration of TOC greater than the linear range of the calibration curve on range 3, the sample must be diluted and re-analyzed. The average of the quadruplicate analyses results is reported as the final result in LIMS.

Note: The laboratory purges the acidified sample during analysis to remove the inorganic and purgeable organic carbon from the sample matrix; therefore, the non-purgeable TOC (nTOC) or non-purgeable DOC (nDOC) concentration is determined.

In order to determine the total carbon (TC) or dissolved carbon (DC) concentration an aliquot of the un-preserved sample is analyzed without purging the inorganic and purgeable organic carbon from the sample.

10.3.4 Example Analytical Sequence

An example analytical sequence is listed below.

Description	Comments
Blank	
Initial Calibration	
ICV	Second Source: 400mg/L
ICB	Used as method blank
Bicarbonate/Carbonate TIC Check Standard	
LCS	
Samples	Up to 10 sample analyses, including MS/MSD
CCV	5mg/L
CCB	
Samples & Batch QC Items	Up to 10 analyses, including MS/MSD
CCV	50mg/L
CCB	
Samples & Batch QC Items	Up to 10 sample analyses, including MS/MSD
CCV	200mg/L
CCB	

11.0 Calculations / Data Reduction

11.1 Data Reduction

Data must be evaluated in accordance with SOP SA-QA-02: *Data Generation and Review.*

11.1.1 Historical Data

Many of the laboratory's clients submit samples for repeat monitoring purposes. Prior to analysis, verify TALS Worksheet Notes and/or use the Historical Data Tracker feature to determine if historical data is available for review.

11.1.2 Chemical Relationships

When available, the following chemical relationships must be evaluated for each sample. If these relationships are not met the Department Manager must be contacted immediately.

- Total Results ≥ Dissolved results
- COD ≥ TOC
- TC ≥ TOC + TIC

11.2 Calculations

11.2.1 The calculations associated with batch QC determinations are given in SOP SA-QA-17. Applicable calculations include accuracy (% recovery) and precision (%RPD).

11.2.2 The calculations associated with initial and continuing calibrations are given in SOP SA-QA-16. Applicable calculations include determination for: calibration factor, standard deviation, relative standard deviation, relative response factor, and relative standard deviation.

11.2.3 The following equations show the relationship between the various forms of carbon that can be measured with this procedure:

$$TC = TOC + TIC$$

Where:
TC = total carbon
TOC = total organic carbon
TIC = total inorganic carbon

$$DC = DOC + DIC$$

Where:
DC = dissolved carbon
DOC = dissolved organic carbon
DIC = dissolved inorganic carbon

11.2.3 The calculation to determine final concentration is given as follows:

$$C(mg/L) = Ccurve \otimes DF$$

Where:
C_{curve}= concentration of TOC from curve
DF = dilution factor

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12.0 Method Performance

12.1 Reporting Limit Verification (RLV)

At a minimum, RLVs must be performed initially upon method set-up in accordance with SOP SA-QA-07: *Determination and Verification of Detection and Reporting Limits*.

For analytes and methods certified by DOD ELAP, RLVs must also be performed quarterly thereafter. For all other analytes and methods, RLVs must also be performed annually thereafter.

12.2 Method Detection Limit (MDL) Study

The 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. 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 current MDLs associated with this procedure are given in the Method Limit Group (MLG) in TALS.

At a minimum, MDL Studies must be performed initially upon method set-up in accordance with SOP SA-QA-07: *Determination and Verification of Detection and Reporting Limits*.

12.3 Method Detection Limit Verification (MDLV)

At a minimum, MDLVs must be performed initially upon method set-up in accordance with SOP SA-QA-07: *Determination and Verification of Detection and Reporting Limits*.

For analytes and methods certified by DOD ELAP, MDLVs must also be performed quarterly thereafter. For all other analytes and methods, MDLVs must also be performed annually thereafter.

12.4 Note: RLVs, MDL Studies, and MDLVs are performed for TOC and TC, only. The TOC and TC values are used to satisfy the DOC and DC values, respectively. TIC and DIC are obtained using a calculation; therefore, these values are defined as the higher of the values between TOC and TC (or DOC and DC).

12.5 Demonstrations of Capability

Initial and continuing demonstration of capability must be performed in accordance with SOP SA-QA-06*: Training Procedures.*

Prior to performing this procedure unsupervised, each new analyst who performs this analysis must demonstrate proficiency per method/analyte combination by successful completion of an initial demonstration of capability. The IDOC is performed by the analysis of 4 consecutive LCSs that meet the method criteria for accuracy and precision. The LCSs must be from a second source than that used to prepare the calibration

standards. The IDOC must be documented on the IDOC Form shown in SOP SA-QA-06 with documentation routed to the QA Department for filing.

Annual continuing demonstrations of capability (CDOCs) are also required per analyst per method/analyte combination. The CDOC requirement may be met by the consecutive analysis of four LCS all in the same batch, by the analysis of four LCS analyzed in four consecutive batches (in different batches on different days), or via acceptable results on a PT study. The CDOC must be documented and routed to the QA Department for filing.

12.6 Training Requirements

All training must be performed and documented in accordance with SOP SA-QA-06: *Training Procedures.*

Note: The SOPs listed in the Reference/Cross-Reference Section are applicable to this procedure. All employees performing this procedure must also be trained on these SOPs, and/or have a general understanding of these procedures, as applicable.

13.0 Pollution Control

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (e.g., examining recycling options, ordering chemicals based on quantity needed, preparing reagents based on anticipated usage and reagent stability, etc.). Employees must abide by the policies in Section 13 of the Environmental Health and Safety Manual and the TestAmerica Savannah Addendum to the EHSM.

This procedure has been evaluated for opportunities to minimize the waste generated. Where reasonably feasible, pollution control procedures have been incorporated.

14.0 Waste Management

Waste management practices must be conducted consistent with all applicable federal, state, and local rules and regulations. All waste (i.e., excess reagents, samples, and method process wastes) must be disposed of in accordance with the TestAmerica Savannah Addendum to the EHSM. Waste description rules and land disposal restrictions must be followed.

14.1 Waste Streams Produced by the Method

The following waste streams are produced when this method is carried out:

- Excess samples, reagents, and standards must be disposed in accordance with the TestAmerica Savannah addendum to the Environmental Health and Safety Manual.
- Excess aqueous samples – Dispose according to characterization on the sample disposal sheets. Neutralize non-hazardous samples before disposal into drain/sewer. Transfer hazardous samples (identified on disposal sheets) to the waste department for disposal.

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15.0 References / Cross-References

- SOP SA-AN-041: *Reagent and Standard Materials Traceability*
- SOP SA-AN-100: *Support Equipment (Verification and Use)*
- SOP SA-QA-02: *Data Generation and Review*
- SOP SA-QA-05: *Preventive and Corrective Action Procedures*
- SOP SA-QA-06: *Training Procedures*
- SOP SA-QA-07: *Determination and Verification of Detection and Reporting Limits*
- SOP SA-QA-16: *Evaluation of Calibration Curves*
- SOP SA-QA-17: *Evaluation of Batch QC Data*
- TestAmerica Savannah Quality Assurance Manual
- TestAmerica Environmental Health and Safety Manual
- TestAmerica Savannah Addendum to the Environmental Health and Safety Manual
- *Method for Chemical Analysis of Water and Wastes;* U.S. EPA Office of Research and Development: Cincinnati, Ohio, March, 1983
- *Test Methods for the Evaluating Solids Wastes, Third Edition, SW-846*; US EPA Office of Solid Waste and Emergency Response: Washington, DC. (including Updates III and IV)
- *Standard Methods for the Examination of Water and Wastewater*; Online Edition; 2000. APHA, AWWA, WEF.

16.0 Method Modifications and/or Clarifications

16.1 Standard Methods 5310B recommends using phosphoric acid to preserve samples and standards. The preservation technique performed by the laboratory has been adopted from EPA Methods 415.1, 9060, and 9060A. These methods use hydrochloric acid to preserve the samples.

16.2 The reference methods do not specify a holding time to be used for TC and TIC. The laboratory defaults to 28 days for these analytes (and DC and DIC).

16.3 The laboratory allows for lab filtration of DOC samples; however, an NCM must be utilized to indicate the 2-hour holding time was not met.

16.4 MDLs are determined for TOC and TC, only. These TOC and TC MDLs are used to satisfy the DOC and DC MDLs, respectively. TIC and DIC are obtained using a calculation; therefore, these MDL values are defined as the higher of the values between TOC and TC (or DOC and DC).

16.5 SM5310B specifies that a blank, and an LCS or MS must be performed per 10 analyses with a 2nd source standard. The laboratory uses a same source standard for the MS; however, a second source LCS is utilized.

17.0 Attachments

The following Tables, Diagrams, and/or Validation Data are included as Attachments:

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Attachment 1: Sample Collection, Preservation, and Holding Time Table
Attachment 2: Preventative Maintenance and Troubleshooting
Attachment 3: SOP Summary
Attachment 4: QC Summary

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Attachment 1:
SOP Summary

Sample Preparation and Analysis Summary

Total organic carbon (TOC) is measured by purging the acid-preserved sample to eliminate the inorganic carbon present in the sample. The acid converts any carbonates present in the sample to CO_2, which is then purged out of the sample. Purging the sample also eliminates any volatile organic compounds (VOC) from the sample matrix; therefore, any direct measurement of TOC is actually non-purgeable TOC. The sample is then combusted at 680°C. The carbon is converted to carbon dioxide and is measured by an infrared (IR) detector. Standards are analyzed under the same instrument conditions as the samples.

Total carbon and total inorganic carbon are performed in the same manner as TOC using unpreserved vials and eliminating the purging step.

Dissolved organic carbon (DOC) is prepared either in the field or immediately upon receipt in the laboratory by filtering an aliquot of unpreserved sample with a 0.45um filter and preserving the filtrate to a pH of <2 with 50% HCl. The dissolved organic carbon is analyzed using the same procedures and equipment as the TOC analysis. The QC samples required for DOC are a filtered method blank and matrix spikes on a filtered sample.

Analytical Sequence

An example analytical sequence is listed below.

Description	Comments
Blank	
Initial Calibration	
ICV	Second Source: 400mg/L
ICB	Used as method blank
Bicarbonate/Carbonate TIC Check Standard	
LCS	
Samples	Up to 10 sample analyses, including MS/MSD
CCV	5mg/L
CCB	
Samples & Batch QC Items	Up to 10 analyses, including MS/MSD
CCV	50mg/L
CCB	
Samples & Batch QC Items	Up to 10 sample analyses, including MS/MSD
CCV	200mg/L
CCB	

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Attachment 2:
Sample Collection, Preservation, and Holding Time Table

Analyte	Routine Bottle Type	Other Field Equipment	Routine Volume	Minimum Volume	Chemical Preservative	Dechlorination Agent	Thermal Preservative	Holding Time
TOC	125mL amber glass	None	25mL	25mL	2mL 50% HCl	None	4°C[1]	28 Days from Collection
TC	40mL VOA Vial	None	25mL	25mL	None	None	4°C[1]	28 Days from Collection
TIC	125mL amber glass	None	25mL	25mL	2mL 50% HCl	None	4°C[1]	28 Days from Collection
	40mL VOA Vial	None			None	None	4°C[1]	28 Days from Collection
DOC	125mL amber glass	10mL syringe 0.45um filter	25mL	25mL	2mL 50% HCl	None	4°C[1]	Filter: 2 Hours from Collection Analysis: 28 Days from Collection
DC	40mL VOA Vial	10mL syringe 0.45um filter	25mL	25mL	None	None	4°C[1]	Filter: 2 Hours from Collection Analysis: 28 Days from Collection
DIC	125mL amber glass	10mL syringe 0.45um filter	25mL	25mL	2mL 50% HCl	None	4°C[1]	Filter: 2 Hours from Collection Analysis: 28 Days from Collection
	40mL VOA Vial	10mL syringe 0.45um filter	25mL	25mL	None	None	4°C[1]	Filter: 2 Hours from Collection Analysis: 28 Days from Collection

[1]Samples must be collected on ice and maintained at 0-6°C until time of analysis.

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Attachment 3:
QC Summary

QC Item	Frequency	Criteria	Corrective Action
Initial Calibration (ICAL) - Minimum of 4 points (with 3 curves)	Upon instrument set-up, and after unsuccessful CCV - Quarterly, at a minimum	r^2>0.995	Re-calibrate
Second Source Initial Calibration Verification (ICV)	After each ICAL	Within ±10% of the true value	Re-calibrate
Continuing Calibration Verification (CCV)	At the beginning and end of the analysis, and every 10 samples	Within ±10% of the true value	Refer to SOP SA-QA-16
Initial and Continuing Calibration Blank (ICB & CCB)	After ICV and every CCV	<1/2RL	Terminate the analysis; correct the problem; and reanalyze the previous 10 samples
Batch Definition	Analyzed together w/in 24-hr timeframe; not to exceed 10 field samples	Not Applicable	Not Applicable
Method Blank (MB)	One per batch	<1/2RL	Refer to SOP SA-QA-17
Laboratory Control Sample (LCS)	One per batch	Within MLG Limits	Refer to SOP SA-QA-17
Laboratory Control Sample Duplicate (LCSD)	One per extraction batch, when insufficient sample is provided for MS/MSD/SD	Within MLG Limits	Refer to SOP SA-QA-17
Matrix Spike (MS)	One per extraction batch	Within MLG Limits	Refer to SOP SA-QA-17
Matrix Spike Duplicate (MSD)	One per extraction batch	Within MLG Limits	Refer to SOP SA-QA-17

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QC Item	Frequency	Criteria	Corrective Action
Initial Demonstration of Capability (IDOC)	Initially, per analyst, per analyte/method/matrix combination	Refer to SOP SA-QA-06	Refer to SOP SA-QA-06 Note: Unsupervised work must not begin until successful IDOC is obtained.
Continuing Demonstration of Capability (CDOC)	Annually, per analyst, per analyte/method/matrix combination	Refer to SOP SA-QA-06	Refer to SOP SA-QA-06
Reporting Limit Verification (RLV)	Upon method/instrument set-up, per analyte/method/matrix combination. Then quarterly thereafter (for DOD ELAP) or annually thereafter (for non-DOD ELAP)	Refer to SOP SA-QA-07	Refer to SOP SA-QA-07
Method Detection Limit Study (MDL)	Upon method/instrument set-up, per analyte/method/matrix combination	Refer to SOP SA-QA-07	Refer to SOP SA-QA-07
MDL Verification (MDLV)	Upon method/instrument set-up, per analyte/method/matrix combination. Then quarterly thereafter (for DOD ELAP) or annually thereafter (for non-DOD ELAP)	Refer to SOP SA-QA-07	Refer to SOP SA-QA-07

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Attachment 4:
Preventative Maintenance and Troubleshooting

Maintenance contracts are carried for most instrumentation and close contact is maintained with service personnel to ensure optimal instrument functioning.

An extensive spare parts inventory is maintained for routine repairs, consisting of combustion tubes, halogen scrubber, sample needles, o-rings, and platinum catalyst., and other common instrumentation components. Since instrumentation is standardized throughout the laboratory network, spare parts and components can be readily exchanged among the network.

Preventive Maintenance

LABORATORY EQUIPMENT PREVENTIVE MAINTENANCE SCHEDULE								
	Service Interval							
Equipment / Item	D	W	M	Q	SA	A	AN	Service
Combustion tube							X	Replace as needed
Carrier gas pressure gauge	X							Check daily and replace gas as necessary
Humidifier water level	X							Check daily and refill as necessary

D = daily; W = Weekly; M = monthly; Q = Quarterly; SA = semi-annually; A = annually; AN = as needed

Desiccator Maintenance

Upright Desiccators with Doors

The following checks must be performed daily:

- Desiccant is active
- Hygrometer is in the low humidity zone
- Door is making an air tight seal

The desiccator door must remain closed and seated whenever possible.

When the desiccant turns from blue to light purple, discard desiccant, in accordance with the TestAmerica Savannah Addendum to the EHSM, and refill pan with fresh desiccant.

Troubleshooting

Troubleshooting should be documented as outlined above. If possible, troubleshooting is best performed in a step-wise manner to systematically isolate instrument components. Refer to the instrument manufacturer's guides for specific information and strategies. Enlist assistance from technical and/or department management as needed.

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Contingency Plan
In general, the laboratory has at least one backup unit for each critical unit. In the event of instrument failure, portions of the sample load may be diverted to duplicate instrumentation, the analytical technique switched to an alternate approved technique (such as manual colorimetric determination as opposed to automated colorimetric determination), or samples shipped to another properly certified or approved TestAmerica location.

Maintenance Log
A maintenance log must be established for each piece of equipment used in the laboratory.

All maintenance that is performed on the instrument must be recorded in the log including:

- analyst or technician performing the maintenance
- date the maintenance was performed
- detailed explanation of the reason for the maintenance
- resolution of the problem and return to control
- all service calls from instrument representatives

Instrument Labeling
Each instrument must be labeled with its name or ID (e.g., MSA, ICP-D, etc.). Additionally, non-operational instruments must be isolated from service or marked as being out of service. Each piece of equipment has an "Operational / Not Operational" sticker that is used for this purpose.

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18.0 Revision History

Summary of Changes from Previous Revision:

- Minor editorial, grammatical, and formatting changes made. Boilerplate text added.
- Updated SOP references to reflect current document control numbers and titles.
- Updated ICAL levels to match current laboratory procedure. Removed the 200 and 500mg/L standards and included a standard at 25mg/L.

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LIQUID PREPARATION PROCEDURES FOR ICP AND ICP/MS

(Methods: EPA 200.7, 200.8, 3005A, 3010A, SM3030C, and Filtration)

Approvals (Signature/Date):

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Andrea Teal Date
Quality Assurance Manager

April 17, 2012
Benjamin Gulizia Date
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Facility Distribution No. 1	Distributed To: QA Navigator

1.0 Scope and Application

This SOP gives the procedures for the preparation of metals in water and leachate samples prior to analysis by ICP (SOP SA-ME-070: *Elements by ICP*) or ICP/MS (SOP SA-ME-074: *Elements by ICP/MS*).

A complete target analyte list, the reporting limits (RL), the method detection limits (MDL), and the accuracy and precision criteria associated with this procedure are provided in the LIMS Method Limit Groups (MLGs).

This SOP was written by and for TestAmerica's Savannah laboratory.

2.0 Summary of Method

2.1 Total Recoverable Metals: A known volume of sample, usually 50mL, is transferred to a digestion vessel. The sample is refluxed with dilute nitric acid and hydrochloric acid at approximately 95°C. After the sample has evaporated to approximately 10-20mL, the sample is brought up to the original volume with reagent water. The laboratory utilizes two versions of the Total Recoverable preparation procedure. One version is equivalent to the EPA 3005A procedure and the second procedure is equivalent to the EPA 200.7 and 200.8 procedures.

2.2 Total Metals and TCLP/SPLP Leachates: A known volume of sample, usually 50mL, is transferred to a digestion vessel. The sample is refluxed with nitric acid at approximately 95°C. After the sample has digested, as evidenced by a clear, pale yellow color, HCl is added and the sample is brought up to the original volume with reagent water. This procedure is equivalent to EPA 3010A, and is utilized as the total metals preparation procedure for the EPA 200.7 and EPA 200.8.

2.3 Acid Extractable Metals: A known volume of sample, usually 50mL, preserved with nitric acid, is transferred to a digestion vessel. Hydrochloric acid is added and the sample is heated on a hot block for 15 minutes. The sample is filtered and the final volume adjusted to the original volume. This procedure is equivalent to Standard Methods 3030C.

2.4 Silica Samples for EPA 200.7 and EPA 6010C: Liquid samples are filtered. The filtered liquid samples are analyzed by ICP.

2.5 Drinking water samples for EPA 200.7 and EPA 200.8 with a turbidity concentration of less than 1NTU may be analyzed with no digestion. The exception to this rule is silver, which requires sample digestion prior to analysis. If the sample turbidity is >1NTU, then the EPA 200.7 or the EPA Method 200.8 preparation procedure is used.

2.6 Samples filtered for the determination of dissolved metals do not require digestion if the following criteria are met:

1) The sample has a low COD (<20mg/L);
2) The sample has a turbidity <1NTU ;
3) The sample is colorless with no significant odor ; ***and***
4) The sample is of one liquid phase and is free of suspended particulates or precipitates after acidification.

Note: Generally the lab will digest samples for dissolved metals, even if the above criteria are met, as the presence of organics in the native sample may lead to false positives for arsenic and selenium.

2.7 This SOP is based on the following methods: EPA 200.7, EPA 200.8, EPA 3005A, EPA 3010A, and Standard Methods 3030C.

3.0 Definitions

Refer to the Glossary Section of the *Quality Assurance Manual* (QAM) for a complete listing of applicable definitions and acronyms.

4.0 Interferences

4.1 Procedural Interferences

4.1.1 Interferences may be caused by contaminants in solvents, reagents, glassware, and other sample processing apparatus and can make identification and/or quantification of the target analytes difficult.

4.1.2 All sample collection containers are single-use disposable containers which limits the potential for contamination. All non-disposable labware must be scrupulously cleaned in accordance with the posted Labware Cleaning Instructions to ensure it is free from contaminants and does not contribute artifacts.

4.1.3 High purity reagents and solvents are used to help minimize interference problems. Hydrochloric acid and nitric acid must be verified prior to use in accordance with the TestAmerica Solvent Lot Testing Program.

4.1.4 Instrument and/or method blanks are routinely used to demonstrate all reagents and apparatus are free from interferences under the conditions of the analysis.

4.2 Matrix Interferences

4.2.1 Matrix interferences may be caused by contaminants that are co-extracted from the sample matrix. The sample may require cleanup or dilution prior to analysis to reduce or eliminate the interferences.

4.2.2 Interfering contamination may occur when a sample containing low concentrations of analytes is analyzed immediately following a sample containing relatively high concentrations of analytes. As such, samples known to be clean should be analyzed first. To prevent carryover into subsequent samples, analysis of reagent blanks may be needed after the analysis of a sample containing high concentrations of analytes.

4.2.3 Turbidity Matrix Interferences

4.2.3.1 The presence of floating debris and coarse sediments which settle out rapidly will give a low reading. Air bubbles will affect the results positively.

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4.2.3.2 The presence of true color, that is the color of water which is due to dissolved substances that absorb light, will cause turbidities to be low, although this effect is generally not significant with drinking waters.

4.2.3.3 Light absorbing materials such as activated carbon in significant concentrations can cause low readings.

5.0 Safety

Employees must abide by the policies and procedures in the TestAmerica Environmental Health and Safety Manual (EHSM), the TestAmerica Savannah Addendum to the EHSM, and this document.

This procedure may involve hazardous materials, 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 to follow appropriate safety, waste disposal, and health practices under the assumption that all samples and reagents are potentially hazardous.

The analyst must protect himself/herself from exposure to the sample matrix. Many of the samples that are tested may contain hazardous chemical compounds or biological organisms. The analyst must, at a minimum, wear protective clothing (lab coat), eye protection (safety glasses or face shield), disposable latex or nitrile gloves, and closed-toe, nonabsorbent shoes when handling samples.

5.1 Specific Safety Concerns or Requirements

Nitric and hydrochloric acids are extremely hazardous as oxidizers, corrosives, poisons, and are reactive. Inhalation of the vapors can cause coughing, choking, irritation of the nose, throat, and respiratory tract, breathing difficulties, and lead to pneumonia and pulmonary edema. Contact with the skin can cause severe burns, redness, and pain. Nitric acid can cause deep ulcers, and staining of the skin to a yellow or yellow-brown color. These acid vapors are irritating and can cause damage to the eyes. Contact with the eyes can cause permanent damage.

Samples that contain high concentrations of carbonates or organic matter, or samples that are at elevated pH can react violently when acids are added. Acids must be added to samples under a hood to avoid splash/splatter hazards and/or possibly toxic vapors that will be given off when the samples are acidified.

5.2 Primary Materials Used

The following is a list of the materials used in this procedure, which have a serious or significant hazard rating, and a summary of the primary hazards listed in their MSDS.

NOTE: This list does not include all materials used in the procedure. A complete list of materials used in this procedure can be found in the Reagents and Standards Section and the Equipment and Supplies Section of this SOP

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 found using the "MSDS" link on the Oasis homepage, on the EH&S webpage on Oasis, and on the QA Navigator.

Material	Hazards	Exposure Limit[1]	Signs and Symptoms of Exposure
Hydrochloric Acid[2]	Corrosive Poison	5ppm 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.
Nitric Acid[2]	Corrosive Oxidizer Poison	2ppm TWA 4ppm STEL	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
[1]Exposure limit refers to the OSHA regulatory exposure limit.			
[2]Always add acid to water to prevent violent reactions.			

6.0 Equipment and Supplies

6.1 Equipment and Instrumentation

Thermometers – Verify in accordance with SOP SA-AN-100: *Laboratory Support Equipment (Verification and Use)*

Digestion block – capable of maintaining a sample digestion temperature of 95±5°C. The temperature of the digestion block must be monitored and recorded for each batch. The temperature is measured in a beaker or digestion vessel containing reagent water.

Turbidimeter – Hach Model 2100AN

6.2 Lab Supplies

Volumetric Containers – various sizes; Class A, where applicable. Verify in accordance with SOP SA-AN-100: *Laboratory Support Equipment (Verification and Use)*

Mechanical Pipettes – various sizes. Verify in accordance with SOP SA-AN-100: *Laboratory Support Equipment (Verification and Use)*

Disposable Graduated Pipettes – various sizes. Verify in accordance with SOP SA-AN-100: *Laboratory Support Equipment (Verification and Use)*

pH paper

Detergent – Alconox or equivalent, used for washing non-disposable labware.

Digestion vessels – appropriate volume for use with digestion block. Verify for volume accuracy per lot in accordance with SOP SA-AN-100: *Laboratory Support Equipment (Verification and Use)*

0.45um polypropylene filters and syringes

Sample Cells for Turbidimeter – clear and colorless. The cells must be kept clean, and discarded when they become scratched or etched.

Disposable Medicine Cups

6.3 Sample Collection Containers

All sample collection containers are single-use disposable containers which limits the potential for contamination.

The routine sample collection containers supplied by the laboratory are:

250mL plastic – purchased with Certificate of Analysis attesting to purity.

7.0 Reagents and Standards

7.1 Expiration Dates

Expiration dates (time from initial use or receipt to final use) for standard and reagent materials must be set according to the guidance in this SOP. Note: These are maximum expiration dates and are not to be considered an absolute guarantee of standard or reagent quality. Sound judgment must be used when deciding whether to use a standard or reagent. If there is doubt about the quality of a standard or reagent material, a new material must be obtained or the standard or reagent material verified. Data quality must not be compromised to extend a standard's life – i.e., when in doubt, throw it out.

The expiration date of any standard or reagent must not exceed the expiration date of the standard or reagent that was used to prepare it; that is, the "children may not outlive the parents".

7.2 Reagents

Reagents must be prepared and documented in accordance with SOP SA-AN-041: *Reagent and Standard Materials Procedures.*

Hydrochloric acid and nitric acid must be verified prior to use in accordance with the TestAmerica Solvent Lot Testing Program.

Laboratory Reagent Water – ASTM Type I

Nitric acid (HNO_3): reagent grade. Stable under ordinary conditions of use and storage.
Storage: Store in a cool, dry, ventilated storage area with acid resistant floors and good drainage. Store away from sunlight, heat, water, and incompatible materials.
Expiration:
Unopened: Manufacturer's expiration date
Opened: 5 years from date of opening

Hydrochloric acid (HCl): reagent grade. Stable under ordinary conditions of use and storage.
Storage: Store in a cool, dry, ventilated storage area with acid resistant floors and good drainage. Store away from sunlight, heat, water, and incompatible materials.
Expiration:
Unopened: Manufacturer's expiration date
Opened: 5 years from date of opening

7.3 Standards

Standards must be prepared and documented in accordance with SOP *SA-AN-041: Reagent and Standard Materials Procedures.* Certificates of analysis or purity must be received with all purchased standards, and scanned and filed in the Data Archival Folder on the G-drive.

7.3.1 Turbidity Standards

7.3.1.1 Formazin Stock Standard – 7500NTU Hach Ampoule
Storage: Store at room temperature
Expiration: Manufacturer's expiration date

7.3.1.2 Formazin Stock Standard – 4000NTU Hach Solution
Storage: Store at room temperature
Expiration: Manufacturer's expiration date

7.3.1.3 Formazin Calibration Standards

Concentration of Formazin Standard (NTU)	Volume of Formazin Standard (mL)	Final Volume (mL)	Concentration of CAL STD (NTU)
4000	0.50	100	20
4000	5.0	100	200
4000	25	100	1000
4000	30	30	4000
7500	30	30	7500

Expiration and storage: These standards must be prepared fresh on each day of use.

7.3.1.4 Second Source Calibration Verification Standards

Gelex turbidity standards, one standard for each instrument range – Stray Light, 0-2NTU, 0-20NTU, 0-200NTU, and 200-4000NTU.
Storage: Store at room temperature
Expiration: Manufacturer's expiration date

7.3.2 ICP Spiking Solutions

7.3.2.1 ICP Spiking Solution 1 is a solution purchased from SPEX (catalogue number SPIKE-1). The concentrations of the analytes in this solution are listed on the accompanying certificate of analysis.
Storage: Store this solution at room temperature.
Expiration: Replace this solution by the manufacturer's expiration date or sooner if needed or required.

7.3.2.2 Single Element Stock Standards – purchased from CPI, Inorganic Ventures, or equivalent vendor, at the concentrations listed below.

Arsenic (As) – 1000mg/L
Barium (Ba) – 1000mg/L
Boron (B) – 1000mg/L
Cadmium (Cd) – 1000mg/L
Calcium (Ca) – 10000mg/L
Chromium (Cr) – 1000mg/L
Lead (Pb) – 1000mg/L
Magnesium (Mg) – 10000mg/L
Molybdenum (Mo) – 1000mg/L
Potassium (K) – 10000mg/L
Selenium (Se) – 1000mg/L
Silver (Ag) – 1000mg/L
Sodium (Na) – 10000mg/L
Strontium (Sr) – 1000mg/L
Tin (Sn) – 1000mg/L
Titanium (Ti) – 1000mg/L

Storage: Store in a cool, dry, ventilated storage area with acid resistant floors and good drainage. Store away from sunlight, heat, water, and incompatible materials.
Expiration: Manufacturer's expiration date.

7.3.2.3 ICP Spiking Solution 2 – Add 1mL of nitric acid and 5mL of hydrochloric acid to a 100-mL volumetric flask containing about 50mL of laboratory reagent water. Add the appropriate volume of each single element stock standard to the flask. Dilute to volume with reagent water, and mix thoroughly. Transfer the spiking solution to a labeled storage container.

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Element	Parent Standard Concentration (mg/L)	Volume Added (mL)	Final Volume (mL)	Final Concentration (mg/L)
Boron (B)	1000	10	100	100
Calcium (Ca)	10000	5.0		500
Magnesium (Mg)	10000	5.0		500
Molybdenum (Mo)	1000	5.0		50
Potassium (K)	10000	5.0		500
Sodium (Na)	10000	5.0		500
Strontium (Sr)	1000	5.0		50
Tin (Sn)	1000	10		100
Titanium (Ti)	1000	10		100

Storage: Store in a cool, dry, ventilated storage area with acid resistant floors and good drainage. Store away from sunlight, heat, water, and incompatible materials.
Expiration: Prepare this solution every 180 days or sooner as needed or required. The expiration date must not exceed the expiration date of any of the components.

7.3.2.4 Silica/Silicon Standards

Silicon (Si) stock solutions are usually purchased for this procedure. The following conversion is used to adjust any volumes or concentrations appropriately:

$$Si = \frac{SiO_2}{2.14}$$

7.3.2.4.1 Stock SiO_2 Standard, 10000mg/L Si / 21400mg/L SiO_2 – purchased from CPI. Store at room temperature. This standard must be used by the manufacturer's expiration date.
Storage: Store in a cool, dry, ventilated storage area. Store away from sunlight, heat, water, and incompatible materials.

7.3.2.4.2 Intermediate SiO_2 Standard, 467mg/L Si / 1000mg/L SiO_2 – Add 20mL to 30mL of reagent water to a clean, plastic 100-mL volumetric flask. Add the volume of the Stock SiO_2 Standard given in the table below to the volumetric flask. Dilute to volume with reagent water.

Element	Conc. Stock SiO_2 Std (mg/L)	Volume Stock SiO_2 Std (mL)	Final Volume (mL)	Final Conc. (mg/L)
Silica (SiO_2) [Silicon (Si)]	21400 [10000]	4.67	100	1000 [467]

Storage: Store in a cool, dry, ventilated storage area. Store away from sunlight, heat, water, and incompatible materials.
Expiration: Prepare this solution every 180 days or sooner as needed or required.

The expiration date must not exceed the expiration date of any of the components.

7.3.3 ICP/MS Spiking Solutions

7.3.3.1 ICP/MS Spiking Solution 1 – Purchased from CPI International (4400-060117RHO2). The concentrations of the analytes in this solution are listed on the accompanying certificate of analysis.
Storage: Store this solution at room temperature.
Expiration: Replace this solution by the manufacturer's expiration date or sooner if needed or required.

7.3.3.2 ICP/MS Spiking Solution 2 – Add 10mL of hydrochloric acid to a 100-mL volumetric flask containing about 50mL of laboratory reagent water. Add the appropriate volume of each single element stock standard to the flask. Dilute to volume with reagent water, and mix thoroughly. Transfer the spiking solution to a labeled storage container.

Element	Parent Standard Concentration (mg/L)	Volume Added (mL)	Final Volume (mL)	Final Concentration (mg/L)
Mercury (Hg)	1000	0.25	500	0.50

Storage: Store in a cool, dry, ventilated storage area with acid resistant floors and good drainage. Store away from sunlight, heat, water, and incompatible materials.
Expiration: Due to mercury requirements, prepare this solution every 28 days or sooner as needed. The expiration date must not exceed the expiration date of any of the components.

8.0 Sample Collection, Preservation, Shipment, and Storage

8.1 Aqueous Samples

8.1.1 Total Metals

Aqueous samples are routinely collected in 250mL plastic containers containing 3mL of a 1:3 nitric acid preservative. The preservative should be sufficient to achieve a sample pH of less than 2.

Although no temperature preservation is required, samples are routinely iced at the time of collection at 4°C (less than 6°C but not frozen). Samples are stored at room temperature until the time of digestion. Samples must be digested and analyzed within 180 days of sample collection. If mercury is requested the samples must be digested and analyzed within 28 days of collection. Digestates are stored at room temperature until the time of analysis.

NCMs must be initiated for samples collected in improper containers and containing improper or insufficient preservatives.

Note: The holding time for samples from North Carolina that require Standard Method 3030C digestion is 72 hours; that is, the digestion must be initiated within 72 hours of collection.

8.1.2 Dissolved Metals

Aqueous samples for dissolved metals are routinely filtered at the time of sampling and collected in 500mL plastic containers containing 3mL of a 1:3 nitric acid preservative. The preservative should be sufficient to achieve a sample pH of less than 2.

Although no temperature preservation is required, samples are routinely iced at the time of collection at 4°C (less than 6°C but not frozen). Samples are stored at room temperature until the time of digestion. Samples must be digested and analyzed within 180 days of sample collection. If mercury is requested the samples must be digested and analyzed within 28 days of collection. Digestates are stored at room temperature until the time of analysis.

Note: If the sample is to be filtered in the laboratory, the sample must be collected in 500-mL plastic container with no preservatives. The sample must be stored at 4°C (less than 6°C but not frozen) until filtered. Once filtered, the laboratory will add nitric acid to obtain a pH of less than 2.

NCMs must be initiated for samples collected in improper containers and containing improper or insufficient preservatives.

Note: The holding time for samples from North Carolina that require Standard Method 3030C digestion is 72 hours; that is, the digestion must be initiated within 72 hours of collection.

8.1.3 Silica Samples

Aqueous samples for silica are routinely collected in 500-mL plastic containers.

Samples must be iced at the time of collection and maintained at 4°C (less than 6°C but not frozen) until the time of filtration and analysis. Samples must be filtered and analyzed within 180 days of collection.

NCMs must be initiated for samples collected in improper containers.

Note: Acid preserved samples routinely collected for ICP analyses and acid digested samples should not be used for the analysis of silica. Also note that glass containers should be avoided.

8.1.4 Preservation Checks – pH Verification

For each sample, prior to sample preparation,
- Place a piece of pH paper in a disposable medicine cup.
- Pour a few drops of sample into the medicine cup and note the color change of the pH paper.
- If the pH is outside the range of less than 2, initiate a Nonconformance Memo. Adjust the sample pH to less than 2 using 1:1 nitric acid.

- Mix well and hold for 24 hours. If pH is still greater than 2 after 24 hours, then repeat this process.

Note: To avoid cross-contamination, use a separate medicine cup and piece of pH paper per sample. Do not dip the pH paper into the sample container. The pH paper dye may bleed into the sample and affect sample results.

Note: Samples that are not at pH <2 upon arrival in the lab may contain cyanide or sulfide or may be highly buffered. Working under a hood minimizes the hazard that may be caused by the evolution of hydrogen cyanide or hydrogen sulfide upon acidification of the sample. Be aware that acid/base neutralization reaction may be violent and evolve a significant amount of heat.

8.2 TCLP/SPLP Leachate Samples

Once the TCLP/SPLP extraction procedure has been performed, the leachate is transferred to a plastic container. The leachate must be acidified with nitric acid to pH <2, upon receipt to the Metals Department. The leachate sample must be digested and analyzed within 180 days of completion of the TCLP/SPLP extraction. If mercury is requested, the leachate sample must be digested and analyzed within 28 days of completion of the TCLP/SPLP extraction.

9.0 Quality Control

SOP SA-QA-17: *Evaluation of Batch QC Data* and the SOP Summary in Attachment 3 of the associated analytical SOPs provide requirements for evaluating QC data.

9.1 Batch QC

A digestion batch consists of up to 20 environmental samples and the associated QC items digested together within a 24 hour period.

EPA 200.7 and EPA 200.8 – Drinking Water

The laboratory's default minimum QC items performed for each digestion batch are: a method blank and a laboratory control sample (LCS), a low-level LCS (LLCS), a matrix spike (MS) to be performed on a minimum of 10% of samples or one per batch – whichever is greater, and a matrix spike duplicate (MSD).

This frequency equates to the following:

- For a batch of 10 or fewer samples, the minimum QC items are a method blank, an LCS, an LLCS, 1 matrix spike, and 1 matrix spike duplicate.
- For a batch of 11-20 samples, the minimum QC items are a method blank, an LCS, an LLCS, 1 matrix spike (from sample 1-10), another matrix spike (from sample 11-20), and a matrix spike duplicate (MSD).

EPA 200.7 and EPA 200.8 – Clean Water Act

The laboratory's default minimum QC items performed for each digestion batch are: a method blank and a laboratory control sample (LCS), a matrix spike (MS) to be performed on a minimum of 10% of samples or one per batch – whichever is greater, and a matrix spike duplicate (MSD).

This frequency equates to the following:

- For a batch of 10 or fewer samples, the minimum QC items are a method blank, an LCS, 1 matrix spike, and a matrix spike duplicate.
- For a batch of 11-20 samples, the minimum QC items are a method blank, an LCS, 1 matrix spike (from sample 1-10), another matrix spike (from sample 11-20), and a matrix spike duplicate.

EPA Methods 3005A and 3010A and Standard Methods 3030C
The laboratory's default minimum QC items performed for each digestion batch are: a method blank, a laboratory control sample (LCS), a matrix spike (MS), and a matrix spike duplicate (MSD) or a sample duplicate.

The routine container supplied for this method is a 250mL container. 50mL is required for extraction. Reduced sample initial volumes may be necessary to achieve the required batch matrix spike frequency; however, the minimum extraction volume to be used for the matrix spike samples is 25mL. Note: Final volumes and spike amounts must be adjusted to compensate for these reduced initial volumes.

If there is insufficient sample volume to perform the required matrix spike(s), the LCS must be prepared in duplicate (i.e., LCSD). An NCM must be initiated on all affected samples to denote this situation. Insufficient sample volume is defined as receiving less than a total of 100mL.

Note: If an LCS and LCSD are performed, both QC items must be evaluated and reported. Acceptable recoveries (as well as %RPD) for both LCS and LCSD are required.

9.2 Instrument QC

Details on instrument QC are given in the associated analytical SOPs listed in Section 1.

9.2.1 Turbidimeter Instrument QC

9.2.1.1 Turbidimeter Initial Calibration (ICAL)

The instrument must be calibrated quarterly (every 3 months) in accordance with SOP SA-QA-16: *Evaluation of Calibration Curves.* This SOP provides requirements for establishing the calibration curve and gives the applicable formulas.

Instrument calibration is performed by analyzing a series of known standards. The calibration curve must consist of a minimum of 5 standards and a blank. The lowest level calibration standard must be at or below the reporting limit, and the remaining standards will define the working range of the analytical system.

The initial calibration standard concentrations currently in use in the laboratory are as follows:

Standard Level	Concentration (NTU)

1	20
2	200
3	1000
4	4000
5	7500

Refer to Section 7.3 for the standard preparation instructions. Other standard concentrations may be used provided they support the reporting limit and are fully documented in accordance with SOP SA-AN-041.

Fill the sample cell with 30mL turbidity-free DI water. Apply a thin bead of silicone oil to the cell and spread uniformly. Remove excess oil with a Kimwipe. Place the sample cell in the instrument and press 'enter'. The instrument will give a result after 60 seconds. Repeat for each Formazin calibration standard.

9.2.1.2 Turbidimeter Second Source Initial Calibration Verification (ICV)

The calibration curve must be verified initially – prior to any sample analyses – in accordance with SOP SA-QA-16 with a standard obtained from a second source.

Determine the turbidity of the Gelex standards by applying silicone oil to the prepared standard and placing in instrument. Place lowest NTU Gelex standard in the instrument first and press 'enter'. The value displayed is used to calculate the true value of the standard. The turbidity of each Gelex standard must be determined three times. The average of these three readings will be used as the true value for the upcoming quarter. The average must be within 15% of the first true value calculated. Repeat for each Gelex standard.

9.2.1.3 Turbidimeter Initial Calibration Blank (ICB) / Continuing Calibration Blank (CCB)

The instrument must be shown to be free from contamination by the analysis of calibration blanks. Initial calibration blanks are analyzed immediately following the initial calibration. Continuing calibration blanks are analyzed after each CCV.

Initial and continuing calibration blanks must be <RL to be acceptable.

If the blank is significantly above 0.50 NTU, the sample cell may be scratched and may need to be replaced.

9.2.1.4 Turbidimeter Continuing Calibration Verification

The initial calibration curve must be verified initially, after every ten samples, and at the end of the analysis with low level Gelex standard (0-2NTU) standard.

The CCV must be within 10% of the true value to be acceptable.

The continuing calibration verification standard concentration currently in use in the laboratory is the low level Gelex standard (0-2NTU) standard.

Note: This continuing calibration verification (CCV) is a second source standard and can also be referred to as the quality control sample (QCS) or the laboratory control sample (LCS).

9.2.1.5 Turbidimeter Reporting Limit Check Standard

The instrument must be verified at the beginning of each batch with a reporting limit check standard.

The RL check standard must be within 50% of the true value to be acceptable.

The RL check standard concentration currently in use in the laboratory is 0.1NTU. Refer to Section 7.3.5 for the standard preparation instructions. Another standard concentration may be used provided the reporting limit for the associated samples are elevated and the standard is fully documented in accordance with SOP SA-AN-041.

9.2.1.6 Turbidimeter Linear Calibration Range

For EPA Method 180.1, the linear calibration range (LCR) must be determined initially as part of an analyst's initial demonstration of capability and verified every 6 months thereafter. The initial demonstration of linearity uses as many standards as necessary to insure the curve is linear. The continued demonstration of linearity consists of a minimum of 3 standards and a blank.

For the linearity to be verified the standards must be within +/- 10% of the initial value to be acceptable. If this criterion is not met, the initial demonstration of linearity must be performed.

Note: This study is not meant to extend the linear range of the calibration. If turbidity is measured at a concentration greater than 40NTU, the sample must be analyzed at a dilution to bring the target analyte into the range of the initial calibration.

9.3 Corrective Action for Out-of-Control Data

When the quality control parameters do not meet the criteria set forth in this SOP, corrective action must be taken in accordance with SOP SA-QA-05: *Preventive and Corrective Action Procedures* the QC Summary Table in Attachment 3 of the associated analytical SOP. SOP SA-QA-05 provides contingencies for out-of-control data and gives guidance for exceptionally permitting departures from approved policies and procedures. Nonconformance Memos must be initiated to document all instances where QC criteria are not met and all departures from approved policies and procedures.

10.0 Procedure

10.1 Sample and QC Sample Preparation

Unless otherwise requested, groundwater and surface waters are to be prepared using the total recoverable metals procedure given in Section 10.1.3. TCLP/SPLP samples must be digested for total metals (Section 10.1.4). Note that there are different spiking solutions for routine and TCLP/SPLP matrix spikes.

10.1.1 Dissolved Metals

10.1.1.1 Results reported as "dissolved metals" are defined as the concentrations of analytes in a liquid sample that will pass through a 0.45um membrane filter prior to acidification/preservation. The filtration step can occur in the field or in the laboratory. In either case, the sample must be unpreserved prior to filtration and then filtered using a 0.45um filter.

10.1.1.2 Field filtered samples are usually preserved in the field, immediately after filtration. Unpreserved samples sent to the laboratory for lab filtration must be preserved immediately after filtration.

10.1.1.3 If the laboratory filters the samples, the reagent water used to prepare the method blank and laboratory control samples associated with the "dissolved metals" samples must be filtered through the same type of filter used to filter the field samples.

Note: The LCS should be spiked prior to filtration.

10.1.1.4 A comparison between total and dissolved results is good practice, and logic should follow that total results should be equal or greater than dissolved results, especially if the dissolved metals are lab filtered. Comparable hits with less than 20% RPD should be considered equal. Any other discrepancies should be discussed with the Department Manager or Supervisor, Technical Manager, or the Project Manager.

10.1.1.5 The laboratory's standard practice is to digest all non-silica liquid samples; however, samples filtered for the determination of dissolved metals do not require digestion if the sample:

- has a low COD (<20mg/L);
- has a turbidity <1 NTU;
- is colorless with no significant odor; ***and***
- is of one liquid phase and free of suspended particulates or precipitates after acidification

10.1.2 Turbidity Determination for Drinking Water Samples

Drinking water samples with a turbidity concentration of less than 1NTU may be analyzed without digestion. The exception to this rule is silver, which requires sample digestion prior to analysis. The turbidity of drinking water samples is checked using the procedures outlined in this section. If the turbidity of the sample is not checked, the digestion procedure must be performed.

10.1.2.1 Turn on turbidimeter and allow it to warm up 15-30 minutes.

10.1.2.2 Verify the instrument is operating properly by the analysis of a continuing calibration verification sample initially, after every 25 samples, and at the end of the run with the lowest available Gelex standard (0-2). The Gelex standard must recover within ±10% of the true value to be acceptable.

10.1.2.3 Analyze a DI water blank (CCB). The DI water blank must be less than 1NTU to be acceptable. If the DI water blank is greater the 1NTU, check to be certain the sample cell is clean. If the blank is significantly above 1NTU, the sample cell may be scratched and may need to be replaced.

10.1.2.4 Determine the turbidities for all samples by filling the cell with sample and placing the cell in the sample well of the turbidimeter. Record these turbidities in the Metals Turbidity Logbook. Be certain to rinse the cells between samples with DI water to avoid contamination.

10.1.3 Total Recoverable Metals (EPA 3005A)

This digestion procedure is used to prepare aqueous samples for total recoverable metals determination by ICP and ICP/MS.

10.1.3.1 Transfer a 50mL aliquot (or an appropriate volume diluted to 50mL with reagent water) of a well-mixed sample to a 50mL digestion block vial.

Note: If there is not sufficient volume to use a 50mL aliquot, the lab can use a smaller volume of sample, proportional volumes of reagents, and adjust the final digestate volume back to the original volume of the sample used. For example, if 25mL of sample is digested, one half of the routine volumes of reagent are used and the final volume of the digestate is brought back to 25mL. When a smaller aliquot is used, the digestion analyst must be careful not to allow the sample digest to evaporate completely.

10.1.3.2 Add 50mL of reagent water to a digestion block vial to serve as the method blank. This QC sample is taken through all digestion and sample preparation steps to monitor for contamination that may be due to glassware, reagents, or sample handling.

10.1.3.3 Add 0.50mL of the appropriate spiking solutions to a 50mL aliquot of reagent water to serve as the laboratory control spike (LCS). If a duplicate laboratory control spike (LCSD) is required, spike a second 50mL aliquot of reagent water with 0.50mL of the appropriate spiking solutions.

Analytical Method	Matrix	QC	Spiking Solutions
6010	Aqueous	LCS/MS/MSD	(Spex) ICP Spike 1 ICP Spike 2
6020	Aqueous	LCS/MS/MSD	ICP/MS Spike 1 ICP/MS Spike 2

10.1.3.4 Add 0.50mL of the appropriate spiking solutions to each of two 50mL aliquots of the client sample designated as the matrix spike samples (MS and MSD).

10.1.3.5 Add 2.5mL of concentrated HCl and 1.0mL of concentrated HNO_3 to each sample. Gently heat the digestion vessel until the sample refluxes. The sample must not be heated to boiling; that is, bubbles are not formed in the liquid in the bottom of the digestion vessel. The sample/acid solution is refluxing when the liquid evaporates and drops of liquid condense on the sides of the digestion vessel and fall back into the digestion vessel. Evaporate the sample until the volume is

approximately 15mL. Do not allow any portion of the vessel bottom to become dry at any time during the digestion.

Note: If a volume of sample smaller than 50mL is digested, the amount of acid should be reduced proportionately.

10.1.3.6 Wash down the inside of the digestion vessel with reagent water. Dilute the sample digestate to 50mL with reagent water.

Note: The digestate may be diluted to a volume less than the original volume if sample pre-concentration is required to meet lower reporting limits. The pre-concentration must be limited to a factor of four.

10.1.3.7 The sample is now ready for analysis via the appropriate analytical SOP.

10.1.4 Total Metals (EPA 3010, EPA 200.7, and EPA 200.8)

This digestion procedure is used for the preparation of aqueous samples for total metal determination by ICP and ICP/MS.

10.1.4.1 Transfer a 50mL aliquot (or an appropriate volume diluted to 50mL with reagent water) of a well-mixed sample to a 50mL digestion block vial.

Note: If there is not sufficient volume to use a 50mL aliquot, the lab can use a smaller volume of sample, proportional volumes of reagents, and adjust the final digestate volume back to the original volume of the sample used. For example, if 25mL of sample is digested, one half of the routine volumes of reagent are used and the final volume of the digestate is brought back to 25mL. When a smaller aliquot is used, the digestion analyst must be careful not to allow the sample digest to evaporate completely.

10.1.4.2 Add 50mL of reagent water to a digestion block vial to serve as the method blank. This QC sample is taken through all digestion and sample preparation steps to monitor for contamination that may be due to glassware, reagents, or sample handling.

10.1.4.3 Add 0.50mL of the appropriate spiking solutions to a 50mL aliquot of reagent water to serve as the laboratory control spike (LCS). If a duplicate laboratory control spike (LCSD) is required, spike a second 50mL aliquot of reagent water with 0.50mL of the appropriate spiking solutions.

Analytical Method	Matrix	QC	Spiking Solutions
6010 / 200.7	Aqueous	LCS/MS/MSD	(Spex) ICP Spike 1 ICP Spike 2
6020 / 200.8	Aqueous	LCS/MS/MSD	ICP/MS Spike 1 ICP/MS Spike 2

10.1.4.4 Add 0.50mL of the appropriate spiking solutions to two 50mL aliquots of the client sample designated as the matrix spike samples (MS and MSD).

10.1.4.5 Add 1.5mL of concentrated HNO_3 to each sample. Gently heat the digestion vessel until the sample refluxes. The sample must not be heated to boiling; that is, bubbles are not formed in the liquid in the bottom of the digestion vessel. The sample/acid solution is refluxing when the liquid evaporates and drops of liquid condense on the sides of the digestion vessel and fall back into the digestion vessel. Evaporate the sample until the volume is approximately 5-10mL. Do not allow any portion of the vessel bottom to become dry at any time during the digestion.

Note: If a volume of sample smaller than 50mL is digested, the amount of acid should be reduced proportionately.

10.1.4.6 Remove the digestion vessels from the digestion block and cool the digestion vessels to room temperature. Add another 1.5mL portion of concentrated HNO_3. Continue heating the sample on the digestion block. Again, at the proper temperature, the sample should gently reflux in the digestion vessel. Do not allow the sample to boil.

10.1.4.7 Continue heating the sample and adding additional 1.5mL portions of concentrated HNO_3 until the digestate is light in color or does not change in appearance after subsequent additions of HNO_3. If a sample requires more than 6mL of acid to digest, contact the Department Manager for guidance.

10.1.4.8 Evaporate the digestate until the volume is approximately 5-10mL.

10.1.4.9 Add 2.5mL of concentrated HCl and warm the sample digestate for 15 minutes.

10.1.4.10 Wash down the inside of the digestion vessel with reagent water. Dilute the sample digestate to 50mL with reagent water.

Note: The digestate may be diluted to a volume less than the original volume if sample pre-concentration is required to meet lower reporting limits. The pre-concentration should be limited to a factor of four.

10.1.4.11 The sample is now ready for analysis via the appropriate analytical SOP.

10.1.5 Total Metals and TCLP/SPLP Samples (EPA 3010)

This digestion procedure is used for the preparation of TCLP/SPLP leachate samples for total metal determination by ICP. Note that the LCS is spiked with the routine analytes to allow the TCLP/SPLP samples to be digested along with aqueous samples.

10.1.5.1 Transfer a 5mL aliquot (diluted to 50mL) for TCLP or a 50mL aliquot for SPLP of a well-mixed sample to a clean 50mL digestion vessel. Larger volumes may be digested at the discretion of the lab. The volume of spike solution added should be adjusted proportionately.

10.1.5.2 Add 5mL of extraction fluid diluted to 50mL for TCLP or 50mL for SPLP to a digestion vessel to serve as the method blank. This QC sample is taken through all digestion and sample preparation steps to monitor for contamination that may

be due to glassware, reagents, or sample handling. A blank for each type of extraction fluid must be digested and analyzed.

10.1.5.3 Add 0.50mL of the appropriate spiking solutions to a 5mL aliquot of extraction fluid diluted to 50mL for TCLP or to a 50mL aliquot of extraction fluid for SPLP. This is designated as the laboratory control spike (LCS). If required, perform a LCSD by following the same procedure as the LCS. The routine ICP spiking solution is used for the TCLP/SPLP LCS because of frequent client requests for reporting more analytes from the TCLP/SPLP leachate than are currently regulated. Preparing an LCS with all of the target analytes eliminates re-digestion and provides QC for the requested analytes.

Note: If both Extraction Fluid 1 and Extraction Fluid 2 are included in the batch, use Extraction Fluid 1 for the LCS and LCSD. Refer to SOP EX15: *Toxicity Characteristic Leaching Procedure (TCLP) and Synthetic Precipitation Leaching Procedure (SPLP)* for information on the TCLP/SPLP extraction fluids.

Analytical Method	Matrix	QC	Spiking Solutions
6010	TCLP/SPLP	MS	(Spex) ICP Spike 1 ICP Spike 2
6010	TCLP/SPLP	LCS	(Spex) ICP Spike 1 ICP Spike 2

10.1.5.4 For the matrix spike, add 0.50mL of each ICP spiking solution (1 and 2) to a separate 5mL aliquot of the client sample (diluted to 50mL) for TCLP or to a separate 50mL aliquot of the client sample for SPLP designated as the matrix spike.

Note: The TCLP/SPLP digestion batch consists of twenty or fewer field samples and the associated QC items. A TCLP/SPLP digestion batch must not exceed 20 field samples. Every TCLP/SPLP digestion batch will have a method blank (MB), a laboratory control sample (LCS), and a matrix spike (MS).

10.1.5.5 Add 1.5mL of concentrated HNO_3 to each sample. Gently heat the digestion vessel until the sample refluxes. The sample is not heated to boiling; that is, bubbles are not formed in the liquid in the bottom of the digestion vessel. The sample/acid solution is refluxing when the liquid evaporates and drops of liquid condense on the sides of the digestion vessel and fall back into the digestion vessel. Evaporate the sample until the volume is approximately 5mL. Do not allow any portion of the vessel bottom to become dry at any time during the digestion.

Note: If a volume of sample smaller than 50mL is digested, the amount of acid should be reduced proportionately.

10.1.5.6 Remove the digestion vessels from the digestion block and cool the digestion vessels to room temperature. Add another 1.5mL portion of concentrated HNO_3. Continue heating the sample on the digestion block. Again, at the proper temperature, the sample should gently reflux in the digestion vessel. Do not allow the sample to boil.

10.1.5.7 Continue heating the sample and adding additional 1.5mL portions of concentrated HNO_3 until the digestate is light in color or does not change in appearance after subsequent additions of HNO_3. If a sample requires more than 6mL of acid to digest, contact the Department Manager for guidance.

10.1.5.8 Evaporate the digestate until the volume is approximately 5-10mL.

10.1.5.9 Add 2.5mL of concentrated HCl and warm the sample digestate for 15 minutes.

10.1.5.10 Wash down the inside of the digestion vessel with reagent water. Dilute the sample digestate to 50mL with reagent water.

10.1.5.11 The sample is now ready for analysis by ICP.

10.1.6 Acid-Extractable Metals (SM3030C)

This digestion procedure is used for the preparation of aqueous samples for acid-extractable metals by ICP and ICP/MS.

10.1.6.1 Transfer a 50mL aliquot (or an appropriate volume diluted to 50mL with reagent water) of a well-mixed sample to a 50mL digestion block vial.

Note: If there is not sufficient volume to use a 50mL aliquot, the lab can use a smaller volume of sample, proportional volumes of reagents, and adjust the final digestate volume back to the original volume of the sample used. For example, if 25mL of sample is digested, one half of the routine volumes of reagent are used and the final volume of the digestate is brought back to 25mL. When a smaller aliquot is used, the digestion analyst must be careful not to allow the sample digest to evaporate completely.

10.1.6.2 Add 50mL of reagent water to a beaker that has been designated as the method blank. This QC sample is taken through all digestion and sample preparation steps to monitor for contamination that may be due to glassware, reagents, or sample handling.

10.1.6.3 Add 0.50mL of the appropriate spiking solutions to a 50mL aliquot of reagent water designated as the laboratory control spike (LCS). If a duplicate laboratory control spike (LCSD) is required, spike a second 50mL aliquot of reagent water with 0.50mL of the appropriate spiking solutions.

Analytical Method	Matrix	QC	Spiking Solutions
6010	Aqueous	LCS/MS/MSD	(Spex) ICP Spike 1 ICP Spike 2
6020	Aqueous	LCS/MS/MSD	ICP/MS Spike 1 ICP/MS Spike 2

10.1.6.4 Add 0.50mL of the appropriate spiking solutions to each of two 50mL aliquots of the client sample designated as the matrix spikes sample (MS and MSD).

10.1.6.5 Add 2.5mL of 1:1 HCl to each sample and QC item.

Note: If a volume of sample smaller than 50mL is digested, the amount of acid should be reduced proportionately.

10.1.6.6 Heat for 15 minutes on a hot block.

10.1.6.7 Allow the digest to cool and filter through a 0.45um filter. Adjust the final volume to 50mL or to the original volume.

10.1.6.8 The sample is now ready for analysis by ICP or ICP/MS.

10.1.7 Total Recoverable Metals (EPA 200.8 and EPA 200.7)

This digestion procedure is used for the preparation of water and drinking water samples for total recoverable metal determination by ICP and ICP/MS.

10.1.7.1 Transfer a 50mL aliquot (or an appropriate volume diluted to 50mL with reagent water) of a well-mixed sample to a clean 50mL block digestion vial.

Note: If there is not sufficient volume to use a 50mL aliquot, the lab can use a smaller volume of sample and bring the final digestate volume back to the original volume of the sample used. That is, if 25mL of sample is digested, the final volume of the digestate should be brought back to 25mL. If a smaller aliquot is used, the digestion analyst must be careful not to allow the sample digest to evaporate completely.

10.1.7.2 Add 50mL of reagent water to a block digestion vial that has been designated as the method blank. This QC sample is taken through all digestion and sample preparation steps to monitor for contamination that may be due to glassware, reagents, or sample handling.

10.1.7.3 Add 0.50mL of the appropriate spiking solutions to a 50mL aliquot of reagent water designated as the laboratory control spike (LCS). If a duplicate laboratory control spike (LCSD) is required, spike a second 50mL aliquot of reagent water with 0.50mL of the appropriate spiking solutions.

Analytical Method	Matrix	QC	Spiking Solutions
200.7 DW and TR	Aqueous	LCS/MS/MSD	(Spex) ICP Spike 1 ICP Spike 2
200.8 DW and TR	Aqueous	LCS/MS/MSD	ICP/MS Spike 1 ICP/MS Spike 2

10.1.7.4 Add 0.50mL of the appropriate spiking solutions to each of two 50mL aliquots of the client sample designated as the matrix spikes sample (MS and MSD).

10.1.7.5 Add 1.0 mL of (1:1) HNO_3 and 0.5 mL of (1:1) HCl to each sample. Gently heat the block digestion vial and reduce the sample volume to about 10mL without boiling; that is, bubbles are not formed in the liquid in the bottom of the block digestion vial. Do not allow any portion of the vessel bottom to become dry at any time during the digestion.

Note: If a volume of sample smaller than 50mL is digested, the amount of acid should be reduced proportionately.

10.1.7.6 Wash down the inside of the block digestion vial with reagent water. Dilute the sample digestate to 50mL with reagent water. Transfer the digest to a labeled storage container, usually a 125mL plastic vial. Allow any undissolved material to settle out or centrifuge the sample to remove particulate matter. A portion of the sample may be filtered, if necessary, to remove particulates but care must be taken to avoid contamination of the sample during filtration.

Note: The digestate may be diluted to a volume less than the original volume if sample concentration is required to meet lower reporting limits. The pre-concentration should be limited to a factor of four.

10.1.7.7 The sample is now ready for analysis by ICP and ICP/MS.

10.1.8 Silica Samples

10.1.8.1 Samples are prepared by filtration of an unpreserved aliquot through a 0.45um syringe filter. The sample must be kept refrigerated until time of analysis. Note: The same number of syringe filters should be used for the method blank, LCS, and the samples.

10.1.8.2 The method blank is prepared by filtering an amount of reagent water equal to the volume of sample that is filtered.

10.1.8.3 The LCS is prepared by spiking 10mL of reagent water with 0.10mL of SiO_2 Intermediate Standard, and passing it through a 0.45um syringe filter. The SiO_2 spike concentration in the LCS is 10mg/L.

10.1.8.5 The matrix spike and/or matrix spike duplicate (MS/MSD) are prepared by adding 0.10mL of SiO_2 Intermediate Standard solution to two separate 10mL aliquots of the sample to be spiked and filtering through a 0.45um filter. The SiO_2 spike concentration in the MS/MSD is 10mg/L.

10.2 Analysis

Details on sample analysis are given in the associated analytical SOPs listed in Section 1.

11.0 Calculations / Data Reduction

11.1 Data Reduction

Data must be evaluated in accordance with SOP SA-QA-02: *Data Generation and Review*.

11.1.1 Historical Data

Many of the laboratory's clients submit samples for repeat monitoring purposes. Prior to analysis, verify LIMS Worksheet Notes and/or use the Historical Data Tracker feature to determine if historical data is available for review.

11.1.2 Chemical Relationships

When available, the following chemical relationships must be evaluated for each sample. If these relationships are not met the Department Manager must be contacted immediately.

- Total results are ≥ Dissolved results

11.2 Calculations

Details on sample calculations are given in the associated analytical SOPs listed in Section 1.

12.0 Method Performance

12.1 Reporting Limit Verification (RLV)

At a minimum, RLVs must be performed initially upon method set-up in accordance with SOP SA-QA-07: *Determination and Verification of Detection and Reporting Limits.*

For analytes and methods certified by DOD ELAP, RLVs must also be performed quarterly thereafter. For analytes and methods certified by NELAC, RLVs must also be performed annually thereafter. Exceptions may be made for project-specific non-routine analytes.

12.2 Method Detection Limit (MDL) Study

The 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. 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 current MDLs associated with this procedure are given in the Method Limit Group (MLG) in TALS.

At a minimum, MDL Studies must be performed initially upon method set-up in accordance with SOP SA-QA-07: *Determination and Verification of Detection and Reporting Limits.*

Note: MDL Studies are not required for non-routine analytes provided results are not reported below the RL (i.e., MDL equals RL in TALS).

12.3 Method Detection Limit Verification (MDLV)

At a minimum, MDLVs must be performed initially upon method set-up in accordance with SOP SA-QA-07: *Determination and Verification of Detection and Reporting Limits.*

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For analytes and methods certified by DOD ELAP, MDLVs must also be performed quarterly thereafter. For analytes and methods certified by NELAC, MDLVs must also be performed annually thereafter.

Note: MDLVs are not required for non-routine analytes provided results are not reported below the RL (i.e., MDL equals RL in TALS).

12.4 Determination of the Instrument Detection Limit (IDL)

The instrument detection limit (IDL) is the concentration of analyte that can be statistically distinguished from the background noise of the instrument. The IDL limit must be determined annually, at a minimum, for each analyte in accordance with SOP SA-QA-07: *Determination and Verification of Detection and Reporting Limits.*

The IDL is defined as three times the standard deviation of seven replicate analyses of a blank solution analyzed over three non-consecutive days. The IDL may be elevated above the background noise (blank levels). The current IDL associated with this procedure is given in the Equipment Limit Group (ELG) in LIMS.

12.5 QC Limit Generation, Control Charting, and Trend Analysis

The control limits for the batch QC items (LCS and MS/MSD) for this procedure are specified in the reference method and cannot be broadened; therefore, the laboratory defaults to the method-defined limits and does not utilize in-house or laboratory-derived limits for the evaluation of batch QC items.

Although the laboratory must default to the method-defined QC limits, control charting is a useful tool and is performed to assess analyte recoveries over time to evaluate trends. Control charting must be performed periodically (at a minimum annually) in accordance with SOP SA-QA-17: *Evaluation of Batch QC Data.*

12.6 Demonstrations of Capability

Initial and continuing demonstration of capability must be performed in accordance with SOP SA-QA-06: *Training Procedures.*

Prior to performing this procedure unsupervised, each new analyst who performs this analysis must demonstrate proficiency per method/analyte combination by successful completion of an initial demonstration of capability. The IDOC is performed by the analysis of 4 consecutive LCSs that meet the method criteria for accuracy and precision. The LCSs must be from a second source than that used to prepare the calibration standards. The IDOC must be documented on the IDOC Form shown in SOP SA-QA-06 with documentation routed to the QA Department for filing.

Annual continuing demonstrations of capability (CDOCs) are also required per analyst per method/analyte combination. The CDOC requirement may be met by the consecutive analysis of four LCS all in the same batch, by the analysis of four LCS analyzed in four consecutive batches (in different batches on different days), via acceptable results on a PT study, or analysis of client samples with statistically indistinguishable results when compared to another certified analyst. The CDOC must be documented and routed to the QA Department for filing.

12.7 Training Requirements

All training must be performed and documented in accordance with SOP SA-QA-06: *Training Procedures.*

Note: The SOPs listed in the Reference/Cross-Reference Section are applicable to this procedure. All employees performing this procedure must also be trained on these SOPs, and/or have a general understanding of these procedures, as applicable.

13.0 Pollution Control

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (e.g., examining recycling options, ordering chemicals based on quantity needed, preparing reagents based on anticipated usage and reagent stability, etc.). Employees must abide by the policies in Section 13 of the Environmental Health and Safety Manual.

This procedure has been evaluated for opportunities to minimize the waste generated. Where reasonably feasible, pollution control procedures have been incorporated.

14.0 Waste Management

Waste management practices must be conducted consistent with all applicable federal, state, and local rules and regulations. All waste (i.e., excess reagents, samples, and method process wastes) must be disposed of in accordance with Section 9 of the TestAmerica Savannah Addendum to the EHSM. Waste description rules and land disposal restrictions must be followed.

14.1 Waste Streams Produced by the Method

The following waste streams are produced when this method is carried out:

- Excess aqueous samples – Dispose according to characterization on the sample disposal sheets. Neutralize non-hazardous samples before disposal into drain/sewer. Transfer hazardous samples (identified on disposal sheets) to the waste department for disposal.
- Acidic sample digestions: Neutralize before disposal into drain/sewer system.

Dispose of all metals standards with concentrations exceeding the TCLP threshold limits as a RCRA aqueous hazardous waste. The TCLP constituents and the respective threshold limits are:

Analyte	Disposal Limit (mg/L)
Arsenic	5.0
Barium	100
Cadmium	1.0
Chromium	5.0
Lead	5.0
Selenium	1.0
Silver	5.0
Mercury	0.20

Neutralize the standard by passing with sodium bicarbonate before transferring the waste to the RCRA aqueous hazardous waste drum.

All other metals standards may be disposed, using copious amounts of water, down a sink that flows to the neutralization pit.

15.0 References / Cross-References

- SOP SA-AN-100: *Laboratory Support Equipment (Verification and Use)*
- SOP SA-AN-41: *Reagent and Standard Materials Procedures*
- SOP SA-QA-02: *Data Generation and Review*
- SOP SA-QA-05: *Preventive and Corrective Action Procedures*
- SOP SA-QA-06: *Training Procedures*
- SOP SA-QA-07: *Determination and Verification of Detection and Reporting Limits*
- SOP SA-QA-15: *Homogenization, Compositing, and Segregation of Samples*
- SOP SA-QA-17: *Evaluation of Batch QC Data*
- TestAmerica Savannah Quality Assurance Manual
- TestAmerica Environmental Health and Safety Manual (CW-E-M-001)
- TestAmerica Savannah Addendum to the Environmental Health and Safety Manual
- Methods 3005A and 3010A: *Test Methods for Evaluating Solid Waste*, Third Edition, SW-846; vs. EPA Office of Solid Waste and Emergency Response: Washington, DC.
- Methods 200.7 and 200.8 (Drinking Water): Methods for the Determination of Metals in Environmental Samples, May 1994, Supplement 1. (EPA 600/R-94/111).
- Standard Methods for the Examination of Water and Wastewater, Online Edition; American Public Health Association: Washington, DC 2004.

16.0 Method Modifications

16.1 The EPA 200.7 and EPA 200.8 reference methods were written specifically for drinking water and source water samples; however, the laboratory may perform other types of water samples using this procedure.

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16.2 The EPA Manual for the Certification of Laboratories Analyzing Drinking Water requires a LFB at the MRL to be performed each day. The laboratory meets this requirement by preparing an LCS at the RL in each EPA 200.7 and EPA 200.8 drinking water batch. The EPA DW Manual does not specify criteria for the low-level LCS; therefore, the laboratory defaults to qualitative identification of the LL-LCS to be acceptable.

16.3 There is no method-defined batch precision requirement for some of these methods. The EPA does require precision for all samples analyzed under the Clean Water Act, and the laboratory routinely performs an MSD to support clients who require precision to be reported. If insufficient sample volume is provided to perform the MS/MSD, the laboratory performs the LCS in duplicate (i.e., LCS/LCSD).

17.0 Attachments

The following Tables, Diagrams, and/or Validation Data are included as Attachments:

Attachment 1: SOP Summary
Attachment 2: Sample Collection, Preservation, and Holding Time Table
Attachment 3: QC Summary
Attachment 4: Instrument Maintenance and Troubleshooting
Attachment 5: Turbidity Curve Form
Attachment 6: Turbidity Form

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Attachment 1
SOP Summary

Sample Preparation Summary
Total Metals and TCLP/SPLP leachates: A known volume, usually 50mL, of sample is transferred to a digestion vessel. The sample is refluxed with nitric acid at approximately 95°C. After the sample has digested, as evidenced by a clear, pale yellow color, HCl is added and the sample is brought up to the original volume with reagent water. The laboratory utilizes two versions of the Total Recoverable preparation procedure. One version is equivalent to the EPA 3005A procedure and the second procedure is equivalent to the EPA 200.7 and 200.8 procedures.

Total Recoverable Metals: A known volume, usually 50mL, of sample is transferred to a digestion vessel. The sample is refluxed with dilute nitric acid and hydrochloric acid at approximately 95°C. After the sample has evaporated to approximately 10-20mL, the sample is brought up to the original volume with reagent water. This procedure is equivalent to EPA Method 3005A, and the EPA Methods 200.7 and 200.8 prep procedures for total recoverable metals.

Acid Extractable Metals: A known volume, usually 50mL of sample preserved with nitric acid, is transferred to a digestion vessel. Hydrochloric acid is added and the sample is heated on a hot block for 15 minutes. The sample is filtered and the final volume adjusted to the original volume. This procedure is equivalent to Standard Methods 3030C.

Silica samples for EPA Methods 200.7 and 6010C: Liquid samples are filtered. The filtered liquid samples are analyzed by ICP.

Drinking water samples for EPA Methods 200.7 and 200.8 with a turbidity concentration of less than 1NTU may be analyzed with no digestion. The exception to this rule is silver, which requires sample digestion prior to analysis. If the sample turbidity is >1NTU, then the EPA Method 200.7 preparation procedure or the EPA Method 200.8 preparation procedure is used.

Samples filtered for the determination of dissolved metals do not require digestion if the sample:
5) has a low COD(<20mg/L);
6) has a turbidity <1NTU ;
7) is colorless with no significant odor ; ***and***
8) is of one liquid phase and free of suspended particulates or precipitates after acidification.

Sample Analysis Summary
ICP: Sample digestates are aspirated and nebulized into a spray chamber. A stream of argon gas carries the sample aerosol through the innermost of three concentric tubes and injects it into the middle of the donut-shaped plasma. The sample elements are dissociated, atomized, and excited to a higher energy level. As the elements fall to a lower energy level, radiation characteristic of the elements present in the plasma is emitted. The light is directed through an entrance slit, dispersed by the diffraction grating, and projected on to the photomultiplier tube (PMT). The PMTs, located behind the exit

slits, convert the light energy to an electrical current. This signal is then digitized and processed by the data system. Background correction is required for trace element determination.

ICP/MS: Sample digestates are aspirated and nebulized into a spray chamber. A stream of argon gas carries the sample aerosol through the innermost of three concentric tubes and injects it into the middle of the donut-shaped plasma. The sample elements are dissociated, atomized, and excited to a higher energy level. The ions that are produced are entrained in the plasma gas and introduced, by means of an interface, into a mass spectrometer. The ions are sorted according to their mass to charge ratios and quantified with a channel mass spectrometer.

Analytical Sequence
See the appropriate analytical SOP for information on the analytical sequence.
ICP: SOP SA-ME-070: *Elements by ICP*
ICP/MS: SOP SA-ME-074: *Elements by ICP/MS*

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Attachment 2:
Sample Collection, Preservation, and Holding Time Table

Matrix	Routine Sample Container	Routine Sample Size	Minimum Sample Size	Dechlorination Agent	Chemical Preservation	Thermal Preservation	Holding Time[2]
Water	250mL plastic	50mL	25mL	Not Applicable	1:1 HNO_3 to pH<2	Not Applicable	Mercury: 28 days from collection Other Metals: 180 days from collection Note: SM3030C has a 72 hour HT.
Water (Silica)	250mL plastic	50mL	25mL	Not Applicable	Not Applicable	4 °C[1]	180 days from collection
Leachate	100mL or 250mL plastic	50mL	25mL	Not Applicable	1:1 HNO_3 to pH<2	Not Applicable	Mercury: 28 days from leaching Other Metals: 180 days from leaching Note: SM3030C has a 72 hour HT.

[1]Samples are collected on ice and maintained at <6 °C with no frozen samples.
[2] Inclusive of digestion and analysis.

Note: If dissolved metals are requested, the sample must be filtered prior to the acid being added to the sample.

Attachment 3: QC Summary

QC Item	Frequency	Criteria	Corrective Action
Batch Definition	Digested together w/in 24-hr timeframe; not to exceed 20 field samples	Not Applicable	Not Applicable
Method Blank (MB)	One per batch	Refer to analytical SOP	Refer to analytical SOP
Lab Control Sample (LCS)	One per batch	Refer to analytical SOP	Refer to analytical SOP
Laboratory Control Sample Duplicate (LCSD)	One per batch, if insufficient sample provided for MS/MSD	LIMS MLG	Redigest and reanalyze batch
Low-Level Laboratory Control Sample (LLCS) – spiked at the RL	EPA 200.7 and EPA 200.8 DW: One per batch	Qualitatively identified	If the "regular" LCS meets criteria, initiate NCM and report data If the "regular" LCS does not meet criteria, redigest and reanalyze batch
Matrix Spike (MS)	EPA 200.7 and EPA 200.8: 10% of samples prepared; i.e., 2 separate matrix spikes per batch of twenty samples Other Methods: 5% of samples prepared; i.e., 1 matrix spike per batch of twenty samples	LIMS MLG	Flag and report data
Matrix Spike Duplicate (MSD) or Sample Duplicate (SD)	One per batch	LIMS MLG	Flag and report data
Initial Demonstration of Capability (IDOC)	Initially, per analyst, per analyte/method/matrix combination	Refer to SOP SA-QA-06	Refer to SOP SA-QA-06 Note: Unsupervised work must not

QC Item	Frequency	Criteria	Corrective Action
			begin until acceptable IDOC is obtained.
Continuing Demonstration of Capability (CDOC)	Annually, per analyst, per analyte/method combination	Refer to SOP SA-QA-06	Refer to SOP SA-QA-06
Reporting Limit Verification (RLV)	Upon method/instrument set-up, per analyte/method/matrix combination. Then quarterly thereafter (for DOD ELAP) or annually thereafter (for NELAC)	Refer to SOP SA-QA-07	Refer to SOP SA-QA-07
Method Detection Limit Study (MDL Study)	Upon method/instrument set-up, per analyte/method/matrix combination	Refer to SOP SA-QA-07	Refer to SOP SA-QA-07
MDL Verification (MDLV)	Upon method/instrument set-up, per analyte/method/matrix combination. Then quarterly thereafter (for DOD ELAP) or annually thereafter (for NELAC)	Refer to SOP SA-QA-07	Refer to SOP SA-QA-07

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Attachment 4:
Instrument Maintenance and Troubleshooting

Instrument Labeling
Each instrument must be labeled with its name or ID (e.g., MSA, ICP-D, etc.). Additionally, non-operational instruments must be isolated from service or marked as being out of service. Each piece of equipment has an "Operational / Not Operational" sticker that is used for this purpose.

Maintenance Log
A maintenance log must be established for each piece of equipment used in the laboratory.

All maintenance that is performed on the instrument must be recorded in the log including:

- analyst or technician performing the maintenance
- date the maintenance was performed
- detailed explanation of the reason for the maintenance
- resolution of the problem and return to control
- all service calls from instrument representatives

Preventive Maintenance
Refer to the instrument manufacturer's guides for trouble-shooting items.

The temperature of the hot plate or digestion block must be monitored with each batch. If the temperature required for sample preparation cannot be maintained, the heating device must be removed from service and repaired or replaced.

Troubleshooting
Troubleshooting should be documented as outlined above. If possible, troubleshooting is best performed in a step-wise manner to systematically isolate instrument components. Refer to the instrument manufacturer's guides for specific information and strategies. Enlist assistance from technical and/or department management as needed.

Contingency Plan
Maintenance contracts are carried for most instrumentation and close contact is maintained with service personnel to ensure optimal instrument functioning. An extensive spare parts inventory is maintained for routine repairs, consisting of thermocouples, digestion tubes, and turbidimeter cells, and other common instrumentation components. Since instrumentation is standardized throughout the laboratory network, spare parts and components can be readily exchanged among the network.

In general, the laboratory has at least one backup unit for each critical unit. In the event of instrument failure, portions of the sample load may be diverted to duplicate instrumentation, the analytical technique switched to an alternate approved technique (such as manual colorimetric determination as opposed to automated colorimetric determination), or samples shipped to another properly certified or approved TestAmerica location.

Attachment 5: Turbidity Curve Form

TURBIDITY CURVE
Method EPA 180.1 / SM 2130B

Analyst Initials: _____ Date: _____ Instrument ID: _____

Formazin Primary Standards Calibration

Check the box when the standard is analyzed and write the LIMS ID # in the blank.

☐ 20NTU _____ ☐ 200NTU _____

☐ 1000NTU _____ ☐ 4000NTU _____

☐ 7500NTU _____

Gelex Secondary Standards Calibration

Each Gelex standard is analyzed in triplicate and the average is used as the true value.

Gelex Criterion: Percent Difference <15%

1. Gelex Concentration: Stray Light LIMS ID #: _____

   a_____NTU First True Value =_____NTU

   b_____NTU Average = _____NTU

   c_____NTU Percent Difference = _____

2. Gelex Concentration: 0 – 2 NTU LIMS ID #: _____

   a_____NTU First True Value =_____NTU

   b_____NTU Average = _____NTU

   c_____NTU Percent Difference = _____

3. Gelex Concentration: 0 – 20 NTU LIMS ID #: _____

   a_____NTU First True Value =_____NTU

   b_____NTU Average = _____NTU

   c_____NTU Percent Difference = _____

4. Gelex Concentration: 0 – 200 NTU LIMS ID #:_____

   a_____NTU First True Value =_____NTU

   b_____NTU Average = _____NTU

   c_____NTU Percent Difference = _____

5. Gelex Concentration: 200 – 4000 NTU LIMS ID #: _____

   a_____NTU First True Value =_____NTU

   b_____NTU Average = _____NTU

   c_____NTU Percent Difference = _____

FGE167:08.06.08:4

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Attachment 6: Turbidity Form

Turbidity for Metals Drinking Water Samples

Date:

Analyst Initials:

CCV LIMS Standard Number:

CCV True Value: NTU

CCV criteria: ±10% of True Value
CCB criteria: <1 NTU
If sample turbidity is <1 NTU and silver is not requested, the sample does not require digestion.

Job Number	Turbidity (NTU)	Comments
CCV		
CCB		
CCV		
CCB		

FME158;09.19.07:1

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18.0 Revision History

Summary of Changes from Previous Revision:

- Minor editorial, grammatical, and formatting changes made. Boilerplate text added.
- Revised standards information to reflect current laboratory practice. Single element stock standards are no longer used; rather standards are no purchased directly from CPI. Revised preparation instructions and removed silver from ICP/MS Spiking Solution 2. Section 7.3.3.1, Section 7.3.3.2, and Section 7.3.3.3
- Added requirement to adjust TCLP/SPLP leachates to pH <2 upon receipt to the department. Section 8.3 (2011 Internal Audit Finding, CAR # 000301)

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ELEMENTS BY ICP

(Methods: EPA 200.7, EPA 6010B, EPA 6010C, and SM3120B)

Approvals (Signature/Date):

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1.0 Scope and Application

This SOP gives the procedures for the determination of metals (elements) by inductively coupled plasma (ICP) atomic emission spectroscopy.

The routine matrices for this procedure are waters and soils; however, this procedure may be adapted to accommodate other matrices as outlined in Section 16.1.

A complete target analyte list, the reporting limits (RL), the method detection limits (MDL), and the accuracy and precision criteria associated with this procedure are provided in the LIMS Method Limit Groups (MLGs).

This SOP was written by and for TestAmerica's Savannah laboratory.

2.0 Summary of Method

Prior to analysis by ICP, the sample must be digested/filtered using the sample preparation method appropriate to the analyte/matrix combination. Sample digestates/filtrates are aspirated and nebulized into a spray chamber. A stream of argon gas carries the sample aerosol through the innermost of three concentric tubes and injects it into the middle of the donut-shaped plasma. The sample elements are dissociated, atomized, and excited to a higher energy level. As the elements fall to a lower energy level, radiation characteristic of the elements present in the plasma is emitted. The light is directed through an entrance slit, dispersed by the diffraction grating, and projected on to the photomultiplier tube (PMT) or onto a charge-coupled device (CCD). The PMTs and CCDs, located behind the exit slits, convert the light energy to an electrical current. This signal is then digitized and processed by the data system. Background correction is required for trace element determination.

Note: Drinking water samples (EPA 200.7) only require digestion if the determination of silver (Ag) is requested or if the turbidity is greater than or equal to 1.0 NTU.

This SOP is based on the following methods: EPA 200.7, EPA 6010B, EPA 6010C, and SM2130B.

3.0 Definitions

Refer to the Glossary Section of the *Quality Assurance Manual* (QAM) for a complete listing of applicable definitions and acronyms.

4.0 Interferences

4.1 Procedural Interferences

4.1.1 Interferences may be caused by contaminants in solvents, reagents, glassware, and other sample processing apparatus and can make identification and/or quantification of the target analytes difficult.

4.1.2 All sample collection containers are single-use disposable containers which limits the potential for contamination. All non-disposable labware must be scrupulously cleaned in accordance with the posted Labware Cleaning Instructions to ensure it is free from contaminants and does not contribute artifacts.

4.1.3 High purity reagents and solvents are used to help minimize interference problems. Hydrochloric acid and nitric acid must be verified prior to use in accordance with the TestAmerica Solvent Lot Testing Program.

4.1.4 Instrument and/or method blanks are routinely used to demonstrate all reagents and apparatus are free from interferences under the conditions of the analysis.

4.1.5 Physical interferences are effects associated with the sample nebulization and transport processes. Changes in viscosity can cause significant inaccuracies, especially in samples containing high concentrations of dissolved solids or high acid concentrations. If physical interferences are present, they must be reduced by diluting the sample digestate, using a peristaltic pump, using the method of standard additions (MSA), or using an internal standard.

4.2 Matrix Interferences

4.2.1 Matrix interferences may be caused by contaminants that are co-extracted from the sample matrix. The sample may require dilution prior to analysis to reduce or eliminate the interferences.

4.2.2 Interfering contamination may occur when a sample containing low concentrations of analytes is analyzed immediately following a sample containing relatively high concentrations of analytes. As such, samples known to be clean should be analyzed first. To prevent carryover into subsequent samples, analysis of reagent blanks may be needed after the analysis of a sample containing high concentrations of analytes.

4.2.3 Spectral interferences are caused by the overlap of a spectral line from another element, unresolved overlap of molecular band spectra, background contribution from continuous phenomena, and stray light from the line emissions of highly concentrated elements.

4.2.3.1 Spectral overlap may be compensated for by the use of inter-element correction factors.

4.2.3.2 Background contribution and stray light can be compensated for by a background correction adjacent to the analyte line.

5.0 Safety

Employees must abide by the policies and procedures in the TestAmerica Environmental Health and Safety Manual (EHSM), the TestAmerica Savannah Addendum to the EHSM, and this document.

This procedure may involve hazardous materials, 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 to follow appropriate safety, waste disposal, and health practices under the assumption that all samples and reagents are potentially hazardous.

The analyst must protect himself/herself from exposure to the sample matrix. Many of the samples that are tested may contain hazardous chemical compounds or biological organisms. The analyst must, at a minimum, wear protective clothing (lab coat), eye protection (safety glasses or face shield), disposable latex or nitrile gloves, and closed-toe, nonabsorbent shoes when handling samples.

5.1 Specific Safety Concerns or Requirements

The ICP plasma emits strong UV light and is harmful to vision. All analysts must avoid looking directly at the plasma.

The plasma generates high temperatures. Analysts must ensure that all equipment is shut down and cooled off before performing maintenance and troubleshooting in the plasma area.

Nitric and hydrochloric acids are extremely hazardous as oxidizers, corrosives, poisons, and are reactive. Inhalation of the vapors can cause coughing, choking, irritation of the nose, throat, and respiratory tract, breathing difficulties, and lead to pneumonia and pulmonary edema. Contact with the skin can cause severe burns, redness, and pain. Nitric acid can cause deep ulcers, and staining of the skin to a yellow or yellow-brown color. These acid vapors are irritating and can cause damage to the eyes. Contact with the eyes can cause permanent damage.

Samples that contain high concentrations of carbonates or organic matter, or samples that are at elevated pH can react violently when acids are added. Acids must be added to samples under a hood to avoid splash/splatter hazards and/or possibly toxic vapors that will be given off when the samples are acidified.

5.2 Primary Materials Used

The following is a list of the materials used in this procedure, which have a serious or significant hazard rating, and a summary of the primary hazards listed in their MSDS.

NOTE: This list does not include all materials used in the procedure. A complete list of materials used in this procedure can be found in the Reagents and Standards Section and the Equipment and Supplies Section of this SOP

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 found using the "MSDS" link on the Oasis homepage, on the EH&S webpage on Oasis, and on the QA Navigator.

Material	Hazards	Exposure Limit[1]	Signs and Symptoms of Exposure
Hydrochloric Acid[2]	Corrosive Poison	5ppm 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.
Nitric Acid[2]	Corrosive Oxidizer Poison	2ppm TWA 4ppm STEL	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
[1]Exposure limit refers to the OSHA regulatory exposure limit.			
[2]Always add acid to water to prevent violent reactions.			

6.0 Equipment and Supplies

6.1 Equipment and Instrumentation

Thermo Jarrell Ash TJA ICAP61E-trace, Varian 730 ES, or other suitable inductively coupled plasma emission spectrometer with data system

Refer to Section 11.1.4 for additional information on MARRS.

Top-loading Balance – Verify in accordance with SOP SA-AN-100: *Laboratory Support Equipment (Verification and Use)*

6.2 Lab Supplies

Argon gas supply and appropriate fittings

Cooling water supply

Peristaltic pump

Volumetric Containers – various sizes; Class A, where applicable. Verify in accordance with SOP SA-AN-100: *Laboratory Support Equipment (Verification and Use)*

Pump-style Pipettes – various sizes. Verify in accordance with SOP SA-AN-100: *Laboratory Support Equipment (Verification and Use)*

Disposable Graduated Pipettes – various sizes. Verify in accordance with SOP SA-AN-100: *Laboratory Support Equipment (Verification and Use)*

pH paper

Detergent – used for washing non-disposable labware.

6.3 Sample Collection Containers

All sample collection containers are single-use disposable containers which limits the potential for contamination.

The routine sample collection containers provided by the laboratory are as follows:

Waters: 250mL Plastic – purchased with Certificate of Analysis attesting to purity.
Solids: 8oz Plastic or Glass Jar – purchased with Certificate of Analysis attesting to purity.

7.0 Reagents and Standards

The standards and reagents listed are those in use at the time this SOP was updated. Other standard and reagent stocks, vendors, and concentrations may be used provided they are fully documented and in compliance with method requirements.

7.1 Expiration Dates

Expiration dates (time from initial use or receipt to final use) for standard and reagent materials must be set according to the guidance in this SOP. Note: These are maximum expiration dates and are not to be considered an absolute guarantee of standard or reagent quality. Sound judgment must be used when deciding whether to use a standard or reagent. If there is doubt about the quality of a standard or reagent material, a new material must be obtained or the standard or reagent material verified. Data quality must not be compromised to extend a standard's life – i.e., when in doubt, throw it out.

The expiration date of any standard must not exceed the expiration date of the standard that was used to prepare it; that is, the "children may not outlive the parents".

Unless listed elsewhere in this SOP, the expiration dates given below apply.

7.1.1 The expiration date for unopened standards and reagents is the manufacturer's expiration date.

7.1.2 The expiration date for opened stock reagents is the manufacturer's expiration date or 5 years from the date opened, whichever is sooner.

7.1.3 The expiration date for opened stock standards is the manufacturer's expiration date.

7.1.4 The expiration date for prepared reagents is 6 months from the date prepared or the expiration date of the parent reagent, whichever is sooner.

7.1.5 The expiration date for prepared standards is 6 months from the date prepared or the expiration date of the parent standard, whichever is sooner.

7.2 Reagents

Reagents must be prepared and documented in accordance with SOP SA-AN-41: *Reagent and Standard Materials Procedures.*

Hydrochloric acid and nitric acid must be verified prior to use in accordance with the TestAmerica Solvent Lot Testing Program.

7.2.1 Laboratory Reagent Water – ASTM Type I. The conductivity must be monitored in accordance with SOP SA-AN-100: *Laboratory Support Equipment (Verification and Use).*

7.2.2 Nitric acid (HNO_3) – reagent grade. Store in a cool, dry, ventilated storage area with acid resistant floors and good drainage. Store away from sunlight, heat, water, and incompatible materials. Stable under ordinary conditions of use and storage.

7.2.3 Hydrochloric acid (HCl) – reagent grade. Store in a cool, dry, ventilated storage area with acid resistant floors and good drainage. Store away from sunlight, heat, water, and incompatible materials. Stable under ordinary conditions of use and storage.

7.3 Standards

Standards must be prepared and documented in accordance with SOP SA-AN-41: *Reagent and Standard Materials Procedures.* Certificates of analysis or purity must be received with all purchased standards, and scanned and filed in the Data Archival Folder on the G-drive.

Refer to Attachments 5 and 6 for standard preparation information.

8.0 Sample Collection, Preservation, Shipment, and Storage

8.1 Aqueous Samples

Aqueous samples are routinely collected in 250mL plastic containers containing 1.5mL 1:1 nitric acid preservative or 500mL plastic containers containing 3.0mL 1:1 nitric acid preservative. The preservative should be sufficient to achieve a sample pH of less than 2.

If dissolved metals are requested the sample must be filtered prior to the addition of the preservative. If the sample is to be filtered by the laboratory, the sample must be collected in a 250mL plastic container without preservative. The samples must be acidified with 1.25mL concentrated nitric acid to a pH<2 after filtration.

Samples are stored at room temperature until the time of digestion. Samples must be digested within 6 months of collection. Digestates are stored at room temperature until the time of analysis and must be analyzed within 6 months of collection.

8.2 Soil Samples

Soil samples are routinely collected in 8oz plastic or glass containers.

Samples must be iced at the time of collection and maintained at 4°C (less than 6°C but not frozen) until the time of digestion. Samples must be digested within 6 months of collection. Digestates are stored at room temperature until the time of analysis and analyzed within 6 months of collection.

9.0 Quality Control

SOP SA-QA-17: *Analytical Batching and Evaluation of QC Data* and the SOP Summary in Attachment 4 provide requirements for evaluating QC data.

9.1 Batch QC

9.1.1 EPA 200.7 – Drinking Water

A digestion batch consists of up to 20 environmental samples and the associated QC items digested together within a 24 hour period.

The laboratory's default minimum QC items performed for each digestion batch are: a method blank, a laboratory control sample (LCS), a low-level LCS (LLCS), a matrix spike (MS) to be performed on a minimum of 10% of samples or one per batch – whichever is greater, and a matrix spike duplicate (MSD).

This frequency equates to the following:

- For a batch of 10 or fewer samples, the default QC items are a method blank, an LCS, an LLCS, 1 matrix spike, and 1 matrix spike duplicate (MSD).
- For a batch of 11-20 samples, the minimum QC items are a method blank, an LCS, an LLCS, 1 matrix spike (from sample 1-10), another matrix spike (from sample 11-20), and a matrix spike duplicate (MSD).

9.1.2 EPA 200.7 – Clean Water Act

A digestion batch consists of up to 20 environmental samples and the associated QC items digested together within a 24 hour period.

The laboratory's default minimum QC items performed for each digestion batch are: a method blank, a laboratory control sample (LCS), a matrix spike (MS) to be performed on a minimum of 10% of samples or one per batch – whichever is greater, and a matrix spike duplicate (MSD).

This frequency equates to the following:

- For a batch of 10 or fewer samples, the default QC items are a method blank, an LCS, 1 MS, and 1 MSD.
- For a batch of 11-20 samples, the default QC items are a method blank, an LCS, 1 MS (from sample 1-10), another MS (from sample 11-20), and an MSD.

9.1.3 EPA 6010B, EPA 6010C, and SM3120B

A digestion batch consists of up to 20 environmental samples and the associated QC items. The laboratory's default minimum QC items performed for each digestion batch are: a method blank, a laboratory control sample (LCS), a matrix spike (MS), and a matrix spike duplicate (MSD).

9.1.4 If there is insufficient sample provided to perform the required matrix spikes, the LCS must be prepared induplicate (i.e., LCS/LCSD). An NCM must be initiated to denote this situation.

Insufficient sample volume is defined as receiving less than a total of 50mL or 2g.

The routine container supplied for this method is a 250mL (water) or 8oz (soil) container. 50mL or 1g is required for digestion. Reduced sample initial volumes may be necessary to achieve the required batch matrix spike frequency; however, the minimum extraction volume to be used for the matrix spike samples is 25mL or 0.5g. Note: Final volumes and spike amounts must be adjusted to compensate for these reduced initial volumes.

9.1.5 Batch QC must meet the criteria given in Attachment 3 of this SOP.

Note: If an LCS and LCSD are performed, both QC items must be evaluated and reported. Acceptable recoveries (as well as %RPD) for both LCS and LCSD are required.

9.1.3 Preparation SOPs

Refer to the following SOPs for specifics on the preparation process:

Matrix	SOP
Water Samples	SA-ME-50
Soil Samples	SA-ME-51

9.2 Instrument QC

9.2.1 Initial Calibration (ICAL)

The instrument must be calibrated in accordance with SOP SA-QA-16: *Evaluation of Calibration Curves.* This SOP provides requirements for establishing the calibration curve and gives the applicable formulas.

Instrument calibration is performed by analyzing a series of known standards. The calibration curve must consist of at least one standard (the high standard) and a blank. If a multi-point curve is requested, the calibration curve must consist of a minimum of 3 standards and a blank. The initial calibration standard concentrations currently in use in the laboratory for a multi-point curve included in Attachment 5.

Note: Refer to Attachment 5 for the standard preparation instructions. Other standard concentrations may be used provided they support the reporting limit and are fully documented in accordance with SOP SA-AN-41.

The correlation coefficient (r) of the regression curve must be greater than or equal to 0.998 for the initial calibration curve to be acceptable.

If a multi-point calibration curve is used, the upper limit of the linear range is the concentration of the High Standard.

9.2.2 High Standard Read Back

The highest concentration calibration standard must be reanalyzed as an "unknown" after the instrument is calibrated. The results for the re-analysis of the highest concentration calibration standard must be within ± 5% of the true value for each target analyte. If the result for any target analyte is outside of this range, the ICP may need to be "profiled" and the standardization/calibration repeated.

9.2.3 Second Source Initial Calibration Verification (ICV)

The calibration curve must be verified initially – prior to any sample analyses – in accordance with SOP SA-QA-16 with a standard obtained from a second source.

The ICV must be within 10% of the true value to be acceptable for EPA 6010B and EPA 6010C. The ICV must be within 5% of the true value to be acceptable for EPA 200.7 and SM3120B, and must also have a %RSD ≤5% to be acceptable for EPA 200.7.

Note: For the Thermo Trace instrument, 2 replicates are analyzed. For the Varian 730 ES instrument, 3 replicates are analyzed.

The initial calibration verification standard concentration currently in use in the laboratory is given in Attachment 5. Another standard concentration may be used provided it is mid-level and fully documented in accordance with SOP SA-AN-41.

9.2.4 Initial Calibration Blank (ICB) / Continuing Calibration Blank (CCB)

The instrument must be shown to be free from contamination by the analysis of calibration blanks. Initial calibration blanks are analyzed immediately following the initial calibration. Continuing calibration blanks must be analyzed immediately following each continuing calibration verification (CCV).

The absolute value of the initial and continuing calibration blanks must be <½RL to be acceptable.

9.2.5 Continuing Calibration Verification

The initial calibration curve must be verified every 10 samples with a mid-level standard.

The CCV must be within 5% of the true value to be acceptable for SM3120B and within 10% of the true value to be acceptable for the other methods.

The continuing calibration verification standard concentration currently in use in the laboratory is equivalent to half the concentration of the High Standard. Refer to Attachment

5 for the standard preparation instructions. Another standard concentration may be used provided it is mid-level and fully documented in accordance with SOP SA-AN-41.

9.2.6 Internal Standard (ISTD)

This procedure is an internal standard (ISTD) procedure. Yttrium is used as the internal standard for the Thermo Trace ICP (ICP-D) and the Varian 730ES ICP (ICP-E). Scandium may be used as an alternate ISTD for the Varian 730ES ICP.

The internal standard must be added to all standards, samples, and QC items prior to analysis. This is accomplished by means of an additional channel on the peristaltic pump and connected to the sample line with a 'T' or 'Y' connector fitting. This ensures constant concentration of the internal standard and eliminates the possibility of human spiking error. The concentration of the internal standard must be the same in all calibration samples, field samples, and QC samples. A concentration of 7mg/L is used for Yttrium and Scandium.

Any sample containing ISTD recoveries greater than 130% must be diluted and re-analyzed. Although ISTD recoveries less than 70% are extremely rare, the analyst should consider further dilution if this situation occurs.

9.2.6.1 Ionization Effects

High concentrations of some elements, such as Na, K, Ca, and Mg, can produce interferences from their ionization effects. The introduction of additional ions in abundance, such as Lithium, will minimize this interference. Therefore, Lithium is added to the internal standard solution, and is continually pumped at a constant rate.

See Attachment 5 for details on the preparation of the internal standard solution.

9.2.7 Reporting Limit Check Standard

A reporting limit (RL) check standard is used to demonstrate that the ICP is capable of detecting the target compounds at or below the reporting limit. EPA 200.7 requires this check only when utilizing a single point calibration. EPA 6010C requires this check for both single point and multi-point calibrations.

The concentrations in the RL check standard must be at levels that are less than or equal to the reporting limit for the samples being analyzed. The determined concentration must be detected within ±50% (for EPA 200.7) or ±30% (for EPA 6010C) of the true concentration. The RL check standard must be analyzed at the beginning and end of an analysis run (i.e., all samples must be bracketed by this check standard).

EPA 6010B and SM3120B do not require a RL check standard; however, it is the laboratory's standard practice to analyze and evaluate this check standard for these methods using the criteria outlined in EPA 200.7.

Note: This RL Check Standard is referred to as the LLICV and LLCCV in EPA 6010C.

9.2.8 Interference Check Standards

The purpose of the Interference Check Standard is to prove that the instrument software is adequately correcting for common interferences through the use interelement correction factors. The concentrations of the target analytes must be within 20% of the true concentrations to be acceptable. The analyst must pay particular attention to false positives and false negatives for elements not present in the interference check solutions.

9.2.9 Serial Dilution

A dilution must be prepared and analyzed on one sample per batch to determine if matrix interferences are present. Compare the results of the diluted and un-diluted aliquots of sample digestate for analytes that are present in the native sample at a concentration ≥50x IDL.

If the results of the dilution are within ±5% of the results of the undiluted sample, for SM3120B, or ±10% of the results of the undiluted sample, for the other methods, no matrix interference is present. If the results differ by greater than the amounts outlined above, a matrix interference should be suspected and the sample digestate should be subjected to a post-digestion spike.

9.2.10 Post-Digestion Spike

A post-digestion spike is performed on one sample per analytical batch to determine if matrix interferences are present. This post-digestion spike is evaluated if the serial dilution fails or if the analyte concentration is not at least 50 times the instrument detection limit. The sample selected as the post-digestion spike should be the same sample selected for serial dilution.

The post-digestion spike must be within 25% of the true value to be acceptable. If these criteria are not met, then the data are qualified.

9.2.11 % RSD of Multiple Exposures

Replicate analyses of a sample should agree within 5% RSD. Replicates that are greater 5% RSD should be investigated for matrix interferences or differences in viscosity of the digest.

9.2.12 Interelement Correction Factors (IEC)

Interelement correction factors (IEC) for all elements must be determined annually using the manufacturer's guidance.

The lab may combine the linearity study with the IEC study, thereby eliminating redundancy. (Single element linearity check solutions for each analyte should be closely evaluated for all non-spiked elements, and applicable correction factors should be applied according to the instrument's software. The analyst must be careful not to correct for any contamination which may be present in the actual solution.)

At a minimum the IECs must be verified at the beginning of each analytical sequence through the analysis of interferent check solutions ICSA and ICSAB. Note: Some protocols and project plans require the analysis of the ICSA and ICSAB standard at the

beginning and end of an analysis sequence. The analyst must be aware of the requirements of the project before beginning the analysis.

9.3 Corrective Action for Out-of-Control Data

When the quality control parameters do not meet the criteria set forth in this SOP, corrective action must be taken in accordance with SOP SA-QA-05: *Preventive and Corrective Action Procedures* the QC Summary Table in Attachment 4. SOP SA-QA-05 provides contingencies for out-of-control data and gives guidance for exceptionally permitting departures from approved policies and procedures. Nonconformance Memos must be initiated to document all instances where QC criteria are not met and all departures from approved policies and procedures.

10.0 Procedure

10.1 Sample Preparation

The sample preparation procedures are given in the following SOPs:

Matrix	SOP #
Water Samples	SA-ME-050
Soil Samples	SA-ME-051

10.2 QC Sample Preparation

Batch QC sample preparation procedures are given in the SOPs listed above. Additional QC items are listed below.

10.2.1 Serial Dilution

Dilute the digestate by a factor of 5 and analyze the dilution using the same procedures used for the un-diluted aliquot.

10.2.2 Post Digestion Spike

Transfer 10mL of a digestate to a suitable vial. Spike the sample with 0.10mL of ~~CLP~~ Spike I and 0.10mL of Spike II. The theoretical concentration of the post digestion spike is the same as the LCS or MS if the volume of spiking solution is discounted.

10.3 Analysis

10.3.1 Instrument Operating Conditions

Turn the ICP on and allow it to become thermally stable before beginning to analyze the calibration standards. It will take about an hour for the instrument to warm up. If the optics were turned off, allow 2 hours warm up time.

Run the "Automatic Profile" program. The "Automatic Profile" of the instrument should be

checked twice a day to compensate for changes in air pressure, humidity, and temperature. If the environment of the instrument is such that daily changes in the instrument profile are extreme, the instrument should be "profiled" every few hours.

Instrument maintenance must be performed in accordance with Attachment 2 of this SOP.

10.3.2 Internal Standard (ISTD)

Prior to analysis, internal standard must be added to all standards, samples, and QC items. The concentration of the internal standard must be the same in all calibration samples, field samples, and QC samples.

10.3.3 Initial and Continuing Calibration

Calibrate the instrument using the standards and criteria described in Section 9.2.1. Once the calibration has been established and verified with a high level standard and an ICV in accordance with Sections 9.2.2 and 9.2.3, sample analysis may proceed.

Verify the calibration curve with a continuing calibration verification using the standards and criteria described given in Section 9.2.5.

10.3.4 Sample Analysis

Samples that are known to be relatively clean should be analyzed first. Samples suspected of containing high concentrations should be analyzed last. Instrument blanks may be analyzed after suspected high concentration samples to allow the complete removal of trace analytes. The instrument blanks would be included in the sample count in determining the maximum number of samples between CCVs/CCBs.

The default procedure is to include QC items (method blank, LCS, MS/MSD, and SD) in determining the maximum number of samples between CCVs/CCBs.

10.3.5 Example Analytical Sequence

An example analytical sequence is listed below.

Analytical Sequence for samples immediately following an initial calibration:

Description	Comments
Instrument Warm-up	
Profile	If torch is changed
Initial Calibration	
High Calibration Standard	Re-analyzed as a sample
ICV	Second Source
ICB	
Reporting Limit Check Standard	
ICP Interference Check Solution A (ICSA)	
ICP Interference Check Solution AB (ICSAB)	
CCV	10-analysis count begins after analysis of the CCB
CCB	
Samples & Batch QC Items	Up to 10 analyses, including QC
CCV	10-analysis count begins after analysis of the CCB
CCB	
Samples & Batch QC Items	Up to 10 analyses, including QC
CCV	10-analysis count begins after analysis of the CCB
CCB	
Samples & Batch QC Items	Up to 10 analyses, including QC
CCV	10-analysis count begins after analysis of the CCB
CCB	
Reporting Limit Check Standard	

Note: The analytical sequence continues as outlined above and must end with the analysis of the CCV and CCB. The 10 samples include all QC samples/standards with the exception of CCVs and CCBs.

11.0 Calculations / Data Reduction

11.1 Data Reduction

Data must be evaluated in accordance with SOP SA-QA-02: *Data Generation and Review*.

11.1.1 Dilutions

If the concentration of a sample is above the linear range of the ICP, as determined in Attachment 7, the sample digestate must be diluted and reanalyzed.

Dilutions must be prepared in reagent water containing 5% hydrochloric acid and 1% nitric acid by volume.

11.1.2 Historical Data

Many of the laboratory's clients submit samples for repeat monitoring purposes. Prior to analysis, the analyst must verify TALS Worksheet Notes and/or use the TALS Historical Data Tracker feature to determine if historical data is available for review.

11.1.3 Chemical Relationships

The analyst must be aware of the following chemical relationships:

- Total Results should be ≥ Dissolved results

11.1.4 Evaluation of Batch QC and Samples for Matrix Interferences

Matrix interference is evaluated using three criteria: the recoveries of the matrix spike (MS) and matrix spike duplicate (MSD), the results of the serial dilution, and the recovery of the post digestion spike. This laboratory uses internal standard calibration and quantification which minimizes the need to use the method of standard additions (MSA) to quantify sample digests with known or suspect matrix interferences. MSA is used when the recoveries of the MS/MSD are less than 50% and there is no obvious reason for the poor recoveries (e.g., viscosity of digest, high dissolved solids, undigested solids, known high concentrations of inter-element interferences, etc.).

The primary indicator of matrix interference is the results of the MS/MSD. If the recoveries and precision of the MS/MSD are within acceptance limits, matrix interferences are not indicated and unacceptable results for the serial dilution and post-digestion spikes are likely due to poor preparation rather than matrix interference.

Use the following table to evaluate for the indication of matrix interferences.

MS	MSD	Serial dilution[1]	Post Digestion Spike[1]	Action
OK	OK	OK	OK	Report
OK	OK	Out	OK	Unlikely to occur; suspect preparation of serial dilution or post digestion spike
OK	OK	OK	Out	
OK	Out	OK	OK	Suspect preparation-qualify results
Out	OK	OK	OK	
Out	Out	Out	OK	Matrix interference confirmed-qualify results if recoveries >50%
Out	Out	OK	Out	
Out	Out	Out	Out	If MS/MSD recoveries <50% and there is no obvious reason, perform MSA as directed in section 11.1.5.

[1]Performed on un-spiked digest of the sample designated as the MS/MSD

11.1.5 Evaluation of Post-Digestion Spikes

Result of Post-Digestion Spikes	Action
Within 75-125% limits	None
>125% recovery	Re-spike and reanalyze if multiple analytes >125%. Repeat analysis of un-spiked sample and re-evaluate recoveries. Qualify results for affected analytes.
<75% recovery but >50% recovery	Qualify results for affected analytes. Dilute digestate and repeat spike for affected analytes. ***Treat all samples associated with spike in the same manner as the spiked sample (i.e., spike or dilute samples).*** If recoveries are not 75-125%, all associated samples may be re-analyzed by single point MSA. Note: High levels of target analytes may inhibit spike recovery. Consult the Department Manager in events where high levels of targets appear to be interfering.
<50% recovery	Dilute and re-spike. Elevate RL accordingly (for all associated samples). Spike and evaluate all associated samples. Spike and evaluate all associated samples by single point MSA. Qualify results for affected analytes.

Note: The >50% recovery of the post digestion spike is a benchmark below which samples may be biased high if corrected for spike recovery.

11.2 Calculations

11.2.1 The calculations associated with batch QC determinations are given in SOP SA-QA-17. Applicable calculations include accuracy (% recovery) and precision (%RPD and % difference).

11.2.2 The calculations associated with initial and continuing calibrations are given in SOP SA-QA-16. Applicable calculations include determination for correlation coefficient, slope, intercept, and percent recoveries.

11.2.3 The calculation to determine final concentration is given as follows:

$$FinalConcentration = CONC_{Sample} \otimes \frac{F}{I \times dw} \otimes D$$

Where:

$CONC_{Sample}$= Concentration of the sample
F = Final volume/weight
I = Initial volume/weight
D = Dilution factor
dw = % Solids decimal equivalent

Note: All dry weight corrections are performed automatically in LIMS.

Note: This calculation assumes all applicable unit correction factors are applied.

11.2.4 Method of Standard Additions

The concentrations of both sample aliquots, Section 9.2.11, are measured and the sample concentration is calculated as follows:

$$C_x = \frac{S_2 V_s C_s}{(S_1 - S_2) V_x}$$

Where:

S_1 = Absorbance or concentration of the spiked aliquot
S_2 = Absorbance or concentration of the un-spiked aliquot
V_s = Volume of spike solution
V_x = Volume of sample aliquots
C_s = Spike solution concentration

Note: This calculation assumes all applicable unit correction factors are applied.

12.0 Method Performance

12.1 Reporting Limit Verification (RLV)

At a minimum, RLVs must be performed initially upon method set-up in accordance with SOP SA-QA-07: *Determination and Verification of Detection and Reporting Limits.*

For analytes and methods certified by DOD ELAP, RLVs must also be performed quarterly thereafter. For all other analytes and methods, RLVs must also be performed annually thereafter. Exceptions may be made for project-specific non-routine analytes.

12.2 Method Detection Limit (MDL) Study

The 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. 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 current MDLs associated with this procedure are given in the Method Limit Group (MLG) in TALS.

At a minimum, MDL Studies must be performed initially upon method set-up in accordance with SOP SA-QA-07: *Determination and Verification of Detection and Reporting Limits*.

Note: MDL Studies are not required for non-routine analytes provided results are not reported below the RL (i.e., MDL equals RL in TALS).

12.3 Method Detection Limit Verification (MDLV)

At a minimum, MDLVs must be performed initially upon method set-up in accordance with SOP SA-QA-07: *Determination and Verification of Detection and Reporting Limits*.

For analytes and methods certified by DOD ELAP, MDLVs must also be performed quarterly thereafter. For all other analytes and methods, MDLVs must also be performed annually thereafter.

Note: MDLVs are not required for non-routine analytes provided results are not reported below the RL (i.e., MDL equals RL in TALS).

12.4 Determination of the Instrument Detection Limit (IDL)

The instrument detection limit (IDL) is the concentration of analyte that can be statistically distinguished from the background noise of the instrument. The IDL limit must be determined annually, at a minimum, for each analyte in accordance with SOP SA-QA-07: *Determination and Verification of Detection and Reporting Limits (RLs, MDLs, and IDLs)*.

The IDL is defined as three times the average of the standard deviation of a minimum of seven replicate analyses of blank solution performed three times over non-consecutive days. The IDL may be elevated above the background noise (blank levels). The current IDL associated with this procedure is given in the Equipment Limit Group (ELG) in LIMS.

12.5 QC Limit Generation, Control Charting, and Trend Analysis

The control limits for the batch QC items (LCS, MS/MSD) for this procedure are specified in the reference method and cannot be broadened; therefore, the laboratory defaults to the method-defined limits and does not utilize in-house or laboratory-derived limits for the evaluation of batch QC items.

Although the laboratory must default to the method-defined QC limits, control charting is a useful tool and is performed to assess analyte recoveries over time to evaluate trends. Control charting must be performed periodically (at a minimum annually) in accordance with SOP SA-QA-17: *Evaluation of Batch QC Data*.

12.6 Lower Limit of Quantitation Check Sample (LLQC)

EPA 6010C requires a lower limit of quantitation check sample (LLQC) analysis to demonstrate the required reporting limit capability. The laboratory shall prepare and analyze this solution on a yearly basis, in accordance with all applicable preparatory SOPs (i.e. the solution must be digested.) It is recommended that the laboratory utilize the RL check solution (Section 9.2.7) by simply carrying it through the entire preparatory procedure for each of the prep methods associated with EPA 6010C. Digesting this

solution on the same prep batch as the yearly MDL studies is preferred. The LLQC must recover within 70-130% to be acceptable, or a new (higher) reporting limit must be established.

12.7 Demonstrations of Capability

Initial and continuing demonstration of capability must be performed in accordance with SOP SA-QA-06*: Training Procedures.*

Prior to performing this procedure unsupervised, each new analyst who performs this analysis must demonstrate proficiency per method/analyte combination by successful completion of an initial demonstration of capability. The IDOC is performed by the analysis of 4 consecutive LCSs that meet the method criteria for accuracy and precision. The IDOC must be documented on the IDOC Form and routed to the QA Department for filing.

Annual continuing demonstrations of capability (CDOCs) are also required per analyst per method/analyte combination. The CDOC requirement may be met by the consecutive analysis of four LCS all in the same batch, by the analysis of four LCS analyzed in four consecutive batches (in different batches on different days), via acceptable results on a PT study, or analysis of client samples with statistically indistinguishable results when compared to another certified analyst. The CDOC must be documented and routed to the QA Department for filing. Note: LCS CDOCs from the *same* source as the calibration standards are permitted.

12.8 Training Requirements

All training must be performed and documented in accordance with SOP SA-QA-06: *Training Procedures*.

Note: The SOPs listed in the Reference/Cross-Reference Section are applicable to this procedure. All employees performing this procedure must also be trained on these SOPs.

13.0 Pollution Control

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (e.g., examining recycling options, ordering chemicals based on quantity needed, preparing reagents based on anticipated usage and reagent stability, etc.). Employees must abide by the policies in Section 13 of the Environmental Health and Safety Manual (EHSM) and the Savannah Addendum to the EHSM.

This procedure has been evaluated for opportunities to minimize the waste generated. Where reasonably feasible, pollution control procedures have been incorporated.

14.0 Waste Management

Waste management practices must be conducted consistent with all applicable federal, state, and local rules and regulations. All waste (i.e., excess reagents, samples, and method process wastes) must be disposed of in accordance with Section 9 of the

TestAmerica Savannah Addendum to the EHSM. Waste description rules and land disposal restrictions must be followed.

14.1 Waste Streams Produced by the Method

The following waste streams are produced when this method is carried out:

- Excess aqueous samples – Dispose according to characterization on the sample disposal sheets. Neutralize non-hazardous samples before disposal into drain/sewer. Transfer hazardous samples (identified on disposal sheets) to the waste department for disposal.

- Excess soil and solid samples – Dispose according to characterization on sample disposal sheets. Transfer non-hazardous samples to TCLP container for characterization in hazardous waste department. Transfer hazardous samples (identified on disposal sheets) to waste department for disposal.

- Acidic sample digestions – Neutralize before disposal into drain/sewer system.

Dispose of all metals standards with concentrations exceeding the TCLP threshold limits as a RCRA aqueous hazardous waste. The TCLP constituents and the respective threshold limits are:

Analyte	Disposal Limit (mg/L)
Arsenic	5.0
Barium	100
Cadmium	1.0
Chromium	5.0
Lead	5.0
Selenium	1.0
Silver	5.0
Mercury	0.20

Neutralize the standard by passing with sodium bicarbonate before transferring the waste to the RCRA aqueous hazardous waste drum.

All other metals standards may be disposed, using copious amounts of water, down a sink that flows to the neutralization pit.

15.0 References / Cross-References

- SOP SA-AN-100: *Laboratory Support Equipment (Verification and Use)*
- SOP SA-AN-41: *Reagent and Standard Materials Procedures*
- SOP SA-QA-02: *Data Generation and Review*
- SOP SA-QA-05: *Preventive and Corrective Action Procedures*
- SOP SA-QA-06: *Training Procedures*
- SOP SA-QA-07: *Determination and Verification of Detection and Reporting Limits*
- SOP SA-QA-15: *Homogenization, Compositing, and Segregation of Samples*

- SOP SA-QA-16: *Evaluation of Calibration Curves*
- SOP SA-QA-17: *Evaluation of Batch QC Data*
- TestAmerica Savannah Quality Assurance Manual
- TestAmerica Environmental Health and Safety Manual
- TestAmerica Savannah Addendum to the Environmental Health and Safety Manual
- *Methods for Chemical Analysis of Water and Waste*; U.S EPA Office of Research and Development: Cincinnati, OHIO, March 1983.
  - EPA 200.7, Revision 4.4, EMMC Version: Determination of Metals and Trace Elements in Water and Wastes by Inductively Coupled Plasma-Atomic Emission Spectrometry; 1994
- *Methods for the Determination of Metals in Environmental Samples*; US EPA Office of Research and Development. Washington, DC.
- *Test Methods for Evaluating Solid Waste, Third Edition*; U.S. EPA Office of Solid Waste and Emergency Response: Washington, D.C., November 1986 (SW-846 Updates III and IV).
  - Method 6010B, Revision 2: Inductively Coupled Plasma-Atomic Emission Spectrometry; December 1996.
  - Method 6010C, Revision 3: Inductively Coupled Plasma-Atomic Emission Spectrometry; February 2007.
- *Standard Methods for the Examination of Water and Wastewater, Online Edition;* American Public Health Association: Washington, DC.
  - SM3020: Quality Assurance/Quality Control
  - SM3210B: *Metals by Plasma Emission Spectroscopy; Inductively Coupled Plasma Method;* 1999

16.0 Method Modifications/Clarifications

16.1 Incorporation of Non-Routine Matrices

This procedure may be modified to analyze other matrices (e.g., wipe, waste, tissue, filter, and TCLP/SPLP leachate samples) upon client request. This will need to be arranged by the Project Manager at the initiation of the project.

Wipe, waste, filter, and tissue matrices are non-routine, and the laboratory is not currently NELAC certified for these matrices. The laboratory uses its routine soil RLs (converted for initial and final volumes, etc.) and soil QC limits to evaluate wipe, waste, filter, and tissue samples. Soil DOCs can be used to satisfy analyst demonstrations of capability for these types of non-routine matrices. The laboratory uses its routine soil RLs (converted for initial and final volumes, etc.) and soil QC limits to evaluate TCLP/SPLP leachate samples. Water DOCs can be used to satisfy analyst demonstrations of capability for TCLP/SPLP matrices. Teflon chips, Ottawa sand, or equivalent is used as the blank matrix for wipes, wastes, filters, and tissues unless specifically requested otherwise by the project.

16.1.1 Collection and Handling Procedures for Non-Routine Matrices

Waste (oil) samples are collected in 500mL plastic containers. Waste (oil) samples are stored at room temperature until the time of digestion. Wipe samples are collected in 20mL scintillation vials. Wipe and filter samples must be iced at the time of collection and maintained at 4°C (less than 6°C but not frozen) until time of digestion. Waste (oil), wipe,

and filter samples must be digested within 6 months of collection.

Tissue samples are collected in plastic containers with the size dependent upon the type of tissue being collected. Plastic jars or plastic baggies can be used. Upon receipt, tissue samples must be placed in the freezer at -10° to -20°C for up to 6 months if digestion cannot be completed that day. Tissue samples must be digested within 6 months of thawing.

Digestates for waste (oil), wipe, tissue, and filter samples are stored at room temperature until the time of analysis and must be analyzed within 6 months of collection.

Once the TCLP/SPLP extraction procedure has been performed, the TCLP/SPLP leachate must be transferred to a 500mL plastic container and preserved with 1.0mL nitric acid to a pH <2. Preserved TCLP/SPLP leachates are stored at room temperature until the time of digestion. The leachate sample must be digested within 6 months of completion of the TCLP/SPLP extraction. Digestates are stored at room temperature until the time of analysis and must be analyzed within 6 months of completion of the TCLP/SPLP extraction.

16.1.2 Preparation and Analytical Procedures for Non-Routine Matrices

Wipe, waste, filter, and tissue samples are prepared in the same manner as routine soil samples as outlined in SOP SA-ME-051. TCLP/SPLP matrices are prepared in the same manner as routine water samples as outlined in SOP SA-ME-050. Refer to the applicable preparation SOPs for more information.

Wipe, waste, filter, tissue, and TCLP/SPLP matrices are analyzed in the same manner as routine samples as outlined in this SOP.

16.2 Other Considerations

16.2.1 The EPA Manual for the Certification of Laboratories Analyzing Drinking Water requires a LFB at the MRL to be performed each day. The laboratory meets this requirement by preparing a low-level LCS (LLCS), spiked at the RL, in each EPA 200.7 batch of drinking water samples. The EPA DW Manual does not specify criteria for the low-level LCS; therefore, the laboratory requires this standard to be qualitatively identified to be acceptable.

16.2.2 The stock standards utilized for the Silica procedure are routinely purchased as Silicon (Si). Both Silica (SiO_2) and Silicon (Si) can be reported using this procedure. The following equation is used to convert between Silicon (Si) and Silica (SiO_2):

$$Si = \frac{SiO_2}{2.14}$$

16.2.3 EPA 6010B does not require an RL check standard; however, it is the laboratory's standard practice to analyze and evaluate this check standard for EPA 6010B using the criteria outlined in EPA 200.7.

16.2.4 The laboratory's default QC items incorporate an MSD to satisfy the Clean Water Act requirements and those clients who batch require precision to be reported. Additionally, if insufficient sample volume is provided to perform the MS/MSD, the LCS is routinely prepared in duplicate (i.e., LCS/LCSD).

16.2.5 EPA Method 6010B specifies that CCBs and ICBs must be less than 3x the IDL or less than the RL, whichever is greater. The laboratory defaults to the requirement to control to 1/2RL which allows for ease of data evaluation and is consistent with the requirement outlined in EPA Method 200.7 and EPA Method 6010C.

16.2.6 Linear Range determinations are performed initially upon instrument set-up and when significant instrument changes are made. Linear Range verifications are performed quarterly as required by DOD QSM. This frequency is greater than that defined by the reference methods.

17.0 Attachments

The following Tables, Diagrams, and/or Validation Data are included as Attachments:

Attachment 1: SOP Summary
Attachment 2: Sample Collection, Preservation, and Holding Time Table
Attachment 3: QC Summary
Attachment 4: Instrument Maintenance and Troubleshooting
Attachment 5: Standard Preparation Tables
Attachment 6: Silica/Silicon Standard Preparation
Attachment 7: Linear Range Determination
Attachment 8: Element Wavelengths
Attachment 9: Hardness Calculation Work Instruction

Attachment 1:
SOP Summary

Sample Preparation Summary

Prior to analysis by ICP, the sample must be digested using the sample preparation method appropriate to the matrix. Samples should be prepared according to the appropriate matrix-specific SOP.

Matrix	SOP #
Aqueous Samples	SA-ME-050
Soil Samples	SA-ME-051

Sample Analysis Summary

Sample digestates are aspirated and nebulized into a spray chamber. A stream of argon gas carries the sample aerosol through the innermost of three concentric tubes and injects it into the middle of the donut-shaped plasma. The sample elements are dissociated, atomized, and excited to a higher energy level. As the elements fall to a lower energy level, radiation characteristic of the elements present in the plasma is emitted. The light is directed through an entrance slit, dispersed by the diffraction grating, and projected on to the photomultiplier tube (PMT) or on to a charge-coupled device (CCD). The PMTs and CCDs located behind the exit slits, convert the light energy to an electrical current. This signal is then digitized and processed by the data system. Background correction is required for trace element determination.

Analytical Sequence

Description	Comments
Instrument Warm-up	
Profile	If torch is changed
Initial Calibration	
High Calibration Standard	Re-analyzed as a sample
ICV	Second Source
ICB	
Reporting Limit Check Standard	
ICP Interference Check Solution A (ICSA)	
ICP Interference Check Solution AB (ICSAB)	
CCV	10-analysis count begins after analysis of the CCB
CCB	
Samples & Batch QC Items	Up to 10 analyses, including QC
CCV	10-analysis count begins after analysis of the CCB
CCB	
Samples & Batch QC Items	Up to 10 analyses, including QC
CCV	10-analysis count begins after analysis of the CCB
CCB	
Samples & Batch QC Items	Up to 10 analyses, including QC
CCV	10-analysis count begins after analysis of the CCB
CCB	
Reporting Limit Check Standard	Method criterion

Note: The analytical sequence continues as outlined above and must end with the analysis of the CCV and CCB. The 10 samples include all QC samples/standards with the exception of CCVs and CCBs.

Attachment 2:
Sample Collection, Preservation, and Holding Time Table

Matrix	Routine Sample Container	Routine Sample Size	Minimum Sample Size	Dechlorination Agent	Thermal Preservation[1]	Chemical Preservation[1]	Holding Time[2]
Water	250mL or 500mL plastic	50mL	25mL	Not Applicable	None required	1:1 Nitric Acid to pH<2	6 months
Water (Silica)	250mL or 500mL plastic	10mL	5mL	Not Applicable	4°C[3]	Not Applicable	6 months
Soil	8oz plastic or glass	1g	0.50g	Not Applicable	4°C[3]	Not Applicable	6 months
Soil (Silica)	8oz plastic or glass	5g	2.5g	Not Applicable	4°C[3]	Not Applicable	6 months

[1]Samples requiring dissolved metals must be filtered prior to preservation.
[2]Inclusive of digestion and analysis.
[3]Samples must be collected on ice and maintained at <6 °C with no frozen samples.

Attachment 3:
QC Summary

QC Item	Frequency	Criteria	Corrective Action
Initial Calibration (ICAL)	Daily	Single Point: 1 standard and 1 blank Multi-point: Minimum 3 standards and 1 blank Correlation coefficient ≥0.998	Recalibrate
High Standard	Immediately after every calibration	Recoveries within ± 5% of expected values	New initial calibration
Initial Calibration Verification Standard (ICV)	At the beginning of the analysis	EPA 6010B and EPA 6010C: within ±10% EPA 200.7: within ± 5%, </= 5%RSD SM3120B: within ± 5%	Recalibrate
Continuing Calibration Verification Standard (CCV)	At the beginning and end of the analysis, and every 10 samples	SM3120B: Within ± 5% of true value Other Methods: Within ±10% of true value </=5%RSD	Terminate the analysis, fix the problem and reanalyze the previous 10 samples.
Calibration Blank (ICB/CCB)	After ICV and every CCV	Absolute value <½RL	Terminate the analysis, correct problem and reanalyze the previous 10 samples
Interference Check Standards (ICSA/ICSAB)	At the beginning of an analysis run	± 20% of the true values Pay attention to false positives and false negatives for elements not present in the solutions.	Terminate the analysis, correct the problem, recalibrate, and reanalyze all samples since the last ICS that was in control.
Batch Definition	Up to 20 field samples digested together within a 24-hr time	Not Applicable	Not Applicable

QC Item	Frequency	Criteria	Corrective Action
	period		
Method Blank (MB)	One per batch of twenty samples or less	|result| <½RL or result <10% of the analyte level in the sample	Refer to SOP SA-QA-17
Laboratory Control Sample (LCS)	One per batch	LIMS MLG	Refer to SOP SA-QA-17
Laboratory Control Sample Duplicate (LCSD)	One per batch, if insufficient sample provided for MS/MSD	LIMS MLG	Refer to SOP SA-QA-17
Low-Level Laboratory Control Sample (LLCS) – at the RL	EPA 200.7 DW Only: One per batch of twenty samples or less for drinking water	Qualitatively detected	If the "regular" LCS meets criteria, initiate NCM and report data If the "regular" LCS does not meet criteria, redigest and reanalyze batch
Matrix Spike (MS)	EPA 200.7: 10% of samples prepared; i.e., 2 separate matrix spikes per batch EPA 6010B & EPA 6010C: 5% of samples prepared; i.e., 1 matrix spike per batch	LIMS MLG	Refer to SOP SA-QA-17
Matrix Spike Duplicate (MSD)	One per batch	LIMS MLG	Refer to SOP SA-QA-17
Serial Dilution (1/5 Dilution)	One per batch	Refer to Section 9.2.9	Refer to Section 9.2.9
Post Digestion Spike (PDS)	One per batch	Refer to Section 9.2.10	Refer to Section 9.2.10
Reporting Limit Check	At the beginning and end of an	EPA 200.7: +/-50% of the true concentration	Opening:

QC Item	Frequency	Criteria	Corrective Action
Solution (also known as LLICV and LLCCV for EPA 6010C)	analysis run (Required for EPA 200.7 when using a single point calibration. Required for EPA 6010C when using both single point and multi-point calibrations.)	EPA 6010C: +/-30% of the true concentration EPA 6010B (Internal Criteria): +/-50% of the true concentration	Stop the analysis, fix the problem, and reanalyze the affected samples. Capping: Qualify affected samples; reanalyze if required by project plan
%RSD (CV) of Multiple Exposures	Evaluate for all Calibration, QC, and samples	Conc. >= RL - Warning: <=20% - Acceptance: <=30%	-if CV>20 but <=30, review data for possible interferences; -if interference present, reanalyze digest -if no interference present, report average -if CV>=30%, reanalyze digest, report the result that has the lower precision value or dilute the digestion and reanalyze
		Conc. < RL -use professional judgment	-use professional judgment
Linear Range	Determined at least every 6 months in accordance with Attachment 7	% difference </=10%	-reanalyze at a lower concentration
Interelement Correction Factors (IEC)	Determined at least annually Verified at the beginning of an analysis run	Refer to ICSA, ICSAB criteria	See ICSA, ICSAB corrective action
Lower Limit of Quantitation Check Sample	Annually	6010C: 70-130%	- reanalyze at a higher concentration - elevate RL accordingly
Initial Demonstration of Capability (IDOC)	Initially, per analyst, per analyte/method/matrix combination	Refer to SOP SA-QA-06	Refer to SOP SA-QA-06 Note: Unsupervised work must not begin until acceptable IDOC is obtained.
Continuing Demonstration of	Annually, per analyst, per analyte/method combination	Refer to SOP SA-QA-06	Refer to SOP SA-QA-06

QC Item	Frequency	Criteria	Corrective Action
Capability (CDOC)			
Reporting Limit Verification (RLV)	Upon method/instrument set-up, per analyte/method/matrix combination. Then quarterly thereafter (for DOD ELAP) or annually thereafter (for non-DOD ELAP)	Refer to SOP SA-QA-07	Refer to SOP SA-QA-07
Method Detection Limit Study (MDL)	Upon method/instrument set-up, per analyte/method/matrix combination	Refer to SOP SA-QA-07	Refer to SOP SA-QA-07
MDL Verification (MDLV)	Upon method/instrument set-up, per analyte/method/matrix combination. Then quarterly thereafter (for DOD ELAP) or annually thereafter (for non-DOD ELAP)	Refer to SOP SA-QA-07	Refer to SOP SA-QA-07
Instrument Detection Limit (IDL)	Upon method/instrument set-up, and then quarterly thereafter	Refer to SOP SA-QA-07	Refer to SOP SA-QA-07

Attachment 4:
Preventative Maintenance and Troubleshooting

Preventive Maintenance

EQUIPMENT ITEM	Service Interval							SERVICE LEVEL
	D	W	M	Q	SA	A	AN	
ICAP Service Schedule								
Pump Tubing							X	Inspect and replace as needed (Recommend daily)
Nebulizer							X	Clean as needed
Filters			X					Inspect monthly, clean or replace as needed
Chiller Water Filter							X	Clean or replace as needed (Recommend every six months)
Injector Tip / Torch		X						Inspect weekly, clean or replace as needed
Tubing Connectors							X	Replace as needed

D = daily; W = Weekly; M = monthly; Q = Quarterly; SA = semi-annually; A = annually; AN = as needed

Troubleshooting

Troubleshooting should be documented as outlined above. If possible, troubleshooting is best performed in a step-wise manner to systematically isolate instrument components. Refer to the instrument manufacturer's guides for specific information and strategies. Enlist assistance from technical and/or department management as needed.

Maintenance Log

A maintenance log must be established for each piece of equipment used in the laboratory. All maintenance that is performed on the instrument must be recorded in the log including:

- analyst or technician performing the maintenance
- date the maintenance was performed
- detailed explanation of the reason for the maintenance
- resolution of the problem and return to control
- all service calls from instrument representatives

Instrument Labeling

Each instrument must be labeled with its name or ID (e.g., MSA, ICP-D, etc.). Additionally, non-operational instruments must be isolated from service or marked as being out of service. Each piece of equipment has an "Operational / Not Operational" sticker that is used for this purpose.

Attachment 5:
Standard Preparation

Note: All standards must be stored at room temperature and have an expiration date of 6 months from date prepared.

Continuing Calibration Verification (CCV)
Final Volume (mL): 2000
Solvent: 5% HCL / 1 % HNO3

Purchased Standards	Concentration (mg/L)	Vendor *	Volume Used (mL)	Final Concentration (mg/L)
Single Element Stocks				
Ag	1000	CPI	1.0	0.50
Al	10000	SPEX	1.0	5.0
B	1000	CPI	10.0	5.0
Fe	10000	CPI	1.0	5.0
Sr	1000	ABSOLUTE	5.0	2.5
Ti	1000	CPI	1.0	0.50
Na	10000	CPI	0.50	2.5
Multi Element Mixes				
CAL STD 2				
As	50	CPI	20	0.50
Mo	50			0.50
Pb	50			0.50
Sb	50			0.50
Se	500			5.0
Tl	500			5.0
CAL STD 3				
Ba	500	CPI	20	5.0
Be	50			0.50
Cd	50			0.50
Co	50			0.50
Cr	500			5.0
Cu	500			5.0
Mn	500			5.0
Ni	250			2.5
Zn	250			2.5
CAL STD 5				
Ca	500	CPI	20	5.0
K	1000			10
Mg	500			5.0
Na	500			5.0
Sn	500			5.0
V	500			5.0

Internal Standard (ISTD)

Final Volume (mL): 10000

Solvent: 30% HNO3

Purchased Standards	Concentration (mg/L)	Vendor *	Amount Used	Final Concentration (mg/L)
Y	10000	CPI	7.0mL	7.0
Lithium Carbonate (powder)	18.78% Li	Mallinckrodt	7.5g	140.85
Sc	10000	CPI	7.0mL	7.0

MDL & IDL Intermediate
Final Volume:
(mL) 100
Solvent: 5% HCL / 1 % HNO3

Purchased Standards	Concentration (mg/L)	Vendor *	Volume Used (mL)	Final Concentration (mg/L)
Single Element Stocks)				
Ag	1000	CPI	0.040	0.40
Al	10000	SPEX	0.10	10
As	1000	CPI	0.20	2.0
B	1000	CPI	0.40	4.0
Ba	1000	CPI	0.020	0.20
Be	1000	CPI	0.0050	0.10
Ca	10000	SPEX	0.10	10
Cd	1000	CPI	0.040	0.40
Co	1000	CPI	0.10	1.0
Cr	1000	CPI	0.10	1.0
Cu	1000	CPI	0.10	1.0
Fe	10000	CPI	0.10	10
K	10000	CPI	0.20	20
Mg	10000	SPEX	0.10	10
Mn	1000	CPI	0.020	0.20
Mo	1000	CPI	0.10	1.0
Na	10000	CPI	2.04	204
Ni	1000	CPI	0.10	1.0
Pb	1000	CPI	0.10	1.0
Sb	1000	CPI	0.20	2.0
Se	1000	CPI	0.20	2.0
Sn	1000	CPI	0.20	2.0
Sr	1000	CPI	0.040	0.40
Ti	1000	CPI	0.050	0.50
Tl	1000	CPI	0.20	2.0
V	1000	CPI	0.060	0.60
Zn	1000	CPI	0.10	1.0

Spike I Solution - Post Spike, LCS, MS/MSD: Purchased from SPEX and at the following concentrations:

Element	Concentration (mg/L)
Ag	5.0
Al	200
As	200
Ba	200
Be	5.0
Cd	5.0
Co	50
Cr	20
Cu	25
Fe	100
Mn	50
Ni	50
Pb	50
Sb	50
Se	200
Tl	200
V	50
Zn	50

SPIKE II - Post Spike, LCS/MS Spike
Final Volume (mL): 100
Solvent: 5% HCL / 1 % HNO3

Purchased Standards	Concentration (mg/L)	Vendor *	Volume Used (mL)	Final Concentration (mg/L)
Single Element Stocks				
B	1000	CPI	10	100
Ca	10000	SPEX	5.0	500
K	10000	CPI	5.0	500
Mg	10000	SPEX	5.0	500
Mo	1000	CPI	5.0	50
Na	10000	CPI	5.0	500
Sn	1000	CPI	10	100
Sr	1000	CPI	5.0	50
Ti	1000	CPI	10	100

RL ICP INTERMEDIATE

Final Volume (mL): 100

Solvent: 5% HCL / 1 % HNO3

Purchased Standards	Concentration (mg/L)	Vendor *	Volume Used (mL)	Final Concentration (mg/L)
Single Element Stocks				
Ag	1000	CPI	0.10	1.0
Al	10000	SPEX	0.20	20
As	1000	CPI	0.20	2.0
B	1000	CPI	1.0	10
Ba	1000	CPI	0.10	1.0
Be	1000	CPI	0.040	0.4
Ca	10000	SPEX	0.50	50
Cd	1000	CPI	0.050	0.5
Co	1000	CPI	0.10	1.0
Cr	1000	CPI	0.10	1.0
Cu	1000	CPI	0.20	2.0
Fe	10000	CPI	0.050	5.0
K	10000	CPI	1.0	100
Mg	10000	SPEX	0.50	50
Mn	1000	CPI	0.10	1.0
Mo	1000	CPI	0.10	1.0
Na	10000	CPI	1.0	100
Ni	1000	CPI	0.40	4.0
Pb	1000	CPI	0.10	1.0
Sb	1000	CPI	0.20	2.0
Se	1000	CPI	0.20	2.0
Sn	1000	CPI	0.50	5.0
Sr	1000	CPI	0.10	1.0
Ti	1000	CPI	0.10	1.0
Tl	1000	CPI	0.25	2.5
V	1000	CPI	0.10	1.0
Zn	1000	CPI	0.20	2.0

RL ICP Working
Final Volume (mL): 1000
Solvent: 5% HCL / 1 % HNO3

Purchased Standards	Concentration (mg/L)	Vendor *	Volume Used (mL)	Final Concentration (mg/L)
From RL ICP Intermediate				
Ag	1.0	CPI	10.0	0.010
Al	20	SPEX		0.20
As	2.0	CPI		0.020
B	10	CPI		0.10
Ba	1.0	CPI		0.010
Be	0.4	CPI		0.0040
Ca	50	SPEX		0.50
Cd	0.5	CPI		0.0050
Co	1.0	CPI		0.010
Cr	1.0	CPI		0.010
Cu	2.0	CPI		0.020
Fe	5.0	CPI		0.050
K	100	CPI		1.0
Mg	50	SPEX		0.50
Mn	1.0	CPI		0.010
Mo	1.0	CPI		0.010
Na	100	CPI		1.0
Ni	4.0	CPI		0.040
Pb	1.0	CPI		0.010
Sb	2.0	CPI		0.020
Se	2.0	CPI		0.020
Sn	5.0	CPI		0.050
Sr	1.0	CPI		0.010
Ti	1.0	CPI		0.010
Tl	2.5	CPI		0.025
V	1.0	CPI		0.010
Zn	2.0	CPI		0.020

ICV

Final Volume (mL): 500

Solvent: 5% HCL / 1 % HNO3

Purchased Standards	Concentration (mg/L)	Vendor *	Volume Used (mL)	Final Concentration (mg/L)
Single Element Stocks				
K	1000	Element	5.0	10
Na	1000	Element	4.5	9.0
Sn	1000	Element	0.50	1.0
Sr	1000	Element	0.50	1.0
Multi Element Stocks				
QC19				
As	100	Element	5.0	1.0
Be	100			1.0
Ca	100			1.0
Cd	100			1.0
Co	100			1.0
Cr	100			1.0
Cu	100			1.0
Fe	100			1.0
Mg	100			1.0
Mn	100			1.0
Mo	100			1.0
Ni	100			1.0
Pb	100			1.0
Sb	100			1.0
Se	100			1.0
Tl	100			1.0
V	100			1.0
Zn	100			1.0
QC7				
Ag	100	Element	5.0	1.0
Al	100			1.0
B	100			1.0
Ba	100			1.0
K	1000			10
Na	100			1.0
Si	50			0.50

ICSA

Final Volume (mL): 1000
Solvent: 5% HCL / 1 % HNO3

Purchased Standards	Concentration (mg/L)	Vendor *	Volume Used (mL)	Final Concentration (mg/L)
Multi Element Mixes				
Interferents A				
Al	5000	SPEX	100	500
Ca	5000			500
Mg	5000			500
Fe	2000			200

ICSAB
Final Volume (mL): 1000
Solvent: 5% HCL / 1 % HNO3

Purchased Standards	Concentration (mg/L)	Vendor *	Volume Used (mL)	Final Concentration (mg/L)
Multi Element Mixes				
Interferents A				
Al	5000	SPEX	100	500
Ca	5000			500
Mg	5000			500
Fe	2000			200
Altany C				
Ag	20	Spectro Pure	10	0.20
As	10			0.10
Ba	50			0.50
Be	50			0.50
Cd	100			1.0
Co	50			0.50
Cr	50			0.50
Cu	50			0.50
Mn	50			0.50
Ni	100			1.0
Pb	5			0.050
Sb	60			0.60
Se	5			0.050
Tl	10			0.10
V	50			0.50
Zn	100			1.0
Trace AB				
Mo	100	CPI	10	1.0
Sn	100			1.0

Trace AB
Final Volume (mL): 100
Solvent: 5% HCL / 1 % HNO3

Purchased Standards	Concentration (mg/L)	Vendor *	Volume Used (mL)	Final Concentration (mg/L)
Single Element Stocks				
Mo	1000	CPI	10	100
Sn	1000	CPI	10	100

High Std
Final Volume (mL): 1000
Solvent: 5% HCL / 1 % HNO3

Purchased Standards	Concentration (mg/L)	Vendor *	Volume Used (mL)	Final Concentration (mg/L)
Single Element Stocks				
Ag	1000	CPI	1.0	1.0
Al	10000	SPEX	1.0	10
B	1000	CPI	10	10
Fe	10000	CPI	1.0	10
Na	10000	CPI	0.50	5.0
Sr	1000	ABSOLUTE	5.0	5.0
Ti	1000	CPI	1.0	1.0
Multi Element Mixes				
CAL STD 2				
As	50	CPI	20	1.0
Mo	50			1.0
Pb	50			1.0
Sb	50			1.0
Se	500			10
Tl	500			10
CAL STD 3				
Ba	500	CPI	20	10
Be	50			1.0
Cd	50			1.0
Co	50			1.0
Cr	500			10
Cu	500			10
Mn	500			10
Ni	250			5.0
Zn	250			5.0
CAL STD 5				
Ca	500	CPI	20	10
K	1000			20
Mg	500			10
Na	500			10
Sn	500			10
V	500			10

Attachment 6:
Silica/Silicon Standard Preparation

Silicon (Si) stock solutions are usually purchased for this procedure. The following conversion is used to adjust any volumes or concentrations appropriately:

$$Si = \frac{SiO_2}{2.14}$$

- Stock SiO_2 Standard, 10000mg/L Si / 21400mg/L SiO_2 – purchased from CPI. Store at room temperature. This standard must be used by the manufacturer's expiration date.

- Stock Second Source SiO_2 Initial Calibration Verification (ICV), 50mg/L Si / 107mg/L SiO_2 QC7 Standard, purchased from Element. Store at room temperature. This standard must be used by the manufacturer's expiration date.

  Note: The ICV must be prepared from a stock standard that is obtained from a different source than the stock standard used to prepare the calibration standard. The second source must be from a separate vendor, unless a second vendor is unavailable, in which case a separate lot from the same vendor may be used.

- Intermediate SiO_2 Standard, 467mg/L Si / 1000mg/L SiO_2 – Add 20mL to 30mL of reagent water to a clean, plastic 100mL volumetric flask. Add the volume of the Stock SiO_2 Standard given in the table below to the volumetric flask. Dilute to volume with reagent water. Store the standard at room temperature. This standard must be used by its parent standard's expiration date or within 6 months of preparation, whichever comes first.

Element	Conc. Stock SiO_2 Std (mg/L)	Volume Stock SiO_2 Std (mL)	Final Volume (mL)	Final Conc. (mg/L)
Silica (SiO_2) [Silicon (Si)]	21400 [10000]	4.67	100	1000 [467]

- Initial Calibration Standards

  - Preparation of the Calibration Blank (ICB, CCB) – The calibration blank is reagent water. The calibration blank is used as the initial calibration blank (ICB) and the continuing calibration blank (CCB).

  - High Level SiO_2 Calibration Standard, 10mg/L SiO_2 – Add 20mL to 30mL of reagent water to a clean, plastic 100-mL volumetric flask. Add the volume of the Intermediate SiO_2 Standard given in the table, below, to the volumetric flask. Dilute to a final volume of 100mL with reagent water. Store the standard at room temperature. This standard must be used by its parent standard's expiration date or within 6 months of preparation, whichever comes first.

Element	Conc. SiO_2 Intermediate Std (mg/L)	Volume SiO_2 Intermediate Std (mL)	Final Volume (mL)	Final Conc. (mg/L)
Silica (SiO_2)	1000	1.0	100	10

- Mid Level SiO_2 Calibration Standard (also used as CCV solution), 5.0mg/L SiO_2 – Add 20mL to 30mL of reagent water to a clean, plastic 100mL volumetric flask. Add the volume of the stock standard given in the table, below, to the volumetric flask. Dilute to volume with reagent water. Store the standard at room temperature. This standard must be used by its parent standard's expiration date or within 6 months of preparation, whichever comes first.

Element	Conc. SiO_2 Intermediate Std (mg/L)	Volume SiO_2 Intermediate Std (mL)	Final Volume (mL)	Final Conc. (mg/L)
Silica (SiO_2)	1000	0.50	100	5

- Low Level SiO_2 Calibration Standard (also used as RL solution), 0.50mg/L – Add 20mL to 30mL of reagent water to a clean, plastic 100mL volumetric flask. Add the volume of the stock standard given in the table, below, to the volumetric flask. Dilute to volume with reagent water. Store the standard at room temperature. This standard must be used by its parent standard's expiration date or within 6 months of preparation, whichever comes first.

Element	Conc. SiO_2 Intermediate Std (mg/L)	Volume SiO_2 Intermediate Std (mL)	Final Volume (mL)	Final Conc. (mg/L)
Silica (SiO_2)	1000	0.050	100	0.50

- Second Source SiO_2 ICV Standard, 0.50mg/L SiO_2 / 1.07mg/L Si – Add 20mL to 30mL of reagent water to a clean, plastic 100mL volumetric flask. Add the volume of the stock standard given in the table to the volumetric flask. Dilute to volume with reagent water. Store the standard at room temperature. This standard must be used by its parent standard's expiration date or within 6 months of preparation, whichever comes first.

Element	Conc. of Stock Std (currently from Element) (mg/L)	Volume Stock Std (mL)	Final Volume of Cal Std mL)	Final Conc. (mg/L)
Silica (SiO_2) [Silicon (Si)]	107 [50]	1.0	100	1.07 [0.50]

- Stock ICP Interference A Check Standard – purchased Interference A Stock Standard, from SPEX. Store the standard at room temperature. This standard must be used by its manufacturer's expiration date.

  Note: This solution does not contain any SiO_2. Concentrations are as follows:

Aluminum (Al)	5000mg/L
Calcium (Ca)	5000mg/L
Magnesium (Mg)	5000mg/L
Iron (Fe)	2000mg/L

- ICP Interference A Check Standard, Working – Add 20mL to 30mL of reagent water to a clean 100-mL plastic volumetric flask. Add 10mL of the Stock ICP Interference A Check Standard to the volumetric flask. Dilute to a final volume of 100mL with reagent water. Store the standard at room temperature. This standard must be used by its parent standard's expiration date or within 6 months of preparation, whichever comes first.

  Note: This solution does not contain any SiO_2. Final concentrations are as follows:

Aluminum (Al)	500mg/L
Calcium (Ca)	500mg/L
Magnesium (Mg)	500mg/L
Iron (Fe)	200mg/L

- ICP Interference Check Solution AB – Add 20mL to 30mL of reagent water to a clean 100-mL plastic volumetric flask. Add 10mL of Stock ICP Interference A Check Standard and 0.50mL of the Intermediate SiO_2 Standard to the volumetric flask. Dilute to a final volume of 100mL with reagent water. Store the standard at room temperature. This standard must be used by its parent standard's expiration date or within 6 months of preparation, whichever comes first.

  The final concentrations of this solution should be as follows:

Aluminum (Al)	500mg/L
Calcium (Ca)	500mg/L
Magnesium (Mg)	500mg/L
Iron (Fe)	200mg/L
Silica (SiO_2)	5.0mg/L

- Linearity Check Solution, 100mg/L – P Add 20mL to 30mL of reagent water to a clean 100-mL plastic volumetric flask. Add 10mL of Intermediate SiO_2 Standard to the volumetric flask. Dilute to a final volume of 100mL with reagent water. Store the standard at room temperature. This standard must be used by its parent standard's expiration date or within 6 months of preparation, whichever comes first.

  Note: Historically, the Linear Dynamic Range for silica has been 100mg/L; however, the instrument is dynamic. The concentration of this standard may need to be adjusted based how the instrument is performing. Prepare other volumes and/or concentrations according to the following equation:

$$Vs = \frac{Vlc \otimes Clc}{Cs}$$

where
V_s = volume of stock standard (mL)
C_s = concentration of stock standard (mg/L)
V_{lc} = volume of linearity check standard to prepare (mL)
C_{lc} = concentration of linearity check standard to prepare (mg/L)

Attachment 7:
Linear Dynamic Range Determination and Verification

Initial Linear Dynamic Range Determination:
The linear dynamic range (LDR) of each instrument must be determined initially, upon instrument set-up, and whenever significant instrument changes are made following the procedure outlined below:

- Prepare and analyze individual standards at concentrations (a minimum of three are recommended) that are expected to define the linear dynamic range of the instrument.

  Note: The calibration standards and the LDR standards must be matrix matched; that is, they must have the same percentage of hydrochloric and nitric acids.

- Compare the concentration of each standard with its true concentration as follows:

$$PercentDifference = \left| \frac{C_{cal} - C_{true}}{C_{true}} \right| \otimes 100$$

  Where:
  C_{cal} = concentration determined from analysis
  C_{true} = true concentration of the standard

- Continue analyzing increasing concentrations of standards (recommend increasing by a factor of two) until the percent difference is greater than 10%. The highest standard that meets the 10% criteria establishes the initial LDR of the instrument.

Note: The laboratory routinely utilizes a "working" LDR that is equivalent to the concentration of the High Standard of the calibration curve. This working LDR allows for consistency between instruments and must not be greater than the LDR established by the initial LDR study.

Linear Dynamic Range Verification:
The working LDR must be verified quarterly by analyzing a standard at this concentration. If the percent difference is less than or equal to 10%, the working LDR is confirmed at that concentration. If the percent difference is greater than 10%, repeat the initial LDR Study as outlined above.

Evaluation of Samples with Relation to the Linear Dynamic Range:
Samples with concentrations greater than 90% of the working LDR must be diluted to bring the concentration below 90% of the working LDR.

Attachment 8:
Element Wavelengths

Element	Wavelength ICP-D (nm)	Wavelength ICP-E (nm)
Aluminum (Al)	308.215	308.215
Antimony (Sb)	206.838	206.834
Arsenic (As)	189.042	188.98
Barium (Ba)	493.409	389.178
Beryllium (Be)	313.042	313.042
Boron (B)	249.678	249.678
Cadmium (Cd)	226.502	226.502
Calcium (Ca)	317.933	370.602
Chromium (Cr)	267.716	267.716
Cobalt (Co)	228.616	228.615
Copper (Cu)	324.753	324.754
Iron (Fe)	271.441	271.441
Lead (Pb)	220.353	220.353
Magnesium (Mg)	279.078	279.078
Manganese (Mn)	257.610	257.610
Molybdenum (Mo)	202.030	202.032
Nickel (Ni)	231.604	231.604
Potassium (K)	766.491	766.491
Selenium (Se)	196.026	196.026
Silver (Ag)	328.068	328.068
Sodium (Na)	330.231	330.237
Strontium (Sr)	421.542	216.596
Thallium (Tl)	190.864	190.794
Tin (Sn)	189.989	189.925
Titanium (W)	334.941	334.941
Vanadium (V)	292.402	292.401
Zinc (Zn)	206.200	206.200

Note: Other wavelengths may be used with DM and TM approval.

Attachment 9:
Hardness Calculation Work Instruction

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

Work Instruction
FME157:05.23.07:0
Page 1 of 1

CALCULATION OF HARDNESS

(as Calcium Carbonate)

Method: SM2340B

Summary of Procedure:

An aqueous sample is analyzed for calcium and magnesium using ICP by methods EPA 200.7 or 6010B (SOP ME70: *Elements by ICP)*, or using ICP/MS by methods EPA 200.8 or 6020 (SOP ME74: *Elements by ICP/MS)*. The concentrations of these elements are converted to equivalents of calcium carbonate using the equation below and added together to give the final result.

Calculation:

Determine the concentration of calcium and magnesium as described in SOP ME70 or ME74.

Calculate the hardness as calcium carbonate using the following equation:

Hardness (as mg/L calcium carbonate) = (4.118 X [Mg]) + (2.497 X [Ca])

where:
[Mg] = Concentration of magnesium (mg/L)
[Ca] = Concentration of calcium (mg/L)

Quality Control:

Report the method blank as the default QC unless otherwise requested.

Formal detection limit studies, as described in 40CFR Part 136B and SOP QA07: *Determination of Detection Limits,* are not required for SM2340B.

This procedure is a calculation based on another analytical procedure; therefore, demonstration of capability (DOC) is accomplished by successful completion of analytical DOCs. (i.e. If the analyst has a successful DOC on file for Ca and Mg by EPA 200.7/6010B or EPA 200.8/6020, proficiency has been shown for SM2340B by default.)

18.0 Revision History

Summary of Changes from Previous Revision:

- Minor editorial and/or grammatical changes made.
- Revised definition of insufficient sample volume received to perform MS/MSD from 100mL to 50mL. Section 9.1.4
- Revised section on IDLs to incorporate use of blanks for IDL determination as opposed to using spiked replicates. Section 12.4
- Revised ISTD preparation instructions to include new amount and final concentration of lithium carbonate powder. Attachment 5
- Revised Stock Second Source SiO_2 ICV Standard vendor from Absolute to Element. Attachment 5
- Revised requirement for ICBs and CCBs for EPA 6010B to match that prescribed in EPA 200.7 and EPA 6010C. Added applicable information to method modification section. Section 9.2.4, Section 16.2.5, and Attachment 3
- Revised internal standard upper limit criteria from 120% to 130% to indicate samples that require dilution, and changed lower limit criteria from 50% to 70% to indicate samples for which dilution should be considered. Section 9.2.6
- Revised procedure for linear dynamic range determinations. Clarified initial LDR procedures versus continuing LDR verification procedure. Attachment 7
- Removed table of corrective actions associated with PDS (including information on MSA). When PDS is not acceptable, samples are qualified and reported. Included additional information in the Method Modifications section. Revised criteria from 20% to 25%. Section 9.2.10
- Revised %RSD criteria from 3% to 5% for ICV. Section 9.2.3
- Revised section on %RSD of multiple exposures. Added 5% criteria requirement. Section 9.2.11
- Included capping RL Check Standard requirement in Attachment 3. Revised criteria for capping to allow reporting samples with qualification (provided project objectives are met). Section 10.3.5 and Attachment 3
- Removed section on MARRS Data Reduction and Reporting as this software is no longer used to evaluate data. Section 11.1.4
- Added section on evaluation of batch QC samples for matrix interferences and revised requirements/frequency for MSA. Section 11.1.4
- Revised criteria and corrective action to be taken for post digestion spikes. Section 11.1.5
- Clarified Waste Disposal instructions. Section 14.1

VOLATILE COMPOUNDS BY GC/MS

(Methods: EPA 8260B, EPA 624, and SM6200B)

Approvals (Signature/Date):

Ammie L. Martin February 21, 2012
Ammie Martin Date
Quality Assurance Specialist, Designee for:
Andrea Teal, Quality Assurance Manager / Interim Environmental Health and Safety Coordinator

December 27, 2011
Benjamin Gulizia Date
Laboratory Director / Lead Technical Director

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1.0 Scope and Application

This SOP contains the procedures for the determination of volatile organic compounds (VOC) by purge and trap gas chromatography/mass spectrometry (GC/MS). This procedure is applicable to a wide variety of low molecular weight compounds that have low aqueous solubility and boiling points less than 200°C.

The routine matrices for this procedure are waters and soils (for EPA 8260B) and waters (for EPA 624 and SM6200B); however, this procedure may be adapted to accommodate other matrices as outlined in Section 16.1. This procedure may also be used to perform Custer Rule analyses for chloroform only (via a modified EPA 624 analysis).

A complete target analyte list, the reporting limits (RL), the method detection limits (MDL), and the accuracy and precision criteria associated with this procedure are provided in the LIMS Method Limit Groups (MLGs).

2.0 Summary of Method

Volatile organic compounds (VOCs) are purged from the sample matrix with helium. The VOCs are transferred from the sample matrix to the vapor phase. The vapor is swept through a sorbent tube where the VOCs are trapped. After the purging is completed, the trap is heated and backflushed with helium to desorb the VOCs onto a GC column. The GC is temperature-programmed to separate the VOCs, which are then detected by a mass spectrometer. Qualitative identification of the target compounds in the sample is based on the relative retention time and the mass spectra of the characteristic masses (ions) determined from standards analyzed on the same GC/MS under the same conditions. Quantitative analysis is performed using the internal standard technique with a single characteristic ion.

Water samples are routinely purged at ambient conditions; however, a heated purge may be used if required by the project. A 5mL purge volume is used as the default. The calibration standards and the associated QC must be analyzed under the same conditions and volume. This sample introduction procedure is based on EPA 5030B, EPA 624, and SM6200B.

Low-level (nominally<1mg/kg) soil samples are purged at 40°C in a purge and trap instrument designed to add water and internal standards to the vial containing the sample without breaking the seal. The sample is stirred during purging to thoroughly mix the soil and water. The calibration standards and associated QC are purged under the same conditions. This sample introduction procedure is based on EPA 5030A (for soil samples collected in bulk) and EPA 5035A (for soil samples collected via Encore/Terracore devices).

High level soils (nominally>1mg/kg) and waste samples are extracted with methanol (1mL of methanol per gram of sample). An aliquot of the methanol extract is injected into reagent water. The methanol extract/reagent water is purged at ambient temperature using the same instrument conditions and calibration used for aqueous samples.

This SOP is based on EPA 8260B, EPA 624, and SM6200B.

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3.0 Definitions

Refer to the Glossary Section of the *Quality Assurance Manual* (QAM) for a complete listing of applicable definitions and acronyms.

4.0 Interferences

4.1 Procedural Interferences

4.1.1 Interferences may be caused by contaminants in solvents, reagents, glassware, and other sample processing apparatus and can make identification and/or quantification of the target analytes difficult.

4.1.2 All sample collection containers are single-use disposable containers which limits the potential for contamination. All non-disposable labware must be scrupulously cleaned in accordance with the posted Labware Cleaning Instructions to ensure it is free from contaminants and does not contribute artifacts.

4.1.3 High purity reagents and solvents are used to help minimize interference problems. Methanol and Hydrochloric Acid must be verified prior to use in accordance with the TestAmerica Solvent Lot Testing Program.

4.1.4 Instrument and/or method blanks are routinely used to demonstrate all reagents and apparatus are free from interferences under the conditions of the analysis.

4.1.5 VOCs commonly used in the laboratory are potential sources of contamination. Methylene chloride, acetone, Freon-113, MEK, hexane, toluene, and isopropanol are used in the laboratory and tend to present the most problems.

4.1.5 The Teflon seals of the purge and trap device can absorb and outgas many of the compounds that are included in this method. These Teflon fittings should be periodically checked for integrity. If the contamination is suspected, the fittings may be heated at 105°C for one hour or replaced.

4.1.6 The addition of acid to the sample during collection will cause the degradation of several target compounds. Acrolein and acrylonitrile recovery may be reduced and 2-chloro-ethyl vinyl ether will be completely degraded. The recovery of 2-chloroethyl vinyl ether will also be reduced as the purge and trap lines become acidic. For this reason, unpreserved vials must be utilized if these analytes are requested.

4.2 Matrix Interferences

4.2.1 Matrix interferences may be caused by contaminants that are purged from the sample matrix.

Contamination by carryover can occur whenever high concentration samples and low concentration samples are analyzed sequentially. As such, samples known to be clean should be analyzed first. Where practical, high concentration samples should be followed by a blank to check for cross-contamination. If the targets found in the highly

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concentrated sample are found in subsequent samples, the analyst must verify that the port and P/T system are not contributing contamination to the subsequent samples. If the target compound(s) are not present in subsequent samples, the analysis of a blank is not required but may be a prudent preventative measure. Frequent trap bakeout and purging of the entire purging system may be necessary when carry-over is suspected. Reagent blanks must be analyzed when contamination is suspected to ensure that the system is free from contamination.

A common type of contamination is from samples containing high concentrations of hydrocarbons such as gasoline or mineral spirits. At high concentrations, these compounds may cause elevated baselines that can obscure the mass ion signals of target compounds with masses similar to the hydrocarbons. For example, a common mass in hydrocarbons is mass 43, which is also present in many ketones. Mineral spirits at high concentrations can be very problematic as it contains hydrocarbons beyond C12, which can linger in the purge and trap unit and carryover for quite a few samples. High concentrations of hydrocarbons can also degrade the trap much more quickly than would be expected.

4.2.3 The volatiles laboratory must be kept as free from contamination as possible. Highly contaminated samples must be segregated from routine samples. Contact with sections of the laboratory where solvents are used should be minimized. Refrigerator and freezer blanks must be prepared, stored, and analyzed to evaluate the sample storage areas for possible contamination. Guidance is provided in SOP SA-QA-15: *Homogenization, Compositing, and Segregation of Samples.*

4.2.4 Matrix interferences may be overcome by the use of the secondary ions for quantitation. An example of this is the use of mass 82 for quantitation with chlorobenzene-d5 internal standard when a potential co-eluter, 1,1,1,2-tetrachloroethane, is a target compound. One of the mass fragments of 1,1,1,2-tetrachloroethane is mass 117, which is the recommended quantitation ion for chlorobenzene-d5. The use of the secondary ions should be used for quantitation in such cases when the laboratory can clearly demonstrate matrix problems. Mass 58 is recommended for quantitation of acetone due to the elution of a hydrocarbon at the same retention time.

5.0 Safety

Employees must abide by the policies and procedures in the TestAmerica Environmental Health and Safety Manual (EHSM), the TestAmerica Savannah Addendum to the EHSM, and this document.

This procedure may involve hazardous materials, 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 to follow appropriate safety, waste disposal, and health practices under the assumption that all samples and reagents are potentially hazardous.

The analyst must protect himself/herself from exposure to the sample matrix. Many of the samples that are tested may contain hazardous chemical compounds or biological organisms. The analyst must, at a minimum, wear protective clothing (lab coat), eye protection (safety glasses or face shield), disposable gloves, and closed-toe,

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nonabsorbent shoes when handling samples. Note: Cut-resistant gloves should be worn, or other hand protection material used, when opening and closing VOA vials.

5.1 Specific Safety Concerns or Requirements

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

The mass spectrometer is under deep vacuum. The mass spectrometer must be brought to atmospheric pressure prior to working on the source.

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.

The exit vent of the split injector must have a carbon trap in-line to collect the volatile compounds that are vented during the injection of the sample. The traps should be changed a minimum of every three months and must be disposed of in accordance with Section 9 of the TestAmerica Savannah Addendum to the EHSM.

Methanol is a flammable solvent. It can cause irritation to the respiratory tract. Overexposure can cause fatigue, confusion, headache, dizziness, and drowsiness.

Hydrochloric acid is extremely hazardous as an oxidizer, a corrosive, a poison, and is reactive. Inhalation of the vapors can cause coughing, choking, irritation of the nose, throat, and respiratory tract, breathing difficulties, and lead to pneumonia and pulmonary edema. Contact with the skin can cause severe burns, redness, and pain. Acid vapors are irritating and can cause damage to the eyes. Contact with the eyes can cause permanent damage. Concentrated acids should be used in a fully functional fume hood.

5.2 Primary Materials Used

The following is a list of the materials used in this procedure, which have a serious or significant hazard rating, and a summary of the primary hazards listed in their MSDS.

Note: This list does not include all materials used in the procedure. A complete list of materials used in this procedure can be found in the Reagents and Standards Section and the Equipment and Supplies Section of this SOP

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 found using the "MSDS" link on the Oasis homepage, on the EH&S webpage on Oasis, and on the QA Navigator.

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Material	Hazards	Exposure Limit[1]	Signs and Symptoms of Exposure
Methanol	Flammable Poison Irritant	200ppm 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.
Hydrochloric Acid	Corrosive Poison	5ppm 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]Exposure limit refers to the OSHA regulatory exposure limit.			
Note: Always add acid to water to prevent violent reactions.			

6.0 Equipment and Supplies

6.1 Equipment and Instrumentation

A list of the instruments, with their basic configuration, is provided in Attachment 8. These instruments were in use at the time of SOP revision. Other instruments and configurations may be used provided they are fully documented and validated in accordance with laboratory procedures.

Top-loading Balance – Verify in accordance with SOP SA-AN-100: *Laboratory Support Equipment (Verification and Use)*

6.2 Analytical Data System / Software / Hardware

Chemstation software is used on a Windows-based PC to schedule and acquire data. Target (UNIX and/or Windows) software is used on a Windows-based PC to store, reduce/evaluate, and output the data to the laboratory's LIMS system (i.e., TALS). Target software has the capability of processing stored GC/MS data by recognizing a GC peak within any given retention time window, comparing the mass spectrum from the GC peak with spectral data in a user-created data base, and generating a list of tentatively identified compounds with their retention times and scan numbers. The software also allows integration of the ion abundance of any specific ion between specified time or scan number limits, calculation of response factors as or construction of a linear regression

calibration curve, calculation of response factor statistics (mean and standard deviation), and calculation of concentrations of analytes using either the calibration curve or the response factors.

6.3 Lab Supplies

Supelco Vocarb 3000 trap or equivalent – Other traps may be used as long as the target compounds can be detected at the required quantitation limit.

Recommended Column: J&W DB-624: 20m x 0.18mm ID, 1.8um film

Volumetric Containers – various sizes; Class A, where applicable. Verify in accordance with SOP SA-AN-100: *Laboratory Support Equipment (Verification and Use)*

Pump-style Pipettes – various sizes. Verify in accordance with SOP SA-AN-100: *Laboratory Support Equipment (Verification and Use)*

Gas-Tight Syringes – various sizes. Verify in accordance with SOP SA-AN-100: *Laboratory Support Equipment (Verification and Use)*

6.4 Sample Collection Containers

All sample collection containers are single-use disposable containers which limits the potential for contamination. Containers are received from vendors with a statement that the containers are suitable for the intended use and have been tested and certified to be free of contamination.

Refer to SOP SA-VO-001: *Preparation, Screening, and Storage of Volatiles Samples* for the containers routinely used to collect field samples.

7.0 Reagents and Standards

7.1 Expiration Dates

Expiration dates (time from initial use or receipt to final use) for standard and reagent materials must be set according to the guidance in this SOP. Note: These are maximum expiration dates and are not to be considered an absolute guarantee of standard or reagent quality. Sound judgment must be used when deciding whether to use a standard or reagent. If there is doubt about the quality of a standard or reagent material, a new material must be obtained or the standard or reagent material verified. Data quality must not be compromised to extend a standard's life – i.e., when in doubt, throw it out.

The expiration date of any standard must not exceed the expiration date of the standard that was used to prepare it; that is, the "children may not outlive the parents".

7.2 Reagents

Reagents must be prepared and documented in accordance with SOP SA-AN-041: *Reagent and Standard Materials Traceability.*

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Methanol and Hydrochloric Acid must be verified prior to use in accordance with the TestAmerica Solvent Lot Testing Program.

7.2.1 Reagent water – ASTM Type II; lab generated water

7.2.2 Blank sand – Macro Chemicals; used as blank matrix for soil samples; Purify by heating at 160°C for four hours or longer in a shallow tray.

7.2.3 Methanol – for purge and trap analysis; J.T. Baker 9077-02 (1L)

7.3 Standards

Standards must be prepared and documented in accordance with SOP SA-AN-041: *Reagent and Standard Materials Traceability*. Certificates of analysis or purity must be received with all purchased standards, and scanned and filed in the Data Archival Folder on the G-drive.

The recipes for the preparation of standards are provided in Attachment 9. The recipes contain the stock standards, preparation steps, storage, and expiration dates for the routine target compounds.

8.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

Refer to Attachment 2 for a summary of the routine containers, holding times, preservation requirements, etc.

Refer to SOP SA-VO-001: *Preparation, Screening, and Storage of Volatiles Samples* for information on the preservation and dechlorination checks required for these methods.

Samples must be iced or refrigerated (at <6°C, with no frozen samples) from the time of collection until analysis.

9.0 Quality Control

SOP SA-QA-17: *Evaluation of Batch QC Data* and the QC Summary in Attachment 3 provide requirements for evaluating QC data.

9.1 Batch QC

All batch QC must meet the criteria given in Attachment 3 of this SOP.

9.1.1 Soil Samples

A batch consists of up to 20 environmental samples and the associated QC items. The laboratory defaults to the following as the minimum QC items required for each batch: a method blank, a laboratory control sample (LCS), a matrix spike (MS), and a matrix spike duplicate (MSD).

If there is insufficient sample to perform the MS/MSD, the LCS must be prepared in duplicate (i.e., LCS/LCSD). An NCM must be initiated on all affected samples to denote

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this situation. Insufficient sample is defined as receiving less than 30g for bulk samples or less than 4 Encores/Terracores.

Note: If an LCS and LCSD are performed, both QC items must be evaluated and reported. Acceptable recoveries (as well as %RPD) for both LCS and LCSD are required.

9.1.2 Aqueous Samples

A batch consists of up to 20 environmental samples and the associated QC items. The laboratory defaults to the following as the minimum QC items required for each batch:

EPA 8260B and SM6200B:
method blank, a laboratory control sample (LCS), a matrix spike (MS), and a matrix spike duplicate (MSD).

EPA 624:
method blank, a laboratory control sample (LCS), a matrix spike (MS) performed per 10% of samples analyzed, and a matrix spike duplicate (MSD). This equates to 1 MS and 1MSD for a batch of 10 or less samples or equates to 1 MS (from sample 1-10), 1 MS (from sample 11-20), and 1 MSD for a batch of 11-20 samples.

If there is insufficient sample to perform the required MS and/or MSD, the LCS must be prepared in duplicate (i.e., LCS/LCSD). An NCM must be initiated on all affected samples to denote this situation. Insufficient sample is defined as receiving less than less than 4 vials.

Note: If an LCS and LCSD are performed, both QC items must be evaluated and reported. Acceptable recoveries (as well as %RPD) for both LCS and LCSD are required.

9.1.3 Poor Performers / Erratic Compounds

As indicated in EPA 8260B and/or via assessment of laboratory control sample recoveries and control charts, the compounds listed in Attachment 10 are Poor Performers and/or behave erratically. These compounds will not be included in the LCS/LCSD/MS/MSD marginal exceedance count, provided their %R is >10%.

Note: An NCM must be initiated to denote this situation.

9.2 Instrument QC

The term "clock time" or "analytical clock" refers to the amount of time that can pass before additional instrument QC items must be performed. The analytical clock begins with the injection of the BFB, and all subsequent injections must be completed before the clock time expires - at which point new instrument QC is performed and a new clock is initiated.

The clock times are defined as follows:

- EPA 8260B and SM6200B= 12 hours
- EPA 624 = 24 hours

- EPA 624 Cluster Rule (chloroform only) = 8 hours

Note: Due to instrument configurations employing dual concentrators, most of the laboratory instruments can analyze more than 20 injections within the designated clock times. An analytical batch is still defined as 20 field samples; therefore, if more than 20 field samples are analyzed within a clock, additional batch QC is required (i.e., another method blank, LCS, and LCSD or MS/MSD must be performed).

9.2.1 BFB Tune Check

9.2.1.1 Fifty nanograms of 4-BFB must be analyzed at the beginning of each clock as a check on the "tune" of the mass spectrometer. Meeting the tuning criteria ensures that the instrument is measuring the proper masses in the proper ratios. The 4-BFB analysis takes place under the same instrument conditions as the calibration standards and samples except that a different temperature program can be used to allow for the timely elution of 4-BFB. All other instrument conditions must be identical - the mass range, scan rate, and multiplier voltage.

If the instrument is configured for direct injection, 50ng of 4-BFB may be injected directly on to the column. If the purge and trap is used to analyze the 4-BFB, the purge and trap conditions must be the same as for the calibration standards and samples.

9.2.1.2 Evaluation of the 4-BFB peak

9.2.1.2.1 The chromatogram must exhibit acceptable baseline behavior and the 4-BFB peak must be symmetrical (i.e., Gaussian). A spectrum of the baseline that shows high abundances of mass 40 (Argon) and mass 44 (carbon dioxide) may indicate a leak or contaminated carrier gas.

9.2.1.2.2 The spectrum of the 4-BFB must meet the criteria listed in Attachment 6. Background subtraction must be straightforward and designed only to eliminate column bleed or instrumental background. Scans ±1 scan from the apex can be evaluated for the 4-BFB criteria. Consecutive scans within this range can be averaged to meet the criteria.

9.2.1.2.3 The 4-BFB analysis should be evaluated as to the relative size of the 4-BFB peak under the m/z 95 profile. A benchmark area window should be established for each instrument. Response outside of this window suggests instrumental problems such as a poor purge, clogged jet separator, leak in the Tekmar purging device, reduced or elevated detector sensitivity, improper electron multiplier voltage selection, wrong tune method or tune file selected for this analysis, PFTBA valve left open, or other anomalies.

9.2.1.2.4 If the 4-BFB fails to meet the acceptance criteria, the instrument may require tuning (manually or automatically with PFTBA). Depending on the nature of the results from the 4-BFB analysis, other corrective measures may include remaking the 4-BFB standard and/or cleaning the mass spectrometer source.

9.2.2 Trap Check Standard

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The trap check standard is used to evaluate the condition of the trap by monitoring the formation of chloromethane and bromomethane. Chloromethane and bromomethane may be formed on a degraded trap by thermal decomposition of halogenated compounds.

9.2.2.1 Prepare the trap check standard by injecting 2uL of a 50ug/mL bromoform standard into 5mL of reagent water. Other sample volumes may be used but the sample must transfer 100ng of bromoform to the column. Add the internal standards and surrogates. Analyze the sample using the same analytical system conditions used for samples and standards.

9.2.2.2 Evaluate the chromatogram for the presence of chloromethane and bromomethane. Compare the response to the 1.0ug/L standard. The response must be less than or equal to one half of the response of the 1.0ug/L standard, and the trap check standard must quantify less than 0.5ug/L when compared to the initial calibration curve.

Note: Ensure sure that the spectra match the reference spectra and that the most abundant ions are present for both compounds - chloromethane (m/z 50, 52) and bromomethane (94, 96).

9.2.2.3 If the trap check standard does not meet the acceptance criteria, the trap must be replaced, conditioned, and the system re-calibrated prior to the analysis of samples.

9.2.3 Initial Calibration (ICAL)

The instrument must be calibrated in accordance with SOP SA-QA-16: *Evaluation of Calibration Curves.* This SOP provides requirements for establishing the calibration curve and gives the applicable formulas.

Instrument calibration is performed by analyzing a series of known standards. The calibration curve must consist of a minimum of 5 standards. The lowest level calibration standard must be at or below the reporting limit, and the remaining standards will define the working range of the analytical system.

The initial calibration standard concentrations currently in use in the laboratory are as follows:

EPA 8260B and SM6200B:

Standard Level	Concentration (ng)	Final Concentration – Waters (ug/L)	Final Concentration – Soils (ug/kg)
1	5	1.0	
2	25	5.0	5.0
3	50	10	10
4	100	20	20*
5	250	50*	50
6	500	100	100
7	1000	200	200

*CCV Level

EPA 624:

Standard Level	Concentration (ng)	Final Concentration – Waters (ug/L)
1	5	1.0
2	25	5.0
3	50	10
4	100	20*
5	500	100
6	1000	200

*CCV Level

Refer to Attachment 9 for the standard preparation instructions. Other standard concentrations may be used provided they support the reporting limit and are fully documented in accordance with SOP SA-AN-041.

9.2.3.2 ICAL Criteria

9.2.3.2.1 EPA 8260B

The initial calibration is evaluated specifically for the calibration check compounds (CCC) and the system performance check compounds (SPCC). The CCC and SPCC criteria are given in Attachment 5 of this SOP. The %RSD criteria for CCC and minimum RRF for SPCC must be met before the analysis of samples can begin.

After the CCC and SPCC initial calibration criteria have been met, each target must be evaluated for linearity. The relative standard deviation of the calibration standards must be <15% for the initial calibration curve to be acceptable.

If one or more compounds do not meet the %RSD criterion, the next option is to evaluate a regression curve. The regression coefficient (r^2) of the regression curve must be greater than 0.990 for the initial calibration curve to be acceptable.

> Note: A minimum of 6 points is required for a quadratic curve. Higher order curves are not permitted. Some programs and agencies (e.g., SC DHEC) do not allow the use of quadratic curves. Refer to the Project Requirement Summary and/or Project Plan to determine if this curve type is prohibited.

SW-846 allows the use of the "grand mean exception" as described below. This exception should only be applied to initial calibration curves in extraordinary circumstances due to the difficulty of maintaining and providing documentation on an on-going basis.

> Grand Mean Exception (GME): If one or more analytes exceed the %RSD criteria, the calibration curve is acceptable if the average of the %RSDs for <u>all</u> of the analytes in the ICAL (i.e., the Grand Mean) is less than or equal to the ICAL %RSD criteria.

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SW-846 does not place a cap on an individual analyte's %RSD as long as the average is within criteria; however, the laboratory has adopted the requirement that no individual analyte can exceed 3X the ICAL criteria. Therefore, the calibration curve is acceptable if the average of the %RSDs is less than or equal 15% with no individual analyte exceeding 45%.

Note: Some programs and agencies do not allow the use of the Grand Mean Exception. Refer to the Project Requirement Summary and/or Project Plan to determine if GME is not allowed.

9.2.3.2.2 EPA 624

The relative standard deviation of the calibration standards must be <35% for the initial calibration curve to be acceptable. If one or more compounds do not meet the %RSD criterion of <35%, the next option is to evaluate a regression curve. The regression coefficient (r^2) of the regression curve must be greater than 0.990 for the initial calibration curve to be acceptable.

9.2.3.2.3 SM6200B

The relative standard deviation of the calibration standards must be <20% for the initial calibration curve to be acceptable. If one or more compounds do not meet the %RSD criterion, the next option is to evaluate a regression curve. The regression coefficient (r^2) of the regression curve must be greater than 0.994 for the initial calibration curve to be acceptable.

Note: If a regression curve is used, all standards must be re-quantitated as samples. The concentration determined must be within +/-20% of the expected concentration to be acceptable.

9.2.4 Second Source Initial Calibration Verification (ICV)

The calibration curve must be verified initially – prior to any sample analyses – in accordance with SOP SA-QA-16 with a standard obtained from a second source.

The initial calibration verification standard concentration currently in use in the laboratory is equivalent to the CCV concentration. Refer to Attachment 9 for the standard preparation instructions. Another standard concentration may be used provided it is mid-level and fully documented in accordance with SOP SA-AN-041.

9.2.4.1 EPA 8260B ICV Criteria

The initial calibration verification (ICV) is acceptable if the average %D of all the analytes in the ICV is less than or equal 20% with no individual analyte exceeding 60%.

The analytes listed in Attachment 10 behave erratically and/or are poor performers; therefore, these analytes may exceed the ICV %D criteria such that their %D may be >60% if the average %D of all the analytes in the ICV is <20%. An NCM must be initiated to denote this situation.

9.2.4.2 EPA 624 ICV Criteria

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The initial calibration verification (ICV) is acceptable if each analyte meets the criteria outlined in Attachment 12.

9.2.4.3 SM6200B ICV Criteria

The initial calibration verification (ICV) is acceptable if the average %D of all the analytes in the ICV is less than or equal 20% with no individual analyte exceeding 60%.

The analytes listed in Attachment 10 behave erratically and/or are poor performers; therefore, these analytes may exceed the ICV %D criteria such that their %D may be >60% if the average %D of all the analytes in the ICV is <20%. An NCM must be initiated to denote this situation.

9.2.5 Continuing Calibration Verification (CCV)

The initial calibration curve must be verified at the beginning of each clock with a mid-level standard.

For SM6200B, the CCV concentration should be varied throughout the range of the calibration curve and must also be performed at the end of the clock (i.e., capping CCV is required).

The continuing calibration verification standard concentration currently in use in the laboratory is specified in Section 9.2.3. Refer to Attachment 9 for the standard preparation instructions. Another standard concentration may be used provided it is mid-level and fully documented in accordance with SOP SA-AN-041.

9.2.5.1 EPA 8260B CCV Criteria

For EPA 8260B, the CCC and SPCC criteria (Attachment 5) must be met for the CCV to be acceptable.

Note: EPA 8260B requires only the CCC analytes to be evaluated for response; however, the laboratory has adopted stricter criteria. Therefore, in addition to the CCC and SPCC criteria listed in Attachment 5, the average %D of all non-CCC and non-SPCC analytes must be <20% with no single analyte's %D >60% for the CCV to be acceptable.

Note: The SPCC criteria must be met even if the regression curve option is used for quantitation.

In addition to the response criteria given in this section, the CCV must be evaluated for internal standard response. The extracted ion current profile (EICP) area for each of the internal standards in the CCV must be within -50% to +100% from the last initial calibration sequence to be acceptable. If these criteria are not met, the analytical system must be inspected for problems and corrective action instituted.

The analytes listed in Attachment 10 behave erratically and/or are poor performers; therefore, these analytes may exceed the CCV %D criteria such that their %D may be >60% if the average %D of all the analytes in the ICV is <20%. An NCM must be initiated to denote this situation.

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9.2.5.2 EPA 624 CCV Criteria

For EPA Method 624, all target analytes must meet the criteria outlined in Attachment 12 to be acceptable.

9.2.5.3 SM6200B CCV Criteria

For SM6200B, all gas compounds must be within 60-140%D and all other compounds must be within 70-130% to be acceptable.

9.2.6 Internal Standard (ISTD)

This procedure is an internal standard (ISTD) procedure. 1,2-dichloroethane-d4, 1,4-difluorobenzene, and chlorobenzene-d5 are the internal standards.

Prior to analysis, the internal standards must be added to all standards, samples, and QC items. The concentration of the internal standards must be the same in all calibration samples, field samples, and QC samples. A concentration of 50ug/L is used for each of the ISTD analytes.

EPA 8260B and EPA 624:
The response of the ISTD in the ICV/CCV must be within -50% to +100% of the response of the ISTD in the CCV-level standard in the initial calibration sequence. If the response is outside of this range, the analysis of the CCV must be repeated and any samples associated with the CCV must also be re-analyzed. Repeated failure of the ISTD response will require re-calibration.

The response of the ISTD in the samples and batch QC items must be within -50% to +100% of the response of the previous CCV. If the response is outside of this range, corrective action must be taken.

SM6200B:
The response of the ISTD in the ICV/CCV must be within +/-30% of the response of the ISTD in the CCV-level standard in the initial calibration sequence. If the response is outside of this range, the analysis of the CCV must be repeated and any samples associated with the CCV must also be re-analyzed. Repeated failure of the ISTD response will require re-calibration.

The response of the ISTD in the samples and batch QC items must be within +/-30% of the response of the previous CCV. If the response is outside of this range, corrective action must be taken.

9.2.7 Surrogates

This procedure uses surrogates to evaluate the analytical process. Dibromofluoromethane, toluene-d8, and p-BFB are the surrogates.

Prior to preparation, these surrogates must be added to all samples and QC items. The concentration of the surrogates must be the same in all field samples and QC samples. A concentration of 50ug/L is used for each of the surrogate analytes.

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The percent recovery of the surrogate in all field samples and QC samples must be within the limits listed in the Method Limit Groups (MLGs) in LIMS. If the percent recovery is outside of this range, the analysis of the sample must be repeated. Repeated failure of the surrogate percent recovery may indicate re-extraction is necessary.

9.3 Corrective Action for Out-of-Control Data

When the quality control parameters do not meet the criteria set forth in this SOP, corrective action must be taken in accordance with SOP SA-QA-05: *Preventive and Corrective Action Procedures* the QC Summary Table in Attachment 4. SOP SA-QA-05 provides contingencies for out-of-control data and gives guidance for exceptionally permitting departures from approved policies and procedures. Nonconformance Memos must be initiated to document all instances where QC criteria are not met and all departures from approved policies and procedures.

10.0 Procedure

10.1 Preparation

10.1.1 Aqueous Sample Preparation

Remove the samples from the refrigerator and allow them to come to room temperature.

Using a 50uL syringe, add 43uL of the ISSU to the sample, injecting the solution through the vial septum. Invert the vial several times to mix. Transfer to the instrument.

10.1.2 Aqueous QC Preparation

The method blank and LCS are prepared in reagent water using a 50mL volumetric flask.

- For the method blank, add 50uL ISSU to 50mL reagent water. Pour into a VOA vial and place on the instrument.
- For the LCS, add 50uL ISTD, 50uL Mega Mix, and 150uL MeOH to 50mL reagent water. Pour into a VOA vial and place on the instrument.
- For the MS/MSD, add 43uL ISTD, 43uL Mega Mix, and 129uL MeOH into each sample vial selected for the MS and MSD. Place on the instrument.

10.1.3 Soil Sample Preparation

Refer to SOP SA-VO-001: *Preparation, Screening, and Storage of Volatiles Samples* for the soil specific sample preparation procedures. This information is summarized below.

Three Terracore/Encore devices and one bulk container are routinely received for each soil sample. Two of the Terracores/Encores are prepared for low level analysis and one is extracted in methanol for medium-level analysis to be used if the low-level samples exceed the calibration range. The bulk container is used for determining the type of preservation for the low-level samples and for screening.

At the time of analysis, the following steps are taken:

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For low-level samples, 5mL reagent water and 5uL ISSU are added directly to the samples vial. The sample is then ready for analysis.

For medium-level samples, a dilution is prepared and analyzed. The default dilution is a DF40 (i.e., using 125uL of sample). 5uL VOA Surrogate Mix is added directly to the sample vial (if 5mL of MeOH were used) or 10uL VOA Surrogate Mix is added (if 10mL of MeOH were used). An aliquot of the sample is removed (to prepare the necessary dilution) and added to 10mL reagent water in VOA vial. 5uL ISTD is added. The sample is then ready for analysis.

10.1.4 Soil QC Preparation

For low-level samples, the method blank and LCS are prepared using blank sand directly in a VOA vial.

- For the method blank, add 5g sand, a stir bar, 10mL reagent water, and 5uL ISSU.
- For the LCS, add 5g sand, a stir bar, 10mL reagent water, 5uL ISTD, 5uL Mega Mix, and 15uL methanol.
- For the MS/MSD, add 5mL water, 5uL ISTD, 5uL Mega Mix, and 15uL methanol directly to the selected sample.

For medium-level samples, the method blank and LCS are prepared using Ottawa sand directly in a VOA vial.

- For the method blank, add 5g blank sand, 5mL methanol, and 5uL VOA Surrogate Mix. Remove a 125uL aliquot and add to 10mL reagent water spiked with 5uL ISTD.
- For the LCS, add 5g blank sand, 5mL methanol and 250uL Mega Mix. Remove a 125uL aliquot and add to 10mL reagent water spiked with 5uL ISTD.
- For the MS/MSD, add 5g blank sand, 5ml methanol and 250uL Mega Mix to the sample containers selected for the MS/MSD. Remove a 125uL aliquot and add to 10mL reagent water spiked with 5uL ISTD.

10.2 Analysis

10.2.1 Instrument Operating Conditions

The instrument conditions listed in this SOP are provided for guidance purposes. The purge time must be 11 minutes. All other parameters are optimized by the lab for the target compounds and documented in the maintenance log. Therefore, the actual conditions used by the laboratory may be slightly different from those listed here and must be documented in the instrument maintenance log, data system, and/or run log.

Instrument maintenance must be performed in accordance with Attachment 4 of this SOP.

The goal is to have maximum separation between the target compounds in the shortest run time while maintaining sufficient sensitivity to detect the target compounds at the reporting limit and MDL (if required).

GC Parameters
Column: 20m x 0.18mm ID x 1.0um
Column: Flow: 0.5-1.0mL/min

Injector: Split/Splitless operated in the split mode with 1mm ID quartz insert
Split ratio (desorb to column flow): 40:1 or 80:1

MS Parameters
Mass spectrometer interface: 240°C (direct column interface)
Mass spectrometer source temperature: 250°C
Mass scan range: 35-300amu, with a minimum scan cycle of 1 scan per second
Injector: 100°C

Temperature Program:
Initial Temperature: 50°C
Program Rate: 18°C/minute
Final Temperature: 200°C

Example Purge and Trap Conditions
The purge and trap conditions listed in this section are for guidance. The lab must document the actual conditions used. The purge time must be 11 minutes. Other parameters may be varied to optimize the detection of the target compounds.

VOCARB 3000 trap
Purge Time: 11 minutes
Purge temperature: aqueous-ambient; soils-heated 40°C
Desorb time: 0.50 minutes
Desorb temperature: 250°C
Bake time: 8 minutes at 260°C
Purge flow: Approximately 30-40mL/minute
Valve temperature: 150°C
Transfer line: 150°C

The purge flow must be balanced for adequate sensitivity of the target compounds. If the purge flow is too high, the response of the gases will be low and not reproducible. The SPCC criteria for chloromethane may not be achieved if the purge flow is too high. If the purge flow is too low, the response of the more water-soluble targets - ketones, ethers, bromoform - may be low and the reporting limit may not be achieved on a routine basis.

10.2.2 Internal Standard (ISTD)

Prior to analysis, internal standard must be added to all standards, samples, and QC items. The concentration of the internal standard must be the same in all calibration samples, field samples, and QC samples. Instructions for the addition of the internal standard spiking mix is given in the previous sections.

10.2.3 BFB Tune Check

A BFB tune check must be analyzed at the beginning of each clock as a check on the "tune" of the mass spectrometer. This check must meet the criteria described given in Section 9.2.1.

If the instrument is configured for direct injection, inject 2uL of the 25ng/uL solution into the GC and analyze the 4-BFB using the instrument conditions listed in Section 10.1. A

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solvent delay should be set to allow the methanol to pass through the mass spectrometer while the MS is "off".

If the purge and trap is used to introduce the 4-BFB into the MS, add 1uL of the 50ng/uL solution to 5.0mL of reagent water. Transfer the 5.0mL to the purge and trap device and purge the sample using the same conditions used for sample analysis.

10.2.4 Trap Check Standard

The trap check standard is used to evaluate the condition of the trap by monitoring the formation of chloromethane and bromomethane. Chloromethane and bromomethane may be formed on a degraded trap by thermal decomposition of halogenated compounds. This check must meet the criteria described given in Section 9.2.2.

10.2.5 Initial and Continuing Calibration

Calibrate the instrument using the standards and criteria described given in Section 9.2.3. Once the calibration has been established and verified with an ICV in accordance with Section 9.2.4, sample analysis may proceed.

Verify the calibration curve with a continuing calibration verification using the standards and criteria described given in Section 9.2.6.

10.2.6 Sample Analysis

The sample/extract must be injected using the same injection volume used for the calibration standards. The samples can only be analyzed after the tune, the trap check, the calibration (initial or continuing), and the method blank and LCS criteria have been met. Samples that are known to be relatively clean should be analyzed first. Samples suspected of containing high concentrations should be analyzed last. Instrument blanks may be analyzed after suspected high concentration samples to allow the detector response to stabilize.

10.2.7 Example Analytical Sequence

A typical example analytical sequence is listed below.

Description	Comments
Blank	
BFB	Clock time begins with injection of the BFB
Initial Calibration	
ICV	Second Source
BFB	Clock time begins with injection of the BFB
CCV	
LCS	
LCSD	If insufficient sample provided for MS/MSD
Trap Check	
MB	
Samples & Batch QC Items	
BFB	Clock time begins with injection of the BFB

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CCV	
LCS	
LCSD	If insufficient sample provided for MS/MSD
Trap Check	
MB	
Samples & Batch QC Items	
CCV	Capping CCV required only for SM6200B

11.0 Calculations / Data Reduction

11.1 Data Reduction

Data evaluation must be performed in accordance with SA-QA-08: *Evaluation of Chromatographic Data.* This SOP includes specific information regarding the evaluation of chromatographic data, including the requirements for performing manual integrations and the evaluation of retention times.

Data review and reporting must be performed in accordance with SA-QA-02: *Data Generation and Review.*

11.1.1 Target Analyte Identification

A target compound is identified by the visual comparison of the sample mass spectrum with the mass spectrum of the target compound from a reference spectrum of the target compound stored in a library generated on the same instrument or a standard spectral library such as the NIST/NBS.

11.1.1.1 Two criteria must be met in order to identify a target compound:

- Elution of the sample component within ±0.06 Relative Retention Time (RRT) units of the daily standard containing that compound. The RRT is calculated as follows:

$$RRT = \frac{retention\ time\ of\ the\ target\ compound}{retention\ time\ of\ the\ associated\ internal\ standard}$$

- Correspondence of the target compound spectrum and the standard component mass spectrum

11.1.1.2 All ions present in the standard component mass spectrum at a relative intensity greater than 10% (most abundant ion = 100%) should be present in the sample component mass spectrum. Other ions may be present in the sample component. Coelution of a non-target compound with a target compound will make the identification of the target compound more difficult. The ions due to the non-target compound should be subtracted from the sample component spectrum as part of the background to account for the discrepancy between the sample spectrum and the standard spectrum.

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11.1.1.3 The relative intensities of the ions present in the sample component spectrum should agree within ± 30% of the relative intensities of the ions in the standard reference spectrum. For example, an ion with an abundance of 50% in the reference spectrum should have a corresponding abundance between 20% and 80% in the sample component spectrum.

11.1.1.4 If the above criteria are not met exactly, the analyst should seek help from a senior analyst or the Department Manager. If there is sufficient evidence to support the identification of the component, then the component is identified, quantified, and reported.

1.1.2 Dilutions

Unless otherwise specified by a client QAPP, results from a single analysis are reported as long as the largest target analyte (when multiple analytes are present) is in the upper half if the calibration range. When reporting results from dilutions, appropriate data flags must be used or qualification in a case narrative provided to the client.

For clients who require we provide lower detection limits, a general guide would be to report the dilution detailed above and one additional run at a dilution factor 1/10 of the dilution with the highest target in the upper half of the calibration curve. For example, if samples analyzed at a 1/50 dilution resulted in a target in the upper half of the calibration curve, the sample would be analyzed at a dilution factor of 1/5 to provide lower reporting limits.

11.1.3 Historical Data

Many of the laboratory's clients submit samples for repeat monitoring purposes. Prior to analysis, verify TALS Worksheet Notes or use the TALS Historical Data Tracker feature to determine if historical data is available for review.

11.1.4 Chemical Relationships

Benzene, toluene, ethylbenzene, and the xylenes are generally present together in samples and indicate the presence of gasoline

m/p-Xylenes are generally higher than o-xylene

Hydrocarbons present is samples containing gasoline generally contain mass 43 and may co-elute with target analytes with mass 43 as the quant or confirmation ion or may skew the spectrum of a compound with mass 43 as part of the spectrum.

Cis-isomers are generally more prevalent than the trans- isomers

Pay particular attention to the retention time of isomer because the only way to positively identify them is by retention time. The isomers are:

1,1-dichloroethane and 1,2-dichloroethane
1,1-dichloroethene, cis-1,2-dichloroethene, and trans-1,2-dichloroethene
1,1,1-trichloroethane and 1,1,2-trichloroethane
ethyl benzene, m/p-ylene, and o-xylene

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1,3-dichlorobenzene, 1,4-dichlorobenzene, and 1,2-dichlorobenzene
1,1-dichloropropene, cis-1,2-dichloropropene, and trans-1,2-dichloropropene
2-chlorotoluene and 4-chlorotoluene
1,2,3-trichlorobenzene and 1,2,4-trichlorobenzene
1.3.5-trimethylbenzene and 1,2,4-trimethylbenzene
4-methyl-2-pentanone (MIBK) and 2-hexanone
n-butylbenzene, sec-butylbenzene, tert-butylbenzene, and isopropylbenzene

Higher chlorinated alkanes and alkenes may have lower chlorinated alkanes or alkenes present due to degradation. The following table lists some common chlorinated compounds and their degradation products. Look for the degradation product(s) when the concentration of the compound in the left column is present at high concentrations.

Analyte	Degration Product
1,1,2,2-tetrachloroethane	trichloroethene (TCE) cis-1,2-dichloroethene (c-1,2-DCE) trans-1,2-dichloroethene (t-1,2-DCE) vinyl chloride 1,1,2-trichloroethane (1,1,2-TCA) 1,2-dichloroethane (1,2-DCA) Chloroethane
1,1,2-trichloroethane (1,1,2-TCA)	1,2-dichloroethane (1,2-DCA) Chloroethane
1,1,1-trichloroethane (1,1,1-TCA)	1,1-dichloroethene (1,1-DCE) 1,1-dichloroethane (1,1-DCA) Chloroethane
Carbon tetrachloride	Chloroform Methylene chloride Chloromethane
Tetrachloroethene (PCE) (PCE = perchloroethylene which is a common name for tetrachloroethene)	trichloroethene (TCE) cis-1,2-dichloroethene (c-1,2-DCE) trans-1,2-dichloroethene (t-1,2-DCE) Chloroethene
1,2,4-trichlorobenzene	1,4-dichlorobenzene (1,4-DCB) 1,2-dichlorobenzene (1,2-DCB) Chlorobenzene

11.2 Calculations

11.2.1 The calculations associated with batch QC determinations are given in SOP SA-QA-17. Applicable calculations include accuracy (% recovery) and precision (%RPD).

11.2.2 The calculations associated with initial and continuing calibrations and are given in SOP SA-QA-16. Applicable calculations include determination for: calibration factor, standard deviation, relative standard deviation, relative response factor, and relative standard deviation.

11.2.3 The calculation to determine final concentration is given as follows:

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$$FinalConcentration = CONC_{Sample} \otimes \frac{F}{I \times dw} \otimes D$$

Where:

$CONC_{Sample}$= Concentration of the sample extract (at the instrument)
F = Final volume of the extract
I = Initial volume/weight
dw = % Solids decimal equivalent
D = Dilution factor

Note: All dry weight corrections are performed automatically in TALS.

Note: This formula assumes all unit conversion factors are applied.

12.0 Method Performance

12.1 Reporting Limit Verification (RLV)

At a minimum, RLVs must be performed initially upon method set-up in accordance with SOP SA-QA-07: *Determination and Verification of Detection and Reporting Limits*.

For analytes and methods certified by DOD ELAP, RLVs must also be performed quarterly thereafter. For all other analytes and methods, RLVs must also be performed annually thereafter. Exceptions may be made for project-specific non-routine analytes.

12.2 Method Detection Limit (MDL) Study

The 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. 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 current MDLs associated with this procedure are given in the Method Limit Group (MLG) in TALS.

At a minimum, MDL Studies must be performed initially upon method set-up in accordance with SOP SA-QA-07: *Determination and Verification of Detection and Reporting Limits*.

Note: MDL Studies are not required for non-routine analytes provided results are not reported below the RL (i.e., MDL equals RL in TALS).

Note: For SM6200B, MDL Studies should be performed over 3-5 days.

12.3 Method Detection Limit Verification (MDLV)

At a minimum, MDLVs must be performed initially upon method set-up in accordance with SOP SA-QA-07: *Determination and Verification of Detection and Reporting Limits*.

For analytes and methods certified by DOD ELAP, MDLVs must also be performed quarterly thereafter. For all other analytes and methods, MDLVs must also be performed annually thereafter.

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Note: MDLVs are not required for non-routine analytes provided results are not reported below the RL (i.e., MDL equals RL in TALS).

12.4 QC Limit Generation, Control Charting, and Trend Analysis

12.4.1 EPA 624 and SM6200B

The control limits for the batch QC items (LCS/LCSD and MS/MSD) for this procedure are specified in the reference method and cannot be broadened; therefore, the laboratory defaults to the method-defined limits and does not utilize in-house or laboratory-derived limits for the evaluation of batch QC items.

Although the laboratory must default to the method-defined QC limits, control charting is a useful tool and is performed to assess analyte recoveries over time to evaluate trends. Control charting must be performed periodically (at a minimum annually) in accordance with SOP SA-QA-17: *Evaluation of Batch QC Data.*

12.4.2 EPA 8260B

The control limits for the batch QC items (LCS/LCSD and MS/MSD) for this procedure are not specified by the reference method; therefore, the laboratory defaults to in-house and/or laboratory-derived limits for the evaluation of batch QC items.

Control charting is a useful tool and is performed to assess analyte recoveries over time to evaluate trends. Control charting must be performed periodically (at a minimum annually) in accordance with SOP SA-QA-17: *Evaluation of Batch QC Data.*

12.5 Demonstrations of Capability

Initial and continuing demonstration of capability must be performed in accordance with SOP SA-QA-06*: Training Procedures.*

Prior to performing this procedure unsupervised, each new analyst who performs this analysis must demonstrate proficiency per method/analyte combination by successful completion of an initial demonstration of capability (IDOC). The IDOC is performed by the analysis of 4 consecutive LCSs that meet the method criteria for accuracy and precision. The LCSs must be from a second source than that used to prepare the calibration standards. The IDOC must be documented on the IDOC Form shown in SOP SA-QA-06 with documentation routed to the QA Department for filing.

Note: SM6200B requires IDOCs to be within 80-120% for the average recovery and <20%RSD.

Annual continuing demonstrations of capability (CDOCs) are also required per analyst per method/analyte combination. The CDOC requirement may be met by the consecutive analysis of four LCS all in the same batch, by the analysis of four LCS analyzed in four consecutive batches (in different batches on different days), via acceptable results on a PT study, or by the analysis of client samples with statistically indistinguishable results when compared to another certified analyst. The CDOC must be documented and routed to the QA Department for filing.

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12.6 Training Requirements

All training must be performed and documented in accordance with SOP SA-QA-06: *Training Procedures*.

Note: The SOPs listed in the Reference/Cross-Reference Section are applicable to this procedure. All employees performing this procedure must also be trained on these SOPs, and/or have a general understanding of these procedures, as applicable.

13.0 Pollution Control

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (e.g., examining recycling options, ordering chemicals based on quantity needed, preparing reagents based on anticipated usage and reagent stability, etc.). Employees must abide by the policies in Section 13 of the Environmental Health and Safety Manual.

This procedure has been evaluated for opportunities to minimize the waste generated. Where reasonably feasible, pollution control procedures have been incorporated.

14.0 Waste Management

Waste management practices must be conducted consistent with all applicable federal, state, and local rules and regulations. All waste (i.e., excess reagents, samples, and method process wastes) must be disposed of in accordance with Section 9 of the TestAmerica Savannah Addendum to the EHSM. Waste description rules and land disposal restrictions must be followed.

14.1 Waste Streams Produced by the Method

The following waste streams are produced when this method is carried out:

- Excess samples, reagents, and standards must be disposed in accordance with the TestAmerica Savannah Addendum to the EHSM.
- Methanolic waste from rinsings and standards – Transfer to satellite container for methanolic (flammable) waste. Transfer to hazardous waste section when satellite container is full.
- Excess aqueous samples – Dispose according to characterization on the sample disposal sheets. Neutralize non-hazardous samples before disposal into drain/sewer. Transfer hazardous samples (identified on disposal sheets) to the waste department for disposal.
- Excess soil and solid samples – Dispose according to characterization on sample disposal sheets. Transfer non-hazardous samples to TCLP container for characterization in hazardous waste department. Transfer hazardous samples (identified on disposal sheets) to waste department for disposal.

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15.0 References / Cross-References

- SOP SA-AN-041: *Reagent and Standard Materials Traceability*
- SOP SA-AN-100: *Laboratory Support Equipment (Verification and Use)*
- SOP SA-QA-02: *Data Generation and Review*
- SOP SA-QA-05: *Preventive and Corrective Action Procedures*
- SOP SA-QA-06: *Training Procedures*
- SOP SA-QA-07: *Determination and Verification of Detection and Reporting Limits*
- SOP SA-QA-08: *Evaluation of Chromatographic Data*
- SOP SA-QA-15: *Homogenization, Compositing, and Segregation of Samples*
- SOP SA-QA-16: *Evaluation of Calibration Curves*
- SOP SA-QA-17: *Evaluation of Batch QC Data*
- SOP SA-VO-001: *Preparation, Screening, and Storage of Volatiles Samples*
- TestAmerica Savannah Quality Assurance Manual
- TestAmerica Environmental Health and Safety Manual
- TestAmerica Savannah Addendum to the Environmental Health and Safety Manual
- Method 8000C: *Test Methods for Evaluating Solid Wastes, Third Edition, SW-846;* U.S. EPA Office of Solid Waste and Emergency Response: Washington, DC.
- Method 8260B: *Test Methods for Evaluating Solid Wastes, Third Edition, SW-846;* U.S. EPA Office of Solid Waste and Emergency Response: Washington, DC.
- EPA Method 624: *Purgeables.* 40 CFR Part 136, Appendix A, July 1, 1995.
- *Standard Methods for the Examination of Water and Wastewater, Online Edition;* American Public Health Association: Washington, DC.
  - SM6020: Quality Assurance/Quality Control
  - SM6200B: *Volatile Organic Compounds; Purge and Trap Capillary-Column Gas Chromatographic/ Mass Spectrometric Method*; 1997

16.0 Method Modifications and Clarifications

16.1 Incorporation of Other Matrices

This procedure may be modified to analyze other matrices (e.g., wipe, waste, and TCLP/SPLP leachate samples) based on the needs of the client. This will need to be arranged by the Project Manager at the initiation of the project.

Wipe and waste matrices are non-routine, and the laboratory is not currently NELAC certified for these matrices. The laboratory uses its routine soil RLs (converted for initial and final volumes, etc.) and default QC limits to evaluate waste samples and its routine water RLs (converted for initial and final volumes, etc.) and default QC limits to evaluate wipe samples. Soil and/or water DOCs can be used to satisfy analyst demonstrations of capability for these types of non-routine matrices, as applicable. The laboratory uses its routine aqueous RLs (converted for initial and final volumes, etc.) and default QC limits to evaluate TCLP/SPLP leachate samples. Water DOCs can be used to satisfy analyst demonstrations of capability for TCLP/SPLP matrices. Ottawa sand is used as the blank matrix for wipes and wastes unless specifically requested otherwise by the project.

16.1.1 Collection and Handling Procedures

Waste (oil) samples are routinely collected in 40mL VOA vials. Waste (oil) samples must be iced at the time of collection and maintained at 4°C (less than 6°C but not frozen) until

time of preparation. Samples must be analyzed within 14 days of collection.

Wipe samples are routinely collected in 40mL VOA vials. Wipe samples must be iced at the time of collection and maintained at 4°C (less than 6°C but not frozen) until time of preparation. Samples must be analyzed within 14 days of collection.

Once the TCLP/SPLP extraction procedure has been performed, the leachate is transferred to a Tedlar bag. TCLP/SPLP leachates must be stored at 4°C (less than 6°C with no frozen samples) until the time of preparation. The leachate sample must be analyzed within 14 days of completion of the TCLP/SPLP extraction.

16.1.2 Preparation and Analytical Procedures

Wipe samples are analyzed in the same fashion as water samples. Refer to Work Instruction FQA088: *Wipe Tests: Sampling and Analysis* for additional information on wipe procedures.

Waste samples are prepared and analyzed in the same fashion as medium-level soil samples. Note: Waste samples often require large dilutions.

TCLP/SPLP matrices are prepared and analyzed in the same manner as routine water samples; however, a default dilution factor of 20 is used.

16.2 Other Considerations

16.2.1 EPA Method 8260B does not contain calibration verification criteria for non-CCC analytes nor does it require non-CCC analytes to be evaluated for response; however, the laboratory has adopted in-house criteria for non-CCC analytes as outlined in this SOP.

16.2.2 EPA Method 8260B does not require the analysis of an ICV. NELAC requires an ICV; however, it does not list specific criteria. The laboratory has adopted in-house criteria for ICVs for EPA 8260B as outlined in this SOP. The SM6200B reference method requires an externally-prepared QCS to be performed quarterly. The laboratory satisfies this requirement via the ICV performed with each ICAL.

16.2.3 EPA Method 8260B allows alternate criteria to be used for the BFB evaluation. As such the laboratory has incorporated criteria the CLP OLMO4.0 (January 1998) method.

16.2.4 The laboratory has defined the analytes listed in Attachment 10 as poor or erratic performers and allows for exceptions to the ICV, CCV, LCS/LCSD, MS/MSD, and Sporadic Marginal Exceedance criteria for these analytes as outlined in this SOP.

16.2.5 EPA Method 8260B does not place a cap on an individual analyte's %D or %RSD when evaluating the Grand Mean Exception. The laboratory has adopted more stringent in-house requirements as outlined in this SOP.

16.2.6 It has been determined that increased methanol concentrations can suppress the response of the gas compounds, leading to erratic recovery and reduced linearity. As such, the laboratory normalizes the volume of methanol in all standards and QC items to minimize this effect. By introducing a constant volume of methanol to initial calibration

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standards, CCV/ICV, LCS, and MS/MSD, better sensitivity and recovery of these analytes is achieved.

16.2.7 The 7-day holding time for samples with pH>2 is listed in EPA 624 and is not included in the SW-846 methods. The laboratory has adopted this as internal guidance for EPA Method 8260B as is common industry practice and will make every effort to analyze samples with pH>2 within 7 days.

16.2.8 Historical standard practice for most laboratories was to combine all analytes into one analysis using a single acid preserved container. This practice is still acceptable in those cases where the compounds of interest are not adversely affected by the addition of the hydrochloric acid (HCl) preservative; however, EPA 624 and EPA 8260B both list special preservation requirements for acrolein, acrylonitrile, and 2-chloroethylvinyl ether (2-CEVE). Although these analytes are rarely found in the environment, preservation at pH<2 (as achieved using HCl preservative) breaks down these analytes and may result in a significantly low bias and/or non-detect value.

The reference methods suggest to preserve these analytes to a pH between 4-5. Achieving this narrow pH range is problematic; therefore, the only other alternative is to use an unpreserved container for these analytes. As such, if acrolein, acrylonitrile, and/or 2-CEVE are on the requested target analyte list, the laboratory now defaults to using an unpreserved container. The other target analytes reported for these methods are not adversely affected by using an unpreserved container provided they are analyzed within the shorter, method-prescribed holding times (HT) defined for unpreserved samples. Specific information on VOA holding times, preservation requirements, and analyte-specific requirements is given in Attachment 2.

16.2.9 The 40mL VOA vials used by the laboratory have been demonstrated to actually contain approximately 43mL. As such, standard spiking amounts have been adjusted accordingly to accommodate for this volume (e.g., spiking 43uL instead of 40uL).

16.2.10 The laboratory has incorporated the batch QC items as outlined in Section 9.1. Some additional QC items are performed which differ from those specified in the reference methods (e.g., LCSD or MSD) to satisfy common state regulatory requirements and/or client requests for precision data and/or to facilitate scheduling and data evaluation. The method-specified batch QC items are as follows:

EPA 8260B:
Method blank, LCS, MS, and sample duplicate or MSD

EPA 624
Method blank, LCS, MS per 10% of samples analyzed
SM6200B:
Method blank, LCS, MS, and MSD

16.2.11 EPA Method 624 specifies a required concentration range criteria for the evaluation of QC items (i.e., the Q-Table). The laboratory has converted these values into percent recovery limits to be used to assess CCV, ICV, LCS/LCSD, and MS/MSD.

16.2.12 The EPA 8260B reference method specifies a 1-week expiration date for the gas standards. The laboratory prepares a 25mL volume of MegaMix standard and splits it

into five 5mL Mini-nert vials. Each vial is assigned a 1-week expiration date from the date that vial is opened, not to exceed 14 days from the preparation date of the 25mL volume. Note: This expiration date is a maximum. Shorter expiration dates may be used based on evidence of gas compound volatility as evaluated via CCVs.

16.2.13 The EPA 8260B reference method recommends 4 surrogates to be used but allows for other surrogates. Due to improved instrument conditions and shorter run times, the laboratory has incorporated the 3 surrogates specified in EPA Method 624.

16.2.14 Due to client requests, the analyte list utilized by the laboratory may be different than those listed in the reference methods.

16.2.15 The SM6200B reference method states that the MDL should be determined annually but does not specify the procedure to be used. The laboratory performs MDL Studies initially, in accordance with 40CFR Part 136B, and re-establishes the MDL annually or quarterly (as dictated by agency-specific requirements) via an MDLV study.

16.2.16 The SM6200B reference method states that the Method Quantitation Limit (MQL) must be 4 times the MDL. The laboratory typically defines the MQL (i.e., the RL) based on the low-point of the calibration curve as opposed to a factor of the MDL.

16.2.17 The SM6200B reference method states that internal standard areas must be within 30% of the ICAL mean. Due to a software limitation, the laboratory evaluates each CCV against the midpoint of the ICAL and all field samples and QC items to their associated CCV. This is consistent with the requirements outlined in SW846 Update IV.

17.0 Attachments

The following Tables, Diagrams, and/or Validation Data are included as Attachments:

Attachment 1: SOP Summary
Attachment 2: Sample Collection, Preservation, and Holding Time Table
Attachment 3: QC Summary
Attachment 4: Preventative Maintenance and Troubleshooting
Attachment 5: EPA 8260B Calibration Criteria: SPCCs and CCCs
Attachment 6: BFB Tune Criteria
Attachment 7: Target Compound Information: Ions and ISTDs
Attachment 8: Instrument Configurations
Attachment 9: Standard Information and Recipes
Attachment 10: Poor Performers
Attachment 11: Evaluation of TICs
Attachment 12: EPA 624 CCV & ICV Criteria (Q-Table)

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Attachment 1:
SOP Summary

Sample Preparation Summary
Aqueous samples are routinely purged at ambient conditions; however, a heated purge may be used if required by the project. A 5mL purge volume is used as the default. The calibration standards and the associated QC must be analyzed under the same conditions and volume.

Low-level soil samples are purged at 40°C in a purge and trap instrument designed to add water and internal standards to the vial containing the sample without breaking the seal. The sample is stirred during purging to thoroughly mix the soil and water. The calibration standards and associated QC are purged under the same conditions.

High level soils and waste samples are extracted with methanol (1mL of methanol per gram of sample). An aliquot of the methanol extract is injected into reagent water. The methanol extract/reagent water is purged at ambient temperature using the same instrument conditions and calibration used for aqueous samples.

Sample Analysis Summary
VOCs are purged from the sample matrix with helium. The VOCs are transferred from the sample matrix to the vapor phase. The vapor is swept through a sorbent tube where the VOCs are trapped. After the purging is completed, the trap is heated and backflushed with helium to desorb the VOCs onto a GC column. The GC is temperature-programmed to separate the VOCs, which are then detected by a mass spectrometer. Qualitative identification of the target compounds in the sample is based on the relative retention time and the mass spectra of the characteristic masses (ions) determined from standards analyzed on the same GC/MS under the same conditions. Quantitative analysis is performed using the internal standard technique with a single characteristic ion.

Description	Comments
Blank	
BFB	Clock time begins with injection of the BFB
Initial Calibration	
ICV	Second Source
BFB	Clock time begins with injection of the BFB
CCV	
LCS	
LCSD	If insufficient sample is provided for MS/MSD
Trap Check	
MB	
Samples & Batch QC Items	
BFB	Clock time begins with injection of the BFB
CCV	
LCS	
LCSD	If insufficient sample is provided for MS/MSD
Trap Check	
MB	
Samples & Batch QC Items	
CCV	Capping CCV required for SM6200B only

Attachment 2:
Sample Collection, Preservation, and Holding Time Table

Aqueous samples are routinely collected in 40mL VOA vials. The table below outlines the laboratory's default procedures for aqueous samples:

Method	Acrolein, Acrylonitrile, or 2-CEVE Requested?	Chemical Preservation	Thermal Preservation	Holding Time
EPA 8260B	Yes	None	<6°C	7 days
EPA 8260B	No	HCl (pH<2)	<6°C	14 days
EPA 624	Yes	None	<6°C	3 days*
EPA 624	No	HCl (pH<2)	<6°C	14 days
SM6200B	Yes	None	<6°C	7 days
SM6200B	No	HCl (pH<2)	<6°C	14 days

*Note: 3-day HT is specific to acrolein. If acrolein is not requested, 7 days is used for the remaining target compounds.

The table below outlines the laboratory's procedures for soil samples:

Sample Container	Chemical Preservation	Thermal Preservation	Holding Time
Terracore Kit: 5g Terracore sampler, 2 x pre-weighed 40-mL VOA w/ H2O, pre-weighed 40-mL VOA w/ MeOH, 2oz. bulk jar	H_2O & MeOH	<-10°C	48 hours to freeze, 14 days to analyze
Encore Kit: 3 x 5g Encore samplers, 4oz. bulk jar	$NaHSO_4$, H_2O, or MeOH (added in lab)	<6°C	48 hours to preserve, 14 days to analyze
Encore Kit: 3 x 5g Encore samplers, 2 x pre-weighed 40-mL VOA w/ NaHSO4, pre-weighed 40-mL VOA w/ MeOH, 4oz. bulk jar	$NaHSO_4$ or MeOH	<6°C	48 hours to preserve, 14 days to analyze

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Attachment 3:
QC Summary

QC Item	Frequency	Criteria	Corrective Action
Clock Time	EPA 8260B: 12 hours EPA 624: 24 hours EPA 624 Cluster Rule (Chloroform Only): 8 hours SM6200B: 12 hours or 20 samples, whichever occurs first	Clock time starts with the injection of the BFB. Analysis of samples and QC items must conclude within expiration of clock time. Subsequent analysis requires new BFB.	Not applicable
Tune Standard (BFB)	At beginning of each clock	Refer to Attachment 6.	- Perform instrument maintenance - Re-tune.
Trap Check Standard	At beginning of each clock	<0.5ug/L (Chloromethane & bromomethane)	- Perform instrument maintenance. - Change trap. - Recalibrate

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QC Item	Frequency	Criteria	Corrective Action
Initial Calibration (ICAL) - Minimum of 3 points for EPA 624. - Minimum of 5 points for EPA 8260B and SM6200B.	Upon instrument set-up, and after unsuccessful CCV	EPA 624: %RSD < 35% If %RSD > 35%, use curve fit w/ r^2>0.990. EPA 8260B: - CCC: %RSD < 30% - SPCC: RRF_{avg} > Attachment 5 - If %RSD >15%, use curve fit with r^2 > 0.990. - GME: Avg %RSD <15%; No single analyte %RSD > 45%. SM6200B: %RSD < 20% If %RSD > 20%, use curve fit w/ r^2>0.994. Requant all levels, w/ 20%D.	-Reanalyze standard(s) -Prepare new standard(s) and reanalyze -Perform injector port maintenance and reanalyze standards -Retune and reanalyze standards -Replace column and reanalyze standards -Clean source and reanalyze standards
Initial Calibration Verification (ICV) - Second Source	After each ICAL	EPA 624: Attachment 12 EPA 8260B and SM6200B: Avg %RSD <20%; No single analyte %RSD > 60%.	-Reanalyze standard -Prepare new standard and reanalyze -Recalibrate

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QC Item	Frequency	Criteria	Corrective Action
Continuing Calibration Verification (CCV)	EPA 8260B and EPA 624: After BFB SM6200B: After BFB and at end of each clock	EPA 624: Attachment 12 EPA 8260B: - CCC: %D < 20% - SPCC: RRF > Attachment 5 - Non-CCC and non-SPCC: Avg %D <20%; No single analyte %D >60% SM6200B: %D < 20%	-Reanalyze standard -Prepare new standard and reanalyze -Recalibrate
Internal Standards (ISTD)	Spiked in all CCVIS, samples, and batch QC items	EPA 8260B & EPA 624: CCVIS: - Area -50% to +100% CCV in ICAL. - RT +/-30 seconds from ICAL. Samples & batch QC items: - Area within -50% to +100% of previous CCVIS. SM6200B: CCVIS: - Area +/-30% CCV in ICAL. Samples & batch QC items: - Area within +/-30% of previous CCVIS.	-Evaluate chromatogram, spectra, and integrations -Reanalyze extract -Perform instrument maintenance and reanalyze extract -Re-extract and reanalyze if sufficient sample available
Surrogate Compounds	Spiked in all samples and batch QC items.	Within MLG limits	-Evaluate chromatogram, spectra, and integrations -Reanalyze sample, if sufficient sample available

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QC Item	Frequency	Criteria	Corrective Action
Analytical Batch Definition	Analyzed together w/in 24-hr timeframe; not to exceed 20 field samples	Not Applicable	Not Applicable
Method Blank (MB)	One per batch	<1/2RL	Refer to SOP SA-QA-17
Laboratory Control Sample (LCS)	One per batch	Within TALS MLG Limits	Refer to SOP SA-QA-17
Laboratory Control Sample Duplicate (LCSD)	One per batch, when insufficient sample is provided for MS/MSD	Within TALS MLG Limits	Refer to SOP SA-QA-17
Matrix Spike (MS)	EPA 8260B and SM6200B: One per batch EPA 624: 1 per 10% of samples	Within TALS MLG Limits	Refer to SOP SA-QA-17
Matrix Spike Duplicate (MSD)	One per batch	Within TALS MLG Limits	Refer to SOP SA-QA-17
Initial Demonstration of Capability (IDOC)	Initially, per analyst, per analyte/method/matrix combination	Refer to SOP SA-QA-06	Refer to SOP SA-QA-06 Note: Unsupervised work must not begin until acceptable IDOC is obtained.
Continuing Demonstration of Capability (CDOC)	Annually, per analyst, per analyte/method/matrix combination	Refer to SOP SA-QA-06	Refer to SOP SA-QA-06

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QC Item	Frequency	Criteria	Corrective Action
Reporting Limit Verification (RLV)	Upon method/instrument set-up, per analyte/method/matrix combination. Then quarterly thereafter (for DOD ELAP) or annually thereafter (for non-DOD ELAP)	Refer to SOP SA-QA-07	Refer to SOP SA-QA-07
Method Detection Limit Study (MDL)	Upon method/instrument set-up, per analyte/method/matrix combination	Refer to SOP SA-QA-07	Refer to SOP SA-QA-07
MDL Verification (MDLV)	Upon method/instrument set-up, per analyte/method/matrix combination. Then quarterly thereafter (for DOD ELAP) or annually thereafter (for non-DOD ELAP)	Refer to SOP SA-QA-07	Refer to SOP SA-QA-07

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Attachment 4:
Preventative Maintenance and Troubleshooting

Instrument Labeling
Each instrument must be labeled with its name or ID (e.g., MSA, ICP-D, etc.). Additionally, non-operational instruments must be isolated from service or marked as being out of service. Each piece of equipment has an "Operational / Not Operational" sticker that is used for this purpose.

Maintenance Log
A maintenance log must be established for each piece of equipment used in the laboratory.

All maintenance that is performed on the instrument must be recorded in the log including:
- analyst or technician performing the maintenance
- date the maintenance was performed
- detailed explanation of the reason for the maintenance
- resolution of the problem and return to control
- all service calls from instrument representatives

Preventive Maintenance

LABORATORY EQUIPMENT PREVENTIVE MAINTENANCE SCHEDULE								
EQUIPMENT ITEM	Service Interval							SERVICE LEVEL
	D	W	M	Q	SA	A	AN	
Injector Port							X	Replace septum, sleeve, inlet seal, and washer (Recommend every 2 weeks)
Sparge Tubes							X	Clean (Recommend every 3 months)
Column							X	Change column (Recommend annually)

D=daily; W=Weekly; M=monthly; Q=Quarterly; SA=semi-annually; A=annually; AN=as needed

Troubleshooting
Troubleshooting should be documented as outlined above. If possible, troubleshooting is best performed in a step-wise manner to systematically isolate instrument components. Refer to the instrument manufacturer's guides for specific information and strategies. Enlist assistance from technical and/or department management as needed.

Contingency Plan
Maintenance contracts are carried for most instrumentation and close contact is maintained with service personnel to ensure optimal instrument functioning. An extensive spare parts inventory is maintained for routine repairs. Since instrumentation is standardized throughout the laboratory network, spare parts and components can be readily exchanged among the network.

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In general, the laboratory has at least one backup unit for each critical unit. In the event of instrument failure, portions of the sample load may be diverted to duplicate instrumentation, the analytical technique switched to an alternate approved technique (such as manual colorimetric determination as opposed to automated colorimetric determination), or samples shipped to another properly certified or approved TestAmerica location.

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Attachment 5:
EPA 8260B SPCC and CCC Criteria

Calibration Check Compounds (CCCs)

Initial Calibration	Continuing Calibration
<=30% RSD	<=20% difference from initial calibration

System Performance Check Compounds (SPCCs)

SPCC	Minimum RRF
Chloromethane	0.10
1,1-Dichloroethane	0.10
Chlorobenzene	0.30
Bromoform	>0.10
1,1,2,2-Tetrachloroethane	0.30

Note: The CCC and SPCC criteria must be met even if the calibration curve option is used for quantitation. If the CCC and SPCC criteria do not pass, a new calibration curve must be prepared and analyzed.

Calibration Check Compounds (CCCs)
Vinyl chloride
1,1-Dichloroethene
Chloroform
1,2-Dichloropropane
Toluene
Ethylbenzene

System Performance Check Compounds (SPCCs)
Chloromethane
1,1-Dichloroethane
Chlorobenzene
Bromoform
1,1,2,2-Tetrachloroethane

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Attachment 6:
BFB Tune Criteria

EPA 8260B

m/e	Abundance Criteria
50	8.0-40.0% of mass 95
75	30.0-66.0% of mass 95
95	Base peak, 100% relative abundance
96	5.0-9.0% of mass 95
173	< 2.0% of mass 174
174	50-120%% of mass 95
175	4.0-9.0% of mass 174
176	93.0-101.0% of mass 174
177	5.0-9.0% of mass 176

*8260 criteria taken from CLP OLMO4.0 (January 1998)

EPA 624 and SM6200B

m/e	Abundance Criteria
50	15.0-40.0% of mass 95
75	30.0-60.0% of mass 95
95	Base peak, 100% relative abundance
96	5.0-9.0% of mass 95
173	<2.0% of mass 174
174	>50% of mass 95
175	5.0-9.0% of mass 174
176	95.0-101.0% of mass 174
177	5.0-9.0% of mass 176

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Attachment 7:
Target Compound Information: Ions and ISTDs

Analyte	Quant Ion	Secondary Ions		ISTD
1,1,1,2-Tetrachloroethane	131	133	119	3
1,1,1-Trichloroethane	97	99	61	2
1,1,2,2-Tetrachloroethane	83	85	168	3
1,1,2-Trichloro-1,2,2-trifluoroethane	101	151	103	1
1,1,2-Trichloroethane	83	97	99	2
1,1-Dichloroethane	63	65	83	1
1,1-Dichloroethene	96	61	98	1
1,1-Dichloropropene	75	110	77	2
1,2,3-Trichlorobenzene	180	182		3
1,2,3-Trichloropropane	110	112		3
1,2,4-Trichlorobenzene	180	182	145	3
1,2,4-Trimethylbenzene	105	120	77	3
1,2-Dibromo-3-Chloropropane	157	75	155	3
1,2-Dichlorobenzene	146	148	111	3
1,2-Dichloroethane	62	49	64	2
1,2-Dichloroethene, Total	Sum of cis and trans isomers			
1,2-Dichloropropane	63	76	65	2
1,3,5-Trimethylbenzene	105	120	77	3
1,3-Dichlorobenzene	146	148	111	3
1,3-Dichloropropane	76	78	41	2
1,3-Dichloropropene, Total	Sum of cis and trans isomers			
1,4-Dichlorobenzene	146	148	111	3
1-Chlorohexane	41	43		3
1-Methylnaphthalene	142	141		3
2,2-Dichloropropane	77	41		1
2-Butanone (MEK)	43	72		1
2-Chloro-1,3-butadiene	53	88		1
2-Chloroethyl vinyl ether	63	65	106	2
2-Chlorotoluene	126	91	63	3
2-Hexanone	43	58		3
2-Methylnaphthalene	142	141		3
3-Chloro-1-propene	76	41		1
3-Methylhexane	43	71		2
4-Chlorotoluene	126	91	63	3
4-Isopropyltoluene	119	134	91	3
4-Methyl-2-pentanone (MIBK)	43	57	58	2
Acetone	58	43		1
Acetonitrile	41	40		1
Acrolein	56	55		1
Acrylonitrile	53	52	51	1
Amyl acetate	70	61		3

Analyte	Quant Ion	Secondary Ions		ISTD
Benzene	78	50		2
Benzyl chloride	91	63		3
Bromobenzene	156	77	158	3
Bromoform	173	171	175	3
Bromomethane	96	94	79	1
Butadiene	54	39		1
Carbon disulfide	76	78		1
Carbon tetrachloride	117	119	121	2
Chlorobenzene	112	77	51	3
Chlorobromomethane	49	128	130	1
Chlorodibromomethane	129	127	131	3
Chloroethane	64	66		1
Chloroform	83	85	47	1
Chloromethane	50	52		1
Chloroprene	53	88		1
cis-1,2-Dichloroethene	96	61	98	1
cis-1,3-Dichloropropene	75	77	110	2
Cyclohexane	56	69	84	2
Cyclohexanone	55	42	98	3
Dibromomethane	93	174	95	2
Dichlorobromomethane	83	85	129	2
Dichlorodifluoromethane	85	87	101	1
Dichlorofluoromethane	67	69		1
Ethyl acetate	43	61	70	1
Ethyl acrylate	55	73		1
Ethyl ether (diethyl ether)	59	74	45	1
Ethyl methacrylate	69	41	39	3
Ethylbenzene	91	106	51	3
Ethylene Dibromide	107	109		2
Ethylene oxide	43	44		1
Furan	68	39		1
Hexachlorobutadiene	225	223	190	3
Hexane	57	41	43	1
Iodomethane	142	127		1
Isobutyl alcohol	43	41		2
Isopropyl acetate	61	59	87	1
Isopropyl ether	45	43		1
Isopropylbenzene	105	120	77	3
m,p-Xylene	106	91	77	3
Methacrylonitrile	67	52		1
Methyl acetate	43	74		1
Methyl acrylate	55	85		1
Methyl methacrylate	69	41		2

Analyte	Quant Ion	Secondary Ions		ISTD
Methyl styrene	117	115	91	3
Methyl tert-butyl ether	73	57		1
Methylcyclohexane	83	55	98	2
Methylene Chloride	84	49	86	1
Naphthalene	128	102	51	3
n-Butyl acetate	43	56		2
n-Butyl acrylate	55	56		2
n-Butylbenzene	91	92	134	3
n-Heptane	43	57	71	1
N-Propylbenzene	120	91	65	3
o-Xylene	106	91	77	3
Pentachloroethane	167	130		3
Propene oxide	58	43		1
Propionitrile	54	55		1
sec-Butylbenzene	105	134	91	3
Styrene	104	78	103	3
Tert-amyl methyl ether	73	43		1
Tert-butyl alcohol	59	41		1
tert-Butylbenzene	119	91	134	3
Tert-butyl ethyl ether	59	87		1
Tetrachloroethene	164	166	168	3
Tetrahydrofuran	42	71		1
Toluene	92	91	65	2
trans-1,2-Dichloroethene	96	61	98	1
trans-1,3-Dichloropropene	75	77	110	2
trans-1,4-Dichloro-2-butene	53	88	89	3
Trichloroethene	130	95	132	2
Trichlorofluoromethane	101	103	105	1
Trichlorotrifluoroethane (Freon 113)	101	151	103	1
Trihalomethanes, Total	Sum of chloroform, dichlorobromomethane, dibromochloromethane, and bromoform			
Vinyl acetate	43	86		1
Vinyl chloride	62	64		1
Xylenes, Total	Sum of m/p- and o- isomers			

Analyte	Quant Ion	Secondary Ions		ISTD
Surrogates				
Dibromofluoromethane	113	81	111	1
Toluene-d8	98	100	70	2
p-Bromofluorobenzene	95	174	176	3
1,2-Dichloroethane-d4	65	67	102	1
Internal Standards				
1,2-Dichloroethane-d4	65	67	102	1
1,4-Difluorobenzene	114	63	88	2
Chlorobenzene-d5	82	117	119	3

Note: For a complete list of target analytes for each method, refer to the LIMS Method Limit Groups (MLGs).

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Attachment 8:
Basic Instrument Configurations

System	GC	MS	P/T	Autosampler	Trap	GC/MS Data system	Methods
MSA	Agilent 6890	Agilent 5973	EST Encon (dual)	EST Centurion	Supelco VOCARB 3000	Agilent Chemstation	EPA 624 EPA 8260B (aqueous) SM6200B
MSB	Agilent 6890	Agilent 5973	Tekmar 3000	Tekmar Aquatek 70	Supelco VOCARB 3000	Agilent Chemstation	EPA 624 EPA 8260B (aqueous) SM6200B
MSL	Agilent 5890	Agilent 5972	Tekmar LSC 2000	Varian Archon	Supelco VOCARB 3000	Agilent Chemstation	EPA 8260B (soils)
MSM	Agilent 5890	Agilent 5972	Tekmar LSC 2000	Varian Archon	Supelco VOCARB 3000	Agilent Chemstation	EPA 8260B (soils)
MSO	Agilent 6890	Agilent 5973	EST Encon (dual)	EST Centurion	Supelco VOCARB 3000	Agilent Chemstation	EPA 624 EPA 8260B (aqueous) SM6200B
MSP	Agilent 6890	Agilent 5973	EST Encon (dual)	EST Centurion	Supelco VOCARB 3000	Agilent Chemstation	EPA 624 EPA 8260B (aqueous) SM6200B

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Attachment 9
Standard Information and Recipes

Store standards in glass vials fitted with Teflon-lined caps in standards-only refrigerator at <-10C. Choose a container volume that will have minimal headspace when standard is added. Mark the level on the outside of the container so that evaporation of the solvent will be apparent.

The expiration dates represent the maximum time a standard can be maintained. Standards that exhibit obvious signs of degradation must be replaced sooner.

Routine Target Compounds

Stock Standard Mixes

Stock/Mix	TALS ID	Vendor/Part Number	Concentration (ug/mL)
O2SI 8260 Additional Compounds	8260 ADD	O2SI 120267-03-02	2500-5000
O2Si Custom VOA 57-1	8260VOA	O2SI 120315-01-02	2500-7500
8260B Surrogates from O2SI	O2SI Surr	O2SI 120005-05-02	2500
O2SI Custom Gases	O2SI-Gases	O2SI 120326-01-02	2500
O2SI 8260 Custom ISTD	O2SI-ISTD	O2SI 120192-02-02	2500

Expiration:
Un-opened ampuls: manufacturer's expiration date
Opened ampuls: 1 month (Note: These ampuls are used to prepare the Working Standards and are typically consumed/disposed open opening.)

8260 Working ISTD
(TALS ID: 8260 ISTD)

Stock/Mix	Initial Volume (uL)	Final Volume (mL)	Final Concentration (ug/mL)
O2SI 8260 Custom ISTD	500	25	50

Solvent: P/T Methanol
Expiration: 1 Month

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O2SI Primary MM + Butadiene/Methylstyrene
(Mega Mix Working Standard)
(TALS ID: O2SI MMix)

Stock/Mix	Initial Volume (uL)	Final Volume (mL)	Final Concentration (ug/mL)
O2SI 8260 Additional Compounds	500	25	50-100
O2Si Custom VOA 57-1	500		50-150
8260B Surrogates from O2SI	500		50
O2SI Custom Gases	500		50

Solvent: P/T Methanol
Expiration: 2 weeks from date prepared
(Note: The 25mL final volume is split between five 5mL Mini-nert vials. Due to the volatility of the gas analytes, each vial is assigned a 1-week expiration date from the date that vial was opened, not to exceed 14 days from the preparation date.)

Calibration Standards

Stock/Mix	1	2	3	4	5	6	7
	Aliquot (uL) to prepare CAL standard						
O2SI MMix	20	100	200	400	1000	2000	4000
8260 ISTD	100	100	100	100	100	100	100
Volume of water (mL)	100	100	100	100	100	100	100
Target Compounds (ng)	5	25	50	100	250	500	1000
Internal Standards (ng)	250	250	250	250	250	250	250

Transfer to 40mL VOA vial containing preservatives.
Expiration: 24 hours

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Appendix IX Target Compounds

Stock Standard Mixes

Stock/Mix	TALS ID	Vendor/Part Number	Concentration (ug/mL)
Appendix 9 Compounds	o2siAP_9	o2si 120314-01-02	1250-50000
Chloroprene Solution	o2siCPS	o2si 020315-030-02	1250
Pentachloroethane	o2siPENTA	o2si 020052-03-02	2500
Acrolein / Acrylonitrile	o2siA/A	o2si 120014-02	1000
Tetrahydrofuran / DEE Mix	o2siT/D	o2si 120312-02-02	2500-25000
O2SI 8260 Custom ISTD	O2SI-ISTD	O2SI 120192-02-02	2500

8260 Working ISTD
(TALS ID: 8260 ISTD)

Stock/Mix	Aliquot Volume (uL)	Final Volume (mL)	Final Concentration (ug/mL)
O2SI 8260 Custom ISTD	500	25	50

Solvent: P/T Methanol
Expiration: 1 month

AP9 Working Standard (TC2)
(TALS ID: o2si_TC2)

Stock/Mix	Aliquot Volume (uL)	Final Volume (mL)	Final Concentration (ug/mL)
Appendix 9 Compounds	500	25	25-1000
Chloroprene Solution	500		25
Pentachloroethane	500		50

Solvent: P/T Methanol
Expiration: 1 month

THF/DEE Working Standard
(TALS ID: XXXXX)

Stock/Mix	Aliquot Volume (uL)	Final Volume (mL)	Final Concentration (ug/mL)
Appendix 9 Compounds	500	25	25-1000
Chloroprene Solution	500		25
Pentachloroethane	500		50

Solvent: P/T Methanol
Expiration: 1 month

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Calibration Standards

Stock/Mix	1	2	3	4	5	6	7
	Aliquot (uL) to prepare CAL standard						
O2SI A/A	1	5	10	20	50	75	100
O2SI TC2	2	10	20	40	100	150	200
THF DEE	1	5	10	20	50	75	100
8260 ISTD	5	5	5	5	5	5	5
Volume of water (mL)	5.0	5.0	5.0	5.0	5.0	5.0	5.0
Target Compounds (ng)	5	25	50	100	250	500	1000
Internal Standards (ng)	250	250	250	250	250	250	250

Note: Record calibration standard preparation in A/D batch.
Expiration: 24 hours

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Attachment 10
Poor Performers

As indicated in EPA 8260B and/or via assessment of laboratory control sample recoveries and control charts, the compounds listed below are poor performers and/or behave erratically. These compounds will not be included in the LCS/LCSD/MS/MSD marginal exceedance count, provided their %R is >10%.

Additionally, these analytes have exceptions to the routine ICV and CCV requirements as outlined in the applicable section of the SOP.

Note: An NCM must be initiated to denote this situation.

Poor Performer List
2-chloroethyl vinyl ether
Acrolein
Carbon disulfide
Methyl Acetate
Pentachloroethane
Vinyl Acetate
Bromomethane
Iodomethane
1,4-dioxane
Chloroethane

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Attachment 11
Procedures for Evaluation of Tentatively Identified Compounds (TICs)

Tentatively identified peaks (TICs) are defined by TestAmerica Savannah as:
1) a calibrated analyte that is not part of the list of analytes requested by the client; or
2) a non-calibrated analyte with a response of 10% or greater than the closest internal standard (ISTD).

The laboratory's default procedure is to report the top 20 TICs with the highest concentration.

Note: Internal standards or surrogates added to the sample, whether they are included in the ICAL or not, must not be identified as TICs. For example, the surrogate o-Terphenyl is added to the low-level 8270 surrogate spiking mix but is used as a surrogate only for LL PAH. This compound would be excluded as a TIC. Also, for semi-volatile analyses, routine target volatile analytes included on the EPA CLP OLM04.2 list (e.g., xylenes) are not included as TICs.

Data Evaluation Steps:
Identification of TICs is made by comparison of the mass spectrum to the reference spectrum (peaks with calibration) or by comparison of the mass spectrum to a reference library such as NIST (peaks without a calibration). Only after visual comparison between the sample spectra and the library-generated reference spectra will the mass spectral analyst assign tentative identification.

The unknown compounds are tentatively identified using a search of the reference library. If the library search produces a match at or above 85%, report that compound. If the library search produces more than one compound at or above 85%, report the first compound (the highest match quality). If the library search produces no matches at or above 85%, report the compound as unknown. If possible, provide a general classification of the unknown – for example, unknown aromatic, unknown hydrocarbon, etc.

TICs should be evaluated within the retention time range from the first eluting target or surrogate (whichever is first in the target list) to the elution of the last target compound.

Relative intensities of the major ions (masses) in the reference spectra (ions >10% of the most abundant ion) should be present in the sample spectrum. The relative intensities of the major ions should agree within approximately ±20%.

Molecular ions present in the reference spectrum should be present in the sample spectrum. Note, however, that differences in the spectra may be attributed to over-lapping or co-eluting peaks. If, in the opinion of the analyst, there is enough evidence to support the tentative identification of a compound even though the above criteria are not met exactly, the peak may be considered tentatively identified. The analyst should consult the Department Manager if there are any questions concerning interpretation of spectra.

The estimated concentration of the tentatively identified compound (TIC) is calculated using the total ion area of the tentatively identified peak and total ion area

of the nearest internal standard that has no interferences. The concentration of TICs with a calibration is the concentration from the calibration curve at the dilution that the target list is reported, even if the concentration is above the calibration range (an "E" value). The concentration of the non-calibrated TIC is as directed in the SOP and as calculated in the Target data system.

Data Processing Steps:

- Evaluate the peaks in the total ion chromatogram for:
  - correct integration
  - peaks that may not have been integrated, paying particular attention to large or odd-shaped peaks.
  - closely eluting peaks
- Manually integrate any peaks that were not detected by the data system and re-process the unknowns.
- Evaluate TICs in Target, as outlined above.
- Merge to TALS.
- Under the TIC tab, reject all "TGT" and "TIC" analytes.
- Right-click and select "Auto-Set TICs Primary". This should set the number of TICs and TGTs requested with the highest concentration to a "Primary" status.
- Highlight all TGT compounds (still under the TIC tab) and right-click.
- Choose "Result Conditions".
- Right-click and choose "Show Assigned Conditions".
- Uncheck all assigned conditions.
- Right-click and choose "Show Flag Suite Conditions".
- Select J, N, and T. Be sure to choose the J-flag defined as "Estimated Result TIC – Manual Flag".

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Attachment 12
EPA 624 CCV and ICV Criteria
(Q Table)

Analyte	Criteria* (ug/L)
Benzene	12.8-27.2
Bromodichloromethane	13.1-26.9
Bromoform	14.2-25.8
Bromomethane	2.8-37.2
Carbon tetrachloride	14.6-25.4
Chlorobenzene	13.2-26.8
Chloroethane	7.6-32.4
2-chloroethyl vinyl ether	D-44.8*
Chloroform	13.5-26.5
Chloromethane	D-40.8
Dibromochloromethane	13.5-26.5
1,2-Dichlorobenzene	12.6-27.4
1,3-Dichlorobenzene	14.6-25.4
1,4-Dichlorobenzene	12.6-27.4
1,1-Dichloroethane	14.5-25.5
1,2-Dichloroethane	13.6-26.4
trans-1,2-Dichloroethene	13.9-26.1
1,1-Dichloroethene	10.1-29.9
1,2-Dichloropropane	6.8-33.2
cis-1,3-Dichloropropene	4.8-35.2
trans-1,3-Dichloropropene	10.0-30.0
Ethylbenzene	11.8-28.2
Methylene chloride	12.1-27.9
1,1,2,2-Tetrachloroethane	12.1-27.9
Tetrachloroethene	14.7-25.3
Toluene	14.9-25.1
1,1,1-Trichloroethane	15.-25.0
1,1,2-Trichloroethane	14.2-25.8
Trichloroethene	13.3-26.7
Trichlorotrifluoromethane	9.6-30.4
Vinyl chloride	0.8-39.2

*These values are given in ug/L (i.e., concentration ranges). The laboratory has converted these values into percent recovery limits to be used to assess CCV, ICV, LCS/LCSD, and MS/MSD.

18.0 Revision History

Summary of Changes from Previous Revision:

SA-VM-020, Rev. 7 (eff. date: 01/06/2011) to SA-VO-004, Rev. 0 (eff. date: 04/13/2012):

- Changed SOP Document Control Number. This was SOP SA-VM-020: *Volatile Compounds by GC/MS*. SOP SA-VM-020 is now obsolete.
- Minor grammatical and/or editorial changes made.
- Updated SOP references to reflect current document control numbers and titles.
- Included the hazards specific to hydrochloric acid, Section 5.1
- Revised poor performer list, Attachment 10.
- Removed requirement that IDOC must be from a second source and IDOC must be documented using the IDOC Form. These items are no longer mandatory. Section 12.5

SA-VO-004, Rev. 0 (eff. date: 04/13/2012) to SA-VO-004, Rev. 0A (eff. date: 06/04/2012):

Minor editorial and boilerplate changes, clarifications, and/or corrections were made as outlined below. These text revisions and additions are administrative in nature. They do not constitute a change to existing laboratory procedures and do not require analyst re-training.

- Revised the medium-level soil QC preparation instructions to match current laboratory practice. The text contained typographical errors. Section 10.1.4.

Approval Signature:

Andrea Teal June 4, 2012

Andrea Teal Date
Quality Assurance Manager

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STANDARD OPERATING PROCEDURE

SEI-10.15.8

THE DETERMINATION OF VOLATILE ORGANIC COMPOUNDS BY GAS CHROMATOGRAPHY / MASS SPECTROMETRY (GC/MS) (SW846 USEPA METHOD 8260C)

SOP Number: SEI-10.15.8
Revision Number: 8

Date Issued: 08/19/04
Date of Revision: 08/22/12

1.0 OBJECTIVE/

This method, based on SW846 USEPA Method 8260C for the analyses of volatile organic compounds, is designed to measure the concentration of specified volatile organic compounds (VOCs) in water, soil and air samples using solid phase micro-extraction (SPME) and a Gas Chromatograph equipped with a capillary column and mass spectrometer. Specific VOCs of interest are outlined in Table 1.

2.0 SUMMARY OF METHOD

This method provides gas chromatographic conditions for the detection of certain aromatic and chlorinated VOCs. Soil and water samples are prepared in 20 mL extraction vials. The SPME fiber is then exposed to the headspace above the sample for a set number of minutes while the sample is agitated via either stirring or orbital shaking. Air samples are collected in Tedlar bags and the SPME fiber is exposed directly to the sample through the syringe septum. The exposure time must be consistent for all standards and samples (typically six or eight minutes). The absorbed analytes are thermally desorbed from the SPME fiber in the injection port of a Gas Chromatograph and transferred to a narrow bore capillary column. A temperature program is used to enhance separation as the analytes are carried through the column to the ion source of the mass spectrometer. The ionized molecules are focused and separated according to their mass/charge (m/z) ratio by a quadrupole analyzer. The signal is amplified by an electron multiplier and interpreted by the mass spectrometer data system to produce a total ion or selected ion chromatogram and mass spectra for every point on the chromatogram. The instrument's mass spectrometer can be set in scan mode or selective ion monitoring mode (SIM). SIM mode is typically used to achieve lower reporting limits.

This method is suitable for analysis of air, water, soils, and wastes extractable in methanol.

Soil and waste samples are collected in approximately 10-12 gram cores directly into methanol (10 mL) in 40 mL VOA vials. Subsequent to sample collection, the methanol/solid mixture is agitated to break up the solids. Following this extraction step, an aliquot of the methanol is diluted into analyte-free water and is further extracted via the headspace SPME technique. Water samples are collected in 40 mL VOA vials with no headspace. All soil and water samples are analyzed as waters on the GC/MS.

This method is based on USEPA 8260C, *Volatile Organic Compounds by Gas Chromatography Mass Spectrometry (GC/MS)* and ASTM Method D6520, *Solid Phase Micro-extraction of water and its headspace for analysis of Volatile and Semi-volatile Organic Compounds.*

3.0 DEFINITIONS

Accuracy: the degree of agreement between a measurement and true or expected value, or between the average of a number of measurements and the true or expected value.

Batch: environmental samples, which are prepared and/or analyzed together with the same process, using the same lot(s) of reagents. A preparation batch is composed of one to 20 environmental samples of a similar matrix, meeting the above-mentioned criteria.

Corrective Action: action taken to eliminate the causes of an existing non-conformance, defect or other undesirable situation in order to prevent recurrence.

Demonstration of Capability (DOC): procedure to establish the ability to generate acceptable accuracy and precision.

Instrument Blank: a blank matrix that is the same as the processed sample matrix (i.e. extract, digestate, condensate) and introduced onto the instrument for analysis. Analyzed typically as a fiber blank.

Laboratory Duplicate: Aliquots of a sample taken from the same container under laboratory conditions and processed and analyzed independently. (NELAC)

Limit of Detection (LOD): An estimate of the minimum amount of a substance that an analytical process can reliably detect. An LOD is analyte- and matrix-specific and may be laboratory-dependent. (DOD QSMV4.2)

Limit of Detection (Clarification): The smallest amount or concentration of a substance that must be present in a sample in order to be detected at a high level of confidence (99%). At the LOD, the false negative rate (Type II error) is 1%. (DOD QSMV4.2)

Limits of Quantitation (LOQ): The minimum levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be reported with a specified degree of confidence. (DOD QSMV4.2)

Limit of Quantitation (Clarification): The lowest concentration that produces a quantitative result within specified limits of precision and bias. For DoD projects, the LOQ shall be set at or above the concentration of the lowest initial calibration standard.

Laboratory Control Sample (LCS): a blank matrix spiked with a known amount of analyte(s), processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure.

Matrix: the substrate of a test sample.

Matrix Duplicate (MD): duplicate aliquot of a sample processed and analyzed independently; under the same laboratory conditions; also referred to as Sample Duplicate; Laboratory Duplicate.

Matrix Spike (MS): field sample to which a known amount of target analyte(s) is added.

Matrix Spike Duplicate (MSD): a replicate matrix spike.

Method Blank: a blank matrix processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure.

Precision: an estimate of variability. It is an estimate of agreement among individual measurements of the same physical or chemical property, under prescribed similar conditions.

Method Detection Limit: One way to establish a Limit of Detection, defined as the minimum concentration of a substance (an analyte) that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte. (DOD QSMV4.2)

Method Detection Limit (MDL) (Clarification): The MDL is one way to establish a Detection Limit, not a Limit of Detection. (DOD QSMV4.2)

Nonconformance: An indication or judgment that a product or service has not met the requirement of the relevant specifications, contract, or regulation; also the state of failing to meet the requirements. (DOD QSMV4.2)

Precision: The degree to which a set of observations or measurements of the same property, obtained under similar conditions, conform to themselves; a data quality indicator. Precision is usually expressed as standard deviation, variance or range, in either absolute or relative terms. (NELAC)

Preservation: Refrigeration and/or reagents added at the time of sample collection (or later) to maintain the chemical and/or biological integrity of the sample. (NELAC)

Proficiency Testing: A means of evaluating a laboratory's performance under controlled conditions relative to a given set of criteria through analysis of unknown samples provided by an external source. (NELAC) [2.1]

Quality Assurance (QA): an integrated system of activities involving planning, quality control, quality assessment, reporting and quality improvement to ensure that a product or service meets defined standards of quality with a stated level of confidence.

Quality Control (QC): the overall system of technical activities, the purpose of which is to measure and control the quality of a product or service.

Quality Control Sample: a control sample, generated at the laboratory or in the field, or obtained from an independent source, used to monitor a specific element in the sampling and/or testing process.

Quantitation Limit (QL): the minimum amount of a substance that can be quantitatively measured with a specified degree of confidence and within the accuracy and precision guidelines of a specific measurement system. The QL can be based on the MDL, and is generally calculated as 3-5 times the MDL, however, there are analytical techniques and methods where this relationship is not applicable. Also referred to as Practical Quantitation Level (PQL), Estimated Quantitation Level (EQL), Limit of Quantitation (LOQ).

Quantitation Range: The range of values in a calibration curve between the LOQ and the highest successfully analyzed initial calibration standard. The quantitation range lies within the calibration range. (DOD QSMV4.2)

Raw Data: Any original factual information from a measurement activity or study recorded in a laboratory notebook, worksheets, records, memoranda, notes, or exact copies thereof that are necessary for the reconstruction and evaluation of the report of the activity or study. Raw data may include photography, microfilm or microfiche copies, computer printouts, magnetic media, including dictated observations, and recorded data from automated instruments. If exact copies of raw data have been prepared (e.g., tapes which have been transcribed verbatim, data and verified accurate by signature), the exact copy or exact transcript may be submitted. (EPA-QAD)

Reporting Limit (RL): The level to which data is reported for a specific test method and/or sample. The RL is generally related to the QL. The RL must be minimally at or above the MDL.

Requirement: Denotes a mandatory specification; often designated by the term "shall". (NELAC)

Retention Time: The time between sample injection and the appearance of a solute peak at the detector. (Skoog, West, and Holler. Fundamentals of Analytical Chemistry. 1992)

Sample: Portion of material collected for analysis, identified by a single, unique alphanumeric code. A sample may consist of portions in multiple containers, if a single sample is submitted for multiple or repetitive analysis. (DOD QSMV4.2)

Selectivity: (Analytical chemistry) The capability of a test method or instrument to respond to a target substance or constituent in the presence of non-target substances. (EPA-QAD)

Selective Ion Monitoring: A configuration of the mass spectrometer in which the intensities of several specific ion beams are recorded rather than the entire mass spectrum.

Sensitivity: The capability of a method or instrument to discriminate between measurement responses representing different levels (e.g., concentrations) of a variable of interest. (NELAC)

Shall: Denotes a requirement that is mandatory whenever the criterion for conformance with the specification requires that there be no deviation. This does not prohibit the use of alternative approaches or methods for implementing the specification so long as the requirement is fulfilled. (ANSI)

Should: Denotes a guideline or recommendation whenever noncompliance with the specification is permissible. (ANSI)

Signal to Noise Ratio: The signal carries information about the analyte, while noise is made up of extraneous information that is unwanted because it degrades the accuracy and precision of an analysis and also places a lower limit on the amount of analyte that can be detected. In most measurements, the average strength of the noise is constant and independent of the magnitude of the signal. Thus, the effect of noise on the relative error of a measurement becomes greater and greater as the quantity being measured (producing the signal) decreases in magnitude. (Skoog, Holler, and Nieman. Principles of Instrumental Analysis. 1998)

Spike: a known amount of an analyte added to a blank, sample or sub-sample.

Standard: (Document) The document describing the elements of laboratory accreditation that has been developed and established within the consensus principles of NELAC and meets the approval requirements of NELAC procedures and policies. (ASQC)

Standard: (Chemical) Standard samples are comprised of a known amount of standard reference material in the matrix undergoing analysis. A standard reference material is a certified reference material produced by the US National Institute of Standards and Technology (NIST) and characterized for absolute content, independent of analytical test method.

Standard Method: A test method issued by an organization generally recognized as competent to do so.

Standard Operating Procedure (SOP): a written document which details the method of an operation, analysis or action whose techniques and procedures are thoroughly prescribed and which is accepted as the method for performing certain routine or repetitive tasks.

SPME – Solid Phase Micro Extraction

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. (QAMS)

Target Analytes: Analytes specifically named by a client (also called project-specific analytes). (DOD QSMV4.2)

Technical Director: Individual(s) who has overall responsibility for the technical operation of the environmental testing laboratory. (NELAC)

Tedlar Bag – Inflatable sample container, used to trap air samples for analysis, equipped with a Teflon®-lined syringe septum that allows a SPME fiber to come in contact with the air sample.

Traceability: The property of a result of a measurement whereby it can be related to appropriate standards, generally international or national standards, through an unbroken chain of comparisons. (VIM - 6.12)

Tuning: A check and/or adjustment of instrument performance for mass spectrometry as required by the method.). (DOD QSMV4.2)

VOC – Volatile Organic Compounds

QA/QC – Quality Assurance/Quality Control

4.0 INTERFERENCES

When analyzing for VOCs, samples can be contaminated by diffusion of volatile organics (particularly chlorofluorocarbons and methylene chloride) through the sample container septum during shipment and storage. A trip blank can be used as a check on this type of interference however, a trip blank is not usually required if the analyses are being conducted onsite. A trip blank is prepared from analyte-free reagent water and carried through sampling and subsequent storage and handling.

Contamination by carryover can occur whenever high-concentration and low-concentration samples are analyzed in sequence. To reduce the potential for carryover, the sample syringe or purging device must be rinsed out between samples with an appropriate solvent. Whenever an unusually concentrated sample is encountered, it should be followed by injection of a method blank to check for cross contamination.

Purging vessels and syringes should be adequately cleaned and flushed prior to use. All glassware must be kept scrupulously cleaned. Clean all glassware as soon as possible after use by rinsing with the last solvent used or analyte-free water. Store clean dry glassware in a clean environment.

5.0 SAFETY ISSUES

1. Laboratory personnel should take extra care when working with standards. When working with neat standards, make certain that there is adequate ventilation and that Nitrile or Latex gloves are worn and if necessary, safety glasses.
2. If necessary and appropriate, a site-specific health and safety plan shall be created for each study site and followed.
3. Field visits may involve accessing remote areas. Health and safety concerns regarding these field visits are minimal, however, hazards such as slip, trip and falls, poisonous plant and dangerous animals, as well as getting vehicles stuck in remote areas, do present considerable health and safety issues. To help ensure field staff's health and safety in remote areas, all field staff are to have daily communication with the project manager or another appropriate SEI employee.
4. All chemicals are required to be received with a Material Safety Data Sheets (MSDS). MSDSs shall be made available to all personnel involved in the sampling and testing.

6.0 EQUIPMENT AND SUPPLIES

1. Gas Chromatograph: Analytical system complete with gas chromatograph and all required accessories, column supplies, gases, syringes and data system to determine peak areas and perform integrations. Hewlett Packard 5890 Series II.
2. Mass Spectrometer: Hewlett Packard 5971 MSD.
3. Typical Analytical Column: RTX-VMS 20m x 0.18mm ID, 1.0 um film or equivalent
4. SPME Autosampler: CTC Combi PAL
5. Array of micro-syringes: range 5-1000 uL.
6. Balance: Top loading, capable of weighing accurately to 0.01 grams (soil samples only).
7. Sample introduction and preparation apparatus: SPME Fiber type 100 µm polydimethylsiloxane (PDMS) and/or 85 µm Carboxen/PDMS fiber, magnetic hot plate and magnetic stirrers, tube clamp and stand.
8. VOA vials: 40 mL collection containers with Teflon®-lined septum and either 24mL or 20mL extraction vials

7.0 REAGENTS AND STANDARDS

Reagents:

1. Analyte-free reagent water demonstrated to be free of compounds of interest (spring water, carbon filtered and/or deionized).
2. Purge and Trap Grade or equivalent grade methanol (demonstrated to be free of analytes and stored away from other solvents).

Stock standards:

Stock standards may either be prepared from pure standard materials or purchased as certified solutions. Secondary dilution standards are prepared using stock standard solutions, which contain the compounds of interest, either as single compounds or mixed together. Typically, for this method the stock standards are obtained from an approved vendor and mixed together. A certificate of analysis is retained by the laboratory and maintained on file or on file with the vendor. The aqueous standards are typically prepared at concentrations as listed in Section 9.0 and are prepared in accordance with Table 3 which is an Example of the Standard Preparation Calculation Sheet. Gas standards are purchased in mixtures of site-specific target analytes.

Calibration standards are prepared at a minimum of five to seven different concentrations levels for VOC analytes.

Stone's SOP No. SEI-4.7.n *Labeling, Preparation and Storage of Reagents, Solutions and Standards* provides procedures on properly labeling, preparing and storing of reagents and standards used in the mobile laboratory for analytical methods. This SOP should be reviewed by the analyst before labeling, preparing and storing standards within the mobile laboratory.

8.0 SAMPLE COLLECTION AND HANDLING

8.1 Sample Collection

1. Aqueous samples are collected in 40 mL Teflon®-lined septum VOA vials filled to ensure that no headspace is present. Upon receipt, and if multiple vials are presented for a given sample, the vials will be uniquely numerically labeled (with the numbers 1, 2, 3, etc.) and at the time of analysis this unique identifier will be recorded in the instrument's run log. If storage of these samples is expected to exceed seven days, aqueous samples should be preserved with hydrochloric acid (HCl) to a pH of < 2.
2. Soil and waste samples are collected in approximately 10-12 gram cores, and placed in 40 mL vials with 10 mL of methanol. Upon receipt, and if multiple vials are presented for a given sample, the vials will be uniquely numerically labeled (with the numbers 1, 2, 3, etc.) and at the time of analysis this unique identifier will be recorded in the instrument's run log. If

required, a separate aliquot of soil from the sample location will be collected in a 40 mL vial (with no methanol preservative) for percent moisture determination.

3. Air samples are collected in Tedlar bags. Upon receipt, and if multiple bags are presented for a given sample, the bags will be uniquely numerically labeled (with the numbers 1, 2, 3, etc.) and at the time of analysis this unique identifier will be recorded in the instrument's run log. The analytes are concentrated on the SPME fiber by exposing the fiber directly to sample through the Teflon®-lined septum. Air samples must be analyzed within 3 days of collection.

8.2 Handling and Holding Times

1. Unpreserved water samples must be analyzed within 7 days of collection. Preserved (preserved with HCl to a pH of < 2) water samples must be analyzed within 14 days of collection. Soil samples collected in methanol must be analyzed within 14 days from collection. All samples shall be stored at < 6°C. Samples are allowed to come to room temperature just prior to analysis. All samples for this program are typically analyzed within one or two days from collection, therefore, holding times should not be a concern.

2. If samples were preserved and analyses are performed after 7 days from collection, pH determination (water samples only) should be performed. Once the sample aliquots have been taken from the VOA vial for analysis, the pH of the water sample must be determined. The purpose of the pH determination is to ensure that all VOA samples were acidified in the field. Test the pH by placing one or two drops of sample on the pH paper (do not add pH paper to the vial). Record the pH of each sample, and report these data on the runlog and in the SDG Narrative.. No pH adjustment is to be performed.

3. If necessary, a separate soil sample aliquot will be collected in a 40 mL vial or equivalent for percent moisture content so that results can be adjusted for %moisture and reported as dry weight rather than as received. Percent moisture is determined as follows: Percent Moisture Determination - Immediately after weighing the sample for analysis, weigh 5-10 g of the soil/sediment into a tared crucible. Determine the percent moisture by drying overnight at 105 °C. If available, allow to cool in a desiccator before weighing. Concentrations of individual analytes will be reported relative to the dry weight of soil/sediment.

$$\text{Percent Moisture} = \frac{\text{Mass of wet sample (g)} - \text{Mass of dry sample (g)}}{\text{Mass of wet sample (g)}}$$

9.0 PROCEDURES

9.1 Typical GC Program

Oven Temp: 50°C (4.00 min) to 160°C at 15°C/min to 200°C at 40°C/min, hold for 1 min.

Flow Rate: Helium, 1.0 mL/min, 8 PSI, set at 45°C

Detector: MSD, SIM mode, ion masses range between 45-190 amu

Injector: 240°C, splitless (close 1.5 min)

Liner: 0.75 mm ID splitless/split injection port

9.2 GC Column

Column: RTX-VMS 20m x 0.18mm ID, 1.0 um film, equivalent

9.3 Material Preparation:

SPME: Fiber Type: 100 µm polydimethylsiloxane (PDMS).

SPME Fiber Type for low level waters or specialized project only: 85 µm Carboxen/PDMS

SPME Extraction: The SPME-absorbent polymer fiber is exposed to the headspace above a sample. The headspace is created by removing approximately 2 mLs from the 20 mL preparation vial. Salt and stir bar (if stirring is used) are added to the mixture and the sample is agitated for 6 or 8 minutes. The fiber is then retracted into the SPME holder and transferred to the GC injection port where the analytes are thermally desorbed and transferred to the head of the GC column. If used, each magnetic stirrer is rinsed three times with analyte free water prior to use. For soil samples; an aliquot of the methanol extract (typically 0.5mL but not more than 1 mL) is prepared in the 20 mL water preparation vial, and prepared and analyzed in the same manner as a water sample above. For air samples, the SPME fiber is exposed directly to the same through the syringe septum located on the Tedlar bag. Preparation and exposure time of the fiber to the site samples is the same for all QC samples and calibration standards.

9.4 Operating Procedure

9.4.1 Tuning with 4-Bromofluorobenzene

The following ion abundances must be met before analysis of calibration standards, environmental samples and at the beginning of a 12-hour analytical sequence by analyzing BFB in a check standard, method blank or as a single injection:

BFB Key Ions and Ion Abundance Criteria	
Mass	Ion Abundance Criteria
50	15.0-40.0 percent of mass 95
75	30.0-60.0 percent of mass 95
95	base peak, 100 percent relative abundance
96	5.0-9.0 percent of mass 95
173	less than 2.0 percent of mass 174

BFB Key Ions and Ion Abundance Criteria	
174	>50.0 percent of mass 95
175	5.0-9.0 percent of mass 174
176	95.0-101.0 percent of mass 174
177	5.0-9.0 percent of mass 176

9.4.2 Calibration Criteria

9.4.2.1 Initial Calibration (ICAL):

1. Five to seven-point calibrations for VOC compounds using a SIM configuration is as follows: 2, 5, 10, 20, 50, 100, 200 ppb. (Scan calibrations are determined as needed by the client's program) The low standard must be set at the quantitation limit (QL) or LOQ. A typical calibration range for air ($ppb^{v}/_{v}$) will be similar to that of water and will be chosen on a site specific basis.

2. Calibration range must encompass the expected sample concentrations; therefore, if site samples are expected to be higher in concentration than the calibration range, adjustments to the calibration range and samples will be documented and made accordingly.

3. The linearity of the calibration curves must be assessed and are used for all quantitation. Volatile organic compounds in soil and water use an internal standard procedure (internal standards; fluorobenzene and chlorobenzene-d5). Average response factors are used for quantitation and the average relative standard deviation (%RSD) of each compound must be $\leq$ 20%. Linear least squares regression must be $r > 0.995$

4. Non-linear regression coefficient of determination (COD) must be $r2 > 0.99$ (6 points shall be used for second order, 7 points shall be used for third order). Depending upon the field on-site program, 25% RSD may be acceptable based on the on-site data quality objectives.

   For DoD Projects (DOD QSM4.2, Table F-4), one of the following must be true for each analyte in the calibration:

   - % RSD for each analyte must be $\leq$15% (method requirement $\leq$ 20%).
   - Linear least squares regression must be $r \geq 0.995$
   - Non-linear regression coefficient of determination (COD) must be $r^2 \geq 0.99$ (6 points shall be used for second order, 7 points shall be used for third order).

Note: The average response factor requirements are only for 8260B and were not included in 8260C, therefore, for DOD projects the laboratory will follow the updated method. (The average response factor requirement will be $\geq$0.30 for chlorobenzene and _1,1,2,2-tetrachlorethane, and

≥0.1 for chloromethane, bromoform, and 1,1-dichloroethane (SPCCs). % RSD must be ≤30% for CCCs if analyzing for 8260B only).

5. VOCs in air use an external calibration procedure. Linear regression is used for quantitation and the correlation coefficient of each compound must be greater than 0.99

9.4.2.2 Continuing Calibration (CCV):

1. A mid-point continuing calibration verification standard for VOC compounds (20 ppb or 50ppb) must be analyzed every 12 hours, prior to sample analysis in order to verify the initial calibration. A percent difference between the expected concentration and actual concentration is calculated. The percent difference (%D) values for each compound must be less than or equal to 20%.

$$\%D = \frac{ExpectedConcentration - ActualConcentration}{ExpectedConcentration}$$

The CCV may also be used to demonstrate that the BFB criteria shown in 10.4.1 have been met when operating in scan mode.

9.4.2.3 Initial Calibration Verification Standard (ICV):

A QC sample from a second (independent) source will be analyzed directly after the ICAL. The ICV sample will contain all the VOC compounds of interest at the mid-point (20-100 ppb or 5 ppb for low level waters). Recovery Limits for the ICV will be 70-130%, if compounds are determined to be poor performers then the recoveries shall be 60-140% for the ICV.

For DoD projects, all project analytes must be within tolerances listsed in Table 2.

$$\%Recovery = \frac{Actual\ Concentration}{Expected\ Concentration}$$

9.4.3 Retention Time Windows and Pattern Recognition

The laboratory shall identify single component target analytes by comparison of its mass spectrum to a reference spectrum. The retention time for each analyte should be within ± 0.04 minutes (DOD QSM4.2, Table F-4 allows +/-0.06 minutes) from the ICAL or daily CCV.

9.4.4 Internal Standard Response

Internal standard areas shall be evaluated based on the value that was observed in the midpoint of calibration curve. The internal standard areas shall be less than ½ or greater than two times the area found in the midpoint of the curve.

9.4.5 Sample Preparation:

Water samples: An aliquot of sample, sufficient to get the highest concentration constituents into the upper half of the linear range, is transferred to a 20 mL extraction vial which contains approximately ¼ tsp. salt. The vial is then spiked with the surrogate (bromofluorobenzene between 4.17 and 41.67 ppb) and internal standard (fluorobenzene between 4.17 and 41.67 ppb). Invert the vial a few (approximately 3) times and remove approximately five mLs and place the vial on the autosampler tray. The autosampler tray is then placed on the CTC autosampler and set for analysis in accordance with the CTC specifications. At that point the CTC autosampler agitates the sample and the SPME fiber is then exposed to the headspace (headspace created by removing approximately 5 mL from preparation vial) above the sample for 6 or 8 minutes while the sample is agitated. The 6 or 8 minutes is measured by either the autosampler. Extraction time may be modified depending on the anticipated equilibration response of the target analytes at given concentrations (if adjusted samples and standards would be adjusted).

Soil samples: Preserved in methanol; 10-12 g core of soil is added to 10 mL of methanol containing an appropriate amount of bromofluorobenzene. Subsequent to sample collection, the methanol/soil mixture is agitated to break up the solids. The level of agitation shall be sufficient enough so that the solids are completely disaggregated as to maximize the contact between the methanol and the soil surfaces. This is typically achieved within 10 minutes using an aggressive orbital shaking procedure. Following this extraction step, an aliquot of the methanol, sufficient to get the highest concentration constituents into the upper half of the linear range but not to exceed 2 mLs, is diluted into analyte-free water and this water/methanol mixture is further extracted via the headspace SPME technique in the same manner as a water sample.

Air samples: Exposure of the SPME fiber to the air sample occurs directly through the syringe septum in the Tedlar bag. Exposure time, measured using a bench-top timer, may be modified depending on the anticipated equilibration response of the target analytes at given concentrations. There is no surrogate or internal standard for air sampling.

9.4.6 Analysis of Samples:

Analysis of samples is performed using SPME followed by analysis on a GC equipped with a narrow bore capillary column and a MSD. For this program the fiber is exposed to the headspace above the sample in the CTC autosampler. A small portion of sample is discarded from the 20 ml vial prior to exposure to generate headspace (approximately 5 mLs). The exposure time needs to be consistent (typically 6 to 8 minutes for this method). To facilitate the release of the analytes from their respective media, the sample is agitated throughout the extraction process (fiber exposure). Following extraction, the fiber adsorbed analytes are

thermally desorbed within the injection port of the GC and are transferred to the capillary column.

9.4.7 Surrogate (SS) and Internal Standard (ISTD) Compounds for VOC compounds

Compound	Amount Spiked
Bromofluorobenzene (SS)	Range: 4.17 to 41.67 ppb
Fluorobenzene (ISTD)	Range: 4.17 to 41.67 ppb
Chlorobenzene-d5 (ISTD)	Range: 4.17 to 41.67 ppb

9.4.8 LCS Preparation

Compounds and spike amounts are on Table 1. For a waters program, compounds and spike amounts are specified according to project specific needs. Waters are spiked at around the mid-point of the calibration.

For the soils program, the soil LCS will be prepared in the same manner as the soil samples, an aliquot (0.5 mL or 1.0 mL) of methanol will be prepared in the SPME preparation vial, spiked with VOCs of interest, and prepared in the same manner as the soil MS/MSD.

9.4.9 Matrix Spike and Matrix Spike Duplicate (MS/MSD) Compounds:

For water samples the MS in SIM configuration will be spiked at 20 ppb; the amount may change to account for the concentration of a particular analyte found in the sample being analyzed as a MS. For soil samples, an appropriate amount of the target analytes are spiked into the 10 mL of methanol. Matrix Spike Analyses contains the VOC compounds of interest in the same manner as the LCS.

9.4.10 Target Compound List Compounds

A Target Compound List with Quantitation Limits can be found on Table 1.

10.0 CALCULATIONS

Identification of the project specific VOCs in the sample chromatogram is based on retention time and on comparison of the sample mass spectrum with characteristic ions in a reference mass spectrum. Retention times must be within ± 0.04 minutes from the ICAL. If a retention time shift is observed in the CCV or daily ICV, then adjustments to the daily retention time windows will be made accordingly. The relative intensities of the characteristic ions must agree within 30% of these ions in the reference spectrum. Quantification of the VOCs

is determined by comparison to the internal standard and average response factor for the target ion of the analyte.

The concentration of each analyte in the sample may be determined by calculating the amount of standard injected, from the target ion peak response, using the calibration curve. The calculations are as noted below.

Concentrations of Unknowns

Method 8260B does not include reporting of tentatively identified compounds (TIC). However, reporting of up to 10 TIC's whose peak heights are ≥ 10% of the nearest internal standard may be requested or ≥ 40% of the nearest internal standard may be requested for low concentration analyses only when the laboratory has analyzed the samples in Scan Mode. Concentrations are calculated using the following formula using an assumed response factor (RF) of 1:

$$C_{(x)} = \frac{C_{(IS)} * A_{(x)}}{A_{(IS)}} * DF$$

Where:

$C_{(x)}$= Concentration of Unknown (µg/L).

$C_{(IS)}$= Concentration of internal standard (µg/L).

DF = Dilution Factor

$A_{(x)}$ = Area of Unknown (from the total ion chromatogram)

$A_{(IS)}$= Area of associated internal standard (from the total ion chromatogram).

Concentrations of Calibrated Compounds (when RRF is used):

$$C_{(x)} = \frac{A_{(x)} * C_{(IS)}}{A_{(IS)} * \overline{RRF}} * DF$$

Where:

$C_{(x)}$ = Concentration of compound (µg/L)

$C_{(IS)}$ = Concentration of associated internal standard (µg/L).

DF = Dilution Factor.

$A_{(IS)}$ = Area of quantitation ion for associated internal standard.

$A_{(x)}$ = Area of quantitation ion for compound.

RRF = Average Relative Response Factor from five-point initial calibration.

Note: for linear and quadratic equations see SW846 Method 8000C.

Data Reporting - Based on the mass spectra and the project specific field program, it is appropriate to report values between the MDL/Limit of detection (LOD) and the QL or LOQ. In this region, an analyte can be qualitatively detected, but not accurately quantified. Any data point reported in this region is flagged with a "J" (estimated) qualifier. Stone reports sample specific QL's. Sample specific QL's are derived by taking into account various sample specific data, which can include the amount of the sample subject to testing, % moisture, dilution factor, interferences and the base QL's for the analysis.

Typical Reporting qualifiers are as follows:

B = Analyte is found in the associated method blank as well as the sample.

D = Compound is identified in an analysis at a secondary dilution factor.

E = Compound quantitation is above the instrument's calibration range for this analysis.

J = Indicates an estimated quantitation value.

Q = One or more quality control criteria failed (e.g., LCS recovery, surrogate spike recovery or CCV).

U = Compound was analyzed for but not detected. Value reported is the method LOQ.

11.0 QUALITY CONTROL AND CORRECTIVE ACTION

11.1 Instrument Blank (SPME Fiber Blank)

To be performed once daily at the start of the analytical day. All target analyte areas detected in the instrument blank must be less than one-half the area of the analyte at the PQL value prior to continuing.

11.2 MS Tuning - 4-Bromofluorobenzene

To be performed every 12 hours, at the start of the analytical day and before beginning acquisition of a calibration curve. Criteria can be found in section 9.4.1.

11.3 Initial Calibration

Prior to the analysis of any samples or QC samples an initial calibration curve for the quantification of analytes of interest is established using criteria as noted in Section 9.0. Minimum of 5 calibration standards with the lowest standard concentration at or below the RL. %RSD for all analytes $\leq$20%RSD. Least squares regression r $\geq$ 0.995; non-linear regression–coefficient of determination (COD) r2 $\geq$ 0.99 (6 points shall be used for second order, 7 points shall be used for third order). If this criteria is not met, then correct problem then repeat ICAL.

Calibration range must encompass the expected sample concentrations; therefore, if site samples are expected to be higher in concentration than the calibration range, adjustments to the calibration range and samples will be documented and made accordingly.

11.4 Continuing Calibration Verification

To be performed every 12 hours, at the start of the analytical day. Percent differences or percent drift must be less than or equal to $\leq 20\%$.

11.5 Method Blank

One per analytical batch (12 hours or 20 samples) directly after the calibration standard and after highly contaminated samples to demonstrate that there is no carry over into subsequent samples. All target analyte concentrations detected in the method blank must be less than one-half the PQL value prior to continuing.

For DoD projects, the method blank will be considered to be contaminated if:

- The concentration of any target analyte in the blank exceeds ½ the reporting limit **and** is greater than 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater);
- The concentration of any common laboratory contaminant in the blank exceeds the reporting limit **and** is greater than 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater); or
- The blank result otherwise affects the samples results as per the test method requirements of the project-specific objectives.

If the method blank is contaminated as described above, then the laboratory shall reprocess affected samples in a subsequent preparation batch, except when sample results are below the limit of detection (LOD). If insufficient sample volume remains for reprocessing, the results shall be reported with appropriate data qualifiers.

11.6 Surrogate Standards

Surrogate recoveries should be 70-130 % in water samples and 60-140 % in soil samples. Samples with surrogate recoveries falling outside of quality control limits should be reanalyzed once. If the reanalysis of the sample is also outside the quality control limits, then it will be determined that the problem may be associated with the sample matrix and noted as such.

For DoD projects, the acceptance limits for 4-Bromofluorobenzene as a surrogate are 75-120%.

11.7 Internal Standards

Internal standard areas shall be evaluated based on the value that was observed in the midpoint of calibration curve. If the internal standard area is less than ½ or greater than two times the area found in the midpoint of the curve, the affected sample(s) should be rerun to confirm a possible matrix affect.

11.8 Initial Calibration Verification Standard (ICV)

A QC sample from a second source independent of the initial calibration will be analyzed directly after the ICAL or CCV. The ICV sample will contain all the VOC compounds of interest at mid point of the calibration curve. Recovery Limits for the ICV will be 70-130% and 40-160% for poor performers. For DoD projects, the ICV Recovery Limits will be 80-120%. This sample may also be used as the laboratory control sample (LCS).

11.9 Laboratory Control Sample (LCS)

For a waters program, compounds and spike amounts are specified according to project specific needs. Waters will be spiked at around the mid-point of the calibration. For the soils program, the soil LCS will be prepared in the same manner as the soil samples, an aliquot (0.5 mL) of methanol will be prepared in the SPME preparation vial, spiked with VOCs of interest, and prepared in the same manner as the soil MS/MSD. Recovery Limits for the LCS are as specified in Table 2 (DOD LCS Limits) and for compounds not in Table 2 the limits will be 60-140%. These shall be analyzed on the frequency of one per analytical batch. For DoD projects, all target analytes must be spiked in the LCS. If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.

11.10 Matrix Spike and Matrix Spike Duplicate (MS/MSD) Analysis

An MS/MSD analysis is required every set of twenty samples or as specified by the client. A sample may be chosen by the client to perform a MS/MSD analysis.

Recovery Limits for the MS/MSD are as specified in Table 2 (DOD LCS Limits) and for compounds not in Table 2 the limits will be 60-140%. For DoD projects, all target analyes must be spiked in the MS/MSD samples.

$$\%\text{Recovery} = \frac{\text{Sample Spiked Concentration} - \text{Un-Spiked Sample Concentration}}{\text{Spiked Concentration}}$$

11.11 CORRECTIVE ACTION

Quality Control Check	*Minimum Frequency*	*Acceptance Criteria*	*Corrective Action*
Initial Calibration (ICAL)	ICAL prior to sample analysis.	Minimum of five to six points for VOCs. Minimum RRF and Maximum %RSD on Table 1 or a correlation factor (R or R^2) of 0.99 for other curve types. Air calibration's linear regression should have correlation factor of 0.99.	Verify solution integrity and check instrument performance. Perform necessary maintenance and recalibrate instrument. Reanalyze all affected samples.
Initial Calibration Verification (ICV) and/or Laboratory Control Sample (LCS)	One ICV per calibration (following acceptable ICAL). One LCS per analytical batch.	QC limits are 70 to 130% for target compounds in water and soil analyses. This same standard may be used as the daily LCS in which the QC limits are set to 70-130% for target compounds and 60-140% for poor performers. All analytes for air analyses should be within 60-140%. For DoD projects, the ICV Recovery Limits will be 80-120%. This sample may also be used as the laboratory control sample (LCS) Note: Limits may be adjusted based on the program.	Reanalyze once; if still out verify solution integrity or ICAL solution integrity, and instrument performance. Re-prepare and reanalyze all associated samples.
Retention Time (RT) Window Study	Every new column installation/per ICAL	All target compounds and surrogates in all standards must fall within the established window of ± 0.04 minutes from ICAL	Perform system maintenance. Reanalyze affected samples.
Continuing Calibration Verification (CCV)	Daily before sample analysis and every 12 hours of analysis time.	See Section 9.0. ± 20%D for VOCs in water and soils programs. ± 30%D for VOCs in air program.	Verify solution integrity and instrument performance. Reanalyze standard once, if still out, recalibrate and reanalyze affected samples. Based on project specific objectives, marginal exceedances may be allowed. If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to all results for the specific analyte(s) in all samples since last acceptable CCV.

Quality Control Check	*Minimum Frequency*	*Acceptance Criteria*	*Corrective Action*
Method Blanks	One per preparation batch	Target Compounds < one-half reporting limit. See section 11.4 above for DOD criteria.	Investigate source of contamination. Re-prepare and reanalyze all associated samples. If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch.
Matrix Spike (MS) and Matrix Spike Duplicate (MSD)	One set per 20 samples of a similar matrix or as specified by the Client.	Recovery Limits for the MS/MSD are as specified in Table 2 (DOD LCS Limits) and for compounds not in Table 2 the limits will be 60-140%. RPD not to exceed 30% for waters RPD not to exceed 50% for soil samples.	Reanalyze only if recoveries indicate analytical error. If LCS or ICV are acceptable, narrate as possible matrix effect. For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and put in the case narrative.
Tuning – 4-Bromofluorobenzene	every 12 hours or at the start of a new analytical day, and before acquisition of a new calibration curve	See section 9.4.1. Not applicable for air program.	Reanalyze once, if still out, MS should be retuned and adjusted to meet all criteria before continuing
Surrogate Recovery	Each sample	70-130% in water and 60-140% for soil samples For DOD projects 75-125% for water and soil samples	Rerun the sample once, if still out verify the solution integrity and instrument performance. Apply Q-flag or other flag to all associated analytes if acceptance criteria are not met.
Internal Standard Verification	Every field sample, standard, and QC sample.	Retention time ± 30 seconds from retention time of the midpoint standard in the ICAL; EICP area within -50% to +100% of ICAL midpoint standard.	Inspect mass spectrometer and GC for malfunctions. Reanalysis of samples analyzed while system was malfunctioning is mandatory. If corrective action fails in field samples, apply Q-flag to analytes associated with the non-compliant IS. Flagging criteria are not appropriate for failed standards.

11.12 Data Review

The analyst is responsible for primary data review of data generated from the sample analysis. Analyses will be documented in the instrument run log. Maintenance is documented in the Instrument Maintenance Logbook. Instrument calibrations and recoveries of all QC samples must be within specified control limits. If instrument calibration or the recoveries of any QC sample exceed specified tolerances, then the affected sample results are evaluated and generally the samples are submitted for

re-analysis. Manual integrations should be kept to a minimum and should be dated and initialed by the analyst. To determine if analytical results are acceptable, a qualified and independent QA/QC program shall conduct a secondary review prior to reporting of the final data package to the client. A QA/QC Checklist (Attachment 1) is used to indicate any problems with the referenced analytical batches. The QA/QC checklist will also address any corrective action taken. All calibrations, calculations, and transcriptions are checked for accuracy and QC sample results are evaluated against specified limits. If instrument calibration and recoveries of all QC samples are within the specified criteria, then the data reports will be submitted to the Project Manager as final results with no qualifiers. If recoveries of any QC samples exceed specified limits and re-analysis is not an option, then the samples will be qualified with a Q-flag or as estimated with a "J" qualifier (J= The analyte was positively identified; the associated numerical value is the approximate concentration of the analyte in the sample.) depending on the corrective action or the QC element that failed. Data will not be reported if significant QC issues affect the batch analyses.

12.0 POLLUTION PREVENTION & WASTE MANAGEMENT

Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. The United States Environmental Protection Agency (USEPA) has established a prevention hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the agency recommends recycling as the next best option.

The quantity of chemical purchased should be based on expected usage during its shelf life and disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.

For information about pollution prevention that may be applicable to laboratories and research institutions, consult "Less is Better: Laboratory Chemical Management for Waste Reduction", available from the American Chemical Society's Department of Government Regulations and Science Policy, 1155 16th Street N.W., Washington, D.C. 20036; (202) 872-4477.

The USEPA requires that laboratory waste management practices conducted be consistent with all applicable rules and regulations. Excess reagents, samples, and method process wastes should be characterized and disposed of in an acceptable manner. The Agency urges laboratories to protect the air, water and land by minimizing and controlling all releases from hoods and bench operations, complying with the letter and spirit of any waste regulations, particularly the hazardous waste identification rules and land disposal restrictions. For further information on waste management consult the "Waste Management Manual for Laboratory Personnel", available from the American Chemical Society.

13.0 REFERENCES

40 CFR Part 136, Appendix A, paragraphs 8.1.1 and 8.2

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USEPA SW846 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, Update IV, 2008, 8000C, March 2003.

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14.0 TABLES, DIAGRAMS, FLOWCHARTS, AND VALIDATION DATA

Attachment 1: Sample GC Laboratory QA/QC Checklist

Table 1: Analyte Details – LOQ/QLs and MS/MSD Spike values shown are for SIM Configuration

Analyte	CAS #	**Minimum RRF	Maximum %RSD	LOQ/QL Water (ug/L)	LOQ/QL Air (ppbv/v)	LOQ/QL Soil (ug/Kg)
Chloromethane	74-87-3	0.01	20%	5	1-50	120
Vinyl Chloride	75-01-4	0.01	20%	2	1-50	80
Chloroethane	75-00-3	0.1	20%	2	1-50	80
1,1,2-Trichloro-1,2,2-trifluoroethane	76-13-1	0.1	20%	2	1-50	80
1,1-Dichloroethene	75-35-4	0.01	20%	2	1-50	80
+Carbon Disulfide	75-15-0	0.1	20%	2	1-50	80
+Methylene Chloride	75-90-2	0.01	20%	2	1-50	80
+t-Butyl-methyl ether	1634-04-4	0.1	20%	2	1-50	80
trans-1,2-Dichloroethene	156-60-5	0.1	20%	2	1-50	80
1,1-Dichloroethane	75-34-3	0.1	20%	2	1-50	80
cis-1,2-Dichloroethene	156-59-2	0.1	20%	2	1-50	80
*+2-Butanone	78-93-3	0.1	20%	50	1-50	2000
Chloroform	67-66-3	0.1	20%	2	1-50	80
1,1,1-Trichloroethane	71-55-6	0.1	20%	2	1-50	80
Cylcohexane	110-82-7	0.1	20%	2	1-50	80
Carbon Tetrachloride	56-23-5	0.1	20%	2	1-50	80
Benzene	71-43-2	0.1	20%	2	1-50	80
1,2-Dichloroethane	107-06-2	0.01	20%	2	1-50	80
Trichloroethene	79-01-6	0.1	20%	2	1-50	80
Methyl Cyclohexane	108-87-2	0.1	20%	2	1-50	80
1,2-Dichloropropane	78-87-5	0.1	20%	2	1-50	80
Bromodichloromethane	75-27-4	0.1	20%	2	1-50	80
cis-1,3-Dichloropropene	10061-01-5	0.1	20%	2	1-50	80
*+4-Methyl-2-pentanone	108-10-1	0.1	20%	50	1-50	2000
Toluene	108-88-3	0.1	20%	2	1-50	80
Trans-1,3-Dichloropropene	10061-02-6	0.1	20%	2	1-50	80
1,1,2-Trichloroethane	79-00-5	0.1	20%	2	1-50	80
Tetrachloroethene	127-18-4	0.1	20%	2	1-50	80
+2-Hexanone	591-78-6	0.1	20%	50	1-50	2000
Dibromochloromethane	124-48-1	0.1	20%	2	1-50	80
1,2-Dibromoethane	106-93-4	0.1	20%	2	1-50	80
Chlorobenzene	108-90-7	0.1	20%	2	1-50	80
Ethylbenzene	100-41-4	0.1	20%	2	1-50	80
M+p-Xylenes	108-38-3 106-42-3	0.1	20%	2	1-50	80
o-Xylene	95-47-6	0.1	20%	2	1-50	80

Analyte	CAS #	**Minimum RRF	Maximum %RSD	LOQ/QL Water (ug/L)	LOQ/QL Air (ppbv/v)	LOQ/QL Soil (ug/Kg)
Styrene	100-42-5	0.1	20%	2	1-50	80
Bromoform	75-252	0.1	20%	2	1-50	80
Isopropylbenzene	98-82-8	0.1	20%	2	1-50	80
Bromofluorobenzene	460-00-4	0.1	20%	2	1-50	80
1,1,2,2-Tetrachloroethane	79-34-5	0.1	20%	2	1-50	80
1,3-Dichlorobenzene	541-73-1	0.1	20%	2	1-50	80
1,4-Dichlorobenzene	106-46-7	0.1	20%	2	1-50	80
1,2-Dichlorobenzene	95-50-1	0.1	20%	2	1-50	80
+1,2-Dibromo-3-chloropropane	96-12-8	0.1	20%	2	1-50	80
1,2,4-Trichlorobenzene	120-82-1	0.1	20%	2	1-50	80
1,3,5-Trimethylbenzene	108-67-8	0.1	20%	2	1-50	80
1,2,4-Trimethylbenzene	95-63-6	0.1	20%	2	1-50	80
Naphthalene	91-20-3	0.1	20%	2	1-50	80

*Ketones or other VOC compounds not listed here may be analyzed for as verified by a successful method verification study. Method validations are performed at time of analysis when other compounds outside this list are added for a project.
**Minimum response factors for a select number of compounds are lower than what is recommended in 8260C due to the fact that this method deploys solid phase microextraction as opposed to purge and trap and these minimum RFs are acceptable as demonstrated with demonstration of an acceptable LOQ standard.

+- Poor Performer

Table 2: LCS Control Limits for DoD Projects (presented in terms of percent recovery)

Analyte	CAS #	Lower DoD Control Limit (Water)	Upper DoD Control Limit (Water)	Lower DoD Control Limit (Solids)	Upper DoD Control Limit (Solids)
Chloromethane	74-87-3	40	125	50	130
Vinyl Chloride	75-01-4	50	145	60	125
Chloroethane	75-00-3	60	135	40	155
1,1,2-Trichloro-1,2,2-trifluoroethane	76-13-1	NA	NA	NA	NA
1,1-Dichloroethene	75-35-4	70	130	65	135
Carbon Disulfide	75-15-0	35	160	45	160
Methylene Chloride	75-90-2	55	140	55	140
t-Butyl-methyl ether	1634-04-4	65	125	NA	NA
trans-1,2-Dichloroethene	156-60-5	60	140	65	135
1,1-Dichloroethane	75-34-3	70	135	75	125
cis-1,2-Dichloroethene	156-59-2	70	125	65	125
2-Butanone	78-93-3	30	150	30	160
Chloroform	67-66-3	65	135	70	125
1,1,1-Trichloroethane	71-55-6	65	130	70	135
Cylcohexane	110-82-7	NA	NA	NA	NA
Carbon Tetrachloride	56-23-5	65	140	65	135
Benzene	71-43-2	80	120	75	125
1,2-Dichloroethane	107-06-2	70	130	70	135
Trichloroethene	79-01-6	70	125	75	125
Methyl Cyclohexane	108-87-2	NA	NA	NA	NA
1,2-Dichloropropane	78-87-5	75	125	70	120
Bromodichloromethane	75-27-4	75	120	70	130
cis-1,3-Dichloropropene	10061-01-5	70	130	70	125
4-Methyl-2-pentanone	108-10-1	60	135	45	145
Toluene	108-88-3	75	120	70	125
Trans-1,3-Dichloropropene	10061-02-6	55	140	65	125
1,1,2-Trichloroethane	79-00-5	75	125	60	125
Tetrachloroethene	127-18-4	45	150	65	140
2-Hexanone	591-78-6	55	130	45	145
Dibromochloromethane	124-48-1	60	135	65	130
1,2-Dibromoethane	106-93-4	80	120	70	125
Chlorobenzene	108-90-7	80	120	75	125
Ethylbenzene	100-41-4	75	125	75	125
M+p-Xylenes	108-38-3 106-42-3	75	130	80	125
o-Xylene	95-47-6	80	120	75	125
Styrene	100-42-5	65	135	75	125
Bromoform	75-252	70	130	55	135
Isopropylbenzene	98-82-8	75	125	75	130
1,1,2,2-Tetrachloroethane	79-34-5	65	130	55	130
1,3-Dichlorobenzene	541-73-1	75	125	70	125
1,4-Dichlorobenzene	106-46-7	75	125	70	125

Analyte	CAS #	Lower DoD Control Limit (Water)	Upper DoD Control Limit (Water)	Lower DoD Control Limit (Solids)	Upper DoD Control Limit (Solids)
1,2-Dichlorobenzene	95-50-1	70	120	75	120
1,2-Dibromo-3-chloropropane	96-12-8	50	130	40	135
1,2,4-Trichlorobenzene	120-82-1	65	135	65	130

Table 3: Example Standard Preparation Calculation Sheet

STONE ENVIRONMENTAL INC

Gas Chromatography Laboratory
HP-5
Standard Prep Log

08/16/05-a1					dmkc	
FID WS						
100	ppm					
x: 8/31/05						
Compound	Vendor	Lot	Exp	Conc (mg/ml)	Amount	
Custom VOC	S	LB18097	8/16/2006	20	20	ul
1,1-DCA	A	B2090306	6/22/2006	20	20	ul
1,1,1-TCA	A	B3050086	6/22/2006	10	40	ul
8020 Mix	A	B1070292	8/31/2005	2	200	ul
1,1-DCE	A	B4050042	1/21/2006	20	20	ul
BFB	A	B4110093	5/17/2006	25	16	ul
MeOH	OmniSolv	44090	3/1/2008	into	3684	ul
				Total	4000	ul

15.0 AUTHORIZATION

Authored by: _____ Date: 8/24/12

David Crosby, Project Scientist

Approved by: _____ Date: 24 AUG 2012

Seth Pitkin, Vice President

16.0 REVISIONS

Revision 1:

1. Included the analytical process to analyze air in a tedlar bag using SPME and SW846 Method 8260.

Revision 2:

1. Allow a range for the concentration of the internal standard from 8.33 to 16.67 ppb.
2. Section 9.4. Included text to allow for the BFB tune evaluation to be performed as part of a check standard prior to running calibration standards and environmental samples.
3. Table 1. Adjust chloromethane and bromomethane reporting limits to 10 ppb for waters and 120 ppb for soils.
4. Include calibration by linear regression in Section 11.9.

Revision 3:

1. Adjust frequency for continuing calibration check standard from one per 15 analytical runs to one at the beginning of each analytical day.
2. Adjustment of reporting limits to reflect latest method detection limit study.

Revision 4:

1. Add reference to SIM mode analysis on the GC/MS in Sections 2.0, 3.0, and 9.4.2., and definition under Section 3.0.
2. Change EPA to USEPA were applicable.
3. Update typical GC Program parameters in Section 9.1
4. Update QC recovery limits for LCS/ICV from 70-130% to 70-130% and 70-160% for poor performers wherever applicable (Section 11.0) and in Attachment I.
5. Update SS/IS spike concentrations to be an allowed range.
6. Add specifics regarding standard preparation procedures in Section 7.0 referencing the SEI-4.7.5 SOP

and added Table 2 Example Standard Preparation Calculation Worksheet in Section 14.0.

7. Added two new paragraphs under Section 8.2 Sample Handling for pH determinations and percent moisture determination.

Revision 5:

1. Take out reference to an LCS in section 9.4.
2. Add clarification to the LCS Section 9.4.7 regarding the preparation and analysis of soils.
3. Change Section 11.0 to read Section 11.6 ICV, Section 11.7 LCS and 11.8 MS/MSD.

Revision 6:

1. Section 6.0: add SPME Autosampler: CTC Combi PAL.
2. Section 9: Add references for DOD exceptions and references
3. Section 11: Add reference to DOD projects and blank requirements.
4. Add Table 2. Reference to DOD project limits and allowances.

Revision 7:

1. Section 8: Add text requiring unique labeling of sample vials when more than one vial is presented for a sample.

Revision 8:

1. Changed document to reflect SW846 USEPA Method 8260C from 8260B
2. Included updates for DOD exceptions.
3. Minor grammatical and typographical errors fixed.
4. Updated extractions times to include 6 minutes for faster methods.
5. Updated columns use to RTX-VMS
6. Correlation Coefficient of 0.99 is now acceptable for calibrations.
7. Added Surrogate Recovery and Internal Standard Verification to Corrective Action Table
8. Added text for IC/LCS to CA Table.
9. Removed references to CCC's and SPCC's which are not used in method 8260C
10. Updated QA/QC Checklist added Table 2.

Attachment I

SAMPLE GC LABORATORY QA/QC CHECKLIST

GC/MS LABORATORY QA/QC CHECKLIST

Instrument:_____ QC Batch:_____

System:_____ VSTD FB Area:_____

Date:_____ VSTD CB(D5) Area:_____

QC Analysis	Criteria	Filename(s):
IC Calibration	%RSD $\leq$ 20%, correlation coefficient >0.99 (other)	
SPME Fiber Blank	< 1/2 reporting limit	
BFB Tune Check	per 8260 Method	
CC Calibration	%D ±20% VOCs, ±30% (other)	
Method Blank	< 1/2 reporting limit	
ICV Standard	See Table 2	
MS/MSD Analysis	See Table 2	
Surrogate Standards	70-130% waters 60-140% soils	

Note: DOD projects may require additional and/or different QC criteria then specified above, see pertinent sections of SOP for details.

Comments/Corrective Action:

Prepared By: _____

Reviewed By:_____

STANDARD OPERATING PROCEDURE

SEI-10.1.6

DETERMINATION OF AROMATIC AND CHLORINATED VOLATILE ORGANICS AND LIGHTWEIGHT PETROLEUM HYDROCARBONS (TYPICAL RANGE C4-C16) COMPOUNDS USING SOLID PHASE MICROEXTRACTION (SPME) AND A GAS CHROMATOGRAPH IN SOIL AND WATER SAMPLES (MODIFIED SW846 METHODS 8021/8015& ASTM D6520)

SOP Number: SEI-10.1.6
Revision Number: 6

Date Issued: 02/21/03
Date of Revision: 5/26/09

1.0 OBJECTIVE

This method is designed to measure the concentration of specified volatiles organic compounds (VOCs) and various classes of petroleum hydrocarbon compounds in water and soil samples using solid phase microextraction (SPME) and a Gas Chromatograph equipped with a capillary column. Specific VOCs of interest may include but are not limited to vinyl chloride, cis-1, 2-dichloroethene, trans-1, 2-dichloroethene, trichloroethene, tetrachloroethene, benzene, toluene, ethylbenzene, m & p xylene, and o-xylene (typically reported as total xylene). The petroleum hydrocarbon compounds of interest include: gasoline, kerosene, No. 2 Fuel Oil, and diesel fuel (specifically hydrocarbons between the ranges of C4-C16).

2.0 SUMMARY OF METHOD

This method provides gas chromatographic conditions for the detection of certain aromatic and chlorinated VOCs and petroleum hydrocarbons (typical range C4-C16). The Gas Chromatograph is equipped with a narrow bore capillary column and a Flame Ionization Detector (FID). Samples are prepared in 24 mL extraction vials. The SPME fiber is then exposed to the headspace above the sample for a set number of minutes while the sample is rapidly stirred. The exposure time must be consistent for all standards and samples (typically eight minutes). The absorbed analytes are thermally desorbed from the SPME fiber in the injection port of a Gas Chromatograph and transferred to the capillary column.

This method is suitable for analysis of water, soils, and wastes.

Soil samples are collected in approximately 10-12 gram cores preserved in methanol (10 mLs) in 40 mL VOA vials and water samples are collected in 40 mL VOA vials with no headspace.

This method is based on EPA SW-846 Methods 8000, 8021B and 8015 and ASTM Method D6520.

3.0 DEFINITIONS

SPME – Solid Phase Microextraction

VOC – Volatile Organic Compounds

QA/QC – Quality Assurance/Quality Control

Lightweight petroleum hydrocarbons- typically gasoline range organic petroleum hydrocarbons in the carbon range of C4-C16.

Laboratory Control Sample (LCS) - A reagent water or method blank matrix spiked with the analytes of interest. The spike recovery is used to evaluate method accuracy and control.

Instrument Blank – Involves only the SPME fiber. The fiber is exposed to laboratory room air (for a time equal to the exposure time for the samples and calibration standards) prior to analysis. The instrument blank is used to measure contamination associated with laboratory room air, the fiber, and the GC.

Method Blank - A reagent water or soil sample spiked with surrogates and subjected to the same preparation procedures as the associated samples. The method blank is used to measure contamination associated with that of the instrument blank and the reagents (dilution water and solvents that contain QC standards) used as part of the program. The surrogate recoveries of the method blank are used as matrix-free laboratory controls.

EPA - U.S. Environmental Protection Agency.

4.0 INTERFERENCES

When analyzing for VOCs, samples can be contaminated by diffusion of volatile organics (particularly chlorofluorocarbons and methylene chloride), through the sample container septum during shipment and storage. A trip blank prepared from organic-free reagent water and carried through sampling and subsequent storage and handling must serve as a check on such interferences.

Sulfur dioxide is a potential interference in the analysis for vinyl chloride.

Contamination by carryover can occur whenever high-concentration and low-concentration samples are analyzed in sequence. To reduce the potential for carryover, the sample syringe or purging device must be rinsed out between samples with an appropriate solvent. Whenever an unusually concentrated sample is encountered, it should be followed by injection of a method blank to check for cross contamination.

Purging vessels and syringes should be adequately cleaned and flushed prior to use. All glassware must be kept scrupulously cleaned. Clean all glassware as soon as possible after use by rinsing with the last solvent used or analyte-free water. Clean dry glassware shall be stored in a clean environment.

The flame ionization detector (FID) is a non-selective detector. There is a potential for many non-target compounds present in samples to interfere with this analysis.

5.0 SAFETY ISSUES

1. Laboratory personnel should take extra care when working with standards. When working with neat standards, make certain that there is adequate ventilation and that Nitrile or Latex gloves are worn and if necessary safety glasses.
2. If necessary and appropriate, a site-specific health and safety plan shall be created for each study site and followed.
3. Field visits may involve accessing remote areas. Health and safety concerns regarding these field visits are minimal, however, hazards such as slip, trip and falls, poisonous plant and dangerous animals, as well as getting vehicles stuck in remote areas, do present considerable health and safety issues. To help ensure field staff's health and safety in remote areas, all field staff are to have daily communication with the project manager or another appropriate SEI employee.
4. All chemicals are required to be received with a Material Safety Data Sheets (MSDS). MSDSs shall be made available to all personnel involved in the sampling and testing.

6.0 EQUIPMENT AND SUPPLIES

1. Gas Chromatograph: Analytical system complete with gas chromatograph and all required accessories, including a detector, column supplies, gases, syringes and data system to determine peak areas and perform integrations. Hewlett Packard 5890 Series II.
2. Analytical Column: RTX-VMS 20m x 0.18 mm ID, 1.1 um film.
3. Detector: Flame ionization detector (FID).
4. Array of Microsyringes: range 5-1000 uL.
5. Balance: Top loading, capable of weighing accurately to 0.01 grams (soil samples only).
6. Sample introduction and preparation apparatus: SPME Fiber type 100 µm polydimethylsiloxane (PDMS) and/or 85 µm Carboxen/PDMS fiber, magnetic hot plate and magnetic stirrers, tube clamp and stand.
7. VOA vials: 40 mL collection containers with Teflon®-lined septum and 24 mL extraction vials

7.0 REAGENTS AND STANDARDS

1. Reagents: Organic-free reagent water demonstrated to be free of compounds of interest (spring water, carbon filtered and/or deionized). Purge and Trap Grade or equivalent grade methanol (demonstrated to be free of analytes and stored away from other solvents).
2. Stock standards – Stock standards may either be prepared from pure standard materials or purchased as certified solutions. Secondary dilution standards are prepared accordingly using stock standard solutions, which contain the compounds of interest, either singly or mixed together. Typically, for this method the stock standards are obtained from an approved vendor mixed together. A certificate of analysis is retained by the laboratory and maintained on file or on file with the vendor. The aqueous standards are prepared at

concentrations as listed in Section 9.0 and prepared in accordance with Table 2 which is an Example of the Standard Preparation Calculation Sheet. Calibration standards at a minimum of five different concentrations for VOC analytes and at a minimum of three for petroleum hydrocarbon compounds are prepared in water.

Stone's SOP No. SEI-4.7.5 *Labeling, Preparation and Storage of Reagents, Solutions and Standards* provides procedures on properly labeling, preparing and storing of reagents and standards used in the mobile laboratory for analytical methods. This SOP should be review by the analyst before labeling, preparing and storing standards within the mobile laboratory.

8.0 SAMPLE COLLECTION AND HANDLING

8.1 Sample Collection

1. Aqueous samples are collected in 40 mL Teflon®-lined septum VOA vials filled to ensure that no headspace is present. If storage of these samples is expected to exceed seven days, aqueous samples should be preserved with hydrochloric acid (HCL) to a pH of < 2.

2. Soil samples are collected in approximately 10-12 gram cores, and placed in 40 mL vials. A soil aliquot of approximately 10-12 grams of sample is placed in a VOA vial with 10 mL of methanol. If available, a separate aliquot of soil from the sample location will be collected in a 40 mL vial (with no preservative) for percent moisture determination.

8.2 Handling and Holding Times

1. Unpreserved water samples must be analyzed within 7 days of collection. Preserved (preserved with HCl to a pH of < 2) water samples must be analyzed within 14 days of collection. Soil samples collected in methanol must be analyzed within 14 days from collection. All samples shall be stored at 4 ° C ± 2 ° C. Samples are allowed to come to room temperature just prior to analysis. All samples for this program are typically analyzed within one or two days from collection, therefore, holding times should not be a concern.

2. If samples were preserved and analyses are performed after 7 days from collection, pH determination (water samples only) should be performed. Once the sample aliquots have been taken from the VOA vial for analysis, the pH of the water sample must be determined. The purpose of the pH determination is to ensure that all VOA samples were acidified in the field. Test the pH by placing one or two drops of sample on the pH paper (do not add pH paper to the vial). Record the pH of each sample, and report these data in the SDG Narrative, following the instructions. No pH adjustment is to be performed.

3. If necessary, a separate soil sample aliquot will be collected in a 40 mL vial or equivalent for percent moisture content so that results can be adjusted for %moisture and reported as dry weight rather than as received. Percent moisture is determined as follows: Percent Moisture Determination - Immediately after weighing the sample for analysis, weigh 5-10 g of the soil/sediment into a tared crucible. Determine the percent moisture by drying overnight at 105 °C. If available, allow to cool in a dessicator before weighing. Concentrations of individual analytes will be reported relative to the dry weight of soil/sediment.

$$\text{Percent Moisture} = \frac{\text{Mass of wet sample (g)} - \text{Mass of dry sample (g)}}{\text{Mass of wet sample (g)}}$$

9.0 PROCEDURES

9.1 Typical GC Program

Oven Temp: 50°C (4.00 min) to 160°C at 15°C/min to 200°C at 40°C/min, hold for 1 min.

Carrier Gas: Helium, 1.0 ml/min (set at 50°C)

Detector: FID

Injector: 240°C, splitless (close 3 min)

Liner: 0.75 mm ID splitless/split injection port

Flow rates: Helium 1.0 ml/min

Air 450 ml/min

Hydrogen 33 ml/min

Helium makeup 30 ml/min

9.2 GC Column

Analytical Column: RTX-VMS 20m x 0.18 mm ID, 1.1 um film

9.3 Material Preparation:

SPME: Fiber Type: 100 µm polydimethylsiloxane (PDMS).

SPME Fiber Type for low level waters only: 85 µm Carboxen/PDMS

SPME Extraction: The SPME-absorbent polymer fiber is exposed to the headspace above a water sample (24 mLs sample preparation vial, sample and ¼ tsp, salt; headspace created by removing approximately 2 mLs from the 24 mL preparation vial) for 8 minutes while the sample is rapidly stirred on a magnetic stirring plate. The fiber is retracted into the holder, and transferred to the GC injection port. Each magnetic stirrer is rinsed three times with volatile free water prior to use. For soil

samples; an aliquot of the methanol extract (1mL but not more than 2 mL) is prepared in the 24 mL water preparation vial, and prepared and analyzed in the same manner as a water sample above. For air samples, the SPME fiber is exposed directly to the same through the Teflon-line septum located on the Tedlar bag. Exposure time of the fiber to the air sample will be the same for all samples and calibration standards. Prior to use, each fiber must be conditioned for at least one hour in the GC inlet at 240°C.

9.4 Reference Standard Preparation

Calibration Standards: Labeling - SOP SEI 4.7.5

9.5 Operating Procedure

9.5.1 Calibration Criteria

9.5.1.1 Initial Calibration (ICAL):

1. Five-point calibration for VOC compounds (10, 40, 100, 200, 500 ppb). The low standard must be set at the practical quantitation limit (PQL). Low level standards (1,2,5,10, 20 ppb)
2. Three-point calibration for petroleum hydrocarbon standards (200, 500, 2,000 ppb of Number 2 Fuel Oil). If appropriate and necessary based on the observed chromatographic pattern of the petroleum hydrocarbons detected in site samples, a three-point calibration for Diesel and No. 6 Fuel Oil (200, 500, 2000 ppb) will be prepared. It should be noted that this headspace methodology will only detect the lightweight petroleum hydrocarbons associated with these fuel oils.
3. Calibration range must encompass the expected sample concentration; therefore, if site samples are expected to be higher in concentration than the calibration range, adjustments to the calibration range and samples will be documented and made accordingly.
4. The linearity of the calibration curves must be assessed and are used for all quantitation. Volatile organic compounds use an internal standard procedure (internal standard; fluorobenzene). Linear regression is used for quantitation and the correlation coefficient (r) of each compound must be greater than 0.995 ($r^2 = 0.990$). Depending upon the on-site program acceptable correlation coefficient may be set at 0.99 in accordance with SW846 8000.
5. For the petroleum hydrocarbon compounds external standard calibration will be performed using a specific window (specifically all peaks encompassing approximately (C4-C16) at approximately 3.0 to 9.0 minutes. The correlation coefficient of the calibration must be greater than 0.99 (> 0.99).

9.5.1.2 Continuing Calibration (CCV):

1. A mid-point continuing calibration verification standard for VOC compounds at 100 ppb (low level: 5 ppb) must be analyzed daily: prior to sample analysis in order to verify the initial calibration; after every 15 analytical runs which include environmental and QC samples; and at the close of an analytical sequence. A percent difference between the expected concentration and actual concentration is calculated. The percent difference (%D) values for each compound must be less than or equal to 20%.

$$\%D = \frac{\text{Expected Conc. - Actual Conc.}}{\text{Expected Conc.}}$$

2. A mid-point CCV standard for petroleum hydrocarbons (500 ppb for diesel or No. 2) must be analyzed daily; prior to sample analysis in order to verify the initial calibration; and if necessary, at the close of an analytical sequence. A percent difference of the daily concentration gasoline range petroleum hydrocarbons found compared to the nominal concentration is calculated. The percent difference values for petroleum hydrocarbons must be less than or equal to ± 30%.

$$\%D = \frac{\text{Expected Conc. - Actual Conc.}}{\text{Expected Conc.}}$$

9.5.1.3 Initial Calibration Verification Standard (ICV):

A QC sample from a second (independent) source will be analyzed directly after the ICAL. The ICV sample will contain all the VOC compounds of interest at the mid-point (40-100 ppb or 5 ppb for low level waters). Recovery Limits for the ICV will be 70-130%, if compounds are determined to be poor performers then the recoveries shall be 60-140% for the ICV.

$$\%\ \text{Recovery} = \frac{\text{Actual Concentration}}{\text{Expected Concentration}}$$

9.5.2 Retention Time Windows and Pattern Recognition

The laboratory shall identify single component target analytes on the basis of retention time windows (± 0.04 minutes from the ICAL or daily CCV). Petroleum hydrocarbons are distinguished on the basis of the ranges of retention times for characteristic components in each type of fuel. During the initial calibration, the retention time window of these components is established and documented. Once the range and retention time window is established, they will be used for the identification of the compounds of interest.

9.5.3 Sample Preparation:

Water samples: An aliquot of sample, sufficient to get the highest concentration constituents into the upper half of the linear range, is transferred to a 24 mL extraction vial and spiked with the surrogate (bromofluorobenzene at 41.67 ppb) and internal standard (fluorobenzene at 41.67 ppb). The SPME fiber is then exposed to the headspace above the sample for eight minutes while the sample is rapidly stirred with a magnetic stirrer on a magnetic plate. The eight minutes is measured using a bench-top timer. Extraction time may be modified depending on the anticipated equilibration response of the target analytes at given concentrations.

Soil samples: Preserved in methanol; 10-12 g core of soil is added to 10 mL of methanol containing an appropriate amount of bromofluorobenzene. After addition of the site soil and prior to analysis, the vial is mixed for five minutes. An aliquot of methanol, sufficient to get the highest concentration constituents into the upper half of the linear range but not to exceed 2 mL, is diluted to volume with analyte free water and ¼ tsp. salt in a 24 mL vial, and analyzed using SPME in the same manner as a water sample.

9.5.4 Analysis of Samples:

Analysis of samples is performed using SPME followed by analysis on a GC equipped with a narrow bore capillary column and a FID. For this program the fiber is exposed to the headspace above the sample. Therefore a small portion is discarded from the 24 ml vial prior to exposure. The exposure time needs to be consistent (8 minutes for this method). To facilitate the release of the analytes from their respective media, the sample is rapidly stirred throughout the extraction. The adsorbed analytes are thermally desorbed in the injection port of the GC and transferred to the capillary column.

9.5.5 Surrogate (SS) and Internal Standard (ISTD) Compounds for VOC compounds

Compound	**Amount Spiked (Low level, Carboxen)**
Bromofluorobenzene (SS)	41.67 ppb (12.48 ppb)
Fluorobenzene (ISTD)	41.67 ppb (4.16 ppb)

9.5.6 Surrogate Spike Compound for Petroleum Hydrocarbons Compounds

Compound	**Amount Spiked**
Fluorobenzene	41.67 ppb (4.16 ppb)

9.5.7 ICV Preparation

Compounds and spike amounts are below (may vary depending on project specific needs). Water samples will be spiked at 40-100 ppb; the amount may change to account for the concentration of a particular analyte found in the sample being analyzed.

9.5.8 LCS Preparation

For a waters program, compounds and spike amounts are specified below or vary depending upon project specific needs. Waters will be spiked at around the mid-point of the calibration.

For the soils program, the soil LCS will be prepared in the same manner as the soil samples, an aliquot (1 mL) of methanol will be prepared in the SPME preparation vial spiked with VOCs of interest, and prepared in the same manner as the soil MS/MSD.

9.5.9 Matrix Spike and Matrix Spike Duplicate (MS/MSD) Compounds:

Compounds and spike amounts are below. Water samples will be spiked at the mid-point of the calibration; the amount may change to account for the concentration of a particular analyte found in the sample being analyzed as a MS. For soil samples, an appropriate amount of the target analytes is spiked into the 10 mL of methanol and grams of site soil when it comes back from the field. Samplers must collect additional sample volume for MS/MSD analyses Matrix Spike Analyses will contain only the VOC compounds of interest.

9.5.10 Matrix Spike and Matrix Spike Duplicate (MS/MSD) and LCS Compounds:

Analyte	CAS #	MS/MSD/ LCS Spiked Amounts
Vinyl Chloride	75-01-4	40-100
1,1-Dichloroethene	75-35-4	40-100
trans-1,2-Dichloroethene	156-60-5	40-100
1,1-Dichloroethane	75-34-3	40-100
cis-1,2-Dichloroethene	156-59-2	40-100
1,1,1-Trichloroethane	71-55-6	40-100
Benzene	71-43-2	40-100
1,2-Dichloroethane	107-06-2	40-100
Trichloroethene	79-01-6	40-100
Toluene	108-88-3	40-100
Tetrachloroethene	127-18-4	40-100
Chlorobenzene	108-90-7	40-100
Ethylbenzene	100-41-4	40-100
M+p-Xylenes	108-38-3 106-42-3	40-100
o-Xylene	95-47-6	40-100
1,3-Dichlorobenzene	541-73-1	40-100

Analyte	CAS #	MS/MSD/ LCS Spiked Amounts
1,4-Dichlorobenzene	106-46-7	40-100
1,2-Dichlorobenzene	95-50-1	40-100
**Total Petroleum Hydrocarbons as Fuel Oil No. 2		500-2000

Note: For water samples the MS will be spiked at 40-100 ppb or at 5 ppb depending upon the program; the amount may change to account for the concentration of a particular analyte found in the sample being analyzed as a MS. For soil samples, an appropriate amount of the target analytes is spiked into the 10 mL of methanol when the sample comes back from the field. Samplers will collect additional samples for MS/MSD analyses.

9.5.11 Target Compound List Compounds

Analyte	CAS #	PQL Water (ug/L)	PQL Soil (ug/Kg)	Approximate MDLs (water)	Approximate MDLs (soil)
Vinyl Chloride	75-01-4	20	480	5.4	330
1,1-Dichloroethene	75-35-4	10	240	3.6	205
trans-1,2-Dichloroethene	156-60-5	10	240	3.4	167
1,1-Dichloroethane	75-34-3	10	240	1.8	72
cis-1,2-Dichloroethene	156-59-2	10	240	4.0	62
1,1,1-Trichloroethane	71-55-6	10	240	4.8	63
Benzene	71-43-2	10	240	0.86	199
1,2-Dichloroethane	107-06-2	10	240	6.0	199
Trichloroethene	79-01-6	10	240	1.6	160
Toluene	108-88-3	10	240	1.6	142
Tetrachloroethene	127-18-4	10	240	4.0	207
Chlorobenzene	108-90-7	10	240	1.3	61
Ethylbenzene	100-41-4	10	240	3.3	223
M+p-Xylenes	108-38-3 106-42-3	10	240	3.4	187
o-Xylene	95-47-6	10	240	3.4	187
1,3-Dichlorobenzene	541-73-1	10	240	3.0	195
1,4-Dichlorobenzene	106-46-7	10	240	2.8	185
1,2-Dichlorobenzene	95-50-1	10	240	3.11	165
**Total Petroleum Hydrocarbons as Fuel Oil No. 2		500	2400	n/a	n/a
Other compounds not listed here may be analyzed for as verified by a successful method verification study.					

* Low Level PQLs can be achieved for all compounds at 1 ppb using the Carboxen Fiber.

**Note: If upon visual inspection of the chromatogram the petroleum hydrocarbon compounds are suspected to be that of No. 6 Fuel Oil or heavier weight hydrocarbons, the PQL will be higher; approximately 200 ug/L for waters and 2400 ug/kg for soils. PQLs are sample dependent and may change based on sample dilutions or sample amounts used during the analysis; adjustments to the PQLs will be made accordingly.

10.0 CALCULATIONS

Identification of the project- specific VOCs in the sample chromatogram is achieved by comparing the retention times generated by the calibration standards, continuing calibration standard and other fortified QC samples. Retention times must be within ± 0.04 minutes from the ICAL. If a retention time shift is observed in the CCV or daily ICV, then adjustments to the daily retention time windows will be made accordingly. Quantification of the VOCs is determined by comparison to the internal standard and linear regression for that analyte from the initial calibration curve.

Quantification of the lightweight petroleum hydrocarbon compounds is determined by summing the peak areas found in the chromatographic window of the sample and using a linear regression of the calibration curve. The SPME gas chromatograph (GC) headspace method will detect only those petroleum hydrocarbon compounds that are lightweight, i.e., those that are typically found in gasoline, kerosene, a portion of No. 2 Fuel Oil and diesel. Therefore, the petroleum hydrocarbon results will be used on a semi-quantitative basis.

The concentration of each analyte in the sample may be determined by calculating the amount of standard injected, from the peak response, using the calibration curve as noted above.

For the analysis of the petroleum hydrocarbons, sum the area of all peaks eluting between approximately 3.0-9.0minutes (set range should be used for both standards and samples). This area is used to calculate petroleum hydrocarbons.

Range concentration = Dilution x (Total FID Area – Surrogate Area).

Use the HP Chemstation software to calculate sample hydrocarbon range concentrations for water in ug/L and soil in ug/Kg.

11.0 QUALITY CONTROL AND CORRECTIVE ACTION

11.1 Instrument Blank (SPME Fiber Blank)

To be performed once daily at the start of the analytical day. All target analyte concentrations detected in the instrument blank must be less than one-half the PQL value prior to continuing.

11.2 Method Blank

One per analytical batch (15 samples) directly after the calibration standard and after highly contaminated samples to demonstrate that there is no carry over into subsequent samples. All target analyte concentrations detected in the method blank must be less than one-half the PQL value prior to continuing.

11.3 Surrogate Standards

Surrogate recoveries should be 70-130% for water samples and 60-140% for soil samples. Samples with surrogate recoveries falling outside of quality control limits should be reanalyzed once. If the reanalysis of the sample is also outside the quality control limits, then it will be determined that the problem may be associated with the sample matrix and noted as such.

11.4 Initial Calibration Verification Standard (ICV)

A QC sample from a second (independent) source than that of the initial calibration will be analyzed directly after the ICAL or CCV. The ICV sample will contain all the VOC compounds of interest at mid point of the calibration curve. Recovery Limits for the ICV will be 70-130%. This sample may also be used as the laboratory control sample (LCS). Daily LCS range 70-130%.

11.5 Laboratory Control Sample (LCS)

For a waters program: a QC sample from a second (independent) source than that of the initial calibration will be analyzed directly after the ICAL or CCV. The LCS sample will contain all the VOC compounds of interest at the mid-point of the calibration range. Recovery Limits for the LCS will be 70-130%.

For the soils program, the soil LCS will be prepared in the same manner as the soil samples, an aliquot (1 mL) of methanol will be prepared in the SPME preparation vial spiked with VOCs of interest at the midpoint of the calibration, and prepared in the same manner as the soil MS/MSD. Recovery Limits for the LCS will be 70-130%.

11.6 Matrix Spike and Matrix Spike Duplicate (MS/MSD) Analysis

An MS/MSD analysis is required every set of twenty samples or as specified by the client. A sample will be chosen by client to perform a matrix spike analysis. The control limits for the matrix spike recoveries will be derived empirically using the most recently acquired group of data. If no data is available, advisory recovery limits (set forth by the USEPA, SW 846 Methodologies) of 70-130% will be used.

$$\% \text{ Recovery} = \frac{\text{Sample Spiked Concentration} - \text{Un-Spiked Sample Concentration}}{\text{Spike Concentration}}$$

11.7 Lab Duplicate

If necessary, a random sample is run in duplicate and the results are compared. The relative percent difference (RPD) should not exceed 30% for waters and 50% for soils.

11.8 CORRECTIVE ACTION

Quality Control Check	*Minimum Frequency*	*Acceptance Criteria*	*Corrective Action*
Initial Calibration (ICAL)	As necessary.	Minimum of five points for VOCs. Correlation coefficient must be > 0.995. (some programs >0.99) Minimum of three points for petroleum hydrocarbon. Low standard must not exceed reporting limit. Petroleum hydrocarbon > 0.99.	Verify solution integrity and check instrument performance. Perform necessary maintenance and recalibrate instrument. Reanalyze all affected samples.
Initial Calibration Verification (ICV) / Laboratory Control Sample (LCS)	One per calibration (following acceptable ICAL) or after CCV or in place of a CCV in the waters program.	QC limits are 70 to 130% for target compounds. Note: Limits may be adjusted based on the program should as required by DOD QMS	Reanalyze once; if still out verify solution integrity or ICAL solution integrity, and instrument performance. Re-prepare and reanalyze all associated samples.
Retention Time (RT) Window Study	Every new column installation	All target compounds and surrogates in all standards must fall within the established window of ± 0.04 minutes from ICAL	Perform system maintenance. Reanalyze affected samples.
Continuing Calibration Verification (CCV)	One after each 15 samples window and must be followed by a method blank if more samples are to be analyzed	See Section 9.0. ≤ ± 20% for VOCs ≤ ±30 % for petroleum hydrocarbons	Verify solution integrity and instrument performance. Reanalyze standard once, if still out, recalibrate and reanalyze affected samples.
Method Blanks	One per preparation batch	Target Compounds < one-half reporting limit	Investigate source of contamination. Re-prepare and reanalyze all associated samples.
Matrix Spike (MS) and Matrix Spike Duplicate (MSD)	One set per 20 samples of a similar matrix	QC limits are 70-130%. RPD not to exceed 30% for waters and 50% for soil samples.	Reanalyze once; if still out, verify solution integrity and instrument performance. If necessary analyze a LCS or ICV and if acceptable, narrate as possible matrix effect.

11.9 Data Review and Data Integrity

The analyst is responsible for primary data review of data generated from the sample analysis. Analyses will be documented in the instrument run log. Maintenance is documented in the Instrument Maintenance Logbook. Instrument calibrations and recoveries of all QC samples must be within specified control limits. If instrument calibration or the recoveries of any QC sample exceed specified tolerances, then the affected sample results are evaluated and generally the samples are submitted for re-analysis. To determine if analytical results are acceptable, a qualified and independent QA/QC program shall conduct a secondary review on a weekly basis. A QA/QC Checklist (Attachment 1) is used to indicate any problems with the referenced analytical batches. The QA/QC checklist will also address any corrective action taken. All calibrations, calculations, and transcriptions are checked for accuracy and QC sample results are evaluated against specified limits. If instrument calibration and recoveries of all QC samples are within the specified criteria, then the data reports will be submitted to the Project Manager as final results with no qualifiers. If recoveries of any QC samples exceed specified limits and re-analysis is not an option, then the samples will be qualified as estimated with a "J" qualifier (J= The analyte was positively identified; the associated numerical value is the approximate concentration of the analyte in the sample.). Data will not be reported if significant QC issues affect the batch analyses.

12.0 POLLUTION PREVENTION & WASTE MANAGEMENT

Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. The United States Environmental Protection Agency (USEPA) has established a prevention hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the agency recommends recycling as the next best option.

The quantity of chemical purchased should be based on expected usage during its shelf life and disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.

For information about pollution prevention that may be applicable to laboratories and research institutions, consult "Less is Better: Laboratory Chemical Management for Waste Reduction", available from the American Chemical Society's Department of Government Regulations and Science Policy, 1155 16th Street N.W., Washington, D.C. 20036; (202) 872-4477.

The USEPA requires that laboratory waste management practices conducted be consistent with all applicable rules and regulations. Excess reagents, samples, and method process wastes should be characterized and disposed of in an acceptable manner. The Agency urges laboratories to protect the air, water and land by minimizing and controlling all releases from hoods and bench operations, complying with the letter and spirit of any waste regulations, particularly the hazardous waste

identification rules and land disposal restrictions. For further information on waste management consult the "Waste Management Manual for Laboratory Personnel", available from the American Chemical Society.

13.0 REFERENCES

Pawliszyn, Janusz, 1999, Applications of Solid Phase Microextraction, *in* RSC Chromatography Monographs; Smith, Roger S., Series Editor, Royal Society of Chemistry, Cambridge, UK, 655 p.

SW846. ASTM D6520.

"Less is Better: Laboratory Chemical Management for Waste Reduction", available from the American Chemical Society's Department of Government Regulations and Science Policy, 1155 16th Street N.W., Washington, D.C. 20036; (202) 872-4477.

14.0 TABLES, DIAGRAMS, FLOWCHARTS, AND VALIDATION DATA

Attachment 1: Sample GC Laboratory QA/QC Checklist

15.0 AUTHORIZATION

Authored by: _____ Date: 5/26/09

David Crosby, Project Geoscientist

Approved by: _____ Date: 5-26-09

Christopher T. Stone, President

16.0 REVISION HISTORY

Revision Number 1

1. Minor wording edits

Revision Number 2

Unknown

Revision Number 3

1. Added Section 12.0 Pollution Prevention
2. Added Section 16.0 Revision History

Revision Number 4

1. Added Section 9.4 Reference Standard Preparation
2. Clarification and verbiage on the Laboratory Control Sample.
3. Corrections to Table 1 under PQLs and concentration of MS/MSD spike.
4. Added two new paragraphs under Section 8.2 Sample Handling for pH determinations and percent moisture determination.

Revision Number 5

1. Redefine and decouple the Laboratory Control Sample from the ICV for a soil sample program. Add Sections: 9.5.8.
2. Change Section 11.0 to read Section 11.4 ICV, Section 11.5 LCS and 11.6 MS/MSD.

Revision Number 6

1. Update Sections 9.5.10 and 9.5.11 to include PQLs and MDLs for both soil and waters for all compounds accredited for under the NELAC program.
2. Removed methanol from the 4th sentence since the magnetic stirrers are just rinsed with VOA free water.

Attachment I

SAMPLE GC LABORATORY QA/QC CHECKLIST

GC LABORATORY QA/QC CHECKLIST

Date:_____

QC Analysis	Criteria	Filename(s):
IC Calibration:	%RSD ± 30% VOCs, >0.99 (other)	
CC Calibration:	%D ± 20 % VOCs, ± 30% (Other)	
SPME Fiber Blank:	< 1/3 reporting limit	
Method Blank:	< 1/2 reporting limit	
ICV Standard:	70-130%	
ICV/LCS:	70-130%	
MS/MSD Analysis:	70-130%	
Surrogate Standards:	70-130% waters, 60-140% for soil samples	
Other (describe):		

Comments/Corrective Action:

Prepared By: _____

Reviewed By:_____

NA = None Available

I:\GLP_NELAC\SOP\SEI_SOPs\Ch10_EnvDrill\SEI_Current\SEI-10.1.6.doc

Attachment 2

EXAMPLE STANDARD PREPARATION CALCULATION SHEET

STONE ENVIRONMENTAL INC

Gas Chromatography Laboratory

HP-5

Standard Prep Log

08/16/05-a1					dmkc
FID WS					
100	ppm				
x: 8/31/05					
Compound	Vendor	Lot	Exp	Conc (mg/ml)	Amount
Custom VOC	S	LB18097	8/16/2006	20	20 ul
1,1-DCA	A	B2090306	6/22/2006	20	20 ul
1,1,1-TCA	A	B3050086	6/22/2006	10	40 ul
8020 Mix	A	B1070292	8/31/2005	2	200 ul
1,1-DCE	A	B4050042	1/21/2006	20	20 ul
BFB	A	B4110093	5/17/2006	25	16 ul
MeOH	OmniSolv	44090	3/1/2008	into	3684 ul
				Total	4000 ul

SOP #BR-0060

Determination of Trace Elements by Inductively Coupled Plasma – Mass Spectrometry using a Perkin-Elmer ELAN DRC II

Brooks Rand Labs

Written 3/24/2005
Revised 05/24/10
Revision 003c

VP of Analytical Services	5/24/10 Date
QA Manager	5/24/10 Date
ICP-MS Analyst	5/24/10 Date

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Determination of Trace Elements by Inductively Coupled Plasma – Mass Spectrometry using a Perkin-Elmer ELAN DRC II

1.0 DESCRIPTION

1.1 This procedure describes a mode of analysis for the determination of dissolved and total recoverable trace elements in waters using inductively coupled plasma – mass spectrometry (ICP-MS). This procedure is intended for samples with total dissolved solids (TDS) of less than 1000 mg/L. Sample dilution may be required in order to achieve an optimum TDS level. The calibration range for the instrument is dependant on the analyte of interest.

1.2 This procedure is written specifically for use with a Perkin-Elmer ELAN DRC II ICP-MS. The instrument software is ELAN Version 3.4 for use on Windows XP®.

1.3 This procedure can be utilized to determine a large list of analytes. However, at this time the method has been fully validated only for Ag, Al, As, B, Ba, Be, Bi, Ca, Cd, Co, Cr, Cs, Cu, Fe, K, Li, Mg, Mn, Mo, Na, Ni, Pb, Pt, Sb, Se, Sn, Sr, Te, Th, Ti, Tl, U, V, W, and Zn. In addition, the following analytes have validated methods developed in DRC Mode: Ag, Al, As, Cd, Co, Cr, Cu, Fe, Ni, Pb, Sb, Se, V, and Zn. Following this method will aid in the method development and validation for the analysis of other analytes.

1.4 Matrices other than waters may be analyzed using this procedure. Sample preparations techniques can be utilized for the analysis of seawaters, municipal and industrial influents and effluents, human biomonitoring samples (such as urine, blood, and plasma/serum), and dilute digestates of sediments, soils, rocks, biota/tissues, or petroleum products.

1.5 This procedure is a modification of Draft EPA Method 1638. The EPA method validation study (reference 11.5) reported that the "EPA intends to revise Method 1638 to recommend use of response factor or weighted linear regression calibrations, allowing unweighted linear regress as an acceptable calibration technique." As the lowest standards are more of an influence in weighted linear regressions than "fit-through-zero" calibrations, Brooks Rand intends to primarily use weighted linear regression calibrations. For samples that are measured at or near the highest standards, a "fit-through-zero" approach may be appropriate.

1.6 This procedure must be followed to assure accuracy and reproducibility of the samples results generated by all of the operating chemists. An analyst must first be fully trained in the use of ICP-MS, including the interpretation of spectral and matrix interferences and procedures for their correction, as well as ultra-clean sample handling.

2.0 SUMMARY OF METHOD

All Brooks Rand Labs (BRL) SOPs are Proprietary Information and protected by WA state law. Proprietary Information shall be kept in the strictest confidence & shall not be used or appropriated to benefit any party without prior written consent from BRL.

2.1 An aliquot of homogenous sample is prepared for analysis. The determination of dissolved elements requires the sample be filtered through a 0.45-µm pre-cleaned filter unit as in BR-0104, and then preserved with acid. The determination of total and total recoverable elements in water samples requires the samples be digested with a combination of heat and acid, or the metals must be extracted from the sample matrix. Solid samples require matrix specific digestion steps before introduction into the instrument. All sample preparation procedures are detailed in depth in separate Brooks Rand Labs SOPs.

2.2 A prepared, aqueous sample is pumped through a nebulizer, where the liquid forms aerosol droplets as it enters a spray chamber. The aerosol separates into a fine aerosol mist and larger aerosol droplets. The larger droplets exit out the bottom of the spray chamber and are pumped off as waste. The finer droplets are transported out of the spray chamber and enter the ICP torch through an injector tube.

2.3 Inside the ICP torch, the aerosol droplets are transported into a high-temperature plasma, where they become atomized and ionized as they are introduced through a radio frequency (RF) load coil. The ions are transported into the plasma through a differentially-pumped interface. The ion stream is then focused by a single ion lens, which focuses the ions through a cylinder with a carefully controlled electrical field. The focused ion stream is directed into the dynamic reaction cell (DRC) where, when operating in DRC mode with a pressurized cell, the ion beam will undergo chemical modifications to reduce elemental interferences. When not operating in DRC mode the ion stream will remain focused as it passes through the cell with no chemical modification taking place.

2.4 The ion stream is then transported to the quadrupole mass filter, where only ions having a specific mass-to-charge ratio (m/z) are passed through at any moment in time. The ions exiting the mass filter are detected by a solid-state detector and processed by the data handling system.

3.0 DEFINITIONS

3.1 A partial list of definitions is presented below. Most definitions can be found for specific terms in the sections where they are first mentioned. Many of the definitions mentioned in this method are based on those found in the glossaries of EPA Methods 1638 and 1640. Please refer directly to these methods for a more detailed list.

3.2 Ambient Water: Waters from the natural environment, such as river or lake water, as opposed to effluents.

3.3 Analyte: A metal tested for by this method.

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3.4 Dissolved Analyte: The concentration of analyte in an aqueous samples that will pass through a 0.45 µm filter membrane prior to the sample being acidified.

3.5 Internal Standard (IS): Pure analyte(s) added to each sample (client and QC) at the instrument in known amounts and used to measure the relative response of other method analytes that are components of the samples. The IS must be an analyte that is not a sample component.

3.6 Linear Dynamic Range: The concentration range over which the instrument response to an analyte is linear.

3.7 m/z: mass-to-charge ratio

3.8 May: This action is optional.

3.9 May Not: This action is prohibited.

3.10 Must: This action is required.

3.11 Should: This action is suggested, but not required.

4.0 EQUIPMENT

4.1 Routine preventative maintenance for the equipment used in this procedure is described in detail in the standard operating procedure BR-1205 (Preventative Maintenance).

4.2 Facility: An ultra-clean laboratory environment is critical for the successful production of quality data at ultra-low levels. All sample preparation must take place in a clean hood (Class-100). Metallic pieces should be kept to a minimum in the lab and coated with an acrylic polymer gel. Adhesive floor mats should be used at entrances to the lab and changed regularly to prevent the tracking of dust and dirt from the outside environment. Clean-room gloves must be worn and changed whenever contact is made with anything non-ultra-clean. The lab floor should be wiped regularly to remove any particles without stirring up dust.

4.3 Instrumentation: ICP-MS (Perkin-Elmer ELAN DRC II) with a free-running 40 MHz RF generator; controllers for nebulizer, plasma, auxiliary, DRC gas A, and DRC gas B flow control; and a getter is equipped for work with ammonia gas. The quadropole mass spectrometer has a mass range of 5 to 270 atomic mass units (amu). The turbo molecular vacuum system achieves 10^{-6} torr or better.

4.4 ESI SC-4 auto-sampler.

4.5 ESI FAST high-flow valve.

4.6 An RF coil designed for axial torch arrangement.

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4.7 Platinum skimmer and sampler cones.

4.8 A Ryton Scott-type spray chamber for analysis with HF and an ESI PC^3 peltier cooled quartz cyclonic spray chamber for all other analysis.

4.9 Alumina injector with 2 mm inner diameter (ID) for HF analysis and a quartz or sapphire injector with 2 mm ID for other matrices.

4.10 ESI PFA ST nebulizer.

4.11 Polyvinyl chloride (PVC) tubing (¼" OD, ⅛" ID) for nebulizer gas and for waste.

4.12 Peristaltic pump tubing: Waste; Pharmed, green-green, 1.855-mm ID, and sample; PVC black-black, 0.76-mm ID, internal standard; PVC orange-green 0.38mm ID.

4.13 Carbon fiber support autosampler probe (0.8mm i.d-ESI N0777285 REV.A) with lines to connect to SC-FAST switching valve.

4.14 Sample loop of desired volume for analysis, typically 1.25mL for a full suite of metals.

4.15 Connection from SC-FAST switching valve to nebulizer with ST fitting.

4.16 Connector for sample and internal standard line.

4.17 Waste unit with tubing holes.

4.18 Gases: high-purity grade liquid argon (> 99.996%), ultra-x grade, 99.9999% minimum purity oxygen, and electronic grade, 99.99% minimum purity ammonia. The gases are stored in a ventilated gas storage cabinet.

4.19 Variable-speed peristaltic pump for solution delivery to the nebulizer.

4.20 Analytical Balance: a standard laboratory balance suitable for sample preparation and capable of measuring to 0.1-mg.

4.21 Clean-room gloves (tested low in trace-metals).

4.22 Sample bottles constructed of fluoropolymer (FEP or PTFE), polyethylene (HDPE or LDPE), polycarbonate (PC), polypropylene (PP), or glass. HDPE bottles are preferred. All bottles must be pre-cleaned by SOP BR-0400.

4.23 Auto-sampler cups (15-mL and 50-mL) pre-cleaned by soaking in 2% nitric acid for at least 24 hours, rinsed three times with DIW, and dried in a clean hood.

4.24 Volumetric flasks, class-A, cleaned by SOP BR-0400.

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4.25 Assorted calibrated pipettes for volumes from 0.005-10 mL. Pipettes are calibrated according to SOP BR-0400. Should include the assorted pipette tips for 2 - 10 mL, 1 - 5 mL, 0.1 - 1 mL and 0.005 - 0.200 mL pipettes.

4.26 Reagent bottles, 10-mL to 2000-mL, must be from any of the plastics listed in 2.20 and cleaned by SOP BR-0400.

4.27 Wash bottles, squeeze type, 500-mL, cleaned by SOP BR-0400.

4.28 Filter units, 150-mL and 500-mL, pre-cleaned with 4% nitric acid in DIW.

4.29 Vacuum pumps for sample filtration.

4.30 Silicon vacuum tubing with 8-mm ID and 14-mm OD.

4.31 pH meter.

4.32 Zip-type bags of various sizes.

4.33 Kimwipes.

4.34 A sonicator to clean cones and RF coils.

5.0 CONTAMINATION and INTERFERENCES

5.1 Contamination

5.1.1 Contamination of the samples during sample collection is a great risk, and extreme care should be taken to avoid this. Potential sources of contamination during sampling include using metallic or metal-containing labware, containers, and sampling equipment.

5.1.2 Sample collection equipment such as tubing, pumps, filters, and homogenization equipment should be non-metallic (whenever possible) and tested for metals through the analysis of equipment blanks.

5.1.3 Contamination of samples by airborne particulate matter is a concern. Sample containers should remain closed as much as possible. Container lids should only be removed briefly and in a clean environment during sample preservation and processing, so that exposure to an uncontrolled environment is minimized.

5.1.4 Laboratory

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5.1.4.1 All labware (including pipette tips, ICP-MS autosampler vials, sample and digestion bottles, extraction apparatus, and reagent bottles) should be tested for the presence of trace-metals. If necessary, the labware should be acid-cleaned, rinsed with DIW, and dried in a clean hood.

5.1.4.2 All auto-sampler vials are cleaned by storing them in 2% (v/v) HNO_3 for at least 24 hours and then rinsed three times with DIW. The vials are then dried in a clean hood before use. Glass volumetric flasks should be soaked in ~5% HNO_3 overnight prior to use.

5.1.4.3 All reagents and gases used for analysis and sample preparation should be tested for the presence of trace metals prior to use in the lab. Due to the ultra-low detection limits of this method, it is imperative that all the reagents and gasses be as low as possible. It is often required to test several different sources of reagents until an acceptable source has been found. Metals contamination can vary greatly from lot to lot. Once an acceptable reagent lot has been identified, several cases of the lot number should be sequestered from the supplier. A logbook should be maintained to better track the acids and is stored in the ICP-MS lab.

5.1.4.4 Metals concentrations in both reagent DIW and HNO_3 are monitored each day at the instrument. These samples are uploaded into every LIMS sequence as IBLs. Although there are no established limits for the blanks, the data is useful for troubleshooting.

5.1.4.5 Great care should be taken to keep the facility free from all sources of metals contamination. Clean hood HEPA filters must be replaced with new filters on a regular basis to reduce the risk of airborne contaminants. Metal corrosion of any part of the facility should be addressed and replaced. Every piece of apparatus that is directly or indirectly used in the collection, preservation, or processing of samples should be trace metals free. Additionally, the lab should be free of clutter to minimize contamination from other sources.

5.2 Elemental interferences

5.2.1 Interference sources that may inhibit the accurate collection of ICP-MS data for trace elements are addressed below.

5.2.2 Isobaric elemental interferences are isotopes of different elements that form singly or doubly charged ions of the same mass-to-charge ratio (m/z) and cannot be resolved by the mass spectrometer.

5.2.3 Data obtained with isobaric overlap must be corrected for by:

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5.2.3.1 Measuring the signal from another isotope of the interfering element and estimating the contribution from the interfering isotope on the isotope of interest. This should not be determined using relative isotopic abundance alone, but instead by spiking actual samples with varying amounts of the interfering element. The relative ratio of counts per second that appear on the interfered mass and a non-interfered isotope for the interfering element can be compared to determine an appropriate correction factor (CF). The correction equation can be entered into the corrections column on the equations page of the analysis method. An example is listed below.

$$CF = I/A$$

Where CF is the correction factor, I is the measured increase on the interfered target isotope in terms of counts per second, and A is the measured increase of an isotope of the interfering element for which no isobaric overlap exists, in terms of counts per second. Analytical data can then be corrected by:

$$T = M - CF*X$$

Where T is the true counts per second of the target analyte, M is the measured counts per second of the target analyte, CF is the correction factor, described above, and X is the measured counts per second of the interfering element at the mass used to determine the correction factor.

5.2.3.2 The correction equation for the example above would look as follows:

Cd 114=mass 114- (Correction Factor)*(Sn 118)

5.2.4 Abundance sensitivity occurs when part of an elemental peak overlaps an adjacent peak. This often occurs when measuring a small mass-to-charge ratio (m/z) peak next to a large m/z peak. The abundance sensitivity is affected by ion energy and quadropole operating pressure. Proper optimization of the resolution during tuning will minimize the potential for abundance sensitivity interferences.

5.2.5 Isobaric Polyatomic Interferences are interferences caused by ions, composed of multiple atoms, which have the same m/z as the isotope of interest, and which cannot be resolved by the mass spectrometer. These ions are commonly formed in the plasma or the interface system from the support gases or sample components. The objective of DRC analysis is to remove these interferences, and as a result, correction factors should not be necessary. If they are determined to be present, then they can be corrected by the following:

5.2.5.1 Spiking a sample, matrix matched to the samples of interest, with varying amounts of a polyatomic parent (i.e., Ca for the production of CaO, which can be detected at mass 56) and calculating a correction factor based on the

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change in counts per second at the parent mass and the change in counts per second at the analyte mass. Once a correction factor has been determined, it can be added to the corrections tab in the method file in the Elan 3.4 software.

5.2.5.2 Polyatomic interferences are highly dependent on the sample matrix. Corrections should be considered at the time of analysis.

5.3 Physical Interferences occur when there are differences in the response of the instrument from the calibration standards and the samples. Physical interferences are associated with the physical processes that govern the transport of sample into the plasma, sample conversion processes in the plasma, and the transmission of ions through the plasma-mass spectrometer interface.

5.3.1 Physical interferences can be associated with the transfer of solution to the nebulizer at the point of nebulization, transport of aerosol to the plasma, or during excitation and ionization processes in the plasma. High levels of TDS in a sample can result in physical interferences. Proper internal standardization (choosing standards that have analytical behavior similar to the associating elements) can compensate for many physical interferences.

5.4 Memory Interferences - Carryover of elements in a previous sample in the sample tubing, cones, torch, spray chamber, connections, and autosampler probe are minimized by rinsing the instrument with a reagent blank after samples high in metals concentrations are analyzed.

5.4.1 If carryover is suspected, the sample should be reanalyzed after a long rinse period or after the analysis of an acceptable blank (less than the MRL. Note: Tighter criteria may apply to different regulatory requirements).

6.0 SAFETY

6.1 Refer to Draft EPA Method 1638, section 5.0, for safety issues associated with the use of the method.

6.2 Hydrofluoric acid (HF) is particularly dangerous and should be used with extreme caution. HF may not cause immediate pain but is still very harmful. If there is any contact, quickly rinse under water followed by an application an unopened tube of calcium gluconate gel. Then go directly to the hospital. There should always be at least one tube in the lab during any procedure involving the use HF. The MSDS should be consulted prior to the use of HF and prior training is required by an experienced staff member.

6.3 Nitric acid and hydrochloric acid are very caustic and adequate protective gear should be worn when working with these acids, including eye protection, gloves with the

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appropriate resistance, and a lab coat. An adequate fume hood should be used for all acids.

6.4 Many of the chemicals have toxicities that are not well established and should be handled with care. For all known chemicals used, the MSDS should be consulted in advance.

6.5 Reference 11.3 in Section 11 lists the local Washington state OSHA regulations. King County local sewer limits should be posted near the ICP-MS waste disposal sinks and no materials higher than the limits should be disposed of.

6.6 The ICP-MS emits UV light when the plasma is on. Goggles should be worn if working near the plasma.

6.7 The instrument generates high levels of radio frequency (RF) energy and is very hot when the plasma is on. In the case of an instrument failure, be aware of these potential dangers.

6.8 DRC gases should be stored safely in a closed cabinet. Adequate caution should always be used with pressurized gasses. Prior training or experience is necessary to change any gas cylinders. Oxygen gas can be explosive with certain materials and ammonia has a very low odor threshold and is corrosive to tissue.

7.0 REAGENTS AND STANDARDS

7.1 Reagents may contain elemental impurities that could negatively affect high data quality. High-purity reagents should always be used. Each reagent lot should be tested and test low for the elements of interest before being used in the laboratory.

7.2 Deionized Water (DIW) (ASTM Type I) demonstrated to be free from the metals of interest and potentially interfering substances.

7.3 Nitric Acid (HNO_3), concentrated, tested low (limits are found in the Bottle Washing SOP BR-0400).

7.3.1 1% (v/v) HNO_3 - Stock standard preparation diluent and sample diluent.

7.3.2 2% (v/v) HNO_3 –Tube soaking solution.

7.3.3 5% (v/v) HNO_3 –Pipette cleaning solution and rinse station solution.

7.4 Stock Standard Solutions - obtained from a reputable and professional commercial source.

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7.4.1 Single element standards are obtained for each determined metal as well as for any metals used as internal standards and interference checks.

7.4.2 A second source is obtained for each determined metal.

7.5 Multi-element stock standard solution-elements must be compatible and stable in solutions together.

7.5.1 "IV-19" Second source stock solution obtained from Inorganic Ventures. 100 mg/L As, Be, Ca, Cd, Co, Cr, Cu, Fe, Mg, Mn, Mo, Ni, Se, Ti, Tl, V, and Zn.

7.5.2 "IV-7" Second source stock solution obtained from Inorganic Ventures. 1000 mg/L K; 100 mg/L Al, Ag, B, Ba, and Na; 50mg/L Si.

7.5.3 "QCS-26" Second source stock solution obtained from High-Purity Standards. 100 mg/L Al, Cd, Pb, Se, Sb, Ca, Mg, As, Cr, Mn, Ag, Ba, Co, Mo, Na, Zn, Be, Cu, Ni, Tl, B, Fe, K, and Ti.

7.5.4 Dilute single element stock standards appropriately in order to prepare multi-element standards in 1% HNO_3. These standards will be stable for six months from date of preparation. These standards may change in composition as the needs for different calibrations change.

7.5.5 "TM-A" Standard (mg/L): 2000 Ca; 1000 Na; 500 Fe; 100 B; 25 Cr; 10 Ti, As, Ni, Cu, Sr; 5.0 Co; 1.0 Cd; and 0.5 Tl.

7.5.6 "TM-B" Standard (mg/L): 10 Se; 5.0 V; 2.5 Sn and Pb; 1.0 Be; and 0.50 Th, and U.

7.5.7 "TM-C" Standard (mg/L): 2000 Mg; 1000 K; 500 Al; 50 Zn; 25 Mn, Ba; and 5.0 Mo.

7.5.8 "ML-1" Standard (mg/L): 0.01 Tl and U; 0.02 Mo, Th, and Cd; 0.05 Be, Mn, Sr, and Pb; 0.1 Co; 0.15 V, Cr and Sn; 0.2 Ba, Ti, As, Ni, Cu, and Zn; 0.5 Se; 1.0 B, Al; 3.0 Mg; 4.0 K; 5.0 Fe; 15 Na; and 30 Ca.

7.5.9 Ag Standard Solutions at 2.0 and 0.02 mg/L

7.5.10 Sb Standard Solutions at 1.0 and 0.02 mg/L

7.5.11 Additional elements can be added to these standard mixes in 1% HNO_3. Attention should be given to the compatibility of the elements added.

7.6 Internal Standard Solution - Dilute stock standard solutions of Ge, In, ^{6}Li, Pt, Rh, Tm, and Sc to give approximately 500,000 counts per second when run at a 4:1 sample to internal standard mix. [redacted]

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[redacted] The solution is stable for six months after the date of preparation. Different internal standards can be chosen based on sample matrix, on-going research, and specific analytical requests. The internal standard solution is added to all standards and samples by passing through a mixing chamber prior to passing through the peristaltic pump device.

7.7 The date, standard information, and preparer's initials must be entered into the Element LIMS at the time of preparation for all standards. The date, preparer's initials, expiration date, elements, and their concentrations must be recorded on all bottles of standards.

7.8 Calibration Standards - Fresh calibration standards should be prepared every four weeks, or at the earliest expiration date for any of the standards in the mix, or as needed. The standards and amounts used, date, and preparer's initials should be recorded in the LIMS each time a fresh calibration is prepared, unless identical stock solutions are being used, and the original calibration standards were made less than 30 days prior.

7.8.1 Dilute the multi-element stock standard solutions into 50-mL pre-cleaned autosampler vials with 1% HNO_3 in the following manner:

	ML1 (µL)	TM-A (µL)	TM-B (µL)	TM-C (µL)	Ag 0.02 (µL)	Ag 2.0 (µL)	Sb 0.02 (µL)	Sb 1.0 (µL)
STD 0	0	0	0	0	0	0	0	0
STD 1	50	0	0	0	50	0	50	0
STD 2	100	0	0	0	100	0	100	0
STD 3	500	0	0	0	500	0	500	0
STD 4	0	25	50	5	0	25	0	50
STD 5	0	50	100	50	0	50	0	100
STD 6	0	250	200	250	0	250	0	200
STD 7	0	500	500	500	0	500	0	500
STD 8	0	1000	1000	1000	0	1000	0	1000

Note #1: The specific element concentrations and the dilution factors for specific standards may be adjusted as needed, such as when element specific PQLs change.

Note #2: Don't use 5-µL aliquots of stock standard when preparing calibration standard mixes. Instead, dilute the stock standard 10x and use a 50-µL aliquot.

7.9 Initial calibration verification (ICV) - ICV solution is made up from secondary source standards in order to verify the validity of the calibration curve.

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7.9.1 The ICV is made by diluting 25 µL of QCS-26 and 25 µL of a 10-fold dilution of QCS-26, to 50-mL of 1% HNO_3. Additional ICVs are prepared as needed.

7.9.2 Alternatively or additionally, a mixed solution standard of all elements at exactly 10-50 times the low calibration standard and may be obtained from CPI.

7.10 "Daily" optimization and tuning solution - [redacted]

7.11 Dual detector calibration solution - [redacted]

7.12 Certified reference material (CRM) - CRMs of various matrices (drinking water, seawater, sediment, soil, tissue, river water, etc).

7.13 Interference check solution - BRL analyzes an aliquot of the river water CRM NIST 1643e with every batch. NIST 1643e is similar in composition to standard interference check solutions for ICP-MS analyses and it offers a real world check of the validity of the interference correction.

8.0 SAMPLE COLLECTION, FILTRATION, PRESERVATION, AND STORAGE

8.1 Samples should be received as described in SOP BR-0300.

8.2 Samples for dissolved metals determination must be filtered (if not field-filtered) through a 0.45-µm or a 0.2-µm pre-cleaned disposable filter unit within 48 hours of collection. Otherwise, all data for the dissolved metals will be qualified "H" for inappropriate sample handling prior to receipt. Filtering procedures are described in BRL SOP BR-0104.

8.3 Samples must be preserved to a pH less than or equal to 2. The following provides a list of common preservation schemes:

8.3.1 Preserve to 0.2% (v/v) with HNO_3 for reductive precipitation (RP) extractions.

8.3.2 Preserve to 0.099% (v/v) with HNO_3 for Co-APDC extractions.

8.3.3 Preserve to 1% (v/v) with HNO_3 for dissolved metals determination and for some total recoverable metals digestions.

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8.3.4 Preserve to 1.8% (v/v) HNO_3 and 0.2% hydrofluoric acid (HF) for some total recoverable metals digestions.

8.4 Store the preserved samples for a minimum of 48 hours before analysis to completely desorb the metals from the container walls. The sample pH should be verified to be less than pH 2 prior to sample preparation.

8.5 Preserved samples can be stored in a secure area of the laboratory at room temperature until analysis.

8.6 Human biomonitoring samples should be shipped frozen and stored frozen until the time of analysis; however, short-term storage of urine and plasma/serum samples at 2-4 °C is acceptable.

9.0 PROCEDURE (PRE-CALIBRATION, CALIBRATION AND STANDARIZATION)

9.1 Sample Preparation

9.1.1 For the determination of dissolved analytes, samples should be preserved with HNO_3 to 1% (v/v) after filtration. Rinse pipette tip two times with a 1% HNO_3 pipette rinsing solution and add 10-mL of a filtered preserved sample to a pre-cleaned 15-mL autosampler vial. If the sample is suspected to be high in metals or total dissolved solids (TDS), then dilute the sample with 1% HNO_3. In the case of a high concentration of metals dilute the sample until the concentration of the metals at the dilution fall within the calibration range of the instrument. In the case of a high TDS, dilute the samples until the TDS count is less than 1000 mg/L.

9.1.2 As a general guideline, normal seawater and sediment digest samples are diluted 50x, tissue samples are diluted 10x, and influent wastewater samples are diluted 5x.

9.1.3 Biomonitoring samples:

9.1.3.1 Urine samples are diluted 10x with 1% (v/v) HNO_3.

9.1.3.2 Whole blood samples are diluted 50x with 1% (v/v) HNO_3.

9.1.4 For the determination of total recoverable analytes, there are several digestion and preservation techniques that can be used. Evaluation of the sample matrix, detection limits, analytes of interest, and the client's needs is required before deciding on an appropriate preparation technique. Current methods include SOP BR-0065 (1% HNO_3 digestion), BR-0066 (RP), BR-0067 (Co-APDC extraction),

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BR-0068 (reverse aqua regia digestion), BR-0069 ($HF/HNO_3/HCl$ digestion), and BR-0070 (tissue digestion).

9.1.5 When the metals concentration of a sample is unknown, the samples should be diluted or analyzed using a total quantification method prior to being run undiluted or with a comprehensive quantitative method. This protects the instrument and the sample introduction system from potential contamination and damage.

9.2 Instrument Startup

9.2.1 Turn on the computer, printer, and monitor. Load Windows XP®. Load the ELAN software by choosing the ELAN icon from the desktop or by choosing the ELAN software from the programs menu in the start menu.

9.2.2 [redacted]

9.2.3 Follow the ICP-MS daily maintenance logbook as you complete the following checks.

9.2.3.1 Change the peristaltic pump tubing as needed. The tubing requires changing if the internal standard shows RSD values above 1.5% for the initial calibration blank. **Note:** The PharMed green-green peristaltic pump tubing for the waste can be used for a month or more.

9.2.3.2 Check that all tubing and connections are in good condition.

9.2.3.3 Check the sample waste container level. Neutralize and dispose of sample waste if needed.

9.2.3.4 Check the sampler and skimmer cones and inspect the o-rings for damage or wear. Replace or clean if needed. Clean the cones by swabbing with DI water. If this is insufficient [redacted]

9.2.3.5 Check the quartz torch, RF load coil, and quartz injector for damage and wear. Replace or clean if needed.

9.2.3.6 Check for the proper alignment of the injector, torch, and RF coil. Perform Z adjustment if not properly aligned and anytime that the spray chamber is changed, the cones are changed, the injector or torch is changed, or the RF coil is cleaned. The following procedure should be followed when performing the Z adjustment.

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9.2.3.6.1 [redacted]

9.2.3.6.2 [redacted]

9.2.3.6.3 [redacted]

9.2.3.7 Check that the Argon pressure to the instrument is [redacted]. The plasma must be on to check this, and it's behind the instrument and difficult to get to. Alternatively, check that the regulator for Ar on the wall above the instrument reads at [redacted] and that all readings are normal on the Instrument Panel in ELAN.

9.2.3.8 If running in DRC mode, check the DRC gases in the safety cabinet. Both should be at around [redacted].

9.2.3.9 Check the recirculator coolant level. Make sure the red light is not on and add coolant if needed. Check the filter, and clean if needed.

9.2.3.10 Check that the instrument vent is on and working properly.

9.2.3.11 Check the pump oil levels and condition of the pump oil. Change the pump oil when it develops the color and transparency of tea or when it appears dirty or turbid. Refer to page 4-34 of the ELAN DRC II hardware guide to change the pump oil. The oil in the high vacuum roughing pump (left) should be changed about every three months. The right roughing pump oil should be changed about every two months or more frequently if high TDS samples or analysis with Ammonia as a DRC gas have been run

9.2.3.12 Enter the instrument window of the ELAN software and choose the front panel tab. The system status indicator should indicate "Ready" if all of the system hardware is running properly and the plasma can be ignited. If the system status indicator displays "Not ready", choose the diagnostic display tab to help determine the reason for this instrument status.

9.2.3.13 Click the plasma "Start" box on the front panel tab to start the plasma ignition process (plasma can also be ignited manually by pushing the green "start" button on the front of the instrument). The plasma ignition process takes about one minute.

9.2.3.13.1 If the plasma fails to ignite or arcing is observed push the yellow stop button on the front of the instrument immediately and wait one minute before opening up the torch box for investigation. Check for damage to the coil, torch, and auxiliary and plasma gas connections to the torch caused from arcing. Determine the reason for the ignition failure.

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9.2.3.13.1.1 Check for moisture in the injector tube and torch assembly.

9.2.3.13.1.2 Check the o-rings on the injector for damage or wear that could lead to loss of vacuum.

9.2.3.13.1.3 Tubing should be checked for damage or poor connections that may be allowing air into the system.

9.2.3.13.1.4 Check for proper installation and alignment of the injector, torch, and RF coil.

9.2.3.13.1.5 Check the drain connection and tubing leaving the spray chamber to ensure a proper connection.

9.2.3.13.1.6 Push the plasma start button again. If the plasma still will not ignite then refer to page 5-2 in the ELAN DRC II hardware guide or to page 2-6 of the ELAN version 3.0 software guide.

9.2.3.14 After the plasma has ignited, the peristaltic pump should automatically begin to rotate counter-clockwise. Alternatively, the pump can be turned on automatically by going to the Devices window, putting "6" in the rpm window, and clicking on the counter-clockwise button.

9.2.3.15 Place the carrier fluid probe into a vial of 1% HNO3 and observe consistent fluid flow. If the flow is inconsistent, determine the cause and continue. Optimize the peristaltic pump levers to a tension that minimizes pump pulsing but keeps a continuous and rapid flow.

9.2.3.16 Let the plasma stabilize for at least [redacted] before beginning optimization.

9.2.3.17 If the instrument has been off for an extended period of time or if it has been moved or had major components replaced, refer to page 2-4 of the ELAN version 3.0 software guide for special startup instructions.

9.3 X-Y Adjustment

9.3.1 The X-Y adjustment of the sample introduction system needs to be performed whenever the cones are cleaned or replaced or after performing maintenance procedures in the torch chamber.

9.3.2 Place the carrier fluid probe into the "Daily" solution (section 6.10).

9.3.3 Open the "1-xy" method and the current daily performance dataset, ie, "Daily Performance IV".

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9.3.4 In the "samples" window, choose the "manual" tab and then click on "Analyze sample".

9.3.5 In the "real-time" window, click on [redacted] in the "analytes available" window, and then click on "Add analyte". Repeat the same steps for [redacted] to observe the background signal. Adjust the setting of the window to optimize the viewing of the signals.

9.3.6 Lift the top cover of the instrument to access the X and Y adjustment knobs. While observing the intensity signal in the real-time graphics window, adjust the Y knob (closer to the operator) to obtain the maximum intensity on the vertical plane. While observing the intensity signal in the real-time graphics window, adjust the X knob to obtain the maximum intensity on the horizontal plane.

9.3.7 Repeat the X and Y adjustments several times until the maximum signal intensity is achieved.

9.4 Tuning

9.4.1 Tuning should be performed whenever there are changes to the instrument's electronics, whenever there is a need to modify the resolution for elements, or monthly (at a minimum).

9.4.2 The tuning file is needed to manage the instrument's quadrupole mass filtering performance. The tuning controls are used to perform either of two operations:

9.4.2.1 A mass calibration is used to adjust the instrument's electronics to ensure accuracy of the mass spectrometer.

9.4.2.2 A resolution measurement and adjustment is used to ensure that the instrument's resolution at each mass of interest is within the defined range.

9.4.3 Place the carrier fluid probe in the "Daily" solution (section 6.10).

9.4.4 In the Smart Tune window, click on the "Edit List" button and add "Mass Calibration and Resolution" to the "Selected" section. Close the "Edit List" tab.

9.4.5 Click on the "Setup" tab and ensure the correct optimization, tuning and dataset files are selected, and that the autosampler position is set to 0.

9.4.6 In the optimization list click on the Mass Calibration and Resolution and ensure that the "1-tuning" method is selected.

9.4.7 Click "Optimize"

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9.4.8 When complete, check to see that the measured masses are ± 0.1 amu of the actual mass value. Check the resolution (measured peak width) to see that it is between 0.650 – 0.750 for all measured masses.

9.4.8.1 The interactive graphics window is used to view the results of the tuning adjustment.

9.4.8.2 If one or more of the measured masses falls outside the acceptable range then retune the instrument. Continue retuning until all the masses are within the acceptable criteria.

9.4.8.3 If one or more of the resolution values falls outside of the acceptable range, adjust the resolution DAC value in the following manner:

9.4.8.3.1 To increase the resolution (narrow the peak width), increase the DAC setting. Increasing the DAC setting by 30 will increase the resolution by 0.1 amu.

9.4.8.3.2 To decrease the resolution (broaden the peak width), decrease the DAC setting. Decreasing the DAC setting by 30 will decrease the resolution by approximately 0.1 amu.

9.4.9 The tuning is optimized when all of the measured masses and resolution values are within the acceptable range. Save the tuning file (default.tun).

9.4.10 Print the tuning report to PDF. Go to the "interactive" window, click the "Analyte" box, and add all the analytes. Printout the spectral data for each analyte. Add the PDF to the Mass Calibration and Resolution file on the server.

9.5 Nebulizer Gas Flow Optimization

9.5.1 The nebulizer gas flow optimization should be performed daily or as needed.

9.5.2 Place the carrier fluid probe in the "Daily" solution (section 6.10).

9.5.3 Open the Smart tune window

9.5.4 Click on the "Edit List" button and bring Nebulizer Gas Flow to the "selected" side.

9.5.5 In the optimization tree box, choose nebulizer gas flow (NEB).

9.5.6 Select the appropriate method, typically "1-Neb Gas Optimization."

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9.5.7 In the criteria section, select the Intensity and Formula boxes. Select In 114.904 as the analyte for intensity and "CeO 155.9" as the first analyte "/" for the operator and "Ce 139.905" as the second analyte for formula.

9.5.8 In the "Range" window, select [redacted].

9.5.9 In the Optimization tree, right click on "Nebulizer Gas Flow (NEB)" and select "Quick Optimize."

9.5.10 When complete go to the interactive window to view the optimization curve. Check that the optimization curve increases, attains a maximum point, then decreases.

9.5.11 Save the optimization file as "default.dac". Printout the optimization curve and add to the ICP-MS optimization logbook.

9.5.12 The DRC Mode NEB is automatically set to the same value as the standard mode NEB, however, it is good practice to re-analyze the nebulizer gas flow for the target analytes, in the exact matrix in which the analysis will be performed.

9.6 Optimizing the Autolens Voltage

9.6.1 The autolens optimization should be performed daily.

9.6.2 Place the carrier solution probe in the "Daily" solution (6.10).

9.6.3 Open the "Smart Tune" window.

9.6.4 Click on the "Edit List" button.

9.6.5 Move "Autolens" to the selected side.

9.6.6 Click on "Autolens" in the optimization tree and then on the "Setup" tab.

9.6.7 Load the correct method file, typically "1-autolens-FAST.mth."

9.6.8 In the range box, select [redacted].

9.6.9 Right click on "Autolens" in the optimization tree and select "Quick Optimize"

9.6.10 Printout the autolens plots in the interactive window by clicking on the box with four little arrows. Add the plot to the ICP-MS logbook. Initial the plot and record the type of spray chamber.

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9.6.11 The lens can also be optimized for a single element by adding "Lens Voltage" to the Optimization tree and performing the above procedure.

9.7 Instrument Performance Check: "Daily"

9.7.1 The instrument performance check is done before the start of each analytical run to ensure proper functioning and sensitivity of the instrument. It is often educational to run a daily before and after optimization in order to observe how performing optimizations change the instrument response.

9.7.2 Place the carrier fluid probe in the "Daily" solution (6.10).

9.7.3 Open the "Smart Tune" window and add "Daily Performance" to the Optimization tree.

9.7.4 Click once on "Daily Performance" in the optimization tree and select the "Setup" tab.

9.7.5 Load the appropriate method file, typically "1-daily-FAST.mth," and ensure that the correct criteria are selected.

9.7.6 Right click on "Daily Performance Check" in the optimization tree and select "Quick Optimize."

9.7.7 Review the results in the report view to check for background counts, sensitivity, double-charged element levels, and oxide levels. Minimum performance specifications are listed on page 4-39 of the ELAN version 3.0 software guide. The specifications should fall within the following guidelines:

Background level at mass 220: < 5 cps
Mg sensitivity: > 8,000 cps/1 ppb
In sensitivity: > 40,000 cps/1 ppb
U sensitivity > 30,000 cps/1ppb
CeO/Ce: ≤ 2.5%
Ba^{++}/Ba: ≤ 2.5%

9.7.8 If the performance check is satisfactory, the instrument is ready to begin sample analysis. Place the autosampler probe in the rinse position to begin flushing the system.

9.7.9 If the performance check is not satisfactory, begin the following steps:

9.7.9.1 Manually adjust the nebulizer gas flow by going into the "Optimize" window and selecting the "Manual adjust" tab. [redacted]

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[redacted]

9.7.9.2 Check the pump tubing, cones, torch, and injector for alignment and cleanliness.

9.7.9.3 Check the nebulizer for blockages and cleanliness.

9.7.9.4 Perform the optimizations steps again (X-Y, tuning, nebulizer gas flow, and autolens), perform a daily after each step to observe the changes.

9.7.9.5 If the daily is still not satisfactory, a full optimization may be necessary.

9.8 Section 9.8 intentionally left blank.

9.9 Full Optimization

9.9.1 A full optimization should be performed when the status of the instrument is unknown, after a major servicing or repair, or when the daily optimizations fail to meet criteria.

9.9.2 Before beginning a full optimization, the daily optimization procedures should be completed. This includes the sample introduction system check, the X-Y adjustment, tuning, nebulizer gas flow optimization, and autolens.

9.9.3 Detector Voltages.

9.9.3.1 Place the carrier fluid and internal standard probes in DIW.

9.9.3.2 Open the Smart Tune window and add detector voltages to the optimization tree.

9.9.3.3 Click once on Detector Voltages in the optimization tree and then on the "setup" tab. Check that the correct methods are loaded in the method window. Typically the Pulse Method File will be "Pulse Stage Optimization" and the Analog Method File will be "Analog Stage Optimization."

9.9.3.4 In the "Criteria" section check that the analyte for the [redacted].

9.9.3.5 In the Range section click "Get Defaults."

9.9.3.6 Right click on detector voltages in the Optimization Tree and select "Quick Optimize."

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9.9.3.7 In the optimization window a plot of the pulse intensity vs. pulse stage voltage is displayed. The diamond symbol indicates the optimum point that the software picked. Evaluate the plot to ensure that the correct value has been chosen. Print the plot and add to the ICP-MS optimization logbook.

9.9.3.8 Save the optimizations file.

9.9.4 Performing Dead Time Correction

9.9.4.1 The dead time correction is a mathematical equation that is used to correct for erroneous readings at high-count rates. The dead time function corrects for the anomaly that two pulses will hit the detector at the same time. The dead time correction should not change as the detector ages and should only need to be adjusted when the detector is replaced.

9.9.4.2 Move the carrier fluid and internal standard probes to DIW.

9.9.4.3 Open the workspace "Setting Dead Time Correction.wrk".

9.9.4.4 In the optimization window, click the dual detector calibration tab, and click "Get analyte list".

9.9.4.5 Ensure that "analog 80" appears in the analyte field and click "Get defaults"

9.9.4.6 Click "Calibrate". When the scanning is complete, note the correlation coefficient (under the heading coefficient).

9.9.4.7 From the options menu, click configuration. In the dialog box that appears, note the value of the dead time correction and increase the value of dead time (ns) by 5. Click OK and Exit.

9.9.4.8 Open the dataset window and highlight the last two samples: the "dual calibration blank" and the "dual calibration". Keep these two samples highlighted while you calibrate at different dead time corrections.

9.9.4.9 Leaving the two samples highlighted in the dataset window, go back to the dual detector tab in the optimization window.

9.9.4.10 Click "Clear calibration", "Get analyte list", and "Calibrate from dataset".

9.9.4.11 Note the correlation coefficient again.

9.9.4.11.1 If the correlation coefficient has increased, access the options menu again and click configuration. Increase the dead time value by another [redacted]. Go back to the dual detector tab in the optimization window and click "Clear

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calibration", "Get analyte list", and "Calibrate from dataset". Continue the above steps until the highest (i.e., closest to 1.000000) correlation coefficient is found.

9.9.4.11.2 If the correlation coefficient has decreased, access the options menu again and click configuration. Decrease the dead time value by another [redacted]. Go back to the dual detector tab in the optimization window and click "Clear calibration", "Get analyte list", and "Calibrate from dataset". Continue the above steps until the highest correlation coefficient is found.

9.9.4.12 The typical dead time correction value is [redacted]. As a guideline, the dead time correction should stay within the range of [redacted]. Even if the calibration yields the best correlation coefficient with a dead time setting of [redacted], do not set the dead time above [redacted]

9.9.4.13 Save the dead time correction value. Record the dead time value in the ICP-MS optimization logbook.

9.9.4.14 On a daily basis and any time the ELAN software has been shut down. The set dead time correction value should be verified as the last recorded value in the optimization logbook. This can easily be done while running the "daily performance check" as the current selected dead time correction value will be listed near the top of the daily performance report.

9.9.5 Dual Detector Cross Calibration

9.9.5.1 The dual detector calibration should be performed only after the above steps of the full optimization have been completed. The dual calibration is used to extend the dynamic range of the detector by normalizing the analog stage voltage of the detector and the pulse stage voltage.

9.9.5.2 The dual detector calibration method file and solution should contain each element that is desired for quantification using the extended dynamic range, including internal standards.

9.9.5.3 Move the carrier fluid and internal standard probes to the dual calibration solution (Section 6.11).

9.9.5.4 Open the "Smart Tune" window and add "Dual Detector Calibration" to the optimization tree.

9.9.5.5 Click once on "Dual Detector Calibration" in the optimization tree and then click on the "Setup" tab.

9.9.5.6 Check that the appropriate method is loaded in the "method" box, typically this is "1-dual.mth."

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9.9.5.7 Check that in the "Range" section, the "Start" value is 2, the "end" value is 13 and the "Step" value is 0.250.

9.9.5.8 Right click on "Dual Detector Calibration" in the optimization tree and select "Quick Optimize."

9.9.5.9 In the "Dual calibration" tab, check the correlation coefficients of all the analytes and the number of counts measured. The correlation coefficient r-value should be at least 0.999 (or closer to 1.000) for each analyte. There should be a minimum of 5 points for each metal. If little or no counts are measured for an analyte, check the dual calibration solution for the presence and the concentration of the analyte(s) in question. Counts for the elements should be in the [redacted] counts range (note: this is counts, and not cps; therefore, to evaluate this, you need to change your settings). The dual detector calibration is only important if counts exceed [redacted].

9.9.5.10 In the interactive window, click on "analytes" and add the analytes five at a time. Review and print the plots for all analytes. Add the plots to the ICP-MS optimization logbook.

9.9.5.11 Save the optimization file.

9.9.6 Optimizing the Autolens Voltage

9.9.6.1 An autolens optimization must be performed following a dual detector calibration.

9.9.6.2 Follow Section 8.6.

9.9.7 Instrument Performance Check: "Daily"

9.9.7.1 A daily performance check will need to be performed following a full optimization. The nebulizer gas flow may need to be adjusted a little manually to optimize the oxides and doubly charged ions.

9.9.7.2 Follow Section 8.7 or 8.8 for automated optimization.

9.10 Cell Rod Offset

9.10.1 If the background is increasing or the detector has been changed, this procedure can be performed.

9.10.2 This procedure is not part of the full optimization and should not be performed as part of a routine optimization. Usually, a new detector is installed in the instrument by a Perkin-Elmer service technician and that person will perform this optimization as part of the installation and maintenance check.

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9.10.3 Open the "Smart Tune" window and add "Cell Rod Offset Std. (CRO)" to the optimization tree.

9.10.4 Click once on "Cell Rod Offset (CRO)" in the optimization tree and click on the "Setup" tab.

9.10.5 Ensure the correct method file is loaded, typically this is "Cell Rod Offset Std.mth."

9.10.6 In the Criteria box, check that "intensity" is selected and that the analyte is "In 114.904." The comparator should be "Maximum."

9.10.7 Click "Get defaults".

9.10.8 In the optimization criteria group, choose "Ramp".

9.10.9 Place the carrier fluid and internal standard probes in the "Daily" solution (6.10).

9.10.10 Right click on "Cell Rod Offset (CRO)" in the optimization tree and select "Optimize".

9.10.11 In the real-time graphics window, find the setting that yields a minimum for the two background signals (8.5 and 220) while obtaining the maximum signal for In. In the intensity vs. time profile, note the point for the optimum In signal.

9.10.12 In the interactive graphics window, click on the intensity point 114.904 in the intensity vs. time profile for In.

9.10.13After clicking on this point, the optimization file will automatically be updated.

9.10.14 In the "Optimization" window, select "File" and "Save".

9.11 Cell Path Voltage

9.11.1 The cell path voltage adjusts the voltage applied to the reaction cell lenses. This is not part of a full optimization procedure and should not be performed as part of routine optimization. In general, this optimization is performed at installation and should not have to be adjusted.

9.11.2 Open the workspace "Optimizing cell path voltage.wrk."

9.11.3 In the optimization window, select the "Auto optimize" tab.

9.11.4 In the parameter description list box, click "Cell path voltage."

9.11.5 Click "Get analyte list" and select In 114.904.

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9.11.6 Click "Get defaults".

9.11.7 In the optimization criteria group, choose "Ramp."

9.11.8 Place the auto-sampler probe in the "Daily" solution (section 6.10).

9.11.9 In the optimization window, click "Optimize."

9.11.10 In the real-time graphics window, find the 3 analyte ions that yield a minimum for the two background signals (8.5 and 220). The signal for these two masses should be below 2 cps while obtaining the maximum signal for In. In the intensity vs. time profile, note the point for the optimum In signal.

9.11.11 In the real-time graphics window, note the In intensity at the optimum point on the intensity vs. time profile.

9.11.12 Click on the optimum In intensity point in the interactive graphics window. The software will automatically update the optimized cell path voltage value.

9.11.13In the "Optimization" window, select "File" and "Save."

9.12 Internal Standardization and Calibration

9.12.1 Following Pre-calibration optimizations, the following calibration table should be used with internal standardization as defined in **BOLD**. The unit of measure for all calibration points in the table is µg/L.

9.12.2 Internal standardization must be used in all analyses to correct for instrument drift and physical interferences. For full mass range scans, 3-5 internal standards should be used. Internal standards must be present in all samples, standards, and blanks at identical levels. Internal standards are added using a second channel of the peristaltic pump at a level of [redacted] counts.

9.12.3 The instrument is typically calibrated using 8 points. Calibration points may be omitted from the calibration, but only at the high or the low end. If low-end calibration points are omitted, the MRL may require adjustment up such that the MRL is ≥ the lowest calibration point used. A minimum of 3 non-zero calibration points for each analyte determined is required.

9.12.4 Multiple isotopes for some analytes may be measured, with only the most appropriate isotope (as determined by the analyst) being reported.

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Typical ICP-MS Calibration Curve Concentrations

Analyte	CAL1	CAL2	CAL3	CAL4	CAL5	CAL6	CAL7	CAL8
^{6}Li								
Be	0.05	0.1	0.5	1	2	4	10	20
B	1	2	10	50	100	500	1000	2000
Na	15	30	150	500	1000	5000	10000	20000
Mg	3	6	30	200	2000	10000	20000	40000
Al	1	2	10	50	500	2500	5000	10000
K	4	8	40	100	1000	5000	10000	20000
Ca	30	60	300	1000	2000	10000	20000	40000
Sc								
Ti	0.2	0.4	2	5	10	50	100	200
V	0.15	0.3	1.5	5	10	20	50	100
Cr	0.15	0.3	1.5	12.5	25	125	250	500
Mn	0.05	0.1	0.5	2.5	25	125	250	500
Fe	5	10	50	250	500	2500	5000	10000
Co	0.1	0.2	1	2.5	5	25	50	100
Ni	0.2	0.4	2	5	10	50	100	200
Cu	0.2	0.4	2	5	10	50	100	200
Zn	0.2	0.4	2	5	50	250	500	1000
Ge								
As	0.2	0.4	2	5	10	50	100	200
Se	0.2	0.4	2	10	20	40	100	200
Sr	0.05	0.1	0.5	5	10	50	100	200
Mo	0.02	0.04	0.2	0.5	5	25	50	100
Ag	0.02	0.04	0.2	1	2	10	20	40
Cd	0.01	0.02	0.1	0.5	1	5	10	20
In								
Sn	0.15	0.3	1.5	2.5	5	10	25	50
Sb	0.02	0.04	0.2	1	2	4	10	20
Ba	0.05	0.1	0.5	2.5	25	125	250	500
Tm								
W	0.02	0.04	0.2	1	2	10	20	40
Tl	0.01	0.02	0.1	0.25	0.5	2.5	5	10
Pb	0.05	0.1	0.5	2.5	5	10	25	50

9.13 Sample Analysis

9.13.1 Creating a method

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9.13.1.1 A method is created to instruct the instrument what analytes (masses) to measure during a run. It is also used to set the data acquisition, processing, and calibration information that will be used to control the determination.

9.13.1.2 A method can either be created new from a blank template or as a modification of an earlier saved method.

9.13.1.3 Under the file menu, click method and new. OR, use the HugeOne-J.mth file as a template.

9.13.1.4 Save the method as a name with the Element sequence number followed by the SOP used for the analysis, followed by the instrument name, followed by the analyst's initial. The method name for the analysis of sequence 0900150, analyzed with SOP BR-0060, using the ICP-MS Darcy, with an analyst with the initials ACM would be "0900150-0060-Darcy-ACM."

9.13.1.5 Click on the Timing tab.

9.13.1.5.1 In the analyte column, enter in the analytes of interest followed by specific mass (i.e.Se78). Right clicking on the analyte column will allow the user to choose from the periodic table of elements which masses to use. Enter in the internal standards to be used. Using the "Edit" menu, define the groups of elements and identify the internal standard for each group. For monitoring elements only, enter the elements near the bottom of the page and do not associate them with a group or internal standard.

9.13.1.5.2 For elements that are to be quantitatively determined and for internal standards, set the dwell time to [redacted]. For monitoring elements, set the dwell time to [redacted].

9.13.1.5.3 Select a sample loop for the analysis based on the approximate sample volume required for each run. [redacted] There should be a buffer of at least 33% when calculating the required sample volume.

9.13.1.5.3.1 [redacted]

9.13.1.6 Click on the Equations tab.

9.13.1.6.1 Correction equations are entered in this page. The software will provide a list of common interferences for each mass.

9.13.1.7 Click on the Calibration tab.

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9.13.1.7.1 Enter in the concentrations of the calibration standards for each element. Leave the rows with the internal standards blank.

9.13.1.7.2 Right click on the units column and choose μg/L from the drop down list. Make all the units µg/L.

9.13.1.7.3 Ensure that the calibration curve type reads "Weighted Linear", unless otherwise requested.

9.13.1.8 Click on the Sampling tab.

9.13.1.8.1 In the "A/S" column, enter in the autosampler location of the calibration standards.

9.13.1.8.2 Set the sample flush times for all the calibration standards to 1 second, as this number is simply a place holder when using the FAST sample introduction system.

9.13.1.8.3 Set the sample flush, wash and delay and analysis speed [redacted].

9.13.1.8.4 Set the read delay to [redacted].

9.13.1.8.5 Set the wash time to [redacted].

9.13.1.8.6 The times above can be altered in selected circumstances. For example, if only As and Se are being analyzed by DRC, a sample uptake time [redacted] is acceptable if the FAST method is adjusted to match.

9.13.1.8.7 If there is no autosampler selected, click on "Select", and choose "As-93plus" from the drop-down menu. In the "Tray" box, click on "Select" and choose "c:\program files\esi\esi sc\esi.try".

9.13.2 Sample Window

9.13.2.1 The sample window is used to identify the samples in a sequence, control the order in which samples are measured, and initiate the measurements.

9.13.2.2 The sample window contains two tabs: "Manual" and "Batch". The manual window is used to individually identify samples for manual control of a determination. The manual window is useful for reanalyzing calibration standards that did not meet criteria during initial analysis. The batch window is used to identify a group of samples for an automated determination, to control the order in which the samples are measured, and to initiate the measurements. Most sample analysis is performed using the batch window.

9.13.2.3 Click the "Batch" tab at the top of the sample window.

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9.13.2.4 Enter the appropriate autosampler location for each sample in the A/S column.

9.13.2.5 In the "Batch ID" column, enter in the batch number of the sample batch to be run. This column should be left blank for rinses, calibrations, CCVs, instrument blanks, etc.

9.13.2.6 In the "Sample ID" column, enter the unique sample name for each sample.

9.13.2.7 The "Measurement Action" column defines when to run standard and blank solutions, in addition to specifying the running of the sample solution. In the measurement action cell, right click the mouse to show a drop down menu with a list of actions to choose from. For the calibration, select "Run blank, stds. and sample". Choosing this action will analyze the calibration blank and the standards that are specified on the calibration tab of the method. For all other samples, choose "Run sample".

9.13.2.8 The method column specifies which method should be used for measuring the sample. In the method cell, right click the mouse to display the "Select method file dialog". From the file list, select the desired method and click OK.

9.13.2.8.1 The description cells provide a field for entering in specific information or notes about the sample. Currently this is used by the Element software to determine the appropriate blank correction factor, for samples diluted 10 times, enter "10x."

9.13.2.9 In the "Aliquot Volume" and "Diluted to Volume" columns, enter the initial and final volume (in milliliters, mL) of sample if the sample is diluted in the autosampler cups. Leave these columns empty if the sample is not diluted.

9.13.3 Dataset window

9.13.3.1 The dataset window records every analysis and the order they have been analyzed. Information in the fields cannot be modified. The dataset should be saved with an identification number that corresponds directly to the Element sequence number i.e. "0900150."

9.13.4 Calibration view

9.13.4.1 The calibration view window is used for post-acquisition viewing and interpretation of calibration curves generated from the measurement of the calibration standards. Statistical information and graphical plots can be evaluated in the calibration window.

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9.13.4.2 To evaluate the effects of eliminating individual calibrations points from the curve, click on the point and it will be removed from the calibration calculation. Click on the point again to introduce it back into the calibration.

9.13.4.3 Evaluate the calibration curve. If all of the calibration points meet the analysis requirements, the calibration should be saved under a file name with a similar identification to the corresponding method file (i.e., 0900150 for analysis consisting of the 150th sequence analyzed in 2009).

9.13.5 Starting an Automated Analysis

9.13.5.1 Highlight the samples to be analyzed in the "Sample" window. Then, click on "Analyze Batch".

9.13.5.2 Immediately following the calibration, an Initial Calibration Blank (ICB should be analyzed. This demonstrates that there is no carryover of the analytes of interest and that the analytical system is free from contamination.

9.13.5.3 Immediately following the ICB, a calibration verification should be analyzed. This is called the Initial Calibration Verification (ICV).

9.13.5.4 A minimum of three reagent blanks should be analyzed following the ICV. These blanks are called Instrument Blanks (IBL).

9.13.5.5 Two certified reference materials (CRM) should be analyzed following the IBWs. Common CRMs used for the analysis of waters are NIST 1640 (fresh water), NIST 1643e (river water), and CRM TMDW (drinking water). If analyzing seawater samples, CRM TMSW can be used. If analyzing for hardness, then CRM WasteWatR Hardness should be analyzed.

9.13.5.6 A continuing calibration verification (CCV) standard should be analyzed every ten samples and at the end of the run. The CCV standard should be the mid-point calibration standard.

9.13.5.7 A blank should be analyzed after each CCV, this blank is called a continuing calibration blank (CCB). This demonstrates that there is not carryover and that the analytical system is free from contamination.

9.13.5.8 For every 10 client samples, prepare and analyze a matrix spike (MS) and a matrix spike duplicate (MSD). Matrix spikes should be spiked at a level two to five times the background concentration or at the practical quantitative limit (PQL), whichever is greater.

9.13.5.9 For every 10 client samples, prepare and analyze a matrix duplicate (MD) sample.

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9.13.5.10 Method of Standard Additions (MSA) curves must also be performed any time that matrix interference is suspected, such as when matrix spikes or post-digestion spikes fail to meet acceptance criteria. In general, a three point MSA curve for solid digests should be prepared.

9.13.5.11 At the end of the run, flush the system with a rinse, DIW, and air. These steps will rinse out the instrument and sample introduction system, remove any acids that could wear down the sample introduction system, and dry the sample introduction system.

9.13.5.12 When the sample information file is complete, highlight all remaining samples to be analyzed. Go to the "Scheduler" window and select the "Analyze Samples" and "Autostop" tabs. In the Details column select the relevant Sample and Dataset file. Click "Start". The instrument will shut down the plasma, stop the peristaltic pump action, and return the autosampler probe to the rinse vial position after the last sample is analyzed. The auto stop can be selected at any time during analysis.

9.13.5.13 The plasma can be extinguished at any time by clicking plasma "Stop" on the front panel of the instrument page or, if need be, manually by pressing the yellow "off" button on the front of the instrument.

9.13.6 Exporting and Reprocessing Instrument Data

9.13.6.1 Reprocess the data in the "Dataset" window by highlighting the runs and clicking "Reprocess" after analysis has finished.

9.13.6.2 Click on the report tab on the far right hand side of the method window to edit the instrument outputs.

9.13.6.3 To print a copy of the instrument data select "Send to printer", and that the appropriate printer is selected. To print a calibration report select "1-CalReport" as the Report Output and reprocess any run. To print a copy of the raw data select "Quant-1" as the Report Output.

9.13.6.4 To export data to LIMS select the "Send to file" box and select "Processforinput1wcalcoeff" as the report output file. Name the produced report by the following: Data\(year_data)\Instrument Files – seq\Sequence Number, i.e., \\Curie\lab\Data\09_data\Instrument Files - seq\0900150.

9.13.6.5 Return to the dataset window and select all relevant runs, click "Reprocess."

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10.0 PROCEDURE FOR ANALYSIS USING DYNAMIC REACTION CELL (DRC)

10.1 For elements that suffer from isobaric or polyatomic interferences (see section 4.2), it is advantageous to utilize the DRC mode operation of the instrument. For non-interfered ions, the DRC mode can be optimized to enhance the standard mode performance.

10.2 When the reaction cell is pressurized with a reactive gas, the polyatomic or isobaric interferants can be converted to a different mass where they no longer interfere with the correct measurement of a desired element or the reaction gas will covert the desired element of interest to a mass which is not interfered upon. Chemical processes that occur in the DRC include charge transfer, proton transfer, and condensation and association reactions. The following procedure describes the process involved with running samples while utilizing the dynamic reaction cell to modify the ion stream chemistry.

10.3 The ELAN DRC II ICP-MS can be operated in three ways.

10.3.1 Using the dynamic reaction cell. The reaction cell is pressurized with a reaction gas (i.e., ammonia or oxygen).

10.3.2 With the dynamic reaction cell vented. In this manner, the instrument will emulate a conventional ICP-MS.

10.3.3 Both modes in sequence. Sample information can sequentially be collected in DRC mode and then in standard mode.

10.4 Interferences in DRC Mode

10.4.1 Isobaric interferences from doubly charged ions at the same m/z as described in section 4.2. can still cause interferences in DRC mode. It is important to ensure that the double-charged ratio associated with Ba^{++}/Ba is less than 2.5% when performing the daily performance check. A daily performance check in DRC mode can be done to verify low levels of Ba^{++}.

10.4.2 Isobaric interferences from polyatomic species are, when optimized properly, reduced to background levels in DRC mode. Reaction chemistry can result in an increase in background levels when not optimized. Specific chemistry within the cell should be understood thoroughly to perform quality analyses.

10.4.3 DRC Gas

10.4.3.1 The DRC gases are separated into two channels. Ammonia is in channel A and is referred to as Cell Gas A. Other gases, typically Oxygen, are in channel B and are called Cell Gas B. Ammonia passes through a "Getter" to dry the ammonia and

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remove trace levels of Oxygen. Cell Gas B bypasses the getter and goes directly to the DRC. Be extra careful to not reverse the gases, as this will contaminate the getter.

10.4.3.2 The type of DRC gas will remove interferences differently. This technology still has not been extensively researched and it is still unknown how certain analytes will behave with certain gases. A guideline of possible DRC gases and their relative effectiveness on certain analytes can be found on the last page of the ELAN DRC Accessory Training Manual.

10.4.4 Optimization using DRC

10.4.4.1 Optimization of the sample introduction system, plasma, and ion optics is the same for DRC mode as in standard mode. Follow sections 8.3 through 8.8. Proper optimization in standard mode will greatly enhance the DRC performance. Further optimization can be performed for a single element if needed (i.e., lens voltage at a single mass instead of using the autolens feature).

10.4.4.2 Following the daily (and full, if needed) optimization in standard mode, the reaction cell should be conditioned prior to optimization in DRC mode.

10.4.4.3 In the optimize window, select manual adjust and enter a value for either cell gas A (ammonia) or cell gas B (oxygen). A typical value will be around [redacted]. If using both gases for analysis, enter a value for cell gas A only. Allow the reaction cell to condition in this manner for at least [redacted]. If the cell has not been pressurized for a long period, or if there are problems, conditioning for [redacted] may be necessary.

10.4.4.4 Performing a DRC mode daily performance check will ensure the proper functioning and sensitivity of the instrument and will verify the sensitivity of isotopes that are interfered with by plasma-based interferences.

10.4.4.4.1 Open the template method file for the desired analysis, possibly "As Se DRC."

10.4.4.4.2 Enter the method window and confirm the presence of the analytes of interest to be determined and save.

10.4.4.4.3 For the analytes added to the method, verify the cell gas flow rate and the RPq are set to what is typical. If these values are unknown (i.e., pre-method development), then do not perform the daily performance.

10.4.4.4.4 Aspirate a solution containing 1 ppb of the analytes of interest.

10.4.4.4.5 Open the sample window and click analyze sample.

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10.4.4.4.6 Compare the intensity of the DRC mode analytes with previous DRC mode instrument performance data to verify that the interference reduction is acceptable.

10.4.4.4.7 If the performance check is satisfactory, the instrument is ready to begin sample analysis.

10.4.4.5 Method development is an integral part of running samples utilizing the DRC feature. Method development includes optimizing a reaction cell gas flow and an RPq parameter. Response to the reaction cell will vary greatly with sample matrix, so it is important that for every new sample matrix ran in DRC mode an appropriate reaction gas, cell gas flow rate, and RPq parameter is found. Once a method has successfully been developed for a particular sample matrix, the cell gas flow and RPq value should not change.

10.4.4.6 Optimizing DRC Parameters

10.4.4.6.1 Optimizing the cell gas flow in DRC mode requires a matrix- matched blank and a matrix matched standard spike [redacted] of the analyte of interest.

10.4.4.6.2 Open the method "1-DRC Method Development.mth". Modify the method so it lists only the analytes of interest. A convenient first approximation for the stability parameter RPq and the cell gas flow rates are [redacted]

10.4.4.6.3 In the "Optimize" window, select the "Auto Optimize" tab and Click "Get Analyte List." In the Parameter Description area, select the Cell Gas or RP value to be optimized. In the "Parameter Range" area select a range of values to be evaluated and the step value used. In the "Optimization Criteria" area select "Ramp."

10.4.4.6.4 Move the sample probe into the desired matrix blank and allow sufficient time for the sample to be aspirated.

10.4.4.6.5 Click "Optimize" to begin the optimization

10.4.4.6.6 When finished, move the sample probe to the matrix matched spiked solution.

10.4.4.6.7 In the sample window, click on the manual tab and enter a spike related ID that will be easily identifiable later.

10.4.4.6.8 Click "Optimize" to begin the optimization.

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10.4.4.6.9 When the second optimization is finished, go to the "Interactive" window, choose "Cell Param Optimize" from the drop-down list and click composite.

10.4.4.6.10 In the cell gas optimize options dialog, click the select buttons to locate and open the blank and spiked sample from the appropriate dataset folder.

10.4.4.6.11 Type in the concentration of the spiked matrix matched sample.

10.4.4.6.12 Click "Calculate".

10.4.4.6.13 Go to the interactive graphics window and choose the analyte of interest from the drop-down list.

10.4.4.6.14 Three plots appear in the interactive window. The matrix blank, matrix spike, and estimated detection limit.

10.4.4.6.15 Select the flow rate that gives the best-estimated detection limit while still maintaining an acceptable number of counts. The counts will depend on the ionization potential of the element. Put the flow rate in method. Alternate between Gas Flow Optimizations and RPq Optimizations until settings have been found which give the required interference resistance and sensitivity.

10.4.4.7 Little or no adjustment is necessary for the RPa value because most sequential chemistry takes place at a lower mass than the analyte. [redacted] The RPa can be used to analyze samples which would normally saturate the detector. This should be done with very careful consideration. Further information can be found in the instrument manual.

10.4.4.8 The Cell Path Voltage (CPV), Cell Rod Offset (CRO), and Quadrupole Rod Offset (QRO) are voltage settings that are optimized at installation and generally do not have to be adjusted. Refer to sections 4-81 through 4-83 of reference 11.1 for procedures on optimizing these settings.

10.4.4.9 Once appropriate values for the reaction cell gas flow and RPq are obtained they should be entered into the method in the timing page and in the cell parameters in the optimize window.

10.4.4.10 In the early stages of method development, it is useful to set up the method to analyze several replicates of a mass using [redacted].

10.5 Current DRC methods – New DRC methods are being developed for different analyte/matrix combinations on a regular basis. The table below is not intended to be all-inclusive, but only a representation of what methods have been fully developed and validated as of the last revision date of this SOP.

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Analyte/Internal Standard	Gas	Matrix	RPq	Cell Gas Flow
^{91}(AsO)/		All Matrices		
^{78}Se/		All Matrices		
^{80}Se/		All Matrices		
^{109}Ag/		All Matrices		
^{26}Al/		All Matrices		
^{52}Cr/		All Matrices		
^{53}Cr/		All Matrices		
^{63}Cu/		All Matrices		
^{62}Ni/		All Matrices		
^{62}Ni/		All Matrices		
^{51}V/		All Matrices		
^{66}Zn/		All Matrices		
^{66}Zn/		All Matrices		

10.5.1 As every matrix may have slight variance, analysis should include more isotopes of the same element if possible and RPq and cell gas flows should be measured for each analyte to ensure best DRC conditions.

10.5.2 The table above lists the general cell conditions optimized for multi-analyte analyses. Cell conditions, especially cell gas flow rates, may be optimized for specific analyte/matrix combinations, as needed. In such cases, the optimized methods will often have slightly different RPq and cell gas flow values.

11.0 QUALITY CONTROL

11.1 All BRL quality control criteria are summarized in Section 16.0. Project specific quality control requirements may differ and always take precedence over BRL requirements. Refer to Addendum A for additional project specific criteria currently used at BRL.

11.2 Preventative maintenance - The ICP-MS daily optimization logbook lists routine maintenance and checks that should be performed on a daily basis. Refer to section 4 of the ELAN DRC II hardware guide for details on daily, weekly, and monthly preventative maintenance. All maintenance should be documented in the ICP-MS daily optimization logbook.

11.3 Instrument optimization is an integral part of quality control. Sometimes optimizing the instrument takes the whole day.

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11.4 Pipettes must be calibrated weekly, or whenever the status of a pipette is unknown, checked daily. Refer to BRL SOP BR-1205 for further instruction on the calibration of pipettes.

11.5 The correlation coefficients of the weighted-linear calibration curves for each element must be $\geq$ 0.995 to proceed with sample analysis. If the correlation coefficient for any element is < 0.995, the results for that element may not be reported from that run. If high standards begin to decrease in sensitivity and do not fall into the linear regression, this might indicate that a cross calibration of the dual detector is needed and a full optimization would be required.

11.6 The percent recovery of the ICV standard must be 85-115% for each element being determined. If the percent recovery is not within the control limits of 85-115%, for any element, the results for that element may not be reported from that run, unless approved by both the Quality Assurance Officer and the Laboratory Manager.

11.7 Instrument rinses should be performed after any samples suspected to be high in metals and before any method blanks to ensure baseline sensitivity has been achieved.

11.8 Each analytical or digestion batch must have at least 3 preparation (or method) blanks associated with it if method blank correction is to be performed (1 method blank may be acceptable if sample results are not being blank corrected). The blanks are treated the same as the samples, and must go through all of the preparative steps. All of the samples should be corrected for the mean concentrations of these blanks. Three times the standard deviation of these blanks is the estimated method detection limit (EMDL) for that batch. The EMDL is evaluated against the MDL determined as per 40 CFR 136. Ideally, the EMDL should be at or below the MDL.

11.9 For every 10 client samples, a matrix duplicate (MD) sample should be analyzed. Generally, the relative percent difference (RPD) for the replicate should be $\leq$ 20% for waters and $\leq$ 30% for solids and seawaters prepared by RP or Co-APDC if the sample concentrations are greater than 5 times the MRL. A duplicate matrix spike (MSD) may be substituted for the MD, with the same control limits unless client-specific data quality objectives require an MD sample.

11.10 For every 10 client samples, a matrix spike (MS) and a matrix spike duplicate (MSD) should be performed. The percent recovery of the spikes should be 75-125% with an RPD $\leq$ 20% for waters and 70-130% with an RPD $\leq$ 30% for solids and seawaters prepared by RP or Co-APDC. If the spike recovery is outside of the control limits, a MSA curve that has been prepared and analyzed may be used to correct for the matrix effect. Samples may be corrected by the slope of the MSA curve if the correlation coefficient of the MSA curve is $\geq$ 0.995. If the sample concentration levels are sufficiently high, the sample may be diluted to reduce the matrix effect. Spike sets should be performed at the same dilution factor as the native sample. Spike at 1-5 times the level of a historical sample of a client, or if unknown, spike 1-5 times a typical value for the matrix; however, spiking levels should also be no

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lower than 5x the MRL. For multi-element analyses, allowances can be made for spiking with mixed standards.

11.11 The percent recoveries of the CRMs should be 75-125%. If the results are not within control limits for each analyte, the sample results may not be reportable. However, it should be noted that for sediment and soil CRMs, nearly all CRMs are certified for "total" metals; whereas, BRL typically prepared these types of samples with a reverse aqua regia (RAR) bomb digestion, which produces results for "total recoverable" metals. For some metals that are less labile (i.e., Cr, Ba, etc.), CRM recoveries may be low and outside of the control limits; however, this would not cause concern for the validity of the data and would not require any corrective action. The Addendum to the Certificates of Analysis for NIST SRMs 2709, 2710, and 2711 do a good job of explaining this issue clearly, and offer a good guide for comparing recovery results for total and total recoverable metals.

11.12 The percent recoveries of the CCV standards should be within 75-125%. If the CCV results are not within the control limits for any analyte, the sample results may not be reportable for that element.

11.12.1 Unless otherwise instructed by the client, sample results may be CCV corrected using the mean recovery of the bracketing CCVs. Only after careful evaluation of the data should this be done. The instrument should show a trending drift of CCV recoveries and not just a few anomalous outliers. The project manager or group leader should be consulted before CCV correcting data.

11.13 CCBs should be monitored for the effects of carry-over and for possible system contamination. If carry-over of the analyte at levels greater than 10 times the MDL is observed, the sample results may not be reportable.

11.14 Results for samples prepared with reductive precipitation (RP) and APDC extractions may be corrected by an efficiency factor that is calculated quarterly based on the previous year's recovery data for CRMs, blank spikes, and MS/MSDs.

11.15 The absolute response of any one internal standard should not vary from the original response in the calibration blank by more than 60-125%. Some analytical samples, such as those containing concentrations of the internal standard and tissue digestates, can have a serious effect on the internal standard intensities, but this does not necessarily mean that the analytical system is out of control. In some situations, it is appropriate to reprocess the samples using a different internal standard monitored in the analysis. Careful evaluation of the data and notification to the project manager should be given before doing this.

12.0 METHOD PERFORMANCE

12.1 All method detection limits (MDLs) were achieved by performing a full MDL study as described in 40 CFR 136, Appendix B. The MDLs for all analytes are listed in the BRL Analytical Services Table and are available upon request, as are the data from the actual

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MDL studies. The quality control acceptance criteria listed in Tables 16.1 and 16.2 are developed from the EPA methods and are validated as achievable at BRL in control charts maintained for the method.

13.0 POLLUTION PREVENTION

13.1 Whenever feasible, lab personnel should use pollution prevention techniques to limit waste generation. The cost involved in purifying used acids makes such recycling unpractical at BRL. Instead, every effort is made to reduce any volumes necessary to still produce the best possible results. This analysis requires very small volumes to analyze sample preparations and standards. Standards should be prepared in volumes consistent with the laboratory use to minimize the volume of disposed standards to be disposed.

14.0 WASTE MANAGEMENT

14.1 All waste is disposed of in accordance with state and federal regulations either by sewer disposal (only if concentrations are less than the King County sewer limits) or through a licensed hazardous waste disposal facility such as Philips Services.

15.0 REFERENCES

15.1 Software Guide-ELAN Version 3.0, Perkin-Elmer part number WE1002821, 2003 Edition Perkin-Elmer/MDS SCIEX instruments, 71 Four Valley Drive, Concord, ON L4K 4V8.

15.2 Hardware Guide-ELAN DRC II, Perkin-Elmer part number WE1002727A, 2001, Perkin-Elmer Instruments LLC, 710 Bridgeport Avenue, Shelton, Connecticut, 06840.

15.3 "Safety and Health Core Rules" OSHA regulation for Washington State, Chapter 296-800 WAC.

15.4 "ELAN DRC Accessory Training Manual". Perkin-Elmer, N0225044, Revision A, 2005.

15.5 EPA SW-846 Method 6020. "Inductively Coupled Plasma –Mass Spectrometry." Revision 2. December 1996.

15.6 EPA Method 1638. "Determination of Trace Elements in Ambient Waters by Inductively Coupled Plasma – Mass Spectrometry." January 1996.

15.7 EPA Method 1640. "Determination of Trace Elements in Ambient Waters by On-Line Chelation Preconcentration and Inductively Coupled Plasma – Mass Spectrometry." January 1996.

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15.8 EPA Method 200.8, Revision 5.5. " Determination of Trace Elements in Waters and Wastes by Inductively Coupled Plasma – Mass Spectrometry." October 1999.

15.9 EPA Method 6020A, Revision 1. "Inductively Coupled Plasma – Mass Spectrometry." SW-846 Methods. February 2007.

15.10 NIST Certificate of Analysis for SRM 2709 – San Joaquin Soil, National Institute of Standards and Technology, Gaithersburg, Maryland, 2003.

15.11 CDC Laboratory Procedure Manual, Urine Multi-Element ICP-DRC-MS, Method No. ITU001B, February 16, 2006.

15.12 CDC Laboratory Procedure Manual, Blood Lead and Cadmium ICP-DRC-MS, Method No. ITB001A, September 9, 2004

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16.0 TABLES

Table 16.1 BRL QA Criteria for Water Samples Prepared by BR-0065 and Analyzed by BR-0060

QC Sample	Measure	Minimum Frequency	Criteria	Corrective Action
Calibration Standards	Linearity of the calibration curve	Analyzed once per analytical day; Minimum of 5 calibration points	Correlation coefficient ≥ 0.995, 1st standard $\leq$ PQL, low standard recovery = 75-125%, all other standard recoveries = 80-120%	Reanalyze suspect calibration standard. High calibration standard(s) may be omitted as long as all samples results are still below the highest calibration standard use. If criteria still not met, then re-prepare standards and recalibrate the instrument.
Internal Standards	Variation in sample properties between samples and standards	Each standard, blank, and sample is spiked with internal standard	60-125% recovery compared to calibration blank	If the responses of the internal standards in the following CCB are within the limit, rerun the sample at an additional 2x dilution. If not, then samples must be re-analyzed on a new calibration.
Interference Check Solution (ICS)/Certified Reference Material (CRM)	Check the validity of the interference correction and method accuracy	At the beginning of each analytical run	All non-certified analytes < RL; other analytes within ± 25% or ± RL if < 5x RL	If CRM true value is $\geq$ 5x the MRL and if the recovery is outside of the control limits, then halt analysis, identify and correct problem, recalibrate if necessary, and reanalyze affected samples.
Independent Calibration Verification (ICV)	Independent check of system performance	1 following instrument calibration	Recovery = 85-115%	Correct problem prior to continuing analysis, recalibrate if necessary
Check Calibration Verification (CCV)	Accuracy	1 per 10 sample preparations and at the end of the analytical run	Recovery = 75-125%	Halt analysis, correct problem, recalibrate, and reanalyze affected samples. CCV-correction may be allowed. Note: If internal standards are not run, then CCV standards must be analyzed at various concentrations.
Method Blanks (MB)	Contamination from reagents, lab ware, etc.	Minimum of 3 per batch	Mean $\leq$ PQL; SD $\leq$ MDL or MBs <1/10th sample result	Reanalyze to confirm results. MBs may be omitted if they meet the Grubb's Outlier test and if a minimum of 3 MB remain. If MBs are still high following rejection of any Grubb's outlier, then a batch specific MDL is estimated (EMDL) by multiplying the standard deviation of the remaining MBs by 3x. The batch specific MRL is estimated (EMRL) by multiplying the EMDL by 3x. All results are evaluated against the EMDL and EMRL, and data is qualified if necessary.
Method Duplicates (MD)	Method precision within a given matrix	Minimum of 1 per 10 client samples (prepared at instrument)	RPD $\leq$ 20% or ±PQL if results $\leq$ 5x PQL	If **RPD** criteria not met, then sample may be reanalyzed, but this is not required. Sample matrix may be inhomogeneous, in which case the results would be qualified.
Matrix Spike/ Matrix Spike Duplicate (MS/MSD)	Method accuracy and precision within a given matrix	Minimum of 1 per 10 client samples (prepared at instrument)	Recovery = 75-125% and RPD $\leq$ 20%	If **MS/MSD** criteria not met, samples must be qualified. Samples may be reanalyzed at a greater dilution, but this is not required. Matrix effects may be present, in which case the results would be qualified. If **RPD** criteria not met, then sample may be reanalyzed, but this is not required. Sample matrix may be inhomogeneous, in which case results would be qualified.

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Table 16.2 BRL QA Criteria for Seawater Samples Prepared by BR-0066/BR-0069 and Analyzed by BR-0060

QC Sample	Measure	Minimum Frequency	Criteria	Corrective Action
Calibration Standards	Linearity of the calibration curve	Analyzed once per analytical day; Minimum of 5 calibration points	Correlation coefficient ≥ 0.995, 1st standard ≤ PQL, low standard recovery=75-125%, all other standard recoveries=80-120%	Reanalyze suspect calibration standard. If criteria still not met, then reprepare standards and recalibrate the instrument
Internal Standards	Variation in sample properties between samples and standards	Each standard, blank, and sample is spiked with internal standard	60-125% recovery compared to calibration blank	If the responses of the internal standards in the following CCB are within the limit, rerun the sample at an additional 2x dilution. If not, then samples must be re-analyzed on a new calibration.
Interference Check Solution (NIST 1643e) (NIST 1640)	Check the validity of the interference correction	At the beginning of each analytical run	All non-certified analytes <RL; other analytes within ±25% or ±RL if <5x RL	Halt analysis, identify and correct problem, recalibrate if necessary, and reanalyze affected samples
Independent Calibration Verification (ICV)	Independent check of system performance	1 following instrument calibration	Recovery = 85-115%	Correct problem prior to continuing analysis, recalibrate if necessary
Check Calibration Verification (CCV)	Accuracy	At beginning and end and 1 per 10 sample preparations	Recovery = 75-125%	Halt analysis, correct problem, recalibrate, and reanalyze affected samples (Note: If internal standards are not run, then CCV standards must be analyzed at various concentrations.)
Method Blanks (MB)	Contamination from reagents, lab ware, etc.	Minimum of 3 per batch	Mean ≤ PQL; SD ≤ MDL or MBs <1/10th sample result	Reanalyze to confirm results. MBs may be omitted if they meet the Grubb's Outlier test and if a minimum of 3 MB remain. If MBs are still high following rejection of any Grubb's outlier, then a batch specific MDL is estimated (EMDL) by multiplying the standard deviation of the remaining MBs by 3x. The batch specific MRL is estimated (EMRL) by multiplying the EMDL by 3x. All results are evaluated against the EMDL and EMRL, and data is qualified if necessary.
Method Duplicates (MD)	Method precision within a given matrix	Minimum of 1 per 10 client samples	RPD ≤ 30% or ±PQL if results ≤ 5x PQL	If **RPD** criteria not met, then sample may be reprepared and reanalyzed, but this is not required. Sample matrix may be inhomogeneous. A post-digestion duplicate (PDD) can be analyzed to evaluate instrument precision.
Matrix Spikes/ Matrix Spike Duplicates (MS/MSD)	Method accuracy and precision within a given matrix	Minimum of 1 per 10 client samples	Recovery = 70-130% and RPD ≤ 30% (may be adjusted to accommodate efficiency factors for RP and APDC preps)	If **RPD** > 25%, results must be qualified. If **RPD** criteria not met, then sample may be reprepared and reanalyzed, but this is not required. Sample matrix may be inhomogeneous. A post-digestion spike (PDS) can be analyzed to evaluate instrument accuracy and also to determine if matrix interference is occurring at the prep stage or at the analytical stage. An MSA curve may be prepared and analyzed along with the samples to quantify and correct for interference.
Post-Digestion Spike (PDS)	Check for matrix interference	When required (samples spiked too low/high, dilution test fails, etc.)	Recovery = 75-125%	Samples must be analyzed using MSA or results flagged accordingly.
Laboratory Fortified Blanks (LFB)	Method accuracy	When no suitable CRM available	Recovery = 75-125%	If LFB recovery is outside of the control limit, then batch must be reprepared and reanalyzed.
Certified Reference Material (CRM)	Method accuracy	Must be matrix matched to samples; Minimum of 1 per batch	Recovery = 75-125% unless limits set by CRM manufacturer are greater or element/CRM specific limits have been established	If CRM true value is ≥5x the PQL and if the recovery is outside of the control limit, then batch must be reprepared and reanalyzed.

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Table 16.3 BRL QA Criteria for Sediment and Biota Samples Analyzed by BR-0060

QC Sample	Measure	Minimum Frequency	Criteria	Corrective Action
Calibration Standards	Linearity of the calibration curve	Analyzed once per analytical day	Correlation coefficient ≥ 0.995, 1st standard ≤ PQL, low standard recovery=75-125%, all other standard recoveries=80-120%	Reanalyze suspect calibration standard. If criteria still not met, then reprepare standards and recalibrate the instrument
Internal Standards	Variation in sample properties between samples and standards	Each standard, blank, and sample is spiked with internal standard	60-125% recovery compared to calibration blank	If the responses of the internal standards in the following CCB are within the limit, rerun the sample at an additional 2x dilution. If not, then samples must be re-analyzed on a new calibration.
Interference Check Solution (ICS)	Check the validity of the interference correction	At the beginning of each analytical run	All non-certified analytes <RL; other analytes within ±25% or ±RL if <5x RL	Halt analysis, identify and correct problem, recalibrate if necessary, and reanalyze affected samples
Independent Calibration Verification (ICV)	Independent check of system performance	1 following instrument calibration	Recovery = 85-115%	Correct problem prior to continuing analysis, recalibrate if necessary
Check Calibration Verification (CCV)	Accuracy	At beginning and end and 1 per 10 sample preparations	Recovery = 75-125%	Halt analysis, correct problem, recalibrate, and reanalyze affected samples
Method Blanks (MB)	Contamination from reagents, lab ware, etc.	Minimum of 3 per batch	Mean ≤ PQL; SD ≤ MDL or MBs <1/10th sample result	As per with sediments (Table 16.3).
Method Duplicates (MD)	Method precision within a given matrix	Minimum of 1 per 10 client samples	RPD ≤ 30% or ± 2x PQL if results ≤ 5x PQL	If **RPD** criteria not met, then sample may be reprepared and reanalyzed, but this is not required. Sample matrix may be inhomogeneous. A post-digestion duplicate (PDD) can be analyzed to evaluate instrument precision.
Matrix Spikes/ Matrix Spike Duplicates (MS/MSD)	Method accuracy and precision within a given matrix	Minimum of 1 per 10 client samples	Recovery = 70-130% and RPD ≤ 30%	If **RPD** > 30%, results must be qualified. If **RPD** criteria not met, then sample may be reprepared and reanalyzed, but this is not required. Sample matrix may be inhomogeneous. A post-digestion spike (PDS) can be analyzed to evaluate instrument accuracy and also to determine if matrix interference is occurring at the prep stage or at the analytical stage. An MSA curve may be prepared and analyzed along with the samples to quantify and correct for interference.
Post-Preparation Spike (PPS)	Check for matrix interference	When required (samples spiked too low/high, dilution test fails, etc.)	Recovery = 75-125%	Samples must be analyzed using MSA or results flagged accordingly.
Laboratory Fortified Blank (LFB) or Blank Spike (BS)	Method accuracy	Minimum of 1 per batch	Recovery = 75-125%	If LFB recovery is outside of the control limit, then batch must be reprepared and reanalyzed.
Certified Reference Material (CRM)	Method accuracy	Must be matrix matched to samples; minimum of 1 per batch	Recovery = 75-125% unless limits set by CRM manufacturer are greater or element/CRM specific limits have been established	If CRM true value is ≥5x the PQL and if the recovery is outside of the control limit, then batch must be reprepared and reanalyzed. See comments in section 10.11.

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Addendum A: Client Specific QA Criteria

Table A1. Department of Defense Quality Systems Manual, Version 4.2, Requirements

QC Sample	Measure	Minimum Frequency	Criteria	Corrective Action
Instrument Tuning	Optimization of the instrument	Prior to ICAL each day	Mass Cal ≤ 0.1 amu from true; resolution < 0.9 amu full width at 10% peak height. Stability, RSD ≤ 5% for 4 replicates.	Retune instrument. Flagging of data is not acceptable. No analysis may be performed without a valid MS tune.
Calibration Standards	Linearity of the calibration curve	Analyze once per analytical day	Correlation coefficient ≥ 0.995, 1st standard ≤ MRL with ± 20% rec.	Reanalyze suspect calibration standard. If criteria still not met, then reprepare standards and recalibrate the instrument
Internal Standards	Variation in sample properties between samples and standards	Each standard, blank, and sample is spiked with internal standard	30-120% recovery compared to calibration blank	If the responses of the internal standards in the following CCB are within the limit, rerun the sample at an additional 2x dilution. If not, then samples must be re-analyzed on a new calibration.
Interference Check Solution ICS-A ICS-AB	Check the validity of the interference correction	At the beginning of each analytical run	All non-spiked analytes < LOQ*; other analytes within ±20% or ±RL if <5x RL	Halt analysis, identify and correct problem, recalibrate if necessary, and reanalyze affected samples
Independent Calibration Verification (ICV)	Independent check of system performance	1 following instrument calibration	Recovery = 90-110%	Correct problem prior to continuing analysis, recalibrate if necessary
Check Calibration Verification (CCV)	Accuracy	At beginning and end and 1 per 10 sample preparations	Recovery = 90-110%	Halt analysis, correct problem, recalibrate, and reanalyze affected samples (Note: If internal standards are not run, then CCV standards must be analyzed at various concentrations.)
Calibration Check Blank (CCB)	Contamination at the instrument	Following each CCV	< LOQ*	Determine and eliminate cause of contamination. Affected samples must be reanalyzed.
Method Blanks (MB)	Contamination from reagents, lab ware, etc.	Minimum of 3 per batch	≤ ½ RL or ≤ 10% of any sample. Or ≤ 10% of regulatory limit.	Determine and eliminate cause of contamination. Affected samples must be reprepared and reanalyzed.
Method Duplicates (MD)	Method precision within a given matrix	Minimum of 1 per 10 client samples	RPD ≤ 20% or results within the PQL if < 5x PQL	If **RPD** criteria not met, then sample may be reprepared and reanalyzed, but this is not required. Sample matrix may be inhomogeneous. A post-digestion duplicate (PDD) can be analyzed to evaluate instrument precision.
Matrix Spikes/ Matrix Spike Duplicates (MS/MSD)	Method accuracy and precision within a given matrix	Minimum of 1 per 10 client samples	Recovery = 80-120%; RPD ≤ 20%	If **RPD** > 20%, results must be qualified. If **RPD** criteria not met, then sample may be reprepared and reanalyzed, but this is not required. Sample matrix may be inhomogeneous. A post-digestion spike (PDS) can be analyzed to evaluate instrument accuracy and also to determine if matrix interference is occurring at the prep stage or at the analytical stage.
Dilution Test	Check for matrix interference	Minimum of 1 per batch, sample > 50x LOQ/MRL	Five-fold dilution must be within ±10% of original result	Perform post-digestion spike addition.
Post-Digestion Spike (PDS)	Check for matrix interference	When dilution test fails or all sample results <100x MDL	Recovery = 75 - 125%	Samples must be analyzed using MSA or apply J qualifier to all samples with same matrix for the specific analyte(s) associated with the post-digestion spike addition.
Laboratory Fortified Blanks (LFB)	Method accuracy	When no suitable CRM available	Recovery = 80 - 120%	If LFB recovery is outside of the control limit, then batch must be reprepared and reanalyzed.
Method of Standard Additions (MSA)	N/A	If post-digestion spike fails	Document use of MSA	N/A
Lab Control Sample (LCS) / Blank Spike (BS)	Method accuracy	One per batch containing all analytes to be reported	QC acceptance criteria specified by DoD, if available. Default of 80-120%	Correct problem and reprepare and reanalyze all samples for failed analytes if available sample mass. If cannot reprepare, then affected results are qualified and explained in the case narrative.
Standard Reference Material (SRM)	Method accuracy	Must be matrix matched to samples; minimum of 1 per batch	Recovery = 75 - 125%, unless limits set by SRM manufacturer are greater	If CRM true value is ≥5x the PQL and if the recovery is outside of the control limit, then batch must be reprepared and reanalyzed. See comments in section 10.11.

* DoD work at BRL is only reported down to the LOQ.

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Appendix A: Analytical Differences between EPA Methods 1638, 200.8, 6020, and Brooks Rand Labs SOP BR-0060.

EPA Method 1638	EPA Method 200.8	EPA Method 6020	SOP BR-0060
Method is performance based. Modifications are allowed if the laboratory can demonstrate that data quality is not affected. Method is in Draft (unpromulgated) status, and QC criteria are based on a limited set of data.	Method is promulgated and is not performance based.	Method is performance based. Modifications are allowed if the laboratory can demonstrate that data quality is not affected.	SOP is performance based. Modifications are allowed to improve data quality or to make allowances for non-routine samples, but all modifications must be clearly documented.
1.1-1.3 Method is written solely for ambient water samples and for a very limited number of metals (9).	1.1 Method applicable for 21 metals in waters, sludges, and soils. 11.3 Solids preparation involves a HNO3/HCl digestion on a hotplate.	1.3 Method applicable for 15 metals. A variety of preparation methods are used to most appropriately analyze different metals in waters, sludges, and soils. Preparation methods are documented in their own EPA methods including 3005, 3010, or 3015 for aqueous samples and 3050 or 3051 for solids.	8.1.2 A variety of preparation methods are used to most appropriately analyze different analytes and matrices. Preparation methods are documented in their own SOPs are not a part of this SOP.
7.7 Tuning solution should contain Be, Mg, Co, In, and Pb. Tuning should be performed daily until it's shown that instrument meets the performance criteria without tuning. Tuning solution should be analyzed at least 5 times with RSD of $\leq$ 10%.	7.7 Tuning solution should contain Be, Mg, Co, In, and Pb. Tuning of instrument is required daily, and tuning report is included with data.	5.8 Tuning solution should contain Li, Co, In, and Tl. Tuning of instrument is required daily, and tuning report is included with data (Resolution < 0.9 amu full width ant 10% peak height.) Tuning solution must be analyzed four times with RSD < 5%.	8.4 Tuning is performed as per the instrument manufacturer recommendations. Tuning solution contains Mg, In, Ce, Pb, and ambient ^{12}C and ^{76}Ar. Tuning is done at least monthly or as needed. Newer instrument do not require tuning as often.
9.7 Ongoing Precision and Recovery prepared with each batch.	7.9 Laboratory Fortified Blank (blank spike) prepared with each batch.	8.7 A Laboratory Control Sample (LCS) should be prepared and analyzed for each sample batch up to 20 samples or less. 8.8.3 Limits within (+/-) 10%.	8.11.5.4 Two Standard reference materials (SRMs) are prepared with each calibration. Additionally, an LFB and CRMs are prepared for solid and seawater matrices.
9.2.4 QCS (2^{nd} source standard) required quarterly.	9.2.3 QCS (2^{nd} source standard) required quarterly.	7.8 ICV (2^{nd} source standard) is required daily. Limits within (+/-) 10%.	10.6 ICV must be analyzed daily.

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EPA Method 1638	EPA Method 200.8	EPA Method 6020	SOP BR-0060
9.6.1 1 or more Laboratory Method Blank (MB) per batch of 20 samples. Blank correction allowed if minimum of 3 blanks are analyzed. Mean MB + 2 SD must be less than the regulatory compliance level.	9.3.1 1 Laboratory Reagent Blank (LRB) per batch of 20 samples. No blank correction performed. LRB must be < 10% of the sample concentration or < 2.2x the MDL.	5.5 One Calibration blank and one preparation blank must be performed. A rinse blank of 2% HNO3 must be analyzed between every calibration standard and sample. 8.8.4 The calibration blank must be < 3 times the current instrument IDL.	10.8 At least 3 Method Blanks (MB) per batch of samples (up to 60 samples). Blank correction performed. Mean MB must be < PQL with a SD < MDL.
9.3 MS/MSD go through entire preparation as a native sample.	9.4 MS/MSD go through entire preparation as a native sample.	8.6 Post Digestion Spike addition should be 75-125% of the known value or within laboratory derived acceptance criteria. 8.10 Analyze one duplicate sample per batch of 20 samples. RPD < 20%.	8.11.5.7 To estimate appropriate spiking levels of water samples, MS/MSD for water samples are spiked post-digestion. Water samples are digested in their original bottle. For solid samples, MS/MSD go through entire preparation process with native samples.
9.3 If there is no monitoring occurring, spike at 1-5 times the sample concentration or the ML, whichever is higher.	9.4 Spiking levels are set at 40-500 μg/L in water and 50 – 100 mg/kg in solids.	8.6 The Post Digestion Spike addition should be based on the indigenous concentration of the sample.	10.10 Spike at 1-5 times the level of a historical sample of a client, or if unknown, spike 1-5 times a typical value for the matrix. For multi-element analyses, allowances can be made for spiking with mixed standards.
9.6 Field and equipment blanks required.	8.5 Field and equipment blanks required.	Field and equipment blanks are not addressed.	Field and equipment blanks are recommended, but this is not included in the ICP-MS analysis SOP.
10.3 Internal standards include at least 3 of the following analytes: Li^6, Sc, Y, Rh, In, Tb, Ho, Lu, Bi. Control limits: 60-125%.	10.3 Internal standards include at least 3 of the following analytes: Li^6, Sc, Y, Rh, In, Tb, Ho, Lu, Bi. Control limits: 60-125%.	3.3 Internal standards include at least 3 of the following analytes: Li^6, Sc, Y, Rh, In, Tb, Ho, Bi. Control limits: 30-120%. If internal standard fails criteria, reanalyze at a fivefold dilution.	6.6 Internal standards typically used are Li^6, Sc, Ge, Rh, Tm, Lu, and In. These may be modified for interferences. DRC mode typically uses Ga and Rh as internal standards. Control limits: 60-125%.
10.4 Calibrations are based on the mean of calibration factors with an RPD < 15%.	10.4 At one or more concentration levels, the calibration is determined from an average response of 3 replicate integrations.	7.6 At one or more concentration levels within the linear dynamic range. The calibration is determined from an average response of 3 replicate integrations.	10.4 Calibrations are based on a weighted linear fit with R > 0.995. Control limits: 75-125% for lowest calibration standard; 80-120% for all other calibration points.

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EPA Method 1638	EPA Method 200.8	EPA Method 6020	SOP BR-0060
10.5 Calibration verification sample criteria is element specific.	9.3.4 CCVs within 15% of the calibration. 10-15% deviation required recalibration.	8.8.2 – 8.8.3 CCVs are run at beginning, after every 10 analyses, and at end. ICV must be 90-110%.	10.6 & 10.12-13 CCV criterion is within 25% of the calibration for all elements. ICV recovery is within 15% of the expected value. Samples may be CCV-corrected, if appropriate.
12.0 Total dissolved metals analysis requires the addition of HCl.	11.3 Samples with high turbidity requires additional HCl.	5.1 Concentrations of Sb and Ag between 50-500ugl/L require 1% HCl for stability.	7.3 All aqueous samples not prepared by reductive precipitation extraction are treated with a nitric acid digestion. No HCl is used.
13.8 Do not perform blank subtraction on sample results.	Blank correction not addressed.	Blank correction not addressed.	Results are routinely instrument and method blank corrected as per standard industry practices.
Interference check solutions not addressed. Sections 4.4.1 & 4.4.2 discuss use of specific isotopes free from isobaric elemental interferences and adjusting the spectrometer resolution to minimize potential wing overlap.	Interference check solutions not addressed. Sections 4.1.1 & 4.1.2 discuss use of specific isotopes free from isobaric elemental interferences and adjusting the spectrometer resolution to minimize potential wing overlap.	5.6 An Interference Check Solution should be analyzed following the calibration, every ten analytical samples and after the last sample.	Tables 12.1 & 12.2 NIST 1640 and NIST 1643e used as interference check solutions. Analyzed with each batch.
9.2.4 MDL should be determined annually, when a new operator begins work or whenever, in the judgment of the analyst, a change in analytical performance caused by either a change in instrument hardware or operating conditions would dictate.	9.2.1 MDLs should be determined when a new operator begins work or whenever, in the judgment of the analyst, a change in instrument hardware or operating conditions would dictate.	8.2 IDLs must be determined at least every 3 months and kept with the instrument log book.	MDL annually for waters and biennially for solids or whenever significant modifications made to the instrument or analytical procedure. IDOCs, including all of the elements of an IDL or MDL study, performed for each new analyst prior to analyzing client samples.
Specific dilution tests not discussed.	Specific dilution tests not discussed.	8.5 Dilution test (run at 5x, result 90-110%) required on 5% of samples.	Not routinely done as part of analysis. Is performed upon request.
Post-digestion spikes and MSA curve not discussed.	Post-digestion spikes and MSA curve not discussed.	8.6 Post-digestion spike with recovery of 75-125% required or MSA curve required.	10.12 As per SW-846 Method 6020.
18.15 Defines what a laboratory duplicate is, but the method does not define any requirements for its analysis.	3.7 Defines what a laboratory duplicate is, but the method does not define any requirements for its analysis.	8.10 One matrix dupe (MD) per 20.	10.9 One MD per every 10 samples, RPD $\leq$ 25% for waters and RPD $\leq$ 30%.
Table 1.2 Approved elements are Ag, Cd, Cu, Ni, Pb, Sb, Se, Tl, and Zn.	Table 1.1 Approved elements are Ag, Al, As, Ba, Be, Cd, Co, Cr, Cu, Hg, Mn, Mo, Ni, Pb, Sb, Se, Th, Tl, U, V, and Zn.	Table 1 Approved elements are Al, Sb, As, Ba, Be, Cd, Cr, Co, Cu, Pb, Mn, Ni, Ag, Tl, and Zn.	1.6 Validated for Ag, Al, As, B, Be, Ca, Cd, Co, Cr, Cu, Fe, K, Mg, Mn, Mo, Na, Ni, Pb, Sb, Se, Sn, Sr, Th, Ti, Tl, U, V, & Zn so far.

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SiREM
Leading Science · Lasting Solutions

130 Research Lane, Ste 2
Guelph ON N1G 5G3
(519) 822-2265

02 November 2012 SiREM Refs: Si-1725-110212

Evan Barman
Watermark Environmental, Inc.
175 Cabot Street
Lowell, MA 01854

via email: evan.barman@watermaekenv.com

Reference: Proposal and Cost Estimate for Laboratory ZVI Column Study

Dear Evan:

This proposal presents the scope of work and estimate of costs for SiREM to conduct a laboratory column study to assess the potential for zero valent iron (ZVI) to degrade chlorinated volatile organic compounds (cVOCs) (mainly cis-1,2-dichloroethene [cDCE] and vinyl chloride [VC]) in groundwater from the Site.

The bench scale column test methodology provides representative site-specific degradation rates for cVOCs in Site groundwater under flowing conditions (Gillham and O'Hannesin, 1994). Degradation rates will be used to calculate the required residence time in the reactive ZVI material. The field flow rate and residence time data will then be used to determine an appropriate treatment zone width. Influent and effluent water samples will be tested for detailed inorganic chemistry to provide information concerning potential mineral precipitation caused by changing redox potential (Eh) and pH conditions in the reactive ZVI material.

The remainder of this proposal presents the study approach and methodology (Section 1); the proposed reporting and schedule (Section 2); and the estimated costs (Section 3). The groundwater for the study will be provided by Watermark Environmental Inc., (Watermark). Sampling protocols, shipping requirements and required sample volumes will be provided upon acceptance of this proposal. The granular ZVI (to be specified by Watermark) for use in this study will be procured by SiREM.

1 STUDY APPROACH & METHODOLOGY

Task 1 – Site Groundwater Collection

Upon acceptance of this proposal, Watermark will collect groundwater representative of the target cVOCs at required treatment concentrations within the treatment zone. Groundwater will be shipped to SiREM in 1 gallon (4 Liter) sample bottles with no headspace. SiREM will require 6 gallons (~ 24L) of Site groundwater for this study.

Upon receipt, Site groundwater will be stored at 39°F (4°C) until required for column set up, at which time it will be siphoned using positive or negative pressure from the sample bottle into a collapsible, sterile Teflon® bag (American Durafilm). All precautions will be made to minimize

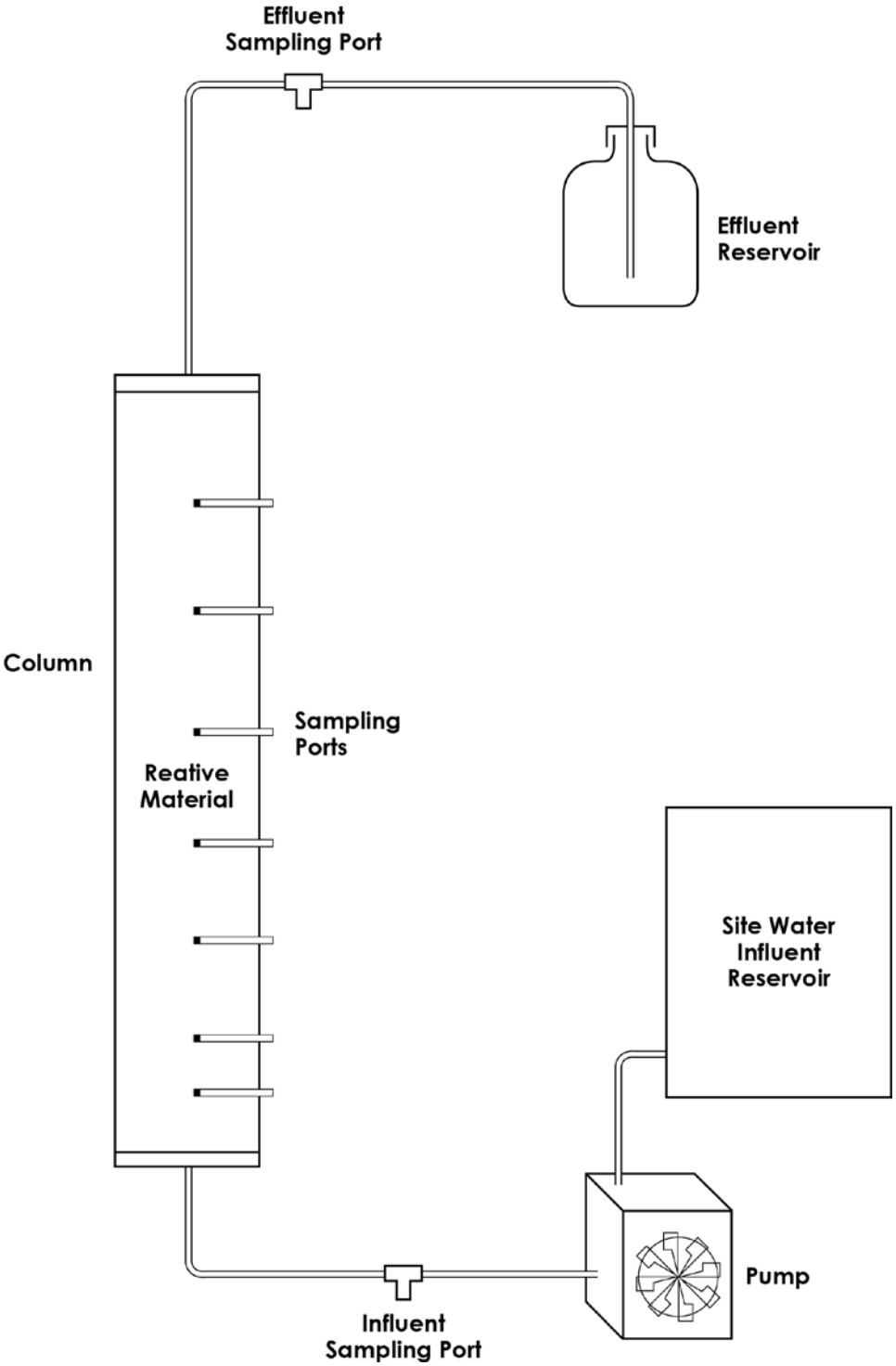
exposure to the ambient air and cVOC losses. Upon arrival, the water will be analyzed for cVOCs.

Task 2 – ZVI Column Construction

The column will be constructed of Plexiglas® with a length of 1.6 feet (ft) (50 centimeter [cm]) and an internal diameter of 1.5 inches (3.8 cm). Seven sampling ports are positioned along the length of the column at distances of 0.08 (2.4 cm), 0.16 (4.9 cm), 0.33 (10.1 cm), 0.50 (15.2 cm), 0.66 (20.1 cm), 1.00 (30.5 cm) and 1.31 ft (39.9 cm) from the inlet end (Figure 1). Sampling ports will be used frequently throughout the study to collect samples for analysis. Each sampling port is constructed of a nylon Swagelok® fitting (0.063 in; 0.16 cm) secured into the side of the column. Within each fitting, a 16-gauge needle (Fischer Scientific) will be tightly secured to allow for sampling from the center axis of the column. To prevent solid materials from entering the needle, glass wool is placed in the needle. Each sample port is fitted with a Luer-Lok™ fitting, such that a glass syringe can be attached to the port to collect a sample. When not in operation the ports are sealed by Luer-Lok™ plugs. Both the top and bottom of the column are sealed by rubber O-ring seals and contain fine (500 micron) mesh to inhibit solid material from entering the influent and effluent hose.

The ZVI material will be packed in vertical lift sections to assure a homogenize mixture. Values of bulk density, porosity and pore volume will be determined gravimetrically. All column experiments will be performed at room temperature unless otherwise specified.

Figure 1: Schematic of column apparatus



Task 3 - ZVI Column Operation

The column will operate for a period of six weeks. The Site water will be supplied to the influent end of the columns at a constant rate using a laboratory pump. The flow velocity will be determined through consultation with Watermark and selected to approximate *in situ* flow conditions for the treatment zone.

Task 4 - Column Sampling and Analysis

Water samples will be collected from the column influent, effluent and individual ports for analysis of cVOCs including cDCE and VC, dissolved hydrocarbon gases (DHGs, e.g., ethene, ethane or methane), Eh and pH on a weekly basis. Anions (including Cl, NO_3, and SO_4) will be collected from the column influent, effluent and individual ports at 3 sampling events. Other analyses will include cations (including total iron, Na, Mg, Ca, K, and Mn) and alkalinity (as milligrams $CaCO_3$/L) on samples collected from the column influent and effluent at the start and end of the study to help predict possible mineral precipitation. At time zero samples for all parameters will be collected from the influent groundwater reservoir to provide the time zero data for the study. Table 1 provides a summary of the sampling parameters and frequency.

Aqueous sampling will be conducted using a 1 milliliter (mL) glass syringe. For cVOC analyses, a 1.0 mL sample will be collected from the sampling port and transferred into a 10 mL auto sampler vial containing 5.0 mL of acidified deionized water (pH ~2). The water is acidified to inhibit microbial activity in the auto sampler vial. The vial will be sealed with an inert Teflon®-coated septum and aluminum crimp cap for immediate automated injection of 3 mL of headspace onto the GC. For anion analysis a 0.5 mL sample will be taken from the sampling port and transferred into a 1 mL eppendorf tube. The sample will be diluted in deionized water and a 25 µL sample will be injected into the ion chromatograph.

Eh will be determined using a combination Ag/AgCl reference electrode with a platinum button and a Corning 313 meter. The electrode is standardized with an Eh standard (Thermo Electron Corporation). Millivolt (mV) readings are converted to Eh, using the electrode reading and the standard potential of the Ag/AgCl electrode at a given temperature. The pH measurements are made using a combination pH/reference electrode and an Oakton™ meter, standardized with the pH buffer 7 and the appropriate buffer of either 4 or 10. A 3.0 mL sample will be collected from the sampling ports with a glass syringe and analyzed immediately for Eh and then pH.

Samples for inorganic parameters (cations and alkalinity) will be sent to a commercial laboratory, Maxxam Analytics Inc., Mississauga, Ontario. Cation analyses will be performed using inductively coupled plasma atomic emission spectroscopy (ICP-AES) (U.S. EPA Method No. 200.7). Alkalinity (as mg $CaCO_3$/L) in water will be determined by automated colorimetry (U.S. EPA Method No. 310.2).

Table 1: ZVI Column Study Sampling and Analyses

Analyte	Number of Sampling events*	Number of Samples (Influent/ Effluent)	Number of Samples (ports)	Total number of analyses per column
cVOCs + DHGs	7	2	7	55
Anions	3	2	7	28
Cations and Alkalinity	2	2	0	3
Eh/pH	7	2	7	55

***at time zero only the influent groundwater reservoir will be sampled**

2 SCHEDULE AND REPORTING

SiREM is prepared to commence work upon award of contract. The columns will be constructed within four weeks of receiving the groundwater samples. SiREM will provide biweekly updates to Watermark via email. SiREM will tabulate the data and will prepare a report containing the methodology, all data generated and interpretation of the results from the column study. A draft report will be provided to Watermark for review and comment, and the final report will be provided after receipt of comments on the draft version. The draft report will be submitted to Watermark within two weeks of receipt of final analytical data and the final report will be delivered two weeks after receipt of the comments on the draft version.

3 COST ESTIMATE AND ASSUMPTIONS

SiREM is prepared to conduct the proposed scope of work for the study for a fixed-fee of $19,500. These costs include set-up and analysis of all reactors, reporting, all professional time, general laboratory disbursements and associated analyses. If a two column study set-up is requested (to evaluate 2 different ZVI products) the cost to conduct two columns would be $32,500.

This cost estimate is based on the following assumptions:

1. No Workplan, other than this proposal will be required for submittal/approval by regulatory agencies.

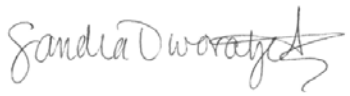
2. Groundwater sample collection and shipment to SiREM are paid for by client (costs not included herein).

The contract terms and conditions applicable to laboratory research studies are unique. To minimize the administrative efforts between our firms and to facilitate this business transaction, SiREM recommends, that this proposal be governed by the attached terms and conditions (Attachment A). Your signature below will confirm your acceptance of SiREM's terms and conditions. If you desire to use your own contract form, please forward it to my attention. As soon as we have reached agreement on the terms, SiREM will proceed with its services.

Please note that SiREM is a division of Geosyntec Consultants, Inc., a corporation organized under the laws of the State of Florida.

If SiREM's proposal is acceptable, please indicate agreement by having an authorized representative sign in the space provided below and returning a complete copy of this proposal and attachments to my attention. Should you have any questions or need additional information regarding this quotation please contact me at 1-866-251-1747 x 236.

Sincerely,



Sandra Dworatzek, M.Sc.
Senior Manager

Attachment A: Terms and Conditions for Research Studies

Agreed and Accepted By:

Proposal # Si-1725-110212	Total Cost $19,500 (plus applicable taxes) (single column test)	
Authorized Signature		
Printed Name		
Fax & Email Address		
Purchase Order	PO #	☐ No PO will be issued
Prime Contract	☐ Applies, Copy Attached	☐ No Prime Contract Applies

References

Gillham, R.W. and O'Hannesin, S.F., 1994. Enhanced Degradation of Halogenated Aliphatics by Zero-Valent Iron. Ground Water, Vol. 32, No. 1, pp. 958-967.

Attachment A: Research Study Terms and Conditions

Assignments. The Order is non-assignable by either party without the prior written consent of the other party.

Client's Prime Contract. In the event that Client is obligated to flow down the provisions of its prime contract, Client shall provide a copy to SiREM concurrent with its execution of this document to which these T&Cs are attached. SiREM reserves the right to review and accept the mandatory flow down provisions of the prime contract prior to its performance. SiREM shall be bound to the Terms and conditions of the prime contract only to the extent that they are expressly imposed upon Client's subcontractors. SiREM's signature on the document to which these T&Cs are attached shall evidence its acceptance of the prime contract flow down requirements.

Confidentiality. SiREM will maintain as confidential any documents or information provided by Client and Research Study reports and will not release, distribute or publish the same to any third party without prior permission from Client, unless compelled by law or order of a court or regulatory body of competent jurisdiction. In such event, SiREM will provide advance written notice to Client prior to release.

Force Majeure/Consequential Damages. Neither party will hold the other responsible for damages or delays in performance caused by force majeure, acts of God, or other events beyond the reasonable control of the delayed party. In no event will either party be liable to the other for any incidental or consequential damages arising in connection with the Research Study.

Integrated Writing. The Order and these T&Cs, together with any Supplemental T&Cs attached hereto, constitute a final and complete agreement between Client and SiREM and supersede all prior or contemporaneous representations or agreements, whether oral or written, and shall only be modified through a mutually acceptable written addendum hereto. It is expressly agreed by the parties that if Client issues a Purchase Order or similar document, it shall be solely for financial authorization purposes and any Terms and conditions contained therein shall not modify these T&Cs.

Invoicing and Payment. SiREM, in order to cover the material cost to set up the Research Study, will invoice Client 50% of the total budget for the Research Study established in the Order upon receipt of the Order. Thereafter, SiREM will invoice Client on a time and materials basis up to the total authorized cost. The balance shall be invoiced upon submission of the Research Study report. Payment is due within 30 days of receipt of SiREM's invoice. In the event the Research Study is pursuant to a prime contract between Client and its client, payment for a Research Study greater than $5,000 shall be made the earlier of Client's receipt of payment from its client or 90 days from receipt of SiREM's invoice. Client shall pay an additional charge of one percent (1%) of the amount of the invoice per month or the maximum percentage allowed by law, whichever is the lesser, for any payment received by SiREM beyond the payment terms set forth herein.

No Third Party Rights. These T&Cs will not create any rights or benefits to parties other than Client and SiREM, provided, however, that if Client is a prime contractor, then Client's client shall be entitled to rely upon the Research Study reports to the same extent as Client, subject to these T&Cs.

Non-discrimination and Affirmative Action. SiREM is an Equal Opportunity and Affirmative Action Employer. Unless exempt, the Equal Opportunity Clauses set forth at 41 CFR §60-1.4(a), 41 CFR §60-250.5(a), 41 CFR §60-300.5(a), and 41 CFR §60-741.5(a); the provisions of 41 CFR §61-250.10 and 41 CFR §61-300.10 (which relate to veterans' employment reports); and the provisions of 29 CFR Part 471, Appendix A to Subpart A (posting of employee notice) are incorporated by reference as terms and conditions of this agreement and are binding on Subcontractors/Vendors. Subcontractors/Vendors may be required to develop written affirmative action programs and/or otherwise comply with the regulations at 41 CFR Part 60.

Notices: Any information or notices required or permitted under the Order will be deemed to have been sufficiently given if in writing and delivered either personally or by mail to the representatives of the parties as set forth in the Order or as otherwise designated by the parties. Notice given by mail will also be transmitted by facsimile or email at the time of mailing.

Order for Research Study. These Terms and Conditions (T&Cs) apply to any research study ("Research Study") performed by SiREM. Such Research Study is initiated by a purchase order, quote or acceptance of a proposal ("Order").

Samples and Reporting. Unless otherwise set forth in the Order, Client will obtain samples and ship to SiREM at Client's expense. At Client's request, SiREM will provide sample containers at no cost. SiREM is responsible for resampling costs while samples are in its care, custody and control only to the extent loss or damage to the samples arises out of SiREM's negligence or willful misconduct. The results of the Research Study will be delivered to Client in SiREM's standard report format. In the event that Client desires a different report format, Client agrees to provide a sample to SiREM prior to the performance of the Research Study. Client understands and agrees that the unit prices in the Order assume standard report format and may be adjusted in the event such special reporting format results in an increased level of effort.

Standard of Care and Indemnity. SiREM will perform the Research Study and will dispose of samples in accordance with the schedule (if any) set forth in the Order, and applicable federal, provincial, state or local laws and regulations. The Research Study will be in accordance with SiREM's general operating quality assurance and control program. SiREM will defend, indemnify and hold harmless Client for losses, claims or causes of action to the extent arising out of SiREM's professional negligence or willful misconduct.

Termination. Client may terminate the Order prior to the commencement of the Research Study by written notice to SiREM.

Validity. These T&Cs will be enforced to the fullest extent permitted by law. If any provision is found to be invalid or unenforceable, the provision will be construed and applied in a way that comes as close as possible to expressing the intention of the parties and that saves the validity and enforceability of the provision.

TC-Research StudiesV6-12-2012.doc

Determination of Volatile Organic Compounds in Ambient Air Using Specially-Prepared Canisters and Analyzed by GC/MS

References: **Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air - Second Edition.** U.S. Environmental Protection Agency. EPA/625/R-96/010b. Office of Research and Development National Risk Management Research Laboratory. Center for Environmental Research Information. Cincinnati, Ohio. January 1999.

Method TO-15: Determination of Volatile Organic Compounds (VOCs) In Air Collected In Specially-Prepared Canisters and Analyzed By Gas Chromatography Mass Spectrometry (GC/MS). U.S. Environmental Protection Agency. EPA/625/R-96/010b. Office of Research and Development National Risk Management Research Laboratory. Center for Environmental Research Information. Cincinnati, Ohio. January 1999.

NJDEP – SRP Low Level USEPA TO-15 Method (NJDEP-LL TO-15 3/2007) March 2009 Revision

1. Scope and Application

Matrices: Air

Definitions: Refer to Section 16 and Alpha Analytical Quality Systems Manual

This SOP describes the procedure for the analysis of volatile organic compounds (VOCS) in ambient air. The whole air samples are collected in fused-silica lined (FSL) stainless steel canisters, or Tedlar® bags. The VOCs are subsequently separated by gas chromatography (GC) and measured by mass selective detector (MSD).

The organic compounds that are amenable to this method are listed in Table 9. Other compounds may also be amenable provided they meet the QA/QC requirements of the method.

The data report packages present the documentation of any method modification related to the samples tested. Depending upon the nature of the modification and the extent of intended use, the laboratory may be required to demonstrate that the modifications will produce equivalent results for the matrix. Approval of all method modifications is by one or more of the following laboratory personnel before performing the modification: Area Supervisor, Department Supervisor, Laboratory Director, or Quality Assurance Officer.

This method is restricted to use by or under the supervision of analysts experienced in the operation of the GC/MS and in the interpretation of GC/MS data. Each analyst must demonstrate the ability to generate acceptable results with this method by performing an initial demonstration of capability, analyzing a proficiency test sample and completing the record of training.

After initial demonstration, ongoing demonstration is based on acceptable laboratory performance of at least a quarterly laboratory control sample or acceptable performance from an annual proficiency test sample. A major modification to this procedure requires demonstration of performance. The identification of major method modification requiring performance demonstration is directed by the Quality Assurance Officer and/or Laboratory Director on a case-by-case basis.

2. Summary of Method

Samples are collected in precleaned, evacuated FSL canisters or Tedlar® bags.

Samples are preconcentrated using the Entech 7100A Cryogenic Concentrator. A specified volume of sample is pulled using a vacuum pump through a mass flow controller. The sample is cryogenically concentrated to a volume of less than one mL in a glass bead trap.

Following preconcentration, the sample is transferred to a Tenax® trap and then refocused on the GC transfer line. This step further reduces the sample volume to less than one microliter for injection.

The sample is then injected into the GC, which is used to separate the compounds of interest. All compounds are detected using an MSD.

2.1 Method Modifications from Reference

Initial Calibration modifications: If a target analyte cannot meet the %RSD criteria for relative response factor calibration, then linear regression may be used. A minimum of five calibration points must be incorporated and a correlation coefficient of 0.995 or greater must be achieved. The calibration plot must be printed and approval by a supervisor must be obtained prior to calibration acceptance. If any compound is calibrated using linear regression then after the ICV and prior to any sample analysis, a low point standard must be analyzed to confirm there is no bias resulting from the linear regression calibration used. Recovery of the low point standard must be 60-140% using the linear regression curve.

Continuing calibration and laboratory controlled spike (LCS) modifications: The recoveries of all analytes must be within 70% to 130% of the true value. If more than 10% of the compounds fail these criteria, or if one compound has a recovery less than 50% or greater than 150% the LCS must be re-analyzed. If failure occurs a second time, the instrument must be re-calibrated. Recoveries greater than 150% may be acceptable, provided analytes are not detected in the samples.

Sample Duplicate modifications: Up to 10% of the target analyte detections may exceed acceptance criteria. If more variation occurs, the sample analysis must be repeated. If an analyte detected in one of the analysis at >5x the reporting limit, and not detected in the duplicate analysis, the analysis must be repeated. If an analyte is detected in one analysis at <5x the reporting limit and not detected in the duplicate analysis, the RPD is not calculable (NC) and the analysis does not have to be repeated. If an analyte is not detected in both the original and duplicate analysis, the RPD is NC.

Section 8.4.1.2 of the TO-15 method requires all canisters to be leak checked for a period of 24 hr. The laboratory conducts the leak check by measuring the vacuum of the canister over a 24 hr. period, not by pressurizing the canister as per the method.

The % RSD for any analyte must be < 30%, as outlined in Section 10.2.2.7 of this SOP.

Humidified nitrogen is used in place of zero air due to the frequency of detection of VOCs in zero air, particularly at SIM detection limits.

There is no NIST-traceable second source standard currently available for the analytes listed in Table 1, Table 3B, and Table A-7.

3. Reporting Limits

Table 9 lists target analytes and Reported Detection Limit information.

4. Interferences

4.1 Contamination may occur in the sampling system if canisters are not properly cleaned before use. Additionally, all other sampling equipment (e.g., pump and flow controllers) must be thoroughly cleaned to ensure that the filling apparatus will not contaminate samples.

4.2 System carryover can be a potential problem, particularly for the heavier molecular weight hydrocarbons. Carryover can occur after the analysis of standards or high-level samples. Measures that must be taken to remove this contamination can include the analysis of multiple blanks, lab air, and the purging of the autosampler with nitrogen.

4.3 High moisture content, methane levels and/or carbon dioxide levels may interfere with the chromatography and trapping of target analytes. Dilutions may be performed on these samples; however, the reporting limits will then be elevated.

5. Health and Safety

The toxicity or carcinogenicity of each reagent and standard used in this method is not fully established; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available in the Chemical Hygiene Plan.

All personnel handling environmental samples known to contain or to have been in contact with municipal waste must follow safety practices for handling known disease causative agents.

All employees performing laboratory procedures must have read and understood the Alpha Analytical Chemical Hygiene Plan. All laboratory procedures must be performed in accordance with the provisions and policies of the manual. All accidents, injuries, spills, or unsafe conditions must be reported immediately to the laboratory manager, and such occurrences must be thoroughly documented.

The analyst must wear a lab coat, gloves, and safety glasses while preparing solutions or handling samples.

Preparation of liquid standards must be performed under a properly functioning fume hood. Preparation and venting of gaseous standards must also be performed under a properly functioning fume hood.

6. Sample Collection, Preservation, Shipping and Handling

6.1 Sample Collection

6.1.1 FSL canister samples can be collected as grab samples or as time-integrated samples. Time-integrated samples can be collected for a maximum of 12 hours using 2.7-liter canisters, or a maximum of 24 hours to 7 days using 6-liter canisters. One liter canisters are typically used for soil vapor sampling with a sampling flowrate of 100-200 ml/min.

6.1.1.1 Grab samples are collected by opening the canister valve and allowing the canister to fill to ambient pressure. This process takes approximately one minute.

6.1.1.2 Time-integrated samples require the use of a properly calibrated flow controller. The flow controller, if provided by Alpha, is calibrated prior to sample collection and is documented in the Alpha ACS LIMs (Refer to Alpha SOP A-007 for Canister and Flow Controller Preparation).

6.1.2 Tedlar® bag samples typically can be collected as grab or composite samples and may require a pumping system or evacuated box.

6.1.3 Upon receipt at the laboratory, all samples are assigned unique laboratory identification numbers, checked for possible discrepancies, etc. (See SOP G-005.)

6.2 Sample Preservation

Canisters-None. Tedlar® bags-should be protected from light.

6.3 Sample Shipping

All samples must be accompanied by a chain of custody form, which documents the date, and time of sample collection.

6.4 Sample Handling

The pressure of all FSL canister samples is measured upon receipt at the laboratory and documented in the ACS LIMs (See Alpha SOP A-007). A pressure gauge is attached to the canister inlet, the canister valve is briefly opened and the pressure is recorded. The gauge apparatus used to measure ambient air samples must be separate from that used to measure soil vapor or other matrices known to have elevated levels of VOCs to avoid cross-contamination.

Samples with pressures greater than -15 inches Hg are considered acceptable for analysis.

Samples with less than -15 inches Hg should be pressurized to > -15 inches Hg in order for the concentrator system to accurately draw the correct volume, resulting in a dilution of the sample. For ambient air samples, the client must be notified prior to sample analysis since this dilution may cause reporting limits to be elevated above project action levels.

Any samples that undergo pressurization prior to analysis are documented in the instrument software. Refer to Section 10.3.3.6 for the calculation of dilution factors due to pressurization of samples.

Refer to SOP G-005 for Sample Management information.

FSL canister and Tedlar® bag samples are stored in the Volatiles Laboratory until analysis has been completed. Tedlar® bag samples are stored in opaque containers.

The recommended holding time for the analysis of FSL canister samples for TO-15 is 30 days. The recommended holding time for the analysis of Tedlar® bag samples for TO-15 is 48-72 hours. Tedlar® bag samples requiring TO-15 analysis may be transferred into canisters upon receipt at the laboratory in order to extend the holding time of the sample to 30 days.

Samples designated by client to be held for subsequent analyses or are "on hold" are to be kept in the Volatiles Laboratory. "Hold" samples are discarded upon client authorization or after holding time expiration date.

7. Equipment and Supplies

7.1 Microliter syringes: 10, 25,and 500 µL

7.2 Gas tight syringes: 1 mL, 5 mL, 25 mL, 50 mL, and 100 mL

7.3 FSL canisters: 1.0, 2.7, 6.0 and 15 Liter

7.4 Tedlar® bags: Various sizes. Alpha supplies 5-Liter sizes. All bags must have polypropylene fittings which are recommended for the analysis of Sulfides and Mercaptans (see App. A).

7.5 Stop watch

7.6 Sample Concentrator

7.6.1 The concentrator system consists of two separate pieces of equipment: (1) Entech Model 7016CA VOC Autosampler, and (2) Entech Model 7100A Cryogenic Concentrator using liquid nitrogen.

7.6.2 A vacuum pump (Vaccubrand Model ME2) delivers the sample from the autosampler to the cryogenic concentrator FSL-lined steel tubing.

7.7 Gas Chromatograph System

7.7.1 Gas chromatograph - Hewlett Packard Model 6890N GC or equivalent

7.7.2 Chromatographic column: Restek RTX-1; 60 meters, 0.25 mm ID, 1 micron film thickness

7.7.3 Transfer line from column to GC injection port: Hydroguard ™ 0.32 mm capillary tubing connected to column with Restek Vu-Union connector.

7.8 Mass Spectrometer System

7.8.1 Mass spectrometer - Hewlett Packard Model 5973 or 5975.

7.8.2 The mass spectrometer must be capable of scanning from 29 to 270 amu every 3 seconds or less, utilizing 70 volts (nominal) electron energy in the electron impact ionization mode and producing a mass spectrum that meets all the criteria in Table 5 when 50 ng of 4- bromofluorobenzene is injected. For SIM (selective ion monitoring) analysis, the system must be capable of simultaneous SIM/full scan acquisition.

7.8.3 Data System - EnviroQuant ChemStation G1701 DA Version D.02.00 SPI or later for data acquisition and version E.02.00 for data processing.

7.9 Dynamic Diluter

7.9.1 Entech 4600A Dynamic Dilution System-for preparing calibration standards in canisters and performing sample dilutions in canisters.

7.10 Primary flow measurement device: BIOS Cell Defender 510 or equivalent

8. Reagents and Standards

8.1 DI Water or Carbon-filtered tap water

8.2 High purity purge and trap grade methanol (Fisher part # A453-1 or equivalent) for MS source cleaning

8.3 Ultra high purity (UHP) helium for the GC/MS system

8.4 Ultra high purity (UHP) nitrogen for standard preparation

8.5 NIST certified TO-15 gas standards, purchased from Linde (formerly Spectra Gases). Standards are stored at room temperature and expire one year from production date, unless re-certified.

8.6 Neat chemicals: Listed in Table 1, Table A-1, and Table 3B, ≥ 98% purity.

8.7 Liquid nitrogen: For the concentrator system and/or GC cooling

8.8 Primary Standards

8.8.1 Primary standard mixtures of TO-15 analytes are purchased certified gaseous standards already prepared as well as gaseous standards prepared in the laboratory by injecting neat chemicals into Tedlar® bags (See Table 1).

8.8.2 Table 1 indicates volumes of neat chemicals that are injected into 20 L of zero air or UHP nitrogen to obtain primary standard concentrations for all analytes.

8.8.3 Purchased primary standards are assigned a CSS # (commercially supplied standard) upon receipt for tracking purposes. Preparation of primary standards must be entered into the primary standard preparation logbook (Form No.: 117-11).

8.8.4 Standards are valid per the manufacturer's expiration date as noted.

8.9 Secondary Standards

8.9.1 Prepare secondary standards in canisters using the Entech 4600A Dynamic Diluter at a minimum of two concentration levels. Table 3A and 3B outlines the preparation steps for each secondary standard.

8.9.2 Prior to preparation of the standards, verify that an appropriate vacuum exists in the canister (>0.5 psia). Figure 3 demonstrates the standard preparation system.

Primary standards prepared in Tedlar bags are injected into a canister (typically 15 L) using an injection tee with a septum or transferred from purchased cylinders via the dynamic diluter.

8.9.3 Attach the transfer lines from the primary standards to the back of the dynamic diluter.

8.9.4 Prior to the injection of the gaseous standards, allow the dynamic diluter to equilibrate for a minimum of 60 minutes by allowing the diluent gas to flow at a rate of 200 mL/min and the primary standard to flow at the rate specified in Table 3A for the appropriate standard being prepared.The flow rate settings are adjusted via the 4600A software, the diluent gas flows through MFC 1 and the calibration standard flows through MFC 2, 3 and 4. Be sure that the isolation valve is open while equilibrating and the vent line is attached to the outlet.

8.9.5 After equilibrating the system, close the isolation valve and attach the canister to the outlet of the diluter. Set stopwatch or timer to the duration specified in Table 3 for the purchased cylinder primary standards. Open the isolation valve and the canister valve and start the timer.

Equation 1: Flow rate calculation:

$$T_f = V_{std} / F_{std}$$

Where:

T_f = standard transfer time, minutes
V_{std} = standard volume, mL
F_{std} = standard flowrate, mL/min

8.9.6 Inject the appropriate amount of Tedlar bag primary standard and the low vapor pressure compounds listed in Table 3B into the injection port tee. This injection must be done while the canister is below atmospheric pressure.

8.9.7 When all the primary standards have been added to the canister, pressurize the canister to 30 psia with humidified nitrogen using the dynamic diluter. This is equivalent to 30 liters of calibration standard in the canister when a 15 L canister is utilized for standard preparation.

NOTE: Standard canisters prepared for analysis using the autosampler must have a maximum pressure of 30 psia to ensure proper and consistent sampling by the instrument.

8.9.8 Label the canister accordingly and record the standard preparation in the secondary standard (SS) preparation logbook (Form No.: 117-12).

8.9.9 Standards are valid for 3 months

8.10 Internal Standard and BFB Tuning Standard/Surrogate Standard

The internal standard and a surrogate standard containing the bromofluorobenzene (BFB) tuning standard are purchased as two separate gas standards at 25 ppbV (2 year expiration date). The internal standards and BFB / surrogates are loaded onto the sample trap prior to the sample via a mass flow controller. The concentration of the internal standard and BFB / surrogates added is based upon the nominal concentration of sample that is analyzed. If the nominal volume of sample is 250 mL, then 100 mL of the 25 ppbV internal standard mix will yield a true value of 10 ppbV for the internal standards and BFB. Using equation 7, the ug/m^3 equivalent of BFB injected 179 ug/m^3 or 179 ng/L (MW of BFB = 175). Thus, the total ng injected is:

Total ng = 179 ng/L x 0.100 L = 17.9 ng

8.11 Instrument Calibration Standards

Calibration standards are prepared by injecting different volumes of the secondary standards into the concentrator/GC/MS system. The low standard will be used to establish the reporting limit for sample analyses. These are described in more detail in Section 10.

9. Quality Control

The laboratory must maintain records to document the quality of data that is generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method.

At a minimum, for each day of analysis, a Continuing Calibration standard, Laboratory Method Blank, Laboratory Control Spike and Laboratory Duplicate must be analyzed. Laboratory Control Spike Duplicate (LCSD) will be analyzed only upon client request.

9.1 Blank(s)

A FSL canister pressurized with humidified nitrogen is utilized as the Laboratory Method Blank. The method blank must be free of target analyte contamination at or above the reporting limit. If it is not, the system must be evaluated for possible sources of contamination. Once the source is determined and eliminated, the Blank must be reanalyzed.

A Laboratory Method Blank must be run after samples suspected of being highly contaminated to determine if sample carryover has occurred. If samples have been analyzed using an autosampler, data must be evaluated for potential carryover and re-analyses conducted, as appropriate.

9.2 Laboratory Control Sample (LCS) / Laboratory Control Spike Duplicate (LCSD)

NOTE: A Laboratory Control Spike Duplicate is only performed when specified by the project requirements and/or upon Client request.

Laboratory Control Spike - A Laboratory Control Spike (LCS) is prepared by spiking an evacuated FSL canister with a different primary standard solution than that used for the calibration or a purchased gaseous standard with the components of interest may be used. The spike recovery must be between 70 and 130%. If the recovery is not within acceptance criteria, the LCS may be analyzed a second time. If the LCS failure continues, the instrument must be recalibrated. Refer to Section 12 for appropriate corrective actions to be taken.

9.3 Initial Calibration Verification (ICV)

A mid-range calibration standard must be analyzed after the initial calibration and prior to sample analysis, and must be a different source than that used for the initial calibration. The %Ds must be ≤30. See section 10.4.2.5 for additional criteria. Otherwise, sample analysis may proceed.

9.4 Continuing Calibration Verification (CCV)

A mid-range calibration standard must be analyzed prior to sample analysis. This standard is of a different source than that used for the LCS/LCSD pair, typically the same source as the initial calibration standards. The %Ds must be ≤30. See section 10.4.2.5 for additional criteria. If repeated failure of the CCV occurs, the instrument must be recalibrated. Otherwise, sample analysis may proceed. The LCS may also be utilized as the continuing calibration.

9.5 Matrix Spike

Not applicable.

9.6 Laboratory Duplicate

A Laboratory Duplicate is a replicate analysis of a sample. The RPD of duplicate analyses must not exceed 25. Up to 10% of the target analyte detections may exceed acceptance criteria. If more variation occurs, the sample analysis must be repeated. If an analyte is detected in one analysis at >5x the reporting limit and not detected in the duplicate analysis, the analysis must be repeated. If an analyte is detected in one analysis at <5x the reporting limit and not detected in the duplicate analysis, the RPD is not calculable (NC) and the analysis does not have to be repeated. If an analyte is not detected in both the original and duplicate analyses, the RPD is NC. Equation 9 is used to calculate the RPD.
The sample chosen for duplicate analysis should not be a trip blank, field blank, or equipment blank. The sample chosen for duplicate analysis must be rotated among clients and/or sites. If possible, field duplicates should not be chosen for duplicate analysis, nor should outside air samples if indoor air samples are also included in the analytical batch.

Equation 9: RPD Calculation

$$RPD = (C_s - C_d) / [(C_s + C_d)/2]*100$$

where:

RPD = relative percent difference
C_s = concentration in original sample analysis

C_d = concentration in duplicate sample analysis

9.7 Method-specific Quality Control Samples

9.7.1 **BFB Tune** - A successful BFB spectrum must meet the criteria in Table 5 prior to sample analysis. If a successful BFB spectrum is not obtained, the MS must be retuned and the BFB spectrum re-evaluated prior to analyzing samples.

9.7.2 **Internal Standards** - The internal standard area counts of each sample, blank, and Laboratory Control Sample are evaluated against the corresponding continuing calibration standard. The internal standard area counts must be within 60-140% of the continuing calibration standard area counts. The retention times of the internal standards must be within +/- 0.33 min. If the internal standards fall outside this range, the sample, blank, or Laboratory Control Sample must be reanalyzed. In addition, area counts for internal standards for continuing calibration must be within 60-140% recovery of initial calibration.

9.7.3 **TIC Internal Standards** - Internal standards used for the quantitation of TICs must be evaluated by comparing the total ion area counts of the internal standards in the samples to the total ion area counts of the internal standards in the blanks. The internal standard area counts must be within 50-200% of the blank area counts. If the internal standards fall outside this range, a different internal standard or an estimated internal standard total ion area must be used to quantitate the TIC. This estimate can be done by using the total ion area from a blank or a clean sample within the analytical batch.

9.8 Method Sequence

- BFB Tune Check
- Calibration Standards (initial) or Continuing Calibration
- Laboratory Control Sample (may be used as the ICV or CC)
- Laboratory Control Sample Duplicate (if needed)
- Laboratory Method Blank
- Samples
- Laboratory Duplicate

Injections may be made until 24 hours after the injection used to check the BFB tune.

All analytical sequences must be recorded in the instrument software and documented in the instrument logbook (Form 117-09)

10. Procedure

10.1 Equipment Set-up

10.1.1 Canister Cleaning and Certification

Refer to Alpha SOP A-007 for canister and flow controller preparation.

10.1.2 Sample Preparation and Concentration

Ensure the integrity of the canister sample as described in Section 6.5.

Connect the canisters or Tedlar® bag(s) to the Entech 7016CA Autosampler. For FSL canisters: Align the tubing from one of the 16 positions to the canister inlet position. Push the inlet line into the orifice of the canister and hold in place while tightening the fitting finger tight. Turn the stainless steel nut ¼ turn more with a wrench. The canister valves must be closed at this point. For Tedlar® bags: Connect the valve of the Tedlar® bag with the autosampler line using an adapter fitting.

For canister samples, leak check all inlet connections using the leak check procedure included with the Entech software. A report will be generated indicating the change in vacuum over a period of 30 sec. The vacuum must not increase more than 2 psia. Analysis cannot begin until the leak check has passed for each canister being tested and/or the source of the leak has been determined. A copy of this report must be available on file.

Open the canister or bag valves.

Set up the sequence of the Entech system to withdraw 250 mL from each sample. If high concentrations are expected, lower volumes can be used (minimum of 25 mL).. Samples suspected to contain elevated concentrations (i.e. soil vapor, sub-slab, landfill gas) should be pre-screened prior to analysis to obtain more precise dilution information. If screening results indicate elevated concentrations of non-target analytes, the sample should be diluted such that the peak height of the non-target analyte is approximately 10X greater than the peak height of the first internal standard. Recommended concentrator operating parameters are provided in Table 6.

General description of procedure: A 3-stage concentration technique called Microscale Purge and Trap is used to analyze VOCs in air. It is analogous to the Purge and Trap used in water analysis, only on a much smaller scale. The air sample is first concentrated to about a 0.5cc volume in a cryogenic trap. The trap is then heated to 10 degrees C and is held there while slowly passing helium or nitrogen through it to transfer these compounds to a secondary trap. The second trap is usually Tenax at –30 °C. Sweeping the VOCs from the first to the second trap with only 40cc of helium results in a transfer of less than 0.5 µl of water (40cc @ 100% RH @ 10 deg. C) which can be easily handled by benchtop mass spectrometers. The 40 cc transfer volume also serves to flush the CO2 through the Tenax trap. After transfer to the second trap, the VOCs are back-flushed while heating to be further focused on an open-tubular focusing trap (cryofocuser) for rapid injection onto the analytical column. Internal standard is added directly to the first stage cryogenic trap prior to the sample by a mass flow controller (MFC). MFC controlled introduction is advantageous over loop injection as it remains consistent with the mechanism used to measure the sample volume. Alternatively, the Cold Trap Dehyrdation procedure may be used as described in Appendix A.

10.2 Initial Calibration

10.2.1 Daily GC/MS Performance Check

10.2.1.1 The first analysis of the day is typically a system blank. The GC/MS system is checked to confirm that acceptable performance criteria for bromofluorobenzene (BFB), which is in surrogate mixture, are achieved. These criteria must be met prior to analyzing further standards, blanks and samples.

10.2.1.2 A maximum injection of 50 ng must successfully meet the BFB spectrum criteria in Table 5.

10.2.1.3 If the spectrum of BFB does not meet the above stated criteria, the analysis must be repeated. If the spectrum of BFB still does not meet these criteria, the GC/MS instrument must be retuned.

10.2.1.4 The Daily GC/MS Performance Check must be analyzed every 24 hours or less.

10.2.2 Initial Calibration

10.2.2.1 Analyze a minimum of five different levels by analyzing various volumes of the secondary standards prepared in Table 3 (Table A-3 for sulfide/mercaptan analysis). The lowest standard will be at or below the reporting limit. If the response is not linear at the lowest level for the higher molecular weight compounds, this point must not be included in the calibration curve for these compounds. As a result, the analysis of more than five levels may be required in order to ensure a minimum of five calibration points for each analyte.

Table 4 lists the calibration standard levels and the volumes of the secondary standards needed to achieve these levels.

10.2.2.2 The true value of each of these calibration points is determined by applying a dilution factor that is based on the volume of sample extracted from the canister for each calibration point. Assuming that a volume of 250 mL will be the maximum volume extracted from the samples; this will be the "1X" volume. A dilution factor can be calculated using Equation 2.

Equation 2: Calculation of Instrument Dilution Factor

$$DF = V_{1X} / V_{actual}$$

where:

DF = dilution factor
V_{1X} = maximum volume sampled, mL
V_{actual} = actual volume sampled for samples and standards

10.2.2.3 Analyze each calibration standard according to the procedures specified in Section 10. The true value of each calibration point is determined by

dividing the concentration of the canister by the dilution factor determined using Equation 2.

10.2.2.4 Tabulate the area response of the characteristic ions against the amount for each analyte and internal standard and calculate relative response factors (RRF) for each compound using Equation 3. Perform this calculation for each calibration standard.

Equation 3: Relative Response Factor for Individual Target Analytes

$$\mathbf{RRF = [(A_{EC}) * (C_i)] / [(A_{EI}) * (C_c)]}$$

where:

RRF = relative response factor
A_{EC} = area count of the extracted ion for the analyte of interest
C_i= amount of internal standard (ppbV)
A_{EI} = area count of the extracted ion for the associated internal standard
C_c = amount of analyte of interest (ppbV)

Table 7 lists all TO-15 analytes, internal standards and the associated quantitation ion.

Table 8 lists the internal standards and the associated TO-15 analytes.

10.2.2.5 Calculate the average response factor for each of the target analytes by the following equation (AVG_x = SUM(RFs) / total # of RFs).

10.2.2.6 Calculate the percent relative standard deviation (%RSD) of the response factors over the secondary range of the curve for each of the target analytes using Equation 4.

Equation 4: Percent Relative Standard Deviation

$$\mathbf{\%RSD = [(SD_n - 1) / (AVG_x)] * 100]}$$

where:

%RSD = percent relative standard deviation
SD_n -1 = standard deviation (n-1 degrees of freedom)
AVG_x = average response factor from the initial calibration curve

This task can also be accomplished using the quantitation software provided by the instrument manufacturer.

10.2.2.7 If the %RSD is <30 for each analyte, linearity can be assumed for the associated target analyte and sample analysis may proceed.

If the %RSD is >30 for any analyte, the integrations must be evaluated and the calculations verified. If a %RSD <30 cannot be achieved, it is acceptable for two (2) of the analytes to be above 30%, but below 40% RSD (applies to analytes flagged with a "C" on the Enviroquant initial calibration summary table).

Alpha, may use the following modified acceptance criteria only for projects that have documentation and approval within the QAPP by the quality assurance project planners, and also for analytes not listed in EPA Method TO-15:

If the %RSD is >30 for any analyte, the integrations must be evaluated and the calculations verified. If a %RSD <30 cannot be achieved, it is acceptable for 10% of the total analytes to be above 30%, but below 50% RSD. Before acceptance of such a Calibration Curve, it must be confirmed with the approval of the Section Supervisor and/or the Project Manager that these analytes are typically and historically "trouble" analytes or "poor performers" (typically compounds listed in Table 3B), and that all Client and Project Data Quality Objectives (DQOs) will still be met when analyzing samples using this calibration.

Calibration points may be removed from the calibration curve to meet the 30% RSD criteria, so long as five consecutive points remain in the calibration curve, and the following procedure is followed:

- Remove high level calibration points
- Remove low-level calibration points; reporting limits will need to be elevated, however.
- If calibration points in the mid-level range need to be removed, the entire calibration level must be removed from the calibration curve.

10.2.2.8 If the %RSD >30, a calibration curve is generated using the EnviroQuant quantitation software. The correlation coefficient (linear) for the calibration curve must be greater than 0.995. If these criteria cannot be met, prepare a new set of calibration standards and recalibrate the instrument. NOTE: Quadratic calibration in any form is not acceptable.

Authorization from the department supervisor is required prior to using linear regression calibration. Linear regression is only allowed if certain criteria listed below are met:

- The minimum number of points for a linear regression curve is five points.
- The curve must be plotted and printed and turned in with the raw data.
- A calibration standard must be analyzed at the low point of the curve. Recovery of the low point standard must be 60-140% using the linear regression curve.
- The recovery of the compound for the continuing calibration / LCS must be within 70-130%.

10.2.2.9 The reference spectra for all target analytes are reviewed for both assignments and purity for all instruments. In addition, this process of reviewing all spectra continues whenever a new calibration is completed. Reference spectra should be updated with each initial calibration performed with the midpoint standard of the calibration.

10.2.2.10 Internal Standard Criteria for Initial Calibration Curve

The mean response for each internal standard compound is calculated over the initial calibration range. The area response at each calibration level must be within 60-140% of the midpoint area response over the initial calibration range for each internal standard. This criteria must be met prior to sample analysis.

All of these criteria must be met prior to sample analysis.

10.3 Equipment Operation and Sample Processing

10.3.1 GC Conditions

Oven program: 25° C, hold for 5.0 minutes, then:

Ramp 1: 100° C at 8.0° C / min.; hold for 0.0 min

Ramp 2: 220° C at 25° C / min.; hold for 4.0 min

Gas Flows

Helium carrier gas flow program: 2.0 mL/min for entire run (23.18 min)

Sample Injection

Injection mode:	split
Injection port temperature:	250° C
Inlet pressure:	27.3 psi
Total flow:	39.3 mL/min
Split ratio:	17.3
Split flow:	34.6 mL/min
Gas saver flow:	OFF

10.3.2 MS Conditions

Temperature of MSD transfer line: 250° C

Temperature of MS Quad: 150° C

Temperature of MS Source: 230° C

Solvent Delay: 3.0 minutes

Scanning Parameters: 29-270 amu until 10 min, scan rate = 5.52 scans/sec; then 35-270 amu, scan rate = 3.1 scans/sec. Threshold = 150. Sampling rate = 2. EM offset-variable to achieve response of 200K area counts (+/- 25K) for the internal standard bromochloromethane.

10.3.3 GC/MS ANALYSIS

10.3.3.1 The Entech 7100A Concentrator is programmed to the specific analytical conditions listed in Table 6 (Entech method alpha_TO15.MPT or Alpha_TO15.CTD) and the GC/MS parameters are set to those listed in Sections 10.3.1 and 10.3.2. (Enviroquant method TO15-SFS.M (SIM and full scan) or TO15_FS.M (full scan only, for sulfide & mercaptan analysis in App. A)).

10.3.3.2 An system blank is performed. The BFB spectrum is evaluated.

10.3.3.3 A continuing calibration and/or a laboratory control spike is analyzed. See sect. 9.2 for acceptable criteria

10.3.3.4 A Laboratory Method Blank is analyzed. The Laboratory Method Blank consists of the analysis of 250 mL from a canister of humidified nitrogen. The method blank must be free of target analyte contamination at or above the reporting limit.

10.3.3.5 A 250-mL aliquot of sample is preconcentrated on the Entech 7100A concentrator and injected onto the GC column.

10.3.3.6 Instrument Dilutions and Sub-Atmospheric Sample dilutions

10.3.3.6.1 For dilutions, smaller sample volumes (<250 mL) are analyzed. The smallest volume that can be analyzed with accuracy is 25 mL. The dilution factor is accounted for by entering the volume analyzed in the sample calculation discussed in Section 10.2.2.2 (Equation 2).

10.3.3.6.2 Samples that arrive at the laboratory with pressures below -15 inches Hg should be pressurized with nitrogen to greater than -15 inches Hg, as discussed in Section 6.4. This pressurization results in a dilution factor. The dilution factor is calculated using Equation 6, and the canister dilution spreadsheet (Form No.: 117-05). Attach a green tag to the canister with the pressurization information (initial pressure and final pressure) recorded on the tag.

Equation 6: Dilution Factor for Pressurization of Subatmospheric Samples: Three Steps

Step 1: Calculate the volume in the canister prior to pressurization (Assume a 2.7-liter canister is used).

$$V_{ci} = 2.7 * P_I / 14.696$$

Step 2: Calculate the volume in the canister after pressurization.

$$V_{cf} = 2.7 * P_F / 14.696$$

Step 3: Calculate the dilution factor.

$$DF = V_{cf} / V_{ci}$$

where:

V_{ci}= volume of air in canister prior to pressurization, L

P_I = pressure reading of canister prior to pressurization (psia)

V_{cf} = volume of air in canister after pressurization, L

P_F = pressure reading of canister after pressurization (psia)

DF = dilution factor

14.696 = atmospheric pressure (psia)

10.3.3.7 If samples require larger dilutions than pressurization and instrument dilutions, a syringe dilution into a Tedlar bag with a known volume of nitrogen or additional canister is required.

10.3.3.8 Fit a VCO adapter with a septa to the pressurized sample canister. With a gastight syringe remove appropriate sample size for dilution. Allow sample to flow through syringe for 1 – 2 seconds to flush syringe prior to volumizing. Inject the sample aliquot into a Tedlar bag. If using an evacuated canister, connect the canister to an injection port tee (see Figure 3) attached to the dynamic diluter. Inject the aliquot of sample while a steady stream of Nitrogen is flowing into the dilution canister. Pressurize this canister to 30 psia. Attach a green tag to the canister with dilution information recorded on the tag. Use the dilution calculation worksheet (Form No.: 117-05) to calculate resulting dilutions.

10.3.4 Qualitative Identifications

10.3.4.1 An analyst competent in the interpretation of mass spectra must identify the target analytes by comparison of the sample mass spectrum to the mass spectrum of the standard. Two criteria must be satisfied to verify the identification: (1) elution of the component in the sample at the same GC relative retention time (RRT) as the component in the standard, and (2) agreement of the sample component and standard component mass spectra.

10.3.4.2 For establishing correspondence of the GC RRT, the RRT of the component in the sample must compare within ± 0.06 RRT units of the RRT of the component in the standard. If co-elution of interfering components prohibits accurate assignment of the sample component RRT from the total ion chromatogram, the RRT must be assigned using extracted ion current profiles for the ion unique to the component of interest.

10.3.4.3 For comparison of the standard and sample component mass spectra, mass spectra of standards obtained on the GC/MS under the same instrument conditions are required. Reference spectra should be updated for each initial calibration performed, using the mid-level standard. Once obtained, these standard spectra may be used for identification and reference purposes.

10.3.4.4 The requirements for qualitative verification by comparison of mass spectra are as follows:

- All ions present in the standard mass spectra at a relative intensity greater than 10% (most abundant ion in the spectrum equals 100%) must be present in the sample spectrum.
- The relative intensities of ions specified must agree within ± 20% between the standard and sample spectra.
- Ions greater than 10% in the sample spectrum must be considered and accounted for by the analyst making the comparison.

Table 7 lists the primary and secondary ions for all analytes.

10.3.4.5 Manual integrations: for peaks that are observed to be not integrated correctly by the quantitation software, manual integrations must be performed. Please refer to the manual integration SOP for further instruction on how to properly perform and document manual integrations (Alpha SOP 08-03).

10.3.4.6 Tentatively identified compounds (TICs)
A library search may be performed for non-target sample components for the purpose of tentative identification, as requested by the client. Mass spectra are compared to the National Institute of Standards and Technology Mass Spectral Library (2002 version), and a qualitative match is determined by the analyst. Computer generated library search routines must not use normalization routines that would misrepresent the library or unknown spectra when compared to each other.

10.3.4.7 Guidelines for making tentative identification:

- Relative intensities of major ions in the reference spectrum (ions greater than 20% of the most abundant ion) must be present in the sample spectrum.
- The relative intensities of the major ions must agree within ± 30%.
- Molecular ions present in the reference spectrum must be present in the sample spectrum.
- Ions present in the sample spectrum but not in the reference spectrum must be reviewed for possible background contamination or presence of coeluting compounds.
- If, in the technical judgment of the mass spectral interpretation specialist, no valid tentative identification can be made, the compound will be reported as "Unknown". The mass spectral interpretation specialist should give additional classification of the unknown compound, if possible (i.e., unknown aromatic, unknown hydrocarbon, unknown acid, unknown chlorinated compound). If probable molecular weights can be distinguished, include them.

10.4 Continuing Calibration

10.4.1 Calibration Verification

10.4.1.1 The initial calibration must be verified through the analysis of an Initial Calibration Verification (ICV) sample. (The ICV may also be used to satisfy LCS requirements.) This analysis must be performed every time an initial calibration is performed.

10.4.1.2 The ICV must be prepared using a purchased gaseous standard (from a different lot # or separate vendor) with the components of interest in an evacuated FSL canister. Follow the standard preparation procedure for the calibration standards outlined in Section 8.0. The standard must be prepared at or below the midpoint of the calibration curve.

See section 10.4.2.5 for acceptable criteria.

10.4.2 Continuing Calibration

10.4.2.1 A continuing calibration check must be performed daily prior to sample analysis. The continuing calibration standard must be one of the initial calibration levels.

10.4.2.2 Analyze a calibration standard that is at the midpoint of the calibration curve.

10.4.2.3 The LCS standard may be utilized as the continuing calibration check, provided that all target analytes of interest are present in the LCS standard.

10.4.2.4 Calculate the percent difference (%D) of the continuing calibration response factor from the initial calibration average response factor using Equation 5.

Equation 5: Percent Difference

$$\%\ D = [(C_{found})-(C_{true}) / (C_{true})] * 100$$

where:

%D = percent difference

C_{found} = amount of the analyte detected in the standard (ppbV)
C_{true} = true amount of the analyte in the standard (ppbV)

This task can also be accomplished using the quantitation software provided by the instrument manufacturer.

10.4.2.5 Acceptance Criteria

The acceptance criteria is less than 30% RSD for any analyte.

Alpha may use the following modified acceptance criteria only for projects that have documentation and approval within the QAPP by the quality

assurance project planners, and also for analytes not listed in EPA Method TO-15:

If the %RSD is <30 for each analyte, linearity can be assumed for the associated target analyte and sample analysis may proceed.

If the %RSD is >30 for any analyte, the integrations must be evaluated and the calculations verified. If a %RSD <30 cannot be achieved, it is acceptable for 10% of the total analytes to be above 30%, but below 50% RSD. Before acceptance of such a Calibration Curve, it must be confirmed with the approval of the Section Supervisor and/or the Project Manager that these analytes are typically and historically "trouble" analytes or "poor performers" (typically compounds listed in Table 3B), and that all Client and Project Data Quality Objectives (DQOs) will still be met when analyzing samples using this calibration.

10.5 Preventive Maintenance

Mass flow controllers should be checked annually for flowrate accuracy using a BIOS cell or other primary flow measurement device.

Ion source cleaning – typically prior to initial calibration.

Electron Mulitiplier (EM)-changed when the voltage setting required to achieve adequate response approaches 1900.

Rough pump oil changed annually.

Transfer lines, concentrator traps, and the GC guard column should be changed semi-annually, or when system repeatedly fails initial calibration.

11. Data Evaluation, Calculations and Reporting

11.1 Calculations

11.1.1 Individual Target Analytes: The average response factor from the initial calibration is used to calculate the amount of analyte detected in the sample analyses. Standards are prepared on a ppbV basis, so if no dilution is performed, values can be reported from the quantitation report without any calculations. Dilution factors are calculated using Equation 2. Equation 7 shows the conversion of ppbV to $\mu g/m^3$.

Equation 7: Conversion of ppbV to $\mu g/m^3$

$\mu g/m^3$ = (ppbV) * MW / 24.45

where:

24.45 = molar gas constant (g/g-mole)

MW = molecular weight of the compound of interest (Table 1 and 2 lists the molecular weights of the target analytes)

11.1.2 TICS: An estimated amount for the TIC is calculated using the total area of the TIC, the total area of the internal standard assigned by the quantitation software, and a response factor of 1.000 (Equation 8). If the internal standard assigned by the quantitation software exhibits significant interference from other analytes, the next closest eluting internal standard will be utilized.

Equation 8: Calculation of TIC Results in ug/m^3

$$ug/m^3 = [(A_T) * (C_{IS})] / [(A_{IS-T}) * (1.000)]$$

where:

A_T = total ion area of the TIC to be measured

C_{IS} = amount of the internal standard

A_{IS-T} = total ion area of the closest eluting internal standard

The integration of target analytes and internal standards must be performed from valley to valley.

11.2 Data Package

11.2.1 Canister Cleaning Information

A copy of the data for the batch certification analysis associated with the FSL canisters must be on file. The raw data must include a sample chromatogram, quantitation report, and spectra of all positive results.

11.2.2 BFB Tune Checks

Tune checks must be included for all days of analysis, including initial calibration. Raw data must include the chromatogram, mass spectra, and summary of relative abundances of the BFB ions.

11.2.3 Calibration Data

- Initial calibration summary (including average response factors, %RSDs, and copies of calibration curves, if appropriate) for target analytes and all calibration chromatograms must be on file.
- Continuing calibration summaries (including %Ds) for individual analytes.
- Chromatograms and quantitation reports associated with all standards used, in the initial and continuing calibrations.

11.2.4 QA/QC

- Internal standard responses and % recoveries vs. the continuing calibration.

- Quantitation report and chromatogram for laboratory control spike (and laboratory control spike duplicate, if requested).
- Quantitation reports, chromatograms, and spectra of positive results for all blanks.
- Copy of the instrument runlog.

11.2.5 Sample Data

- Quantitation reports, chromatograms, spectra of positive results, negative proofs, and pre- and post-manual integrations for all LCSs, samples, and duplicates.
- A copy of the canister dilution worksheet, Form No.: 117-05 (if any canister pressurizations or canister dilutions are performed).

12. Contingencies for Handling Out-of-Control Data or Unacceptable Data

The laboratory must maintain records to document the quality of data that is generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method. When results indicate atypical method performance, a calibration verification standard is used to confirm the measurements were performed in an in-control mode of operation.

Holding time exceedence and/or container damage is noted on the Sample Delivery Group form.

Perform routine preventative maintenance following manufacturer's specification. Record all maintenance in the instrument logbook.

Review of standards, blanks and standard response for acceptable performance occurs for each batch of samples. Record any trends or unusual performance on a nonconformance action form.

If the CV, LCS or LCSD recovery of any parameter falls outside the designated acceptance range, the laboratory performance for that parameter is judged to be out of control, and the problem must be immediately identified and corrected. The analytical result for that parameter in the unspiked samples is suspect and is only reported for regulatory compliance purposes with the appropriate Narratives. Immediate corrective action includes reanalyzing all affected samples by using any retained sample before the expiration of the holding time.

13. Method Performance

13.1 Method Detection Limit Study (MDL) / Limit of Detection Study (LOD) / Limit of Quantitation (LOQ)

The laboratory follows the procedure to determine the MDL, LOD, and/or LOQ as outlined in Alpha SOP/08-05. These studies performed by the laboratory are maintained on file for review.

13.2 Demonstration of Capability Studies

Refer to Alpha SOP/08-12 for further information regarding IDC/DOC Generation.

13.2.1 Initial (IDC)

The analyst must make an initial, one-time, demonstration of the ability to generate acceptable accuracy and precision with this method, prior to the processing of any samples.

13.2.2 Continuing (DOC)

The analyst must make a continuing, annual, demonstration of the ability to generate acceptable accuracy and precision with this method.

14. Pollution Prevention and Waste Management

Refer to Alpha's Chemical Hygiene Plan and Waste Management and Disposal SOP for further pollution prevention and waste management information.

15. Referenced Documents

Chemical Hygiene Plan

SOP/08-05 MDL/LOD/LOQ Generation

SOP/08-12 IDC/DOC Generation

SOP G-006 Hazardous Waste & Sample Disposal

SOP 08-03 Manual Integration

Form 117-05: Canister Dilution Worksheet Template

Form 117-09: Instrument Run Log

Form 117-11: Primary Standard Preparation Log

Form 117-12: Secondary Standard Preparation Log

16. Attachments

Table 1	TO-15 Tedlar® Bag Stock Standard Preparation
Table 2A	TO-15 Purchased Stock Standard Cylinders
Table 2B	TO-15 Custom Mix Purchased Stock Standard Cylinder
Table 3A	Summary of Working Standard Preparation
Table 3B	Preparation of Calibration Standards for Low Vapor Pressure Compounds
Table 4	Calibration Standard Levels
Table 5	BFB Key Ions and Abundance Criteria
Table 6	Entech7100A/7016CA Operating Parameters
Table 7	Quantitation and Secondary Ions for TO-15 Analytes and Internal Standards
Table 8	Internal Standards and the Associated Target Analytes
Table 9A	TO-15 Target Analytes and Reporting Limits-Standard List

Table 9B TO-15 Target Analytes and Reporting Limits-Additional Analytes

Figure 3 FSL Canister Standard Preparation System

Appendix A Cold Trap Dehydration technique (CTD) for Analysis of Sulifides and Mercaptans

Appendix B Data Acquisition Parameters and Analysis Modifications for Conducting SIM Analysis

Appendix C Modifications to Data Review and Case Narrative to Comply with MADEP MCP-TO-15 Method

Appendix D Method Modification to Perform the NJDEP TO-15 Low Level Method

DEFINITIONS

Absolute canister pressure - Pg + Pa, where Pg = gauge pressure in the canister (psig) and Pa = barometric pressure.

Absolute pressure - Pressure measured with reference to absolute zero pressure (as opposed to atmospheric pressure), usually expressed as kPA, mm Hg, or psia (pounds per square inch absolute).

Cryogen - The refrigerant used to obtain very low temperatures in the cryogenic trap of the analytical system. A typical cryogen is liquid nitrogen (bp = -196^{0}C).

Gauge pressure - Pressure measured above atmospheric pressure (as opposed to absolute pressure). Zero gauge is equal to ambient atmospheric (barometric) pressure. Units = psig (pounds per square inch gauge).

ppmV – parts per million on a volume basis.

ppbV – parts per billion on a volume basis

psia – pounds per square inch absolute

Relative retention time (RRT)– retention time (RT) ratio of the target analyte and the internal standard used to quantitate (RT target / RT internal standard).

Table 1

TO-15 Tedlar® Bag Stock Standard Preparation

COMPOUND (liquids)	MOL WGT	Density ug/uL	uL injected*	FINAL ppmV
Indan	118.18	965	5.0	50.0
Indene	116.16	996	4.8	50.4
1,2,3-Trimethylbenzene	120.19	894	5.5	50.1
Thiophene	84.14	1051	3.3	50.4
2-Ethylthiophene	112.19	990	4.7	50.7
2-Methylthiophene	98.17	1014	4.0	50.6
3-Methylthiophene	98.17	1016	4.0	50.6
Acetaldehyde	44.05	785	11.5	250.7
Halothane **	197.38	1872	4.3	49.9

* All neat chemicals are injected into a Tedlar® bag containing 20 Liters of zero air or UHP nitrogen.

** Halothane reported via SIM analysis only.

See Table A-1 & Table A-2 for sulfide/mercaptan stock standard preparation

Table 2A

TO-15 Purchased Primary Standard Mix

TO-15 Primary Standard Mix #1

COMPOUND	MOL WGT	Conc. ppmV	COMPOUND	MOL WGT	Conc. ppmV
dichlorodifluoromethane	120.92	1.0	cis-1,3-dichloropropene	110.97	1.0
chloromethane	50.49	1.0	trans-1,3-dichloropropene	110.97	1.0
Freon-114	170.92	1.0	1,1,2-trichloroethane	133.41	1.0
vinyl chloride	62.5	1.0	toluene	92.14	1.0
bromomethane	94.94	1.0	1,2-dibromoethane	187.87	1.0
chloroethane	64.52	1.0	tetrachloroethene	165.83	1.0
trichlorofluoromethane	137.37	1.0	chlorobenzene	112.56	1.0
1,1-dichloroethene	96.94	1.0	ethylbenzene	106.17	1.0
methylene chloride	84.93	1.0	m-xylene	106.17	1.0
Freon-113	187.38	1.0	p-xylene	106.17	1.0
trans-1,2-dichloroethene	98.96	1.0	styrene	104.15	1.0
1,1-dichloroethane	96.94	1.0	1,1,2,2-tetrachloroethane	167.85	1.0
cis-1,2-dichloroethene	96.94	1.0	o-xylene	106.17	1.0
chloroform	119.38	1.0	1,3,5-trimethylbenzene	120.2	1.0
1,2-dichloroethane	98.96	1.0	1,2,4-trimethylbenzene	120.2	1.0
1,1,1-trichloroethane	133.41	1.0	1,3-dichlorobenzene	147.0	1.0
benzene	78.11	1.0	1,4-dichlorobenzene	147.0	1.0
carbon tetrachloride	153.82	1.0	1,2-dichlorobenzene	147.0	1.0
1,2-dichloropropane	113	1.0	1,2,4-trichlorobenzene	181.45	1.0
trichloroethene	92.14	1.0	hexachlorobutadiene	260.76	1.0

All mixes currently purchased from Linde (formerly Spectra Gases)

TO-15 Primary Standard Mix #2

COMPOUND	MOL WGT	Conc. ppmV
Propylene	42.08	1.0
1,3-butadiene	54.09	1.0
Vinyl bromide	106.96	1.0
Acetone	58.08	1.0
Isopropyl alcohol	60.1	1.0
Carbon disulfide	76.14	1.0
3-chloropropene	76.53	1.0
Trans-1,2-dichloroethene	96.94	1.0
Methyl-tert butyl ether	88.15	1.0
Vinyl acetate	86.09	1.0
2-butanone (MEK)	72.11	1.0
Hexane	86.18	1.0
Ethyl acetate	88.11	1.0
Tetrahydrofuran	72.11	1.0
Cyclohexane	84.16	1.0
Bromodichloromethane	163.83	1.0
1,4-dioxane	88.11	1.0
2,2,4-trimethylpentane	114.23	1.0
Heptane	100.21	1.0
4-methyl-2-pentanone (MIBK)	100.16	1.0
2-hexanone	100.16	1.0
Dibromochloromethane	208.29	1.0
Bromoform	252.75	1.0
Benzyl chloride	126.59	1.0
4-ethyl toluene	120.2	1.0

Table 2B

TO-15 Purchased Custom Mix

TO-15 Custom Standard Mix					
COMPOUND	**MOL WGT**	**Conc. ppmV**	**COMPOUND**	**MOL WGT**	**Conc. ppmV**
Propane	44.10	1.0	n-Octane	114.23	1.0
Chlorodifluoromethane	86.47	1.0	1,1,1,2-Tetrachloroethane	167.85	1.0
Methanol	32.04	5.0	1,2,3-Trichloropropane	147.43	1.0
n-Butane	58.12	1.0	Nonane	128.26	1.0
Dichlorofluoromethane	102.92	1.0	Isopropylbenzene	120.19	1.0
Ethanol	46.07	5.0	Bromobenzene	157.01	1.0
Acetonitrile	41.05	1.0	2-Chlorotoluene	126.58	1.0
Acrolein	56.10	1.0	n-Propylbenzene	120.19	1.0
n-Pentane	72.20	1.0	4-Chlorotoluene	126.58	1.0
Acrylonitrile	53.10	1.0	tert-Butylbenzene	134.20	1.0
Ethyl Ether	74.12	1.0	n-Decane	142.28	1.0
tert-Butyl Alcohol	74.12	1.0	sec-Butylbenzene	134.20	1.0
2,2-Dichloropropane	112.99	1.0	p-Isopropyltoluene	134.22	1.0
Di-Isopropyl Ether	102.17	1.0	n-Butylbenzene	134.20	1.0
Tert-Butyl Ethyl Ether	102.20	1.0	1,2-Dibromo-3-chloropropane	236.33	1.0
1,1-Dichloropropene	110.97	1.0	n-Undecane	156.31	1.0
Tert Amyl Methyl Ether	102.17	1.0	Naphthalene	128.17	1.0
Dibromomethane	173.83	1.0	n-Dodecane	170.33	1.0
1,3-Dichloropropane	112.99	1.0	1,2,3-Trichlorobenzene	181.45	1.0
n-Butyl Acetate	116.16	1.0	Methyl methacrylate	100.12	1.0

All mixes currently purchased from Linde (formerly Spectra Gases)

Table 3A

Summary of Secondary Standards Preparation

Primary Standard	Primary Standard Conc. ppmV	Volume of Primary Standard Injected into canister	Primary Standard Transfer Data	Final Volume canister (L)	Final Concentration ppbV
Secondary standards prepared using dynamic dilution system (Entech 4600A)					
Tedlar® bag primary standard	50	60 mL	Syringe Injection	30	100
TO-15 #1 & #2, & Custom Mix	1.0	3000 mL	50 mL/min for 60 min.	30	100
Tedlar® bag primary standard	50	12 mL	Syringe Injection	30	20
TO-15 #1 & #2, & Custom Mix	1.0	600 mL	50 mL/min for 12 min.	30	20
Tedlar® bag primary standard	50	3 mL	Syringe Injection	30	5.0
TO-15 #1 & #2, & Custom Mix	1.0	150 mL	50 mL/min for 3 min.	30	5.0
Secondary standards prepared via serial dilution					
100 ppbV ICAL mix	1.0	300 mL	Syringe Injection	30	1.0
100 ppbV ICAL mix	0.1	60 mL	Syringe Injection	30	0.2
100 ppbV ICAL mix	0.1	12 mL	Syringe Injection	30	0.04 *

* This calibration standard is used for TO-15 SIM analysis only (see Appendix B).

Table 3B

Preparation of Calibration Standards for Low Vapor Pressure Compounds

COMPOUND (solids)	Vapor Pressure* (P), atm	Molecular Weight	Volume (V) extracted, mL	Gas Constant (R) (L atm/gm mol K)	T, °K	n**	Final Volume, L	mg	ug/m³	ppbV
1-methylnaphthalene	7.11E-05	142.20	2.1	0.082057	298.1	6.101E-09	30	0.0008675	28.9	5.0
1-methylnaphthalene	7.11E-05	142.20	8.4	0.082057	298.1	2.44E-08	30	0.00347	115.7	20
1-methylnaphthalene	7.11E-05	142.00	42	0.082057	298.1	1.22E-07	30	0.0173255	577.5	100
2-methylnaphthalene	8.96E-05	142.00	1.7	0.082057	298.1	6.228E-09	30	0.0008843	29.5	5.08
2-methylnaphthalene	8.96E-05	142.00	6.8	0.082057	298.1	2.491E-08	30	0.0035373	117.9	20.3
2-methylnaphthalene	8.96E-05	142.00	34	0.082057	298.1	1.246E-07	30	0.0176867	589.6	101.6
1,2,4,5-tetramethylbenzene	4.23E-04	134.22	1.0	0.082057	291.5	6.182E-09	30	0.0008298	27.7	5.0
1,2,4,5-tetramethylbenzene	4.23E-04	134.22	4.2	0.082057	291.5	2.473E-08	30	0.0033191	110.6	20
1,2,4,5-tetramethylbenzene	4.23E-04	134.22	21	0.082057	291.5	1.236E-07	30	0.0165954	553.2	101
benzothiophene	7.70E-05	134.20	1.9	0.082057	291.5	6.115E-09	30	0.0008206	27.4	5.0
benzothiophene	7.70E-05	134.20	7.6	0.082057	291.5	2.446E-08	30	0.0032824	109.4	20.0
benzothiophene	7.70E-05	134.20	38	0.082057	291.5	1.223E-07	30	0.016412	547.1	100

Approximately 5.0 g of solid material was allowed to stand in a 250 mL jar w/ septa cap for 30 min prior to removal of vapor phase aliquot. The aliquot was then spiked directly into secondary standard.

All vapor pressure values from Lange's Handbook of Chemistry & Physics w/ the exception of 1,2,4,5-tetramethylbenzene. The initial value was derived using Antoine Equation below, and then normalized using the PIANO vapor phase standard:

Antoine Equation used to calculate vapor pressure

	p, mm Hg	log p	A	B	C	T, C	T, K
1,2,4,5-tetramethylbenzene	0.32108333	-0.4933822	7.08	1672.43	201.43	19.4	292.55

Table 4

Calibration Standard Levels

Calibration Level	Amount (ppbV)	Volume / Secondary Standard
1	0.20	250 mL of 0.2 ppbV sec. standard
2	0.50	125 mL of 1.0 ppbV sec. standard
3	1.0	250 mL of 1.0 ppbV sec. standard
4	2.5	125 mL of 5.0 ppbV sec. standard
5	5.0	250 mL of 5.0 ppbV sec. standard
6	10	125 mL of 20 ppbV sec. standard
7	20	250 mL of 20 ppbV sec. standard
8	50	125 mL of 100 ppbV sec. standard
9	100	250 mL of 100 ppbV sec. standard

Table 5

BFB Key Ions and Abundance Criteria

Mass	Ion Abundance Criteria
50	8.0-40.0 percent of the base peak
75	30.0-66.0 percent of the base peak
95	Base peak, 100 percent relative abundance
96	5.0-9.0 percent of the base peak
173	Less than 2.0% of mass 174
174	50.0 to 120.0% of mass 95
175	4.0-9.0 percent of mass 174
176	Greater than 93.0 percent but less than 101.0 percent of mass 174
177	5.0-9.0 percent of mass 176

Table 6

ENTECH 7016CA/7100A Operating Parameters

Module 1 (Glass Bead Trap)	
Parameter	**Setting**
Trapping Temperature	-150° C
Internal standard volume	100 mL
Internal standard flow rate	50 mL / min
Sample volume (may vary depending on sample concentrations)	250 mL
Sample flow rate, mL / min	150 mL / min
Preheat Temperature	10° C
Desorb Temperature	10° C
Bake Temperature	220° C
Bake Time	10 min
Module 1 to Module 2 transfer volume / rate	40 cc @ 10 cc/min
Module 2 (Tenax trap)	
Parameter	**Setting**
Trapping Temperature	-10° C
Desorb Temperature	220° C
Bake Temperature	220° C
Module 2 to Module 3 desorb time	3.5 min
Module 3 (Cryofocusser)	
Parameter	**Setting**
Cryofocusing Temperature	-130° C
Desorb Temperature	Approx. 70° C
Module 3 to GC desorb time	3 min
Bake temperature / event	Approx. 90° C / event 3
Delay time	13 – 17 min

Table 7
Quantitation and Secondary Ions for TO-15 Analytes and Internal Standards

Compound	Quant. Ion	Sec. Ion(s)	Compound	Quant. Ion	Sec. Ion(s)	Compound	Quant. Ion	Sec. Ion(s)
bromochloromethane	49	130	1,4-difluorobenzene	114	63	bromoform	173	171, 175
chlorodifluoromethane	51	67	n-hexane	57	43, 86	styrene	104	103, 78
propylene	41	39, 42	diisopropyl ether	87	45, 59	1,1,2,2-tetrachloroethane	83	85
propane	29	43,39	ethyl acetate	61	43, 70	o-xylene	91	106
dichlorodifluoromethane	85	87	2,2-dichloropropane	77	41, 97	1,2,3-trichloropropane	75	39, 110
chloromethane	50	52	tetrahydrofuran	42	71, 72	nonane	43	57, 128
Freon-114	85	87, 135	tert-butyl ethyl ether	59	87, 57	bromofluorobenzene	95	75, 174
methanol	31	32,29	1,2-dichloroethane-D4	65	67, 102	isopropylbenzene	105	120
vinyl chloride	62	64,	1,1,1-trichloroethane	97	61, 119	bromobenzene	77	156
1,3-butadiene	54	39	1,1-dichloropropene	75	39,110	2-chlorotoluene	126	91
butane	43	41,58	benzene	78	52	n-propylbenzene	120	91
bromomethane	94	96	carbon tetrachloride	117	119, 82	4-chlorotoluene	91	126
chloroethane	64	66	cyclohexane	56	84, 41	4-ethyl toluene	105	91, 120
dichlorofluoromethane	67	69, 47	tert-amyl methyl ether	73	43, 87	1,3,5-trimethylbenzene	105	91, 120
ethanol	31	45	dibromomethane	93	95, 174	tert-butylbenzene	119	134
acetonitrile	41	40	1,2-dichloropropane	63	39, 62	1,2,4-trimethylbenzene	105	91, 120
vinyl bromide	106	108	bromodichloromethane	83	85, 129	decane	57	43, 142
acrolein	56	55,29	trichloroethene	130	132, 97	benzyl chloride	91	126
acetone	43	58	1,4-dioxane	88	58	1,3-dichlorobenzene	146	75, 111
trichlorofluoromethane	101	103	2,2,4-trimethylpentane	57	41, 99	1,4-dichlorobenzene	146	75, 111
isopropyl alcohol	45	59	n-heptane	43	57, 100	sec-butylbenzene	105	134
acrylonitrile	53	52,51	cis-1,3-dichloropropene	75	39, 77	p-isopropyltoluene	119	134
pentane	43	57,72	4-methyl-2-pentanone	43	58, 100	1,2-dichlorobenzene	146	75, 111
ethyl ether	31	59,45	methyl methacrylate	41	69, 100	n-butylbenzene	91	134
1,1-dichloroethene	61	96, 63	trans-1,3-dichloropropene	75	39, 77	1,2-dibromo-3-chloropropane	75	39, 157
Tertiary butyl Alcohol	59	41, 43	1,1,2-trichloroethane	97	61, 83	undecane	57	43, 71, 156
methylene chloride	49	84	Thiophene	84	45, 58	dodecane	57	43
3-chloropropene	41	39, 76	chlorobenzene-D5	54	82, 117	1,2,4-trichlorobenzene	180	109, 145
carbon disulfide	76	44	toluene	91	92	naphthalene	128	102
Freon 113	101	85, 151	toluene-D8	98	100	1,2,3-trichlorobenzene	180	109, 145
trans-1,2-dichloroethene	61	96, 98	1,3-dichloropropane	76	41,49	hexachlorobutadiene	225	118, 260
1,1-dichloroethane	63	65	2-hexanone	43	58, 100	2-methylthiophene	97	45, 98
MTBE	73	57, 43	dibromochloromethane	129	127, 131	3-methylthiophene	97	45, 98
vinyl acetate	43	86	1,2-dibromoethane	107	109	2-ethylthiophene	97	45, 112
2-butanone	43	72	butyl acetate	73	43, 56	1,2,3-trimethylbenzene	105	120
cis-1,2-dichloroethene	61	96, 98	octane	85	43, 57, 114	indan	117	91, 118
chloroform	83	85, 47	tetrachloroethene	166	94, 131	indene	115	89, 116
1,2-dichloroethane	62	49, 63, 64	1,1,1,2-tetrachloroethane	131	95, 133	1,2,4,5-tetramethylbenzene	119	91, 134
acetaldehyde	29	43, 44	chlorobenzene	112	77, 114	benzothiophene	134	63, 89
			ethylbenzene	91	106	2-methylnaphthalene	142	115, 141
			m+p-xylene	91	106	1-methylnaphthalene	142	115, 141

Table 8

Internal Standards and the Associated Target Analytes

bromochloromethane		1,4-difluorobenzene	chlorobenzene-D5	
chlorodifluoromethane	trans-1,2-dichloroethene	hexane	toluene	decane
propylene	1,1-dichloroethane	diisopropyl ether	toluene-D8	benzyl chloride
propane	MTBE	tert-butyl ethyl ether	1,3-dichloropropane	1,3-dichlorobenzene
dichlorodifluoromethane	vinyl acetate	1,2-dichloroethane-D4	2-hexanone	1,4-dichlorobenzene
chloromethane	2-butanone	1,1,1-trichloroethane	dibromochloromethane	sec-butylbenzene
Freon-114	cis-1,2-dichloroethene	1,1-dichloropropene	1,2-dibromoethane	p-isopropyltoluene
methanol	chloroform	benzene	butyl acetate	1,2-dichlorobenzene
vinyl chloride	1,2-dichloroethane	carbon tetrachloride	Octane	n-butylbenzene
1,3-butadiene	trans-1,2-dichloroethene	cyclohexane	tetrachloroethene	1,2-dibromo-3-chloropropane
butane	1,1-dichloroethane	tert-amyl methyl ether	1,1,1,2-tetrachloroethane	undecane
bromomethane	acetaldehyde	dibromomethane	Chlorobenzene	dodecane
chloroethane	ethyl acetate	1,2-dichloropropane	Ethylbenzene	1,2,4-trichlorobenzene
dichlorofluoromethane	2,2-dichloropropane	bromodichloromethane	m+p-xylene	naphthalene
ethanol	tetrahydrofuran	trichloroethene	Bromoform	1,2,3-trichlorobenzene
acetonitrile		1,4-dioxane	Styrene	hexachlorobutadiene
vinyl bromide		2,2,4-trimethylpentane	1,1,2,2-tetrachloroethane	2-methylthiophene
acrolein		heptane	o-xylene	3-methylthiophene
acetone		cis-1,3-dichloropropene	1,2,3-trichloropropane	2-ethylthiophene
trichlorofluoromethane		4-methyl-2-pentanone	Nonane	1,2,3-trimethylbenzene
isopropyl alcohol		methyl methacrylate	bromofluorobenzene	indan
acrylonitrile		trans-1,3-dichloropropene	isopropylbenzene	indene
pentane		1,1,2-trichloroethane	bromobenzene	1,2,4,5-tetramethylbenzene
ethyl ether		thiophene	2-chlorotoluene	benzothiophene
1,1-dichloroethene			n-propylbenzene	2-methylnaphthalene
Tertiary butyl Alcohol			4-chlorotoluene	1-methylnaphthalene
methylene chloride			4-ethyl toluene	
3-chloropropene			1,3,5-trimethylbenzene	
carbon disulfide			tert-butlybenzene	
Freon 113			1,2,4-trimethylbenzene	

Table 9A

TO-15 Target Analytes and Reporting Limits
Standard List

COMPOUND	CAS #	Standard Reporting Limit, ppbV	Standard Reporting Limit, ug/m^3
1,1,1-trichloroethane	71-55-6	0.2	1.09
1,1,2,2-tetrachloroethane	79-34-5	0.2	1.37
1,1,2-trichloroethane	79-00-5	0.2	1.09
1,1-dichloroethane	75-34-3	0.2	0.81
1,1-dichloroethene	75-35-5	0.2	0.79
1,2,4-trichlorobenzene	120-82-1	0.2	1.48
1,2,4-trimethylbenzene	95-63-6	0.2	0.98
1,2-dibromoethane	106-93-4	0.2	1.54
1,2-dichlorobenzene	95-50-1	0.2	1.2
1,2-dichloroethane	107-06-2	0.2	0.81
1,2-dichloropropane	78-87-5	0.2	0.92
1,3,5-trimethylbenzene	108-67-8	0.2	0.98
1,3-butadiene	106-99-0	0.2	0.44
1,3-dichlorobenzene	541-73-1	0.2	1.2
1,4-dichlorobenzene	106-46-7	0.2	1.2
1,4-dioxane	123-91-1	0.2	0.72
2,2,4-trimethylpentane	540-84-1	0.2	0.93
2-butanone	78-93-3	0.2	0.59
2-hexanone	591-78-6	0.2	0.82
3-chloropropene	107-05-1	0.2	0.63
4-Ethyltoluene	622-96-8	0.2	0.98
Acetone	67-64-1	1.0	0.94
benzene	71-43-2	0.2	0.64
Benzyl Chloride	100-44-7	0.2	1.03
bromodichloromethane	75-27-4	0.2	1.34
bromoform	75-25-2	0.2	2.07
bromomethane	74-83-9	0.2	0.78
carbon disulfide	75-15-0	0.2	0.62
carbon tetrachloride	56-23-5	0.2	1.26
chlorobenzene	108-90-7	0.2	0.92
chloroethane	75-00-3	0.2	0.53
chloroform	67-66-3	0.2	0.98

COMPOUND	CAS #	Standard Reporting Limit, ppbV	Standard Reporting Limit, ug/m^3
chloromethane	74-87-3	0.2	0.41
cis-1,2-dichloroethene	156-59-2	0.2	0.79
cis-1,3-dichloropropene	10061-01-5	0.2	0.91
cyclohexane	110-82-7	0.2	0.69
dibromochloromethane	124-48-1	0.2	1.7
dichlorodifluoromethane	75-71-8	0.2	0.99
ethanol	64-17-5	2.5	4.71
ethyl acetate	141-78-6	0.5	1.8
ethylbenzene	100-41-4	0.2	0.87
Freon-113	76-13-1	0.2	1.53
Freon-114	76-14-2	0.2	1.4
hexachlorobutadiene	87-68-3	0.2	2.13
hexane	110-54-3	0.2	0.7
isopropyl alcohol	67-63-0	0.5	1.23
methylene chloride	75-09-2	1.0	3.48
MIBK	108-10-1	0.2	0.82
MTBE	1634-04-4	0.2	0.72
m+p-xylene	108-38-3 106-42-3	0.4	1.74
n-heptane	142-82-5	0.2	0.82
o-xylene	95-47-6	0.2	0.87
propylene	115-7-1	0.5	0.85
styrene	100-42-5	0.2	0.85
tetrachloroethene	127-18-4	0.2	1.36
tetrahydrofuran	109-99-9	0.2	0.59
toluene	108-88-3	0.2	0.75
trans-1,2-dichloroethene	156-60-5	0.2	0.79
trans-1,3-dichloropropene	10061-02-6	0.2	0.91
trichloroethene	79-01-6	0.2	1.07
trichlorofluoromethane	75-69-4	0.2	1.12
vinyl acetate	108-05-4	0.2	0.70
vinyl bromide	593-60-2	0.2	0.87
vinyl chloride	75-01-4	0.2	0.51

Table 9B

TO-15 Target Analytes and Reporting Limits
Additional Analytes

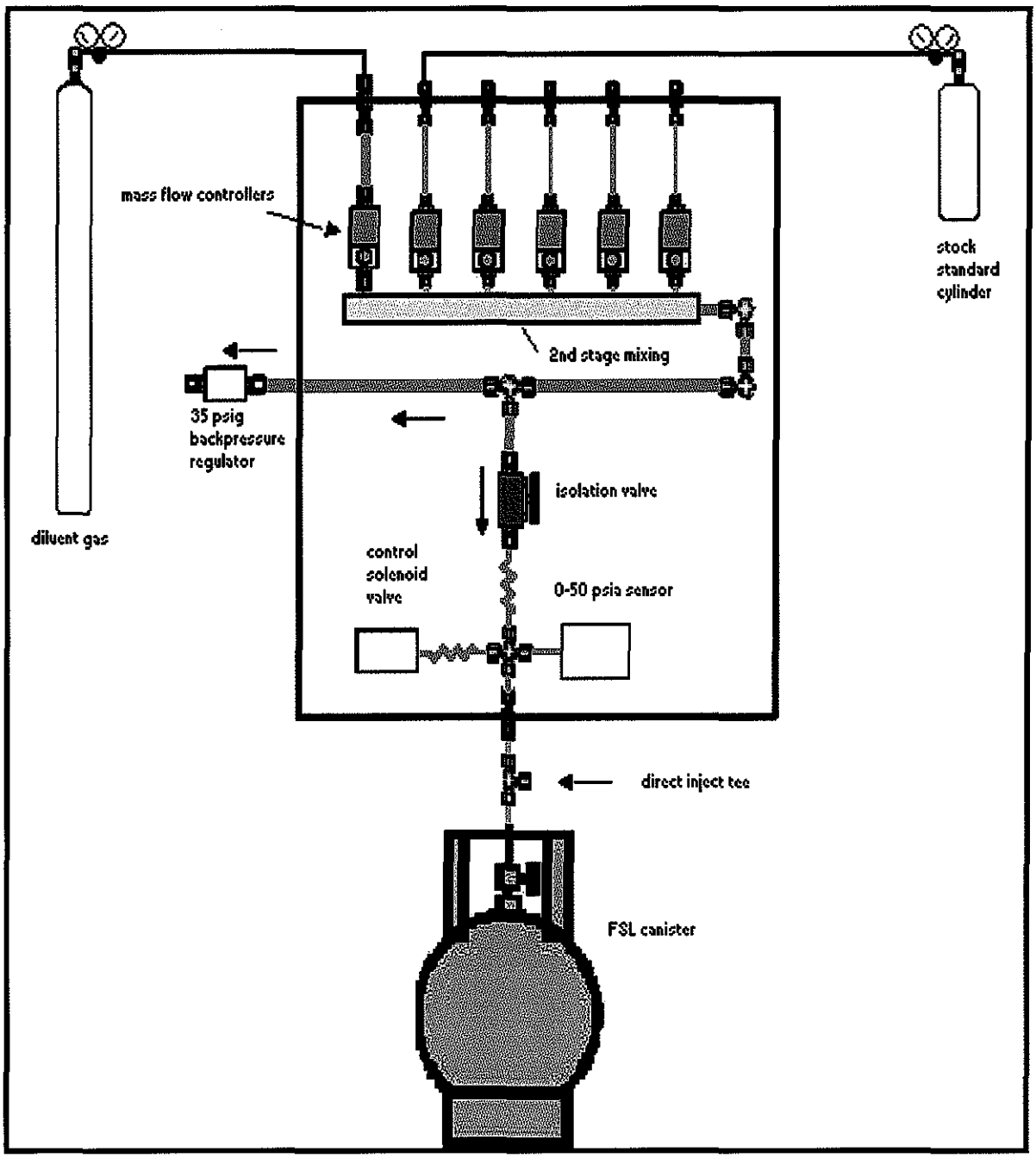
COMPOUND	CAS #	Standard Reporting Limit, ppbV	Standard Reporting Limit, ug/m3
AP-42 Analytes			
acrolein	107-02-8	0.50	1.15
acrylonitrile	107-13-1	0.20	0.43
butane	106-97-8	0.20	0.48
Chlorodifluoromethane	75-45-6	0.20	0.71
Dichlorofluoromethane	75-71-8	0.20	0.84
n-Pentane	109-66-0	0.20	0.59
Propane	74-98-6	0.20	0.36
MADEP MCP 8260 Analytes			
1,1,1,2-tetrachloroethene	630-20-6	0.20	1.37
1,1-dichloropropene	563-58-6	0.20	0.91
1,2,3-trichlorobenzene	87-61-6	0.20	1.48
1,2,3-Trichloropropane	96-18-4	0.20	1.20
1,3-dichloropropane	142-28-9	0.20	0.92
2,2-dichloropropane	594-20-7	0.20	0.92
2-chlorotoluene	95-49-8	0.20	1.03
4-chlorotoluene	106-43-4	0.20	1.03
bromobenzene	108-86-1	0.20	0.79
1,2-dibromo-3-chloropropane	96-12-8	0.20	1.93
dibromomethane	74-95-3	0.20	1.42
diisopropyl ether	108-20-3	0.20	0.84
isopropylbenzene	98-82-8	0.20	0.98
isopropyltoluene	99-87-6	0.20	1.10
naphthalene	91-20-3	0.20	1.05
n-butylbenzene	104-51-8	0.20	1.10
n-propylbenzene	103-65-1	0.20	0.98
sec-butylbenzene	135-98-8	0.20	1.10
tert-amyl methyl ether	994-05-8	0.20	0.84
tert-Butyl ethyl ether	637-92-3	0.20	0.84
tert-butylbenzene	98-06-6	0.20	1.10

COMPOUND	CAS #	Standard Reporting Limit, ppbV	Standard Reporting Limit, ug/m3
NYDEC Petroleum Indicator Compounds			
nonane	111-84-2	0.20	1.05
octane	111-65-9	0.20	0.93
undecane	1120-21-4	0.20	1.28
decane	124-18-5	0.20	1.16
dodecane	112-40-3	0.20	1.39
indene	95-13-6	0.20	0.95
Indan	496-11-7	0.20	0.97
thiophene	110-02-1	0.20	0.69
2-methylthiophene	554-13-3	0.20	0.80
3-methylthiophene	616-44-4	0.20	0.80
2-ethyl thiophene	872-55-9	0.20	0.92
benzothiophene	934-80-5	0.50	13.7
1,2,3-trimethylbenzene	526-73-8	0.20	0.98
1,2,4,5-tetramethylbenzene	95-93-2	0.20	1.10
2-methylnaphthalene	91-57-6	1.0	5.8
1-methylnaphthalene	90-12-0	1.0	5.8
Project Specific Analytes			
acetaldehyde	75-07-0	5.0	9.01
Acetonitrile	75-05-8	0.20	0.34
butyl acetate	123-86-4	0.50	0.95
ethyl ether	60-29-7	0.20	0.61
methanol	67-56-1	5.0	6.55
tert-butyl alcohol	75-65-0	0.50	1.25
Methyl methacrylate	80-62-6	0.50	2.05

Figure 3

Standard Preparation System

Entech 4600A Dynamic Diluter



Appendix A

Cold Trap Dehydration technique (CTD) for Analysis of Sulfides and Mercaptans by EPA TO-15

Target analytes:

Compound	CAS#	Reporting Limit, ppbV
Hydrogen sulfide	7783-06-4	2.0
Carbonyl sulfide	463-58-1	2.0
Methyl mercaptan	74-93-1	2.0
Ethyl mercaptan	75-08-1	0.5
Dimethyl sulfide	75-18-3	0.5
carbon disulfide	75-15-0	0.5
Isopropyl Mercaptan	75-33-2	0.5
tert-Butyl Mercaptan	75-66-1	0.5
n-Propyl Mercaptan	107-03-9	0.5
Ethyl Methyl Sulfide	624-89-5	0.5

Compound	CAS#	Reporting Limit, ppbV
Thiophene	110-02-1	0.5
Isobutyl Mercaptan	513-44-0	0.5
diethyl sulfide	352-93-2	0.5
Butyl Mercaptan	109-75-5	2.0
dimethyl disulfide	624-92-0	0.5
3-Methylthiophene	616-44-4	0.5
Tetrahydrothiophene	110-01-0	0.5
2-Ethylthiophene	872-55-9	0.5
2,5-Dimethylthiophene	638-02-8	0.5
Diethyl Disulfide	110-81-6	0.5

The cold trap dehydration method requires removal of the glass bead trap installed in module 1 of the Entech 7100A concentrator and installing a blank trap (i.e. no trapping material). This trap is cooled and a 250 mL aliquot of sample is allowed to pass through this trap and then directly onto the Tenax trap in module 2, which is also cooled (see Table A-5 for setpoints). The sample is then transferred to module 3 (cryofocusser) via ballistic heating. All requirements stated in this SOP must be applied to any TO15-Sulfide/Mercaptan analysis conducted in the laboratory.

SOP modifications:

Standard preparation and calibration procedures for these analytes are listed in Table A-1 through A-4. Quantitation parameters are listed in Table A-7. Table A-6 lists modified GC conditions.

Section 9.2.5 and 9.5.3.1: Use the Entech method alpha_H2S&SULF.CTD in place of the alpha_TO15.MPT method.

A second source laboratory check standard (LCS) is not readily available for these analytes. An LCS standard is prepared at a different concentration than the initial calibration standards using the same stock standard.

Table A-1

Sulfide/Mercaptan Liquid Cocktail Primary Standard

COMPOUND (liquids)	Initial Conc.	CAS#	Molecular Weight	Density ug/uL	uL injected into cocktail *
ethyl mercaptan	neat	75-08-1	62.14	839	37.0
dimethyl sulfide	neat	75-18-3	62.14	846	36.7
carbon disulfide	neat	75-15-0	76.1	1266	30.1
Isopropyl Mercaptan	neat	75-33-2	76.2	820	46.5
tert-Butyl Mercaptan	neat	75-66-1	90.19	800	56.4
n-Propyl Mercaptan	neat	107-03-9	76.16	841	45.3
Ethyl Methyl Sulfide	neat	624-89-5	76.16	842	45.2
Thiophene	neat	110-02-1	84.1	1051	40.0
Isobutyl Mercaptan	neat	513-44-0	90.19	831	54.3
diethyl sulfide	neat	352-93-2	90.2	837	53.9
Butyl Mercaptan	neat	109-79-5	90.19	842	107.1
dimethyl disulfide	neat	624-92-0	94.2	1046	45.0
3-Methylthiophene	neat	616-44-4	98.17	1016	48.3
Tetrahydrothiophene	neat	110-01-0	88.17	1000	44.1
2-Ethylthiophene	neat	872-55-9	112.19	990	56.7
2,5-Dimethylthiophene	neat	638-02-8	112.19	985	57.0
Diethyl Disulfide	neat	110-81-6	122.25	993	61.6
				Total volume of cocktail mix, uL	**865**

* **Volumes determined using Entech ESP software**

Table A-2

TO15-CTD Tedlar® Bag Stock Standard Preparation

COMPOUND (liquids)	MOL WGT	Density ug/uL	uL injected *	FINAL ppmV
Liquid Cocktail Primary Standard (Table A-1)	NA	NA	71.5	50 / 100 (n-butyl merc)
COMPOUND (gases)	**MOL WGT**	**CONC ppmV**	**mL injected**	**Conc ppmV**
hydrogen sulfide	34.08	1.00E+06	5.0	250.0
carbonyl sulfide	60.08	1.00E+06	5.0	250.0
methyl mercaptan	48.11	1.00E+06	5.0	250.0

*** Volume determined using Entech ESP software**

Table A-3

Summary of Secondary Standards Preparation for Sulfides and Mercaptan Analysis

Primary Standard	Primary Standard Conc. ppmV	Volume of Primary Standard Injected into canister	Primary Standard Transfer Data	Final Volume Tedlar® Bag (L)	Final Concentration ppbV
Secondary standards prepared using gas-tight syringes					
Tedlar® bag primary standard-Low	50	0.32 mL	Syringe Injection	4.0	4.0 / 8.0 / 20
Tedlar® bag primary standard-High	50	8.0 mL	Syringe Injection	4.0	100 / 200 / 500
Tedlar® bag primary standard-LCS	50	0.8	Syringe Injection	4.0	10 / 20 / 50

Table A-4

Calibration Standard Levels

Calibration Level	Amount (ppbV)	Volume / Secondary Standard
1	0.40	25 mL of 4.0 ppbV sec. standard
2	0.80	50 mL of 4.0 ppbV sec. standard
3	4.0	250 mL of 4.0 ppbV sec. standard
4	10	25 mL of 100 ppbV sec. standard
5	20	50 mL of 100 ppbV sec. standard
6	50	125 mL of 100 ppbV sec. standard
7	80	200 mL of 100 ppbV sec. standard
8	100	250 mL of 100 ppbV sec. standard

Table A-6

ENTECH 7016CA/7100A Operating Parameters for CTD Method

Module 1 (Glass Bead Trap)	
Parameter	**Setting**
Trapping Temperature	-40° C
Internal standard volume	100 mL
Internal standard flow rate	60 mL / min
Sample volume (may vary depending on sample concentrations)	250 mL
Sample flow rate	60 mL / min
Preheat Temperature	10° C
Desorb Temperature	10° C
Bake Temperature	220° C
Bake Time	10 min.
Module 2 (Tenax trap)	
Parameter	**Setting**
Trapping Temperature	-40 –to -50° C
Desorb Temperature	180° C
Bake Temperature	190° C
Module 2 to Module 3 desorb time	3 min
Module 3 (Cryofocusser)	
Parameter	**Setting**
Cryofocusing Temperature	-160° C
Desorb Temperature	Approx. 70° C
Module 3 to GC desorb time	2 min
Bake temperature / event	Approx. 90° C / event 3
Delay time	13-15 min

Table A-7

Internal Standard (IS) Assignments and Quantitation Ions for Sulfides & Mercaptans

Compound	Quant Ion	Sec. Ion(s)
Bromochloromethane (IS)	49	130
Hydrogen sulfide	34	33, 36
Carbonyl sulfide	60	62, 32
Methyl mercaptan	47	48, 45
ethyl mercaptan	62	47, 29
dimethyl sulfide	62	45, 47
carbon disulfide	76	44, 78
Isopropyl Mercaptan	43	41, 76
tert-Butyl Mercaptan	41	57, 90
n-Propyl Mercaptan	76	47, 43
Ethyl Methyl Sulfide	61	76, 48

Compound	Quant Ion	Sec. Ion(s)
1,4-difluorobenzene (IS)	114	63
Thiophene	84	58, 45
Isobutyl Mercaptan	41	56, 90
diethyl sulfide	75	90, 47
Butyl Mercaptan	56	41, 90
dimethyl disulfide	94	79, 45
1,2-Dichloroethane-D4	65	102

Compound	Quant Ion	Sec. Ion(s)
Chlorobenzene-D5 (IS)	54	82, 117
3-Methylthiophene	97	98, 45
Tetrahydrothiophene	60	88, 45
2-Ethylthiophene	97	112, 45
2,5-Dimethylthiophene	111	112,97
Diethyl Disulfide	122	66, 94
Toluene-D8	98	100
Bromofluorobenzene	95	174

Appendix B

Data Acquistion Parameters and Analysis Modifications for Conducting SIM Analysis

SIM analysis is conducted when full scan sensitivity does not meet the data quality objectives (DQO) of the project and/or regulatory criteria. The acquisition method used to acquire full scan data simultaneously acquires SIM data using the SIM ions and windows specified in Table B-1. The following modifications to the full scan SOP must be done to generate data using the SIM signal:

- SIM level calibration standards must be analyzed w/ the full scan curve (0.02 and 0.04 ppbV)
- A calibration curve is generated using the SIM signal utilizing the lower level calibration standards and must meet the same criteria as the full scan calibration criteria.
- The continuing calibration and/or LCS should be analyzed at a lower concentration (5.0 ppbV)
- Method blanks must be evaluated for the SIM reporting limit as listed in Table B-2.

The SIM signal only acquires data for a limited target analyte list. These target analytes and reporting limits are listed in Table B-2. Additional ions have been added to allow for more analytes to be added, if requested by client. All requirements stated in this SOP must be applied to any TO15-SIM analysis conducted in the laboratory.

Table B-1

Calibration Standard Levels for SIM Analysis

Calibration Level	Amount (ppbV)	Volume / Secondary Standard
1	0.02	125 mL of 0.04 ppbV sec. standard
2	0.04	250 mL of 0.04 ppbV sec. standard
3	0.20	250 mL of 0.2 ppbV sec. standard
4	0.50	125 mL of 1.0 ppbV sec. standard
5	1.0	250 mL of 1.0 ppbV sec. standard
6	2.5	125 mL of 5.0 ppbV sec. standard
7	5.0	250 mL of 5.0 ppbV sec. standard
8	10	125 mL of 20 ppbV sec. standard
9	20	250 mL of 20 ppbV sec. standard
10	50	125 mL of 100 ppbV sec. standard

Table B-2

Seletive Ion Monitoring (SIM) Groupings

Group ID Start Time (may vary)	Compounds in Group	Ions Scanned
Auto_2 4.00 min	Dichlorodifluoromethane Chloromethane Freon-114 vinyl chloride 1,3-butadiene	29, 39, 41, 43, 44, 50, 52, 54, 58, 62, 64, 85, 87, 135
Auto_3 4.95 min	bromomethane	31, 45, 64, 66, 94, 96
Auto_5 5.80 min	chloroethane Acetone	29, 45, 55, 43, 56, 58, 59, , 106, 108
Auto_6 6.30 min	trichlorofluoromethane	43, 45, 57, 58, 59, 101, 103
Auto_7 6.87 min	Acrylonitrile	43, 45, 51, 52, 53, 57, 59, 72
Auto_8 7.27 min	1,1-dichloroethene methylene chloride	39, 41, 43, 49, 59, 61, 63, 76, 84, 96
Auto_9 7.80 min	Freon-113	39, 41, 44, 76, 85, 101, 151
Auto-10 8.38 min	trans-1,2-dichloroethene Halothane	61, 96, 98, 117, 198
Auto-11 8.95 min	1,1-dichloroethane MTBE	43, 57, 63, 65, 73
Auto-12 9.50 min	2-butanone	43, 72
Auto-13 9.95 min	cis-1,2-dichloroethene bromochloromethane chloroform	43, 47, 49, 57, 61, 83, 85, 86, 96, 98, 130
Auto-14 10.94 min	1,2-dichloroethane 1,2-dichloroethane-D4	49, 61, 62, 63, 64, 65, 67, 102
Auto-15 11.45 min	1,1,1-trichloroethane	61, 97, 119

Group ID Start Time (may vary)	Compounds in Group	Ions Scanned
Auto-16 11.85 min	benzene carbon tetrachloride 1,4-difluorobenzene	41, 45, 52, 56, 58, 63, 78, 82, 84, 114, 117, 119
Auto-17 12.68 min	1,2-dichloropropane bromodichloromethane trichloroethene	39, 41, 43, 57, 58, 62, 63, 83, 85, 88, 99, 97, 129, 130, 132
Auto-18 13.91 min	cis-1,3-dichloropropene 4-methyl-2-pentanone	39, 43, 57, 58, 75, 77, 100
Auto-19 14.60 min	trans-1,3-dichloropropene 1,1,2-trichloroethane Toluene Toluene-D8	39, 45, 61, 75, 77, 83, 91, 92, 97, 98, 100
Auto-20 15.45 min	Dibromochloromethane	43, 45, 58, 97, 98, 100, 127, 129, 131
Auto-21 15.85 min	1,2-dibromoethane	107, 109
Auto-22 16.10 min	Tetrachloroethene	43, 57, 85, 94, 114, 131, 166
Auto-23 16.72 min	1,1,1,2-tetrachloroethane Chlorobenzene-D5 chlorobenzene	54, 77, 82, 95, 112, 114, 117, 131, 133
Auto-24 17.12 min	ethylbenzene p & m-xylene bromoform styrene 1,1,2,2-tetrachloroethane o-xylene	43, 45, 57, 97, 78, 83, 85, 91, 103, 104, 106, 112, 171, 173, 175
Auto-25 17.95 min	Bromofluorobenzene isopropylbenzene	43, 57, 75, 95, 105, 120 128,174
Auto-26 18.42	4-ethyl toluene 1,3,5-trimethylbenzene	91, 105, 120, 126
Auto-27 18.89 min	1,2,4-trimethylbenzene dichlorobenzene isomers sec-butylbenzene p-isopropyltoluene n-butylbenzene	43, 57, 75, 89, 91, 105, 111, 115, 116, 117, 118, 119, 120, 134, 142, 146
Auto-28 20.50 min	naphthalene 1,2,4-trichlorobenzene 1,2,3-trichlorobenzene hexachlorobutadiene	43, 57, 63, 71, 89, 102, 109, 118, 119, 128, 134, 145, 156, 180, 225, 260
MENAPH 21.80	1 & 2-methylnaphthalene	115, 141, 142

Table B-3

TO15-SIM Target Analytes and Reporting Limits

COMPOUND	CAS #	SIM Reporting Limit, ppbV	SIM Reporting Limit, ug/m^3
1,1,1-trichloroethane	71-55-6	0.020	0.109
1,1,2,2-tetrachloroethane	79-34-5	0.020	0.137
1,1,2-trichloroethane	79-00-5	0.020	0.109
1,1-dichloroethane	75-34-3	0.020	0.081
1,1-dichloroethene	75-35-5	0.020	0.079
1,2,4-trimethylbenzene	95-63-6	0.020	0.098
1,2-dibromoethane	106-93-4	0.020	0.154
1,2-dichlorobenzene	95-50-1	0.020	0.120
1,2-dichloroethane	107-06-2	0.020	0.081
1,2-dichloropropane	78-87-5	0.020	0.092
1,3,5-trimethylbenzene	108-67-8	0.020	0.098
1,3-butadiene	106-99-0	0.020	0.044
1,4-dichlorobenzene	106-46-7	0.020	0.120
1,4-dioxane	123-91-1	0.10	0.360
benzene	71-43-2	0.10	0.223
bromodichloromethane	75-27-4	0.020	0.134
bromoform	75-25-2	0.020	0.207
bromomethane	74-83-9	0.020	0.078
carbon tetrachloride	56-23-5	0.020	0.126
chlorobenzene	108-90-7	0.020	0.092
chloroethane	75-00-3	0.020	0.053
chloroform	67-66-3	0.020	0.098
chloromethane	74-87-3	0.500	1.03
cis-1,2-dichloroethene	156-59-2	0.020	0.079
cis-1,3-dichloropropene	10061-01-5	0.020	0.091
dibromochloromethane	124-48-1	0.020	0.170
dichlorodifluoromethane	75-71-8	0.050	0.247
ethylbenzene	100-41-4	0.020	0.087
Freon-113	76-13-1	0.050	0.383
Freon-114	76-14-2	0.050	0.349
hexachlorobutadiene	87-68-3	0.050	0533
methylene chloride	75-09-2	1.0	1.74
MTBE	1634-04-4	0.020	0.072
m+p-xylene	108-38-3 106-42-3	0.040	0.174
o-xylene	95-47-6	0.020	0.087
styrene	100-42-5	0.020	0.085
tetrachloroethene	127-18-4	0.020	0.136
toluene	108-88-3	0.050	0.188
trans-1,2-dichloroethene	156-60-5	0.020	0.079
trans-1,3-dichloropropene	10061-02-6	0.020	0.091
trichloroethene	79-01-6	0.020	0.107
1,2,4-trichlorobenzene	120-82-1	0.050	0.371
vinyl chloride	75-01-4	0.020	0.051
CT RSR Additional Analytes			
1,1,1,2-tetrachloroethane	630-20-6	0.020	0.137
acrylonitrile	107-13-1	0.500	1.08
isopropyltoluene	99-87-6	0.500	2.74
n-butylbenzene	104-51-8	0.500	2.74
sec-butylbenzene	135-98-8	0.500	2.74
isopropylbenzene	98-82-8	0.500	2.46
2-butanone (MEK)	78-93-3	0.500	1.48
Acetone	67-64-1	2.000	4.70
2-methyl-2-pentanone (MIBK)	108-10-1	0.500	2.05
Project Specific Analytes			
Naphthalene	91-20-3	0.050	0.262
Halothane	151-67-7	0.050	0.403
1,2,3-trichlorobenzene	87-61-6	0.0.050	0.371

Appendix C

Modifications to Data Review and Case Narrative to Comply with MADEP MCP TO-15 Method

This addendum addresses modifications to this SOP required to be in compliance with the MADEP MCP TO-15 method, specifically "Quality Control Requirements and Performance Standards for the ***Analysis of Volatile Organic Compounds in Air Samples (TO-15) by Gas Chromatography/Mass Spectrometry (GC/MS)*** in Support of Response Actions under the Massachusetts Contingency Plan (MCP)" Revision No. 0, Section IX B.

1) For duplicate analyses, the 25% RPD criteria stated in Sect. 9.6 of this SOP does not need to be applied to concentrations less than 5X the reporting limit.

2) Samples cannot be analyzed if any of the target analytes if the LCS recovery criteria stated in sect. 9.2 of this SOP is below 70% recovery. For compounds listed as difficult analytes (hexachlorobutadiene, 1,2,4-trichlorobenzene, naphthalene, acetone, and 1,4-dioxane), LCS recovery cannot be less than 50%.

3) Any analyte exceeding %RSD criteria of 30% during initial calibration that is a not listed in the MCP TO-15 method, but may still be reported to the client, must be noted in the case narrative.

Appendix D

Method Modifications to Perform the NJDEP TO-15 Low Level Method

The NJDEP-SRP Low Level USEAP TO-15 Method (NJDEP-LL TO-15-3/2007) requires modifications to data acquisition, data processing, and quality control requirements to be compliant with the method.

- **Data Acquisition**
  - scan range has been changed to 35-300 amu and no SIM signal is acquired.
  - The acquisition method is identified as TO15-NJ.M in the Chemstation software.
  - Calibration levels need to be modified from the current SOP. Maximum concentration of the linear range is 40 ppbV (80 ppbV for m+p-Xylene). See table D-1A and D-1B for preparation of secondary standards and required calibration levels.
- **Data Processing**
  - The quantitation ion for ethanol must be changed to 45 due to the change in scan range (normal quantitation ion is 31).
  - Methyl Methacrylate is a target analyte, and the quantitation ion for this compound is 41, with 69 and 100 utilized as secondary ions. It is associated with the internal standard 1,4-Difluorobenzene.
  - Tentatively Identified Compounds (TICs) – a maximum of 30 non-alkane and non-alkene TICs is to be reported for method blanks, canister certifications, and samples. However, a summation of the concentration of all the alkenes and alkanes detected must be reported.
- **Quality Control Requirements**
  - BFB Tune Check –same requirements as stated in this SOP, both ion abundances and frequency.
  - Initial calibration acceptance criteria-minimum 5 point calibration. %RSD for response factors must be less than 30%, with only two analytes allowed to exceed to 40% RSD.
  - Initial Calibration Verification (ICV) - average response factor must be within +/- 30% of initial calibration, and must be a second-source standard or standard prepared from a different lot. If any exceedences occur, repeat the ICV analysis. If second ICV is not within criteria, investigate possible causes for failure and/or re-calibrate.
  - Continuing Calibration Verification (CCV)- average response factor must be within +/- 30% of initial calibration. If any exceedences occur, repeat the CCV

analysis. If second CCV is not within criteria, investigate possible causes for failure and/or re-calibrate.

- A daily LCS check is not required, however a Reporting Limit Laboratory Control Sample (RLLCS) is required. The required concentration to be analyzed is in Table D-2. Acceptance criteria: the percent recovery must be within 60-140% of the known value for 90% of the compounds.

- A sample duplicate analysis is NOT required for this method.

- A closing calibration check must be analyzed within the 24 hour tune window established by the BFB injection time. If any exceedances occur, repeat the closing CCV analysis; this second injection must also be within the 24 hour tune window. If the second closing CCV fails acceptance criteria, laboratory must note exceedance(s) in the case narrative of the report.

Table D-1A

Calibration Standard Levels for NJ-TO15 Analysis

Primary Standard	Primary Standard Concentration	Volume of Primary Standard Injected into canister	Primary Standard Transfer Data	Final Volume Canister (L)	Final Concentration ppbV
TO-15 Mix #1, TO-15 Mix #2, and TO-15 NJ Custom Mix *	1.0 ppmV	1200 mL	50 mL/min for 24 min	12	100
TO-15 Mix #1, TO-15 Mix #2, and TO-15 NJ Custom Mix	1.0 ppmV	120 mL	50 mL/min for 2.4 min	12	10.0
100 ppbV secondary standard	100 ppbV	60 mL	Syringe Injection	12	0.50

* The TO-15 NJ mix is a NIST-tracable standard purchased from Linde. It contains butane, methyl methacrylate, ethanol, tert-butyl alcohol, and 2-chlorotoluene at 1.0 ppmV. Refer to sect. 8.8 & 8.9 of the SOP for detailed instructions of preparation of secondary standards.

Table D-1B

Calibration Standard Levels for NJ-TO15 Analysis

Calibration Level	Amount (ppbV)	Volume / Secondary Standard *
1	0.2	100 mL of 0.5 ppbV sec. standard
2	0.5	250 mL of 0.5 ppbV sec. standard
3	1.0	25 mL of 10.0 ppbV sec. standard
4	5.0	125 mL of 10.0 ppbV sec. standard
5	10.0	250 mL of 10.0 ppbV sec. standard
6	20.0	125 mL of 40.0 ppbV sec. standard
7	40.0	250 mL of 40.0 ppbV sec. standard

Table D-2

True Values for RLLCS Analyses

Analyte	True Value (ppbV)
m+p-xylene	0.40
isopropanol	0.50
tert-butyl alcohol	0.50
methylene chloride	0.50
methyl ethyl ketone	0.50
Tetrahydrofuran	0.50
1,4-dioxane	0.50
methyl methacrylate	0.50
methyl isobutyl ketone	0.50
ethanol	1.0
acetone	1.0
All other analytes listed in Table D-3	0.20

Table D-3

NJ-TO-15 Target Analytes and Reporting Limits

COMPOUND	CAS #	Standard Reporting Limit, ppbV	Standard Reporting Limit, ug/m^3
1,1,1-trichloroethane	71-55-6	0.20	1.09
1,1,2,2-tetrachloroethane	79-34-5	0.20	1.37
1,1,2-trichloroethane	79-00-5	0.20	1.09
1,1,2-trichloro-1,2,2-trifluoroethane (Freon-113)	76-13-1	0.20	1.53
1,1-dichloroethane	75-34-3	0.20	0.81
1,1-dichloroethene	75-35-5	0.20	0.79
1,2,4-trichlorobenzene	120-82-1	0.20	1.48
1,2,4-trimethylbenzene	95-63-6	0.20	0.98
1,2-dibromoethane	106-93-4	0.20	1.54
1,2-dichlorobenzene	95-50-1	0.20	1.20
1,2-dichloroethane	107-06-2	0.20	0.81
1,2-dichloropropane	78-87-5	0.20	0.92
1,3,5-trimethylbenzene	108-67-8	0.20	0.98
1,3-butadiene	106-99-0	0.20	0.44
1,3-dichlorobenzene	541-73-1	0.20	1.20
1,4-dichlorobenzene	106-46-7	0.20	1.20
1,4-dioxane	123-91-1	0.50	1.80
2,2,4-trimethylpentane	540-84-1	0.20	0.93
2-chlorotoluene	95-49-8	0.20	1.03
4-ethyltoluene	622-96-8	0.20	0.98
acetone	67-64-1	1.0	2.34
allyl chloride (3-chloropropene)	107-05-1	0.20	0.63
butane	106-97-8	0.20	0.48
benzene	71-43-2	0.20	0.64
bromodichloromethane	75-27-4	0.20	1.34
bromoform	75-25-2	0.20	2.07
bromomethane	74-83-9	0.20	0.78
carbon disulfide	75-15-0	0.20	0.62
carbon tetrachloride	56-23-5	0.20	1.26
chlorobenzene	108-90-7	0.20	0.92
chloroethane	75-00-3	0.20	0.53
chloroform	67-66-3	0.20	0.98

COMPOUND	CAS #	Standard Reporting Limit, ppbV	Standard Reporting Limit, ug/m^3
chloromethane	74-87-3	0.20	0.41
cis-1,2-dichloroethene	156-59-2	0.20	0.79
cis-1,3-dichloropropene	10061-01-5	0.20	0.91
cyclohexane	110-82-7	0.20	0.69
dibromochloromethane	124-48-1	0.20	1.7
dichlorodifluoromethane	75-71-8	0.20	0.99
1,2-Dichlorotetrafluoroethane (Freon-114)	76-14-2	0.20	1.4
ethanol	64-17-5	1.0	1.88
ethylbenzene	100-41-4	0.20	0.87
hexachlorobutadiene	87-68-3	0.20	2.13
hexane	110-54-3	0.20	0.7
isopropanol	67-63-0	0.50	1.23
methylene chloride	75-09-2	0.50	1.74
methyl ethyl ketone (2-butanone)	78-93-3	0.50	1.47
methyl isobutyl ketone	108-10-1	0.50	2.05
Methyl methacrylate	80-62-6	0.50	2.05
methyl tert-butyl ether	1634-04-4	0.20	0.72
m+p-xylene	108-38-3 106-42-3	0.40	1.74
n-heptane	142-82-5	0.20	0.82
o-xylene	95-47-6	0.20	0.87
styrene	100-42-5	0.20	0.85
tert-butyl alcohol	75-65-0	0.50	1.52
tetrachloroethene	127-18-4	0.20	1.36
tetrahydrofuran	109-99-9	0.50	1.47
toluene	108-88-3	0.20	0.75
trans-1,2-dichloroethene	156-60-5	0.20	0.79
trans-1,3-dichloropropene	10061-02-6	0.20	0.79
trichloroethene	79-01-6	0.20	0.91
trichlorofluoromethane	75-69-4	0.20	1.07
vinyl bromide	593-60-2	0.20	0.70
vinyl chloride	75-01-4	0.20	0.87

APPENDIX B: HEALTH AND SAFETY PLAN/ACTIVITY HAZARD ANALYSES

PRE-DESIGN INVESTIGATION PROJECT PLAN

FINAL

HEALTH AND SAFETY PLAN AND ACTIVITY HAZARD ANALYSIS

DEFENSE NATIONAL STOCKPILE CENTER SCOTIA DEPOT SITE
GLENVILLE, NEW YORK

CERCLIS Number: NYN000204534
P2 Project Number: 397651

Contract Number: W912WJ-11-D-0001
Stone Project ID 102305-R-C
April 2013

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1. SIGNATURE SHEET

HEALTH AND SAFETY PLAN SIGNATURE SHEET
for Pre-Design Investigation at the
Defense National Stockpile Center Scotia Depot Site
Glenville, New York

Prepared under the direction of: _____
Kim Watson, Corporate Health and Safety (802-229-2196) Date:

Adopted by: _____
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Prepared for:

U.S. Army Corps of Engineers
New England District

Prepared by:

Stone Environmental, Inc. Montpelier, Vermont
Task Order: 0005, Contract: W912WJ-11-D-0001

2. BACKGROUND INFORMATION

2.1. APPLICABILITY

This Health and Safety Plan/Activity Hazard Analysis (HASP/AHA) has been developed by Stone Environmental, Inc. (Stone) for the US Army Corps of Engineers New England District (USACE) under Task Order 0005 of Contract W912WJ-11-D-0001. It establishes the health and safety procedures to minimize any potential risk to Stone personnel and their subcontractors conducting Stone field tasks at the Defense National Stockpile Center Scotia Depot Site in Glenville, New York. The proposed field work will support the ongoing groundwater evaluation of the nature and extent of chlorinated solvent contamination in groundwater and soil vapor due to former military activities, to support a pre-design investigation. Stone is conducting this work on behalf of USACE.

The provisions of this plan apply to all Stone personnel and Stone subcontractor personnel who may potentially be exposed to safety and/or health hazards related to activities described in this document. This HASP/AHA only addresses the specific tasks being implemented by Stone and Stone subcontractors.

This HASP/AHA has been written to comply with the requirements of the Occupational Safety and Health Administration (OSHA) Hazardous Waste Operations and Emergency Response Standard (29 CFR 1910.120) and the USACE Safety and Health Requirements Manual (EM 385-1-1 2008). This HASP/AHA also directly incorporates the USACE requirements for an Accident Prevention Plan as outlined in Appendix A of the USACE Safety and Health Requirements Manual. All activities covered by this HASP/AHA must be conducted in complete compliance with this HASP/AHA and with all applicable federal, state, and local health and safety regulations. Personnel covered by this HASP/AHA who cannot or will not comply will be excluded from site activities.

This plan will be distributed to each employee involved with the proposed investigative program being implemented by Stone. Each employee must sign a copy of the attached health and safety plan receipt and acceptance form (see Attachment A).

2.2. SITE LOCATION

The Scotia Naval Depot is adjacent to the north side of New York State (NYS) Route 5 in the Town of Glenville, Schenectady County, New York (see Figure 1: Location Map). The Site and adjacent properties are zoned for commercial use. The Mohawk River is located approximately 1,500 feet west-southwest of the Scotia Naval Depot and represents the major drainage feature in Schenectady County (see Figure 2: Site Map). The Site overlies an USEPA designated Sole Source Aquifer referred to as the Schenectady or Great Flats Aquifer system. Relative to a series of four aquifer protection zones established to protect municipal water supplies relying on the aquifer system, the Site lies in Zone III, or the General Aquifer Recharge Area (see Figure 2: Site Map).

Portions of the original Scotia Naval Depot have been subdivided and sold since 1972 by the United States Government. The Site now consists of several large privately held parcels in addition to a portion of land still administered by the GSA. The private parcels contain a variety of industrial tenants; while the GSA leases its remaining portion to the DLA/DNSC and the Navy.

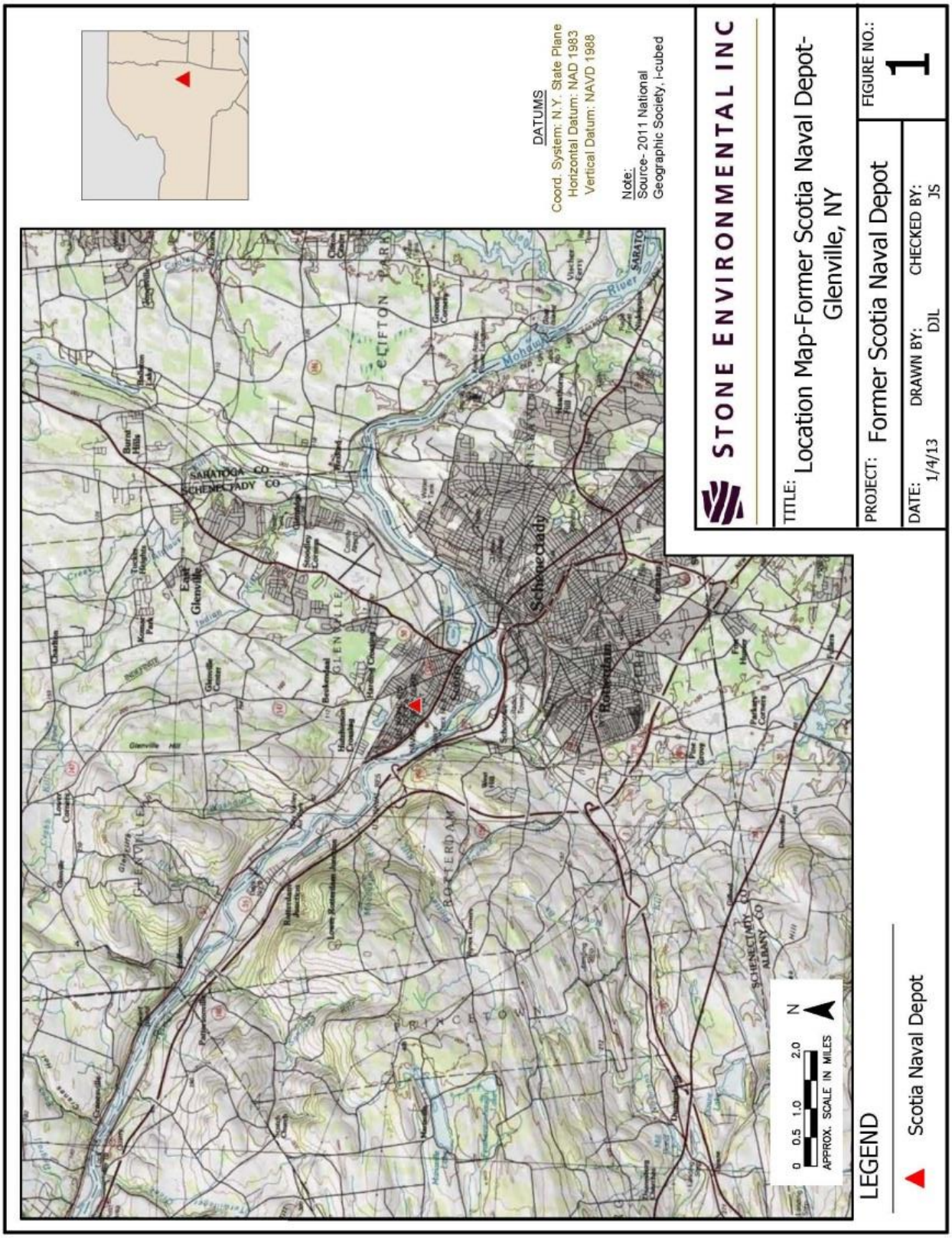


Figure 1: Location Map

STONE ENVIRONMENTAL INC

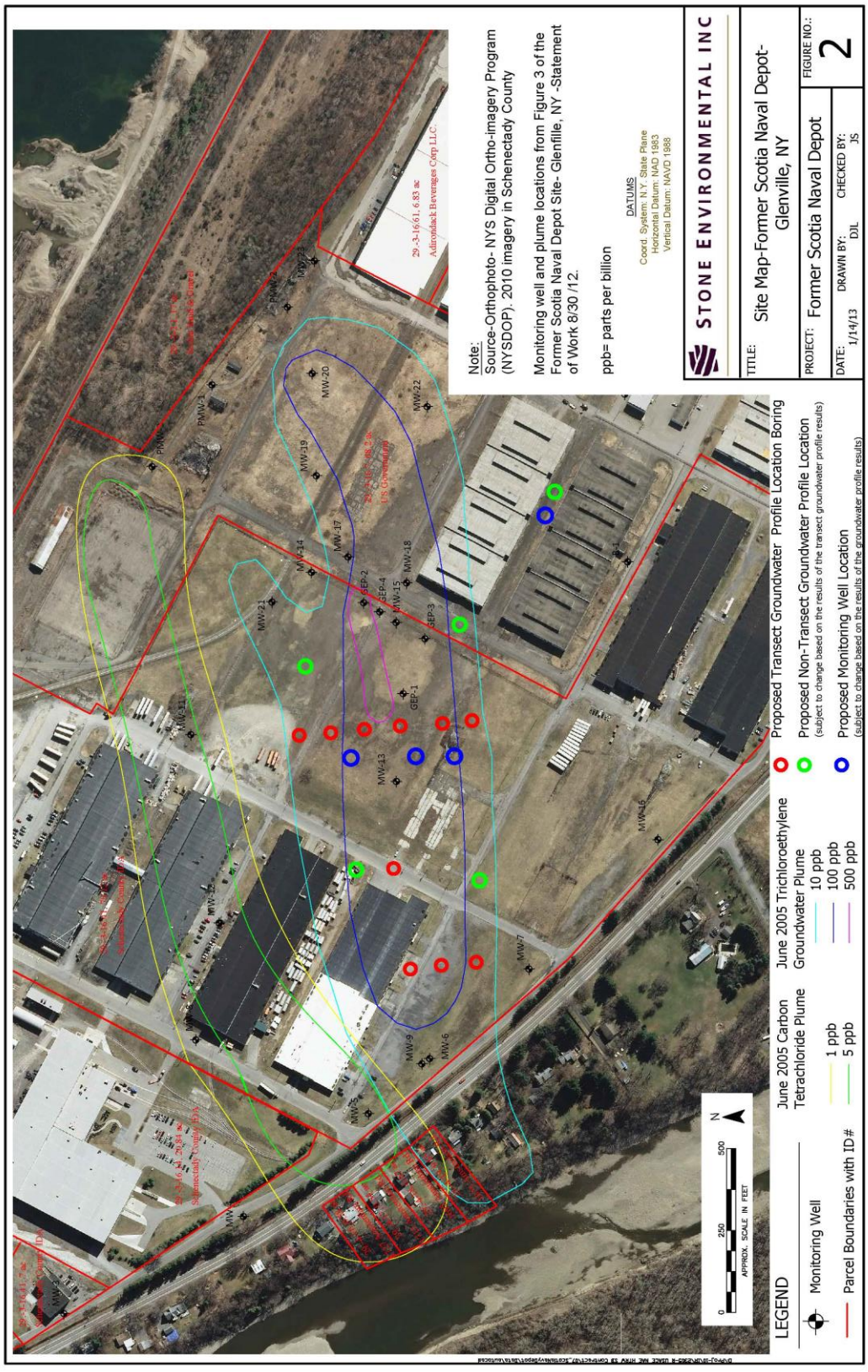


Figure 2: Site Map

STONE ENVIRONMENTAL INC

2.3. HISTORICAL INFORMATION

The primary contaminants of concern at the Site have been identified during previous investigations as chlorinated volatile organic compounds (CVOCs). Of particular interest is the distribution of the primary CVOCs detected in groundwater: carbon tetrachloride, tetrachloroethene (PCE); 1,1,1-trichloroethane (1,1,1-TCA); trichloroethene (TCE); cis-1,2 dichloroethene (cis-1,2-DCE); 1,1 dichloroethene (1,1- DCE); and chloroform.

Groundwater at the Property and off-Property has been impacted by a number of CVOCs including: PCE, TCE, 1,1,1 TCA, cis-1,2 DCE, 1,1 DCE, and chloroform. The CVOC with the highest reported concentration is TCE. PCA degrades into TCE, cis-1,2 DCE, and chloroform and many of the other observed CVOCs on-Property. Maximum reported historical concentrations of these chemicals in groundwater in 2012 are as follows:

Table 1: Maximum Reported Historical CVOC Concentrations in Groundwater

Compound	Maximum Concentration (µg/L)
Carbon tetrachloride	3.5
PCE	28
TCE	70
1,1,1-TCA	22
cis-1,2-DCE	6.2
1,1-DCE	1.6
Chloroform	1.2

2.4. CONTRACTOR ACCIDENT EXPERIENCE

Stone maintains a file of a summary of work-related injuries and illnesses via OSHA's Form 300A. Completed forms are maintained on file at Stone's Corporate office in Montpelier, Vermont. A summary for the 2011 and 2012 calendar years maintains that out of the 140,587 total hours worked, with an average number of employees at 42, the total number of days away from work due to an injury or illness was zero (0).

2.5. PURPOSE OF INVESTIGATION

The primary goal of the work is to collect additional data regarding the nature and extent of the CVOC plume, to support the design of a permeable reactive barrier. The work will use a combination of screening results from a mobile laboratory and fixed laboratory analyses.

2.6. FIELD TASKS

The field work will include:

1. Perform site reconnaissance, collect surface geophysical data; perform surveying;

2. Collect and analyze groundwater samples from approximately 26 monitoring wells;
3. Perform vertical groundwater profiling with drilling equipment;
4. Conduct subsurface soil sampling with drilling equipment;
5. Chemical analysis in Stone's Mobile Field Laboratory;
6. Install and develop groundwater monitoring wells;
7. On-site handling of Investigation-Derived Waste;
8. Equipment and personnel decontamination.
9. Sub-Slab Soil Vapor and Indoor Air Sampling

3. STATEMENT OF SAFETY AND HEALTH POLICY

Stone has provided quality engineering and environmental consulting services since 1992. The safety and health of employees has always been, and will continue to be, a top priority of Stone. Stone has an excellent safety record, and intends to maintain that record.

Stone has developed and implemented a Health and Safety Program (HASP) to ensure continuation of safe work practices, further emphasize health and safety awareness, and comply with Occupational Safety and Health Administration (OSHA) workplace regulations and guidelines.

The HASP/AHA presents a general description of health and safety risks associated with the type of work performed by employees of Stone along with Standard Operating Procedures to be followed for prevention and protection.

When work tasks are performed at a specific project site other than Stone's office or other fixed facilities (equipment and material storage), an HASP/AHA shall be prepared and used in conjunction with the corporate HASP.

4. RESPONSIBILITIES AND LINES OF AUTHORITY

4.1. ORGANIZATION/RESPONSIBILITIES

The implementation of health and safety at this project location will be the shared responsibility of the Stone PM, Stone Health and Safety Officer, Site Safety Officers (SSO) and other staff and contractors participating in this field program.

Table 2: Stone Contacts

Title	SSO	Name, Firm	Phone Number	Cell Number	Email
CENAN Project Manager		Gregory Goepfert	917-790-8235		Gregory.J.Goepfert@usace.army.mil
CENAE Project Manager		Dean Brammer	978-318-8144	603-732-8930	Dean.D.Brammer@usace.army.mil
Project Manager	X	Joseph Schmidl	802-229-5377	603-455-0946	jschmidl@stone-env.com
Site Safety Officer	X	David Linari	802-229-6434	802-578-5817	dlinari@stone-env.com
Corporate Health and Safety Officer		Kim Watson	802-299-2196	802-249-7753	kwatson@stone-env.com

Project Manager (PM)

The Stone PM for this project is Joseph Schmidl.

The PM(s) are individuals who are primarily responsible for ensuring the overall health and safety of this project. As such, the PM(s) are responsible for ensuring that the requirements of this HASP/AHA are implemented. Some of the PM's specific responsibilities include:

- Assuring that all personnel to whom this HASP/AHA applies have received a copy;
- Providing the team Health and Safety Officers with updated information regarding conditions at the Site and the scope of site work;
- Supporting the decisions made by the SSO and team health and Safety Officers;
- Maintaining regular communications with the SSOs and, if necessary, the team health and Safety Officers; and,
- Coordinating the activities of all team subcontractors and ensuring that they are aware of the pertinent health and safety requirements for this project.

Corporate Health and Safety Officer

Stone's Corporate Health and Safety Officer for this project is Kim Watson. Her credentials are provided in Attachment E.

As the Corporate Health and Safety Officer, she is responsible for the preparation, interpretation and modification of this HASP/AHA. Modifications to this HASP/AHA which may result in less stringent precautions cannot be undertaken by the PM or the SSO without the approval of the Health and Safety Officer. Specific duties of the team Health and Safety Officers include:

- Develop a site specific HASP/AHA designed to provide the necessary safeguard to sufficiently protect employees working at the site;

- Ensure that the HASP/AHA addresses all compliance issues associated with the work at the site, to include both regulatory and contractual requirements;
- Review the final HASP/AHA to ensure that all foreseeable hazards have been identified and that reasonable hazard control systems are in place;
- Advising the PM and SSOs on matters relating to health and safety on this site;
- Recommending appropriate personal protective equipment (PPE) to protect team personnel and team subcontractors from potential site hazards;
- Conducting accident investigations; and,
- Maintaining regular contact with the PM and SSO to evaluate site conditions and new information which might require modifications to the HASP/AHA.

Site Safety Officer (SSO)

The SSO for this project is David Linari, and Joseph Schmidl may serve as an alternate SSO. Their credentials are provided in Attachment E. All field staff are responsible for implementing the safety requirements specified in this HASP/AHA; however, only one field staff member will serve as the SSO. The SSO will be appointed by the PM, and will be onsite at all times while work is being conducted. The SSO for any given day of field work will be on-site during all team activities covered by this HASP/AHA. The SSO is responsible for enforcing the requirements of this HASP/AHA once work begins. The SSO has the authority to immediately correct all situations where noncompliance with this HASP/AHA is noted and to immediately stop work in cases where an immediate danger is perceived. Some of the SSO's specific responsibilities include:

- Assuring that all personnel to whom this HASP/AHA applies have submitted a completed copy of the HASP/AHA receipt and acceptance form;
- Assuring that all personnel to whom this HASP/AHA applies have attended a pre-entry briefing and any subsequent safety meetings that are conducted during the implementation of this program;
- Interfacing and coordinating site access and work activities with the USACE site representative and, if necessary, other on-site contractors working for the USACE;
- Maintaining a high level of health and safety consciousness among team members and team contractors at the work site;
- Procuring and distributing the PPE needed for this project for team field members;
- Verifying that all PPE and health and safety equipment used by team members is in good working order;
- Verifying that the selected team contractors are prepared with the PPE and safety equipment required by this HASP/AHA;
- Notifying the PM of all noncompliance situations and stopping work in the event that an immediate danger situation is perceived;
- Monitoring and controlling the safety performance of team personnel and team contractors within the established restricted areas to ensure that required safety and health procedures are being followed;

- Conducting accident/incident investigations and preparing accident/incident investigation reports;
- Conducting the pre-entry briefing as required by the HASP/AHA; and,
- Initiating emergency response procedures in accordance with this HASP/AHA.

Field Staff

All team field personnel covered by this HASP/AHA are responsible for following the health and safety procedures specified in this HASP/AHA and for performing their work in a safe and responsible manner. Some of the specific responsibilities of the field personnel are as follows:

- Reading the HASP/AHA in its entirety prior to the start of on-site work;
- Submitting a completed HASP/AHA Acceptance Form to the team SSO prior to the start of work;
- Attending the required pre-entry briefing prior to beginning on-site work and any subsequent safety meetings that are conducted during the implementation of the proposed field program;
- Bringing forth any questions or concerns regarding the content of the HASP/AHA to the PM or the SSO prior to the start of work;
- Immediately correct all situations where noncompliance with this HASP/AHA is noted and to immediately stop work in cases where an immediate danger is perceived;
- Reporting **all** accidents, injuries and illnesses, regardless of their severity, to the team SSO; and
- Complying with the requirements of this HASP/AHA and the requests of the SSO.

4.1.1. Lines of Authority

Project Managers and Site Safety Officers are responsible for project-specific health and safety issues. The Site Health and Safety Plan identifies the roles regarding project-specific site health and safety issues, and when a Site Health and Safety Plan is required or recommended. No external contacts should be made prior to notification of CENAE/CENAN Project Managers.

Stone's organizational chart identifies each employee as working within a Group, with a Group Officer. The Group Officers are responsible for general health and safety issues within their group. The Corporate Health and Safety Officer and Safety Committee also have responsibilities regarding health and safety and can be contacted directly by employees.

4.2. Noncompliance

Any overt noncompliance with health and safety procedures is treated as non-performance under the terms of employment, which is dealt with promptly through a personal meeting between the employee and executive management of Stone. The employee is warned that another such noncompliance incident could lead to termination of employment depending on the circumstances. If the incident was inadvertent or occurred due to a lack of understanding or knowledge, the meeting will be with the Corporate Health and Safety Officer, and will be used to develop strategies to avoid future incidents.

5. SUBCONTRACTORS AND SUPPLIERS

Stone has retained subcontractors in order to complete the requirements of the project. A description of the subcontractors who will be working in the field and their responsibilities is presented below. All subcontractors are responsible for their own safety and concurrence with OSHA and project specific requirements. The roles of the Stone subcontractors are as follows:

Subsurface Utility Clearance

Subsurface utility clearance services will be provided by:

New York Leak Detection, Inc.
Mark Manzari
P.O. Box 269
Jamesville, NY 13078
Phone: 315-469-4601
mmanzari@nyld.com

Drilling

Drilling services will be provided by:

Stone Environmental, Inc.
Platform Environmental Drilling and Remediation Services
535 Stone Cutters Way
Montpelier, Vermont 05602
Phone: 802-229-1883
mjordan@stone-env.com

Geologic NY
P.O. Box 350
37 Copeland Avenue
Homer, NY 13077
Phone: 607-749-5000
forrest@geologic.net

Parratt-Wolff, Inc.
5879 Fisher Road
P.O. Box 56
East Syracuse, NY 13057
Phone: 800-782-7260
spepling@pwinc.com

Surveyor

Surveying services will be provided by:

C.T. Male, Inc.
50 Century Hill Drive
Latham, New York 12110

Phone 518-786-7605
j.cook@ctmale.com

On-site Subcontractors involved in intrusive work hired by Stone are responsible for:

- Reading the HASP/AHA in its entirety prior to the start of on-site work;
- Attending the required pre-entry briefing prior to beginning on-site work and any subsequent safety meetings that are conducted during the implementation of the program;
- Ensuring, via daily inspections, that their equipment is in good working order;
- Operating their equipment in a safe manner;
- Appointing an on-site safety coordinator to interface with the Stone SSO;
- Providing the SSO with copies of training certifications and material safety data sheets (MSDS) for all hazardous materials brought on-site; and,
- Providing all the required PPE, respiratory equipment and safety supplies to their employees.

6. TRAINING

6.1. Medical monitoring

All personnel performing activities covered by this HASP/AHA, except for the field reconnaissance, surface geophysics and surveying, must be active participants in the medical monitoring program that complies with 29 CFR 1910.120(f). Each individual must have completed an annual surveillance examination within the last year (if exposure criteria are met) and an initial baseline examination prior to performing any work on the site covered by this HASP/AHA.

6.2. Health and Safety Training

6.2.1. HAZWOPER

All personnel performing on-site activities, except for the field reconnaissance, surface geophysics and surveying, covered by this HASP/AHA must have completed the appropriate training requirements specified in 29 CFR 1910.120(e). Each individual must have completed an annual 8-hour refresher-training course and/or initial 40-hour training course within the last year prior to performing any work on the sites covered by this HASP/AHA. Additionally, each employee will have completed at least three days of on-site training under the direction of a trained and experienced team supervisor from the member's employer. Also, on-site managers and supervisors directly responsible for supervising individuals engaged in hazardous waste operations must have completed the specified 8-hour managers training course.

6.2.2. Hazard Communication

Hazard communication training is incorporated into the Stone HAZWOPER training program. The specific hazards associated with the calibration gases and equipment decontamination solutions will be reviewed during the pre-entry briefing. MSDSs for these materials will be maintained on site.

6.2.3. First Aid/CPR

At least two members of the field team will be certified in First Aid/CPR. In addition, staff have had training on controlling exposure to blood-borne pathogens and the use of universal precautions.

6.3. Pre-Entry Briefing

The SSO will conduct a pre-entry briefing to all personnel on-site before site activities begin, to include subcontractor employees from the onsite laboratories. Specific topics that will be discussed during the pre-entry briefing include:

- Discussion of site history
- Discussion of work scope
- Review of the potential hazards associated with contaminants of concern and how these potential hazards will be controlled
- Review of air monitoring requirements and action limits
- Review of PPE and respiratory protection requirements
- Discussion of the potential physical hazards associated with implementing the scope of work

- Review of emergency egress and hospital location/directions
- Review of decontamination procedures

HASP/AHA receipt and acceptance sheets will be collected at this meeting. Short safety refresher meetings will be conducted daily during on-site field work throughout the duration of the project. Attendance of the pre-entry meeting and daily safety refreshers is mandatory and will be documented by the SSO. An attendance form is presented in Attachment C.

7. SAFETY AND HEALTH INSPECTIONS

As discussed in other places in this HASP/AHA, all employees have the right to stop work if deficiencies are noted and only to resume work once these deficiencies have been corrected. In most cases, identified issues will be easy to resolve on site.

Field inspections (audits) will be conducted by the PM and/or the Corporate Health and Safety Officer or designee. For short duration jobs (i.e., one week or less), the safety inspection should be conducted shortly after the work has commenced. For longer duration jobs, the inspection should be conducted shortly after commencement and monthly thereafter, if applicable.

Audits should be documented on the PM Audit Checklist. The auditor should print out a copy of the checklist before leaving for the job site, unless a computer and printer will be available at the site. Once the inspection is complete, the form should be placed in the project files.

In the event that deficiencies are noted during the audit, the PM, SSO and the Health and Safety Officer will confer as necessary to determine how to correct the deficiencies and how to determine when work can resume. The SSO will provide follow-up to insure that the deficiencies have been corrected.

8. SAFETY AND HEALTH GOALS AND OBJECTIVES, INCENTIVE PROGRAMS, AND COMPLIANCE

The objectives of the safety and health program at Stone, specifically as required under the contract with the USACE, are to develop and provide the necessary documents which describe the health and safety criteria, procedures, practices and equipment to be implemented and utilized during the performance of field tasks to sufficiently protect on-site personnel from chemical, physical, and/or biological hazards that may be encountered. Specific goals of the safety and health program and site-specific health and safety plans are to prevent unacceptable exposure to chemicals, protect against physical and environmental hazards, and avoid accidents that could injure personnel.

Strict adherence to Stone's health and safety program, and site-specific health and safety plans, is considered part of each employee's obligations working at Stone. Any overt noncompliance with health and safety procedures is treated as non-performance under the terms of employment, which is dealt with promptly through a personal meeting between the employee and executive management of Stone. The employee is warned that another such noncompliance incident could lead to termination of employment depending on the circumstances. If the incident was inadvertent or occurred due to a lack of understanding or knowledge, the meeting will be with the Corporate Health and Safety Officer, and will be used to develop strategies to avoid future incidents.

Managers and Senior Management are held accountable for the proper implementation of all health and safety procedures for projects/employees they are in charge of by ensuring that their role and responsibilities are well defined in the HASP and SSHP. Their awareness of the level of accountability they maintain is enhanced by ensuring they read (and sign where appropriate) the appropriate plans, and through periodic health and safety meetings with Company Management, the Corporate Health and Safety Officer, and other PMs.

9. ACCIDENT REPORTING

9.1. Stone Environmental, Inc. Procedures

Any work related injury or illness must be reported by the worker to the Corporate Health and Safety Officer and executive management within 24 business hours of the accident/incident by completing the reporting section of Stone's Incident/Accident/Injury/Illness Reporting Form (Attachment D) . The Corporate Health and Safety Officer in conjunction with the QA representative shall investigate either incident/accident/injury/ illness/ and fill out the investigation section of the form with the injured employee. The Safety Committee will review this report. If the accident/incident may have been preventable the Corporate Health and Safety Officer and QA representative shall indicate by what means future accidents/incidents of a similar nature could be avoided. The Corporate Health and Safety Officer and QA representative shall also investigate if applicable health and safety procedures were followed, whether safety and/or personal protective equipment was used and was the usage proper. The completed form will be kept on file for at least five years.

9.2. USACE Reporting Requirements

All accidents and near misses shall be investigated by the SSO. All work-related recordable injuries, illnesses and property damage accidents (excluding on-the-road vehicle accidents), in which the property damage exceeds $2,000 shall be reported verbally to the USACE by the SSO or PM within 24 hours of the incident. Serious accidents as described in EM 385-1-1 Section 01.D.02 shall be immediately reported to the USACE. ENG Form 3394 shall be completed and submitted to the USACE within five working days of the incident.

The PM shall complete the USACE Contractor Monthly Summary Record of Injuries/Illness and Work Hour Exposure (for the prime contractor and its subcontractors) and forward the completed form to the USACE no later than close of business on the 10th calendar day of the following month. The method of transmission by the prime contractor to the USACE will be electronic.

10. MEDICAL SUPPORT

Fire extinguishers, eye wash equipment, and a first aid kit shall be maintained on-site at all times that personnel are performing tasks on-site. The location of these facilities will be shown to all on-site personnel during the health and safety briefings described above.

An ambulance, the fire department, and the police can be contacted by dialing 911 on a cell phone.

The nearest hospital is approximately 4.8 miles away:

Ellis Hospital
1101 Nott Street
Schenectady NY 12308
518-243-4000

Ellis Hospital provides emergency, inpatient medical/surgical and psychiatric care for Albany, Saratoga and Schenectady Counties in upstate New York.

The SSO or his designee is responsible for arranging off-site emergency medical care and transport. David Linari, Joseph Schmidl, and Will Waterstrat are trained in CPR and 1st Aid (see Attachment E).

Directions to Hospital (see Figure 3: Hospital Route Map):

From the Site, depart via Y Street onto Route 5 / Amsterdam Road and turn LEFT.

In 3 miles, turn left onto Erie Boulevard.

In 0.7 mile, bear right onto Nott Street.

In 0.9 mile, arrive at 1101 Nott Street, Schenectady, NY.

11. MONITORING INSTRUMENTS AND PERSONAL PROTECTIVE EQUIPMENT

An Activity Hazard Analysis (AHA) has been prepared for each of the anticipated tasks (see Attachment B).

11.1. Direct Reading Instrumentation

11.1.1. Photoionization Detector (PID)

An Ionscience Phocheck PID (model 1000+), with a 10.6 eV lamp will be used to monitor the breathing zone by the SSO during the proposed subsurface investigations to determine the presence of VOCs. If the PID indicates sustained (15-minute) breathing zone vapor concentrations in excess of 1 ppmV or more in the breathing zone above background concentrations, engineering controls will be implemented or respiratory protection, as described this document, will be donned.

The PID will be calibrated daily to 100 parts per million (ppm) isobutylene span gas and to a zero level (clean air reference) in accordance with Stone's standard operating procedure *SEI-6.19.2 Use, Maintenance and Calibration of the Ionscience Phocheck 1000+ Photionization Detector (PID)*. Daily calibration information will be recorded in the field notebook or on appropriate forms.

11.2. Personal Protective Equipment (PPE)

As required by OSHA 1910.120, Personal Protective Equipment (PPE) must be selected that will protect team members from the specific hazards they are likely to encounter on-site. Selection of the appropriate PPE shall be in general compliance with 29 CFR 1910.132. In areas where the potential for contact with the contaminants of concern exist, Modified Level D will be worn unless air monitoring indicates that the action level set for respiratory exposure is exceeded. If this occurs, engineering controls will be implemented, and/or Level C will be worn as long as the respirator and cartridge is capable of reducing the level of potential exposure to workers to below the Permissible Exposure Level (PEL) or Threshold Limit Value (TLV).

Stone’s Corporate Health and Safety Officer has reviewed the contaminants of concern and the proposed tasks and have completed PPE assessments for each task and have reviewed applicable manufacturer’s charts to determine that the selected chemical protective clothing is appropriate for the contaminants of concern at this site

11.2.1. Chemical Protective Clothing

PPE will be worn during all field activities to prevent on-site personnel from being injured by the safety hazards or exposed to hazardous chemicals activities. The following subsections describe the different circumstances under which PPE will be worn during performance of site activities.

11.2.1.1. Level D or Modified Level D

Level D or Modified Level D PPE are the most likely PPE levels that will be required during this work. It should be cautioned that PPE selection and evaluation is an ongoing process and may be adjusted as additional information about the hazards at the job site and PPE performance is obtained.

The Level D ensemble includes long pants, shoes and a shirt. The Level D protection may be modified as follows:

- Type I or Type II (ANSIZ89.1) hard hat (when overhead hazards are present)
- Safety glasses with permanently attached sideshields (when splash, dust, flying particles or other eye hazards are present)
- Safety-toed boots compliant with the ANSI Z41 standard (when working with heavy machinery)
- Cut-resistant gloves (when using knives or blades, or working with power tools and machinery)
- Chemically-resistant gloves when collecting samples, handling investigation derived wastes, or performing work with potential contact hazards with chemicals (i.e. nitrile for short exposure times of less than five minutes and Viton for longer exposure times). Gloves should not be re-used.
- Hearing protection (when excessive noise levels exist)
- Traffic Vest (when working around heavy machinery such as loaders, excavators, trucks or backhoes)
- Tyvek coverall (as need to protect clothing and skin from splash or chemical contact hazards)
- Lab coats for personnel working in the on-site laboratory
- Personal floatation device when working from small boat or near water hazard

11.2.1.2. Level C or Modified Level C

Based on detectible contaminant concentrations and the expected locations and types of activities, it is not currently anticipated that Level C PPE will be necessary in the conduct of the field work. However, Level C PPE is included in this HASP/AHA to protect workers in the event unanticipated vapor levels (in excess of the Action Level) are encountered during intrusive field activities at known or suspected source areas.

The Level C ensemble includes the following:

- Half mask or full mask air purifying respirators (NIOSH approved) with organic vapor cartridges (safety glasses required if half mask respirator is used).
- Tyvek coverall
- Gloves, outer, chemical-resistant.(Viton)
- Gloves, inner.
- Inner boots, chemical-resistant safety toe and shank.
- Disposable boot-covers, outer, chemical-resistant (optional)
- Hard hat (when overhead hazards exist).
- Hearing protection (when excessive noise levels exist).

11.2.1.3. Respiratory Protection

If the PID indicates sustained (15-minute) breathing zone VOC vapor concentrations in the breathing zone in excess of 1 ppmV above background levels, engineering controls will be implemented and/or respiratory protection will be donned.

Engineered air exchanges are used to provide controls for vapors in the mobile laboratory.

Level C Specification: Half- mask or full-mask, air-purifying respirator with organic vapor cartridges

All employees who are expected to don respiratory protection must have been successfully fit-tested for the model, brand and size respirator they plan to wear at this site within the past year.

11.2.1.4. Other Protective Equipment

The following additional safety equipment will be brought on-site by team personnel:

- Portable hand-held eyewash bottles that will deliver at least 0.4 gallons of water per minute with a minimum of six gallons of water available on-site
- First aid kit
- Type A-B-C fire extinguisher (on drilling equipment and in vehicles)
- Portable walkie-talkies and/or cell phones
- Class 3 lime yellow traffic vest (compliant with ANSI/ISEA 107 Class 3 high visibility apparel).

Potable water and sanitary facilities will be provided on-site for hand washing, eye washing (if needed), etc.

11.2.1.5. Summary of Required PPE for Specific Tasks

Table 3 presents a summary of the minimum required PPE level on an activity-specific basis.

Table 3: Required Personal Protective Equipment by Activity

Activity No.	Activity Description	PPE Level
1	Reconnaissance, surface geophysics and surveying	Level D
2	Collect and analyze groundwater samples from monitoring wells	Modified Level D
3	Perform vertical groundwater profiling with drilling equipment	Modified Level D (with hard hats, hearing protection, safety boots, gloves and safety glasses)
4	Perform subsurface soil sampling with drilling equipment	Modified Level D (with hard hats, hearing protection, safety boots, gloves and safety glasses)
5	Chemical Analysis in Mobile Field Laboratory	Modified Level D (with lab coats, gloves and safety glasses)
6	Install and develop groundwater monitoring wells	Modified Level D
7	On-site handling of Investigation-Derived Wastes	Modified Level D
8	Equipment and Personal Decontamination	Modified Level D
9	Sub-Slab Soil Vapor and Indoor Air Sampling	Modified Level D

By participating in OSHA's 40-hr HAZWOPER safety training, as well as subsequent 8-hr HAZWOPER refresher courses, each employee who is required to wear PPE for this field program has been properly instructed as to how to inspect and maintain their safety gear. Prior to mobilizing to the site, each employee

will inspect their safety gear. Any equipment that is defective, such as scratched safety glasses or cracked hard hats, will be replaced. Chemical protective clothing such as gloves will be inspected by each wearer for holes, rips and tears. Defective clothing will be thrown away.

12. PLANS REQUIRED BY THE SAFETY MANUAL

The following sections of this HASP/AHA incorporate details of the plans required by the safety manual, including:

- An emergency response plan, (18.0)
- Spill and firefighting plans, (18.4)
- Emergency phone numbers, (10.0)
- Hazard communication, (18.3)
- Heat and Cold Stress Monitoring Plan (15.7)
- Respiratory protection, (11.2)
- Lifting procedures, (15.6)
- Severe weather contingencies, (15.7)
- Prohibitions on alcohol and drug use, (16.3)
- Slips, trips and falls, and (15.1)
- Site sanitation. (16.0 and 17.0)

13. CONTRACTOR INFORMATION

The requirements of applicable sections of the EM 385-1-1 are addressed throughout this document. The HASP/AHA has been reviewed along-side-of EM 385-1-1, and adjusted as necessary to be in accordance with the manuals requirements.

14. SITE-SPECIFIC HAZARDS AND CONTROLS

14.1. Chemical Hazards and Contaminants of Concern

During its years of operation, the Navy and its contractors reportedly used solvents, including TCE. Based on standard practices at the time, it is assumed that waste solvents were disposed of on the ground at the various locations where they were used and eventually migrated into the groundwater.

Solvents have been detected in groundwater and soils on the Property. The potential exists for exposure to carbon tetrachloride, PCE, 1,1,1-TCA, TCE, cis-1,2-DCE, 1,1-DCE, and chloroform during intrusive work. MSDS sheets for these compounds are presented in Attachment F.

Overexposure to the chlorinated organic solvents detected in groundwater may result in depression of the central nervous system, symptoms of which include dizziness, headache, giddiness and drunken-like behaviors. Chronic overexposures can result in liver and kidney damage.

A summary of the Contaminants of Concern and their associated exposure limits is presented in Table 4.

Table 4: Contaminants of Potential Concern and Associated Exposure Threshold and Physical Properties

Chemical	PEL[1] (ppmV)	TLV[2] (ppmV)	Odor Thres-hold (ppmV)	IDLH (ppmV)	Density (water = 1) @ 20°C (g/ml)	Henry's Constant @ 25°C (atm-m^3/mol)	Vapor Pres-sure @ 25°C (mmHg)	Vapor Density @ 20°C (Air = 1) (g/cm^3)	Solubility in H_20@ 25°C (mg/L)	Log K_{ow}	Maximum Reported Conc. in Groundwater (µg/L)
Carbon Tetrachloride	25	5	40.7	200	1.59	$8.71x10^{-3}$	91.3	5.32	80	2.64	3.5
Tetrachloroethene	100	25	47	500	1.62	$1.8x10^{-2}$	18.5	1.62	$1.5x10^{2}$	3.40	28
Trichloroethene	100	50	82	1,000	1.46	$1.1x10^{-2}$	74	1.47	$1.366 x10^{3}$	2.42	70
1,1,1-trichloroethane	10	500	400	700	1.34	$4.90x10^{-3}$	100	4.54	2.5	0	22
cis-1,2-dichloroethene	200	200	0.08-17	1,000	1.28	$3.37x10^{-3}$	180	1.28	$3.5 x10^{3}$	1.86	6.2
1,1-dichloroethene	100	100	49-1,359	3,000	1.18	$4.2x10^{-2}$	230	1.17	$5.5 x10^{3}$ *	1.79	1.6
Chloroform	50	10	85	500	1.48	$1.10x10^{-3}$	21.1	4.36	8	1.97	1.2

[1] Permissible Exposure Limit – OSHA; 8-hour, time-weighted average (8-hr TWA)
[2] Threshold Limit Value – American Conference of Governmental Industrial Hygienists; 8-hr TWA
Sources: ATSDR Toxic Profiles, *NIOSH Pocket Guide to Chemical Hazards*, *3M Respirator Guide*

14.2. Hazardous Substances Brought Onsite by Team Members

In accordance with OSHA's Hazard Communication Standard (29 CFR 1910.1200) as well as Section 01.B.06C of EM385-1-1, a material safety data sheet (MSDS) must be available for each hazardous substance that team members bring on the Site. This includes solutions/chemicals that will be used to decontaminate sampling equipment and any calibration gases or calibration reagents that team members may use for field instruments. Once on site, the SSO will create an inventory of all hazardous substances brought on site. This inventory will include the name of the material, the estimated quantity on site and a general description of where these materials are being stored. In accordance with Stone's Hazard Communication Program, team members will be informed of the hazardous properties of the chemicals with which they may work, safe handling procedures, and measures to take to protect them from chemical hazards. MSDS sheets for these materials are located within the facilities and/or mobile units, which will be maintained current during all site work. All containers of hazardous materials must be properly labeled in accordance with OSHA's Hazard Communication Standard.

14.3. Chemical Exposure and Control

The potential exists for workers to be exposed to contaminants of concern through inhalation of vapors and direct dermal contact with contaminated soils, rock, and/or groundwater.

The following chemical exposure control measures will be implemented during the proposed investigations:

- To protect against exposure to COCs through inhalation of vapors, a photoionization detector (PID) will be utilized to record sustained readings. Should readings exceed 1ppmV as an average of three measurements over a fifteen-minute interval, engineering controls will be implemented to reduce the level to less than 1 ppmV, and/or a respirator will be used by personnel within the vapor zone. Should peak readings exceed 100 ppmV, workers will evacuate the area and notify the SSO and/or the PM.
- To avoid direct dermal contact with contaminated media, protective clothing will be required when collecting samples, decontaminating sampling equipment, and installing or removing data loggers.
- To avoid direct dermal contact with contaminated media, protective clothing, including lab coats, chemical resistant gloves and eye protection will be required when analyzing samples in the field laboratory.
- Although highly unlikely, exposure to all of the contaminants of concern may occur via ingestion (hand-to-mouth transfer). The decontamination procedures address personal hygiene issues that will limit the potential for contaminant ingestion.
- The field work program does not involve activities that will expose workers to contaminated air-borne particulates, with the possible exception of the air rotary drilling.

14.4. Fire Prevention Plan

14.4.1. Introduction

Fire protection, fire safety and emergency planning are important aspects of worker Health and Safety. Stone Environmental, Inc. has developed an overall strategy and policy for fire control practices and emergency evacuation. Of primary concern is the safety of our workers, customers and the general public.

14.4.2. Purpose and Implementation

This plan describes the actions that must be taken immediately by Stone personnel in response to fires, explosions, or any unplanned sudden or non-sudden release of hazardous substances or hazardous wastes at the Site. The plan establishes the procedure to follow in the event an emergency situation arises which involves fire or hazardous substances stored, used, or located at the site.

The purpose of this plan is to minimize hazards to human health, property damage or the environment and to outline the provisions that must be carried out immediately whenever there is a fire, explosion or release of hazardous substance constituents to air, soil, or surface water.

Evacuation procedures are included for whenever evacuation maybe necessary. These procedures include signals to be used to begin evacuation of the facilities, and routes out of the building or, if blocked by releases,

alternate routes. This plan outlines emergency action procedures and complies with regulations outlined in by OSHA.

14.4.3. Policy Statement

Stone's policy is to evacuate all employees in the event of a fire, explosion or release of hazardous material. Activate fire alarm system. Use of fire extinguishing equipment will be allowed only in those cases where employees trained in the use of fire extinguishing equipment recognize there to be a minimum danger to the Health and Safety of response employees.

The SSO will be responsible for coordinating all emergency response measures and are thoroughly familiar with:

- All operations and activities at the facility;
- The location and characteristics of hazardous substances present;
- The location of all records within the facility; and
- The physical layout of the facility.

The SSO has the authority to commit company resources or to contact outside contractors in emergency situations. Should a chemical release, fire or explosion involving hazardous substances occur at the site, the SSO or his/her alternate will respond and provide coordination for control of hazardous substance releases, collective emergency actions and assistance to local responding emergency services personnel.

14.4.4. Reporting

The Emergency Coordinator will notify the Fire Department/Police Department if the emergency involves a fire or the need to evacuate areas outside of the site unless not already done. Notify the Fire/Police Department by calling 911 and give the following information:

- Identify yourself
- State nature of emergency
- State location of emergency
- Do not hang up until told to do so
- Remain in the designated area by the lawyers office parking area near scene until emergency help arrives.

14.4.5. Assessment

The SSO shall assess the possible hazards to human health, property and the environment, which may result from the release of hazardous substances, fire, flood or explosion. The following will be considerations used during an assessment:

Identity, quantity, source and extent of the release;

- Effects of hazardous surface water runoff from water or chemical agents used to control fire and heat induced explosions;
- Downwind hazard exposure in relation to wind direction; and

- The most effective techniques to be employed for providing containment.

14.4.6. Emergency Response and Fire Control Procedures

It is important that emergencies be dealt with in a calm, logical and safe manner. Containment of fire or hazardous materials flow is of primary importance, but not to the jeopardy of employee Health and Safety.

A Stone employee shall follow the following procedures after discovering a fire:

- Activate fire alarm system
- Notify the Emergency Coordinator or Alternate
- Following notification of the SSO, any attempt to put out the fire should be made only in its early stage. (Do not attempt to put out a fire larger than a trash can fire. Use the "Two Second Rule.")
- In situations where control cannot be established, initiate evacuation of the facility. Facility personnel will report to their designated reassembly locations for directions by their Supervisor/Manager.

The SSO shall conduct his/her activities during a fire to include the following:

- Identify the character, source, amount and the extent of any released materials. Determine the source and extent of the fire.
- Stop the flow of materials that may cause the fire to escalate or spread to other areas of the facility. Ensure the release of hazardous materials including runoff has been contained or stopped.
- Direction and control should be exercised to prevent the spread or recurrence of fires, explosions, or other hazardous materials releases.
- Use Emergency Response Checklist to Imminent Threats, (see below).
- The SSO shall make him/herself known to the fire chief or fire supervisor upon their arrival. The Fire Department will have primary responsibility for coordinating the response or any evacuation.
- Following notification that the facility is "all clear" by the Fire Department, the SSO shall inspect the facility. Only upon completion of this inspection shall employees be allowed to return to the building or work area.

14.4.7. Fire Classifications

Class "A" Fires - Occur in ordinary combustible materials such as wood, rubbish, paper and cloth and must be extinguished by a wetting agent such as water. After initial application of wetting agent, material should be stirred to make certain that the fire is out.

Class "B" Fires - Occur in flammable liquids such as paint, thinners, grease, oil, gasoline and some chemicals. Fires of this sort will be aggravated by the use of water. Therefore, class B fires must be extinguished using a dry chemical extinguisher. B-type, C-type, ABC-type and CO2-type extinguishers are all effective in putting out a class "B" fire. Caution: Fires extinguished by CO2-type extinguishers may reignite.

Class "C" Fires - Occur in electrical equipment and use of water on an electrical fire can cause electrocution. CO2-type extinguishers are effective on a class "C" fire and the fog created by their use will disappear. BC-type and ABC-type extinguishers are also effective; however, they will leave a powder residue on the equipment. Caution: The gas produced by CO2 extinguishers is extremely cold and, if in contact with skin, will cause burns.

Class "D" Fires - Occur in combustible metals such as magnesium, titanium, zirconium, sodium, lithium and potassium and can be extinguished utilizing special extinguishing agents approved by recognized testing laboratories.

14.4.8. Care and Use of Fire Extinguishers

- Always store extinguishers as close as possible to the hazard they're intended to control.
- Extinguishers are to be kept charged at all times and must be inspected by an outside source on an annual basis.

14.4.9. Additional Safeguards

Special care must be taken to insure that flammable materials are stored properly. All gasoline, thinners, paints, etc. will be stored in metal cans or approved containers with tight lids. Only cans designed to store flammable liquids and prevent explosions and accidental spills are to be used. Following are additional safeguards that are to be adhered to:

- Whenever possible, keep flammable liquids and paints in closed storage cabinets, closets or lockers.
- Keep all flammable liquids away from sparks, heat and open flames.
- Compressed oxygen and gas cylinders must be secured. Cylinders must be capped and stored away from heat, sparks and open flames.
- Oxygen, though itself nonflammable, is essential to combustion. Never refer to oxygen as air nor use oxygen from cylinders to eliminate dust or dirt.
- Always dispose of product cartons in an appropriate manner.
- Always follow manufacturer's instructions when using welding equipment, torches, hoses and electrical devices.
- Flammables are never to be stored in glass containers.

14.4.10. Emergency Response Checklist to Imminent Threats

- Hazardous Substance Releases
    - Determine Source
        - Shipping papers
        - Inventory
        - Container labels
    - Stop release/ensure containment

- Determine extent of contamination
- Initiate cleanup/decontamination
- Containerize recovered material
- Label and segregate containers of incompatible materials
- Place in storage facility
- Verify complete removal and cleanup

- Sudden Incidents
  - Implement procedures for spills, fires, or toxic gases if needed
  - Spill Procedures
    - Determine materials and quantities
    - Determine fire and breathing hazards
    - Implement fire procedures, if needed
    - Distribute protective equipment
    - Determine if ample storage capacity exists
    - Notify spill contractor, if needed
    - Contain liquid
    - Vacuum and absorb material
    - Remove contaminated earth
    - Containerize and store recovered material
    - Label and segregate vessels of incompatible hazardous waste
    - Clean equipment and replace protective clothing
- Fire Procedures
  - Determine the extent of involvement
  - Determine presence of toxic gases
  - Make evacuation decision
    - Prevailing winds
    - Extent of fire involvement
    - Evolution of toxic gases
    - Threat to main plant
    - Notify Fire Department, if needed
    - Appraise fire fighters of special hazards
    - Coordinate firefighting action
    - Extinguish fire

        - Implement spill procedures, if needed
        - Implement flooding procedures, if needed
- Fumes Emanating From Storage Areas
    - Evacuate local area/room
    - Determine fire/breathing hazard
    - Determine source of gases
    - Safe entry
        - Implement fire procedures, if needed
        - Correct problem directly
        - Implement spill or flooding procedures, if needed
    - Unsafe entry
        - Correct problem indirectly
        - Cut power to area
        - Cut water to area
        - Flood area
        - Inject fire-suppressing foam into area
    - Ventilate area to eliminate breathing hazard
    - Verify facility cleanup and operation

14.5. Hazardous Energy Control Plan

The Hazardous Energy Control Plan is intended to reduce the number of injuries by accidental startup of a machine or piece of equipment while undergoing service or routine maintenance. In addition, injuries from the release of stored energy could occur. This plan establishes requirements for minimum performance for control of such hazardous energy.

A “lockout device” is a locking device that provides a positive means for rendering a switch, valve, or energy source inoperable. The device may be a padlock, restraining bar, chain, or any device that positively prevents a machine or piece of equipment from becoming “energized” or from releasing stored energy.

A “tagout device” serves as a lockout and is a means of identifying who locked out the machinery, the date and time of day the lockout took place, and the group for which the person works. There is additional information that may be placed on the tag such as a phone number, extension number, etc. Tags are durable nature and can be securely fastened to the locking mechanism so as not to fall off.

Tags are never to be removed by anyone except the technician who is responsible for the lockout/tagout procedure.

14.5.1. Policy

Stone employees shall follow Lockout/Tagout (LOTO) procedures:

- During servicing and/or maintenance of machines and equipment.
- During removal or bypassing of a machine guard or other safety device.
- When placing any part of their body into an area where work is actually performed (point of operation) including danger zones with respect to a machine's normal operating cycle.

14.5.2. General Safety Guidelines

It is the responsibility of each technician to initiate all lockout/tagout procedures. The Technician completes the attached Lockout/Tagout Schedule Form for machinery of equipment requiring LOTO procedures.

14.5.3. Procedures

Using the proper form, the following sequence shall be used by the Technician whenever LOTO is required:

- Identify the piece of machinery or equipment requiring servicing or maintenance on the LOTO Schedule Form.
- Note the number and location of energy sources that require locks and/or tags for the piece of equipment or machinery being services.
- Note the hazards identified for the piece of equipment or machinery.
- Note the date
- Note the equipment Name and Location
- Not the LOTO start time.
- Note the LOTO end time.
- The Technician will perform the final inspection of the energy sources and operating controls to make certain they are disconnected, and check that the operating controls are turned to the OFF or NEUTRAL positions.
- The equipment is now locked out and tagged out. The I&R Technician will notify the Group Officers and employees in the immediate area of the machinery or equipment's "down" condition.

The following sequence shall be used to restore machines and equipment to normal operations:

- When maintenance or servicing has been completed and machinery or equipment is ready to be placed into normal operation, it is the responsibility of the Facilities Technician to check out the immediate area to confirm that no one is exposed to any danger.
- Remove or check that all tools have been removed from the machinery or equipment.
- Confirm that all guards, pulleys, and safety devices have been reinstalled and are secure.
- Remove all locks and tags only after a final check to ensure that all employees are free from hazards.
- Calibrate, monitor, and perform quality control checks on the machine or equipment as needed and appropriate.

15. PHYSICAL HAZARDS AND CONTROLS

15.1. Slips, Trips, and Falls

On any work area, it is expected that the ground may be uneven. The ground surface may be unreliable due to settling. Surface debris may be present and wet or swampy areas may exist.

Extension cords and hoses may be present. Cords and hoses should be routed so that they lay flat on the ground, and are long enough that they do not cross heavily traveled areas.

Tools such as shovels, rakes, and other hand tools will be used. Tools and equipment should not be left on the ground when not in use. They should be placed in a tool box, on a tool belt, or in specially designated and clearly marked areas. Orange or similar bright colored survey flagging may be attached to the tool handles to make them clearly visible.

Employees should walk around, not over or on top of debris or trash piles. When carrying equipment, identify a path that is clear of any obstructions. It may be necessary to remove obstacles to create a smooth, unobstructed access point to the work areas.

15.1.1. Working in Steep Terrain

Workers may be walking over rough terrain to perform the proposed sampling. This work may occur when temperatures, high winds and inclement weather, may make traversing the area difficult. Employees must be cautious when working not to become overzealous and try to quickly access areas on steep slopes.

All work will be conducted by a two-person team. The team will be equipped with appropriate communications equipment to facilitate contact between the team and the USACE on-site representative and/or the local emergency responders in the event of an emergency.

During winter months, each team member will be equipped with appropriate cold weather gear, as well as shoes equipped with strap-on, spike-like attachments that provide traction on ice during the winter, such as Yaktrax™.

15.1.2. Good Housekeeping

The active portion of the work area should be maintained in a neat and clean condition to reduce the opportunity for tripping when walking. Maintaining a work environment that is free from accumulated debris is the key to preventing slip, trip and fall hazards at construction sites. Essential elements of good housekeeping include:

- orderly placement of materials, tools and equipment;
- placing trash receptacles at appropriate locations for the disposal of miscellaneous rubbish;
- prompt removal and secure storage of items that are not needed to perform the immediate task at hand;
- awareness on the part of all employees to walk around, not over or on, equipment that may have been stored in the work area.

15.2. Working in Close Proximity to Operating Machinery

Stone will be working around and in close proximity to drill rigs. The use of such equipment poses a potential hazard to the support crew working around the equipment. Use of heavy equipment at the site requires all employees working in the exclusion zone to wear ANSI-approved Type I or Type II hard hats, safety-toed shoes/boots, safety glasses and hearing protection.

15.2.1. Working in Close Proximity to Drilling Equipment

Onsite personnel working in close proximity to drilling equipment will be required to observe the following safety rules as well as wear the PPE discussed in the paragraphs above.

- Personnel shall not remain in the vicinity of operating equipment unless it is required for their work responsibilities.
- All drill rigs and other machinery with exposed moving parts must be equipped with an operational emergency stop device. Drillers and geologists must be aware of the location of this device. This device must be tested prior to job initiation and periodically thereafter.
- Drillers, helpers and geologists must secure all loose clothing when in the vicinity of drilling operations to avoid entanglement.
- Make eye or verbal contact with operator to ensure that the rig is idle when entering work area, when approaching the drill string or hoist, when placing or removing blocking, and when operating auger racks to avoid being struck, entangled, or pinched by operating equipment

15.3. Noise

Working in close proximity to drill rigs may expose Stone employees to noise levels that exceed the OSHA PEL of 90 dBA for an 8-hour day. Exposure to noise can result in the following:

- Temporary hearing losses where normal hearing returns after a rest period;
- Interference with speech communication and the perception of auditory signals;
- Interference with the performance of complicated tasks; and
- Permanent hearing loss due to repeated exposure resulting in nerve destruction in the inner ear.

Although personal noise monitoring will not be conducted during the proposed activities, it is well established that drilling rigs and other heavy equipment can emit sound levels beyond the OSHA established limits. The table below outlines likely noise levels that can be experienced during site activities. Disposable earplugs or earmuffs with a minimum noise reduction rating (NRR) of 27 db will be worn anytime Stone employees are subjected to 85dBA or above in accordance with Table 5 below.

Table 5: RCNM Default Noise Emission Reference Levels and Usage Factors

Equipment Description	Impact Device?	Acoustical Usage Factor (%)	Spec. 721.560 L_{max} @ 50 feet (dBA, slow)	Actual Measured L_{mx} @ 50 feet (dBA, slow) (Samples Averaged)	Number of Actual Data Samples (Count)
All Other Equipment > 5 HP	No	50	85	N/A	0
Auger Drill Rig	No	20	85	84	36
Backhoe	No	40	80	78	372
Bar Bender	No	20	80	N/A	0
Blasting	Yes	50	85	N/A	0
Boring Jack Power Unit	No	50	80	83	1
Chain Saw	No	20	85	84	46
Clam Shovel (dropping)	Yes	20	93	87	4
Compactor (ground)	No	40	80	78	18
Compressor (air)	No	50	85	N/A	0
Concrete Batch Plant	No	15	83	N/A	0
Concrete Mixer Truck	No	40	85	79	40
Concrete Pump Truck	No	20	82	81	30
Concrete Saw	No	20	90	90	55
Crane	No	16	85	81	405
Dozer	No	40	85	82	55
Drill Rig Truck	No	20	84	79	22
Drum Mixer	No	50	80	80	1
Dump Truck	No	40	84	76	31
Excavator	No	40	85	81	170
Flat Bed Truck	No	40	84	N/A	0
Front End Loader	No	40	80	79	96
Generator	No	50	82	81	19
Generator(<25KVA, VMS Signs)	No	50	85	N/A	0
Gradall	No	40	85	83	70
Grader	No	40	85	N/A	0
Grapple (on backhoe)	No	25	80	82	6
Hydra Break Ram	Yes	10	90	N/A	0
Impact Pile Driver	Yes	20	95	101	11
Jackhammer	Yes	20	85	89	133

Equipment Description	Impact Device?	Acoustical Usage Factor (%)	Spec. 721.560 L_{max} @ 50 feet (dBA, slow)	Actual Measured L_{mx} @ 50 feet (dBA, slow) (Samples Averaged)	Number of Actual Data Samples (Count)
Man Lift	No	20	85	75	23
Mounted Impact Hammer (hoe ram)	Yes	20	90	90	212
Pavement Scarifier	No	20	85	90	2
Paver	No	50	85	77	9
Pickup Truck	No	40	55	75	1
Pneumatic Tools	No	50	85	85	90
Pumps	No	50	77	81	17
Refrigerator Unit	No	100	82	73	3
Rivet Buster/Chipping Gun	Yes	20	85	79	19
Rock Drill	No	20	85	81	3
Roller	No	20	85	80	16
Sand Blasting (single nozzle)	No	20	85	96	9
Scraper	No	40	85	84	12
Sheers (on backhoe)	No	40	85	96	5
Slurry Plant	No	100	78	78	1
Slurry Trenching Machine	No	50	82	80	75
Soil Mix Drill Rig	No	50	80	N/A	0
Tractor	No	40	84	N/A	0
Vacuum Excavator (Vac-Truck)	No	40	85	85	149
Vacuum Street Sweeper	No	10	80	82	19
Ventilation Fan	No	100	85	79	13
Vibrating Hopper	No	50	85	87	1
Vibratory Concrete Mixer	No	20	80	80	1
Vibratory Pile Driver	No	20	95	101	44
Warning Horn	No	5	85	83	12
Welder/Torch	No	40	73	74	5

Reference: Reherman, Clay M., Rochat, Judith L., Thalheimer, Erich S., Lau, Michael C. Fleming, Gregg C., Ferroni, Mark and Corbisier, Christopher. Roadway Construction Noise Model (RCNM). Federal Highway Administration, Washington, DC: January 2006.

15.4. Air Compressor Use

Compressed air or nitrogen may be used to drill and develop wells, for geophysical work, or to inflate packers. Compressed air may also be used to inflate bladder pumps used for groundwater sampling. When using an air compressor or compressed nitrogen, the following safety requirements must be implemented:

- Operators shall perform a pre-operational check of all air hoses, couplings and connections to determine if leakage or other damage exists. Be familiar with operator's manual. Do not operate unsafe equipment.
- All pipes, hoses, and fittings must have a rating of the maximum pressure of the compressor.
- Air supply shutoff valves should be located (as near as possible) at the point-of-operation.
- Air hoses should be kept free of grease and oil to reduce the possibility of deterioration.
- Hose ends must be secured to prevent whipping if an accidental cut or break occurs.
- Pneumatic impact tools should never be pointed at a person.
- Before a pneumatic tool is disconnected (unless it has quick disconnect plugs), the air supply must be turned off at the control valve and the tool bled.
- Compressed air must not be used under any circumstances to clean dirt and dust from clothing or off a person's skin. When directed against the skin, even 30 psi of compressed air can be driven into the flesh or eyes. If there is a break in the skin, air can be driven into tissue, causing swelling and pain. Bubbles of air can be driven into blood vessels, which can be serious, even fatal.
- Goggles, face shields or other eye protection must be worn by personnel using compressed air for cleaning equipment.

15.5. Cuts and Lacerations

Minor wounds are the most likely of the risks to persons moving about the project site. Cuts and abrasions are most likely to occur as individuals move through trees and underbrush and contact thorny plants, sharp stones, sharp ends of branches, and similar objects. Many of these injuries can be prevented by wearing appropriate clothes, gloves, and footwear. Eye injuries can be prevented by wearing safety glasses, or goggles. Most of these types of injuries require only minor first aid administered on site. Medical attention should be sought for more serious injuries.

Employees are at an increased risk of cutting themselves with the knives used to cut tubing for groundwater sampling. Tube-cutters are available and should be used to eliminate this hazard. If knives or blades must be used, follow the safety precautions listed below:

- Keep your free hand out of the way
- Secure your work if cutting through thick material
- Use only sharp blades; dull blades require more force which results in less knife control
- Don't put your knife in your pocket
- Use a self-retracting blade
- Wear leather or Kevlar® gloves when using knives or blades.

15.6. Back Safety

The manual moving and handling of materials on a site poses a risk to workers in the form of muscle strains and minor injuries due to dropped containers. Using the proper techniques to lift and move heavy pieces of equipment is important to reduce the potential for back injury. The following precautions should be implemented when lifting or moving heavy objects (generally defined as fifty pounds or more under ”perfect” conditions, and less if loading or unloading vehicles, if the weight is not evenly distributed in the object, or if the ground conditions are not level, smooth and dry):

- Use mechanical devices to move objects that are too heavy to be moved manually.
- If mechanical devices are not available, ask another person to assist you.
- Bend at the knees, not the waist. Let your legs do the lifting.
- Do not twist while lifting.
- Bring the load as close to you as possible before lifting.
- Be sure the path you are taking while carrying a heavy object is free of obstructions and slip, trip and fall hazards.

15.7. Heat and Cold Stress Monitoring Plan

Work in New York could present both cold and heat stress concerns.

15.7.1. Cold Stress

15.7.1.1. Types of Cold Stress

Cold injury is classified as either localized, as in frostbite, frostnip or chilblain; or generalized, as in hypothermia. The main factors contributing to cold injury are exposure to humidity and high winds, contact with wetness and inadequate clothing. Hypothermia may appear at temperatures above freezing, especially if the person becomes wet from precipitation, sweating, or immersion in water.

The likelihood of developing frostbite increases when the face or extremities are exposed to a cold wind in addition to cold temperatures. The freezing point of the skin is about 30° F. When fluids around the cells of the body tissue freeze, skin turns white. This freezing is due to exposure to extremely low temperatures. As wind velocity increases, heat loss is greater and frostbite will occur more rapidly.

15.7.1.2. Symptoms of Cold Stress

Symptoms of hypothermia, a condition of abnormally low body temperature, include uncontrollable shivering and sensations of cold. The heartbeat slows and may become irregular, the pulse weakens and the blood pressure changes. Pain in the extremities and severe shivering can be the first warning of dangerous exposure to cold.

Maximum severe shivering develops when the body temperature has fallen to 95° F. Productive physical and mental work is limited when severe shivering occurs. Shivering is a serious sign of danger. Immediately remove any person who is shivering from the cold.

The first symptom of frostbite is usually an uncomfortable sensation of coldness, followed by numbness. There may be a tingling, stinging or aching feeling in the affected area. Patches of white may be visible on exposed skin. The most vulnerable parts of the body are the nose, cheeks, ears, fingers and toes.

15.7.1.3. Cold Stress Monitoring Techniques

- Mental Function: Monitor personnel for decreases in mental function as exhibited by fatigue, forgetfulness, reduced alertness, increase in minor injuries, etc. Any worker showing signs of decreased mental function shall be placed in a warm environment for a minimum period of 10 minutes. If symptoms persist, the worker shall be released of work that further exposes them to cold until approved by the SSO.
- Physical Signs: If any worker complains of serious discomfort or exhibits discoloration of lips or extremities, numbness, or excessive shivering, the worker shall be moved to a warm location until symptoms abate. The worker shall not be allowed to work on any task that further exposes them to the cold until approved by the SSO.

15.7.1.4. Methods to Prevent Cold Stress

When the ambient temperature, or a wind chill equivalent, falls to below 40° F (American Conference of Governmental Industrial Hygienists recommendation), site personnel who must remain outdoors should wear insulated coveralls, insulated boot liners, hard hat helmet liners and insulated hand protection. Wool mittens are more efficient insulators than gloves. Keeping the head covered is very important, since 40% of body heat can be lost when the head is exposed. If it is not necessary to wear a hard hat, a wool knit cap provides the best head protection. A facemask may also be worn.

Persons should dress in several layers rather than one single heavy outer garment. The outer piece of clothing should ideally be wind and waterproof. Clothing made of thin cotton fabric or synthetic fabrics such as polypropylene is ideal since it helps to evaporate sweat. Polypropylene is best at wicking away moisture while still retaining its insulating properties. Loosely fitting clothing also aids in sweat evaporation. Denim is not a good protective fabric. It is loosely woven which allows moisture to penetrate. Socks with high wool content are best. If two pairs of socks are worn, the inner sock should be smaller and made of polypropylene or similar types of synthetic material that wick away moisture. If clothing becomes wet, it should be taken off immediately and a dry set of clothing put on.

If wind conditions become severe, it may become necessary to shield the work area temporarily. The SSO and the PM will determine if this type of action is necessary. Heated break trailers or a designated area that is heated should be available if work is performed continuously in the cold at temperatures, or equivalent wind chill temperatures, below 20° F.

Dehydration occurs in the cold environment and may increase the susceptibility of the worker to cold injury due to significant change in blood flow to the extremities. Drink plenty of fluids, but limit the intake of caffeine.

15.7.2. Heat Stress

15.7.2.1. Types of Heat Stress

Heat related problems include heat rash, fainting, heat cramps, heat exhaustion and heat stroke. Heat rash can occur when sweat isn't allowed to evaporate, leaving the skin wet most of the time and making it subject to irritation. Fainting may occur when blood pools to lower parts of the body and as a result, does not return to the heart to be pumped to the brain. Heat related fainting often occurs during activities that require standing erect and immobile in the heat for long periods of time. Heat cramps are painful spasms of the muscles due to excessive salt loss associated with profuse sweating. Heat exhaustion results from the loss of large amounts of fluid and excessive loss of salt from profuse sweating. The skin will be clammy and moist and the affected individual may exhibit giddiness, nausea and headache.

Heat stroke occurs when the body's temperature regulatory system has failed. The skin is hot, dry, red and spotted. The affected person may be mentally confused and delirious. Convulsions could occur. EARLY RECOGNITION AND TREATMENT OF HEAT STROKE ARE THE ONLY MEANS OF PREVENTING BRAIN DAMAGE OR DEATH. A person exhibiting signs of heat stroke should be removed from the work area to a shaded area. The person should be soaked with water to promote evaporation. Fan the person's body to increase cooling.

15.7.2.2. Early Symptoms of Heat-Related Problems

- decline in task performance
- excessive fatigue
- loss of coordination
- reduced vigilance
- decline in alertness
- muscle cramps
- unsteady walk
- dizziness

Susceptibility to Heat Stress Increases due to:

- lack of physical fitness
- obesity
- lack of acclimation
- drug or alcohol use
- increased age
- sunburn
- dehydration
- infection

People unaccustomed to heat are particularly susceptible to heat fatigue. First timers in PPE need to gradually adjust to the heat.

15.7.2.3. The Effect of Personal Protective Equipment

Sweating normally cools the body as moisture is removed from the skin by evaporation. However, the wearing of certain personal protective equipment (PPE), particularly chemical protective coveralls (e.g., Tyvek), reduces the body's ability to evaporate sweat and thereby regulate heat buildup. The body's efforts to maintain an acceptable temperature can therefore become significantly impaired by the wearing of PPE.

15.7.2.4. Measures to Avoid Heat Stress

The following guidelines should be adhered to when working in hot environments:

- Establish work-rest cycles (short and frequent are more beneficial than long and infrequent).
- Identify a shaded, cool rest area.
- Rotate personnel, alternate job functions.
- Water intake should be equal to the sweat produced. Most workers exposed to hot conditions drink less fluid than needed because of an insufficient thirst. DO NOT DEPEND ON THIRST TO SIGNAL WHEN AND HOW MUCH TO DRINK. For an 8-hour workday, 50 ounces of fluids should be drunk.
- Eat lightly salted foods or drink salted drinks such as Gatorade to replace lost salt.
- Save most strenuous tasks for non-peak heat hours such as the early morning or at night.
- Avoid double shifts and/or overtime.

The implementation and enforcement of the above mentioned measures will be the joint responsibility of the PM and SSO. Potable water should be made available each day for the field team.

15.7.2.5. Heat Stress Monitoring Techniques

Site personnel should regularly monitor their heart rate as an indicator of heat strain by the following method: Check radial pulse rates by using fore-and middle fingers and applying light pressure to the pulse in the wrist for one minute at the beginning of each rest cycle. If the pulse rate exceeds 110 beats/minute, shorten the next work cycle by one-third and keep the rest period the same. If, after the next rest period, the pulse rate still exceeds 110 beats/minute, shorten the work cycle by one-third.

Table 6 provides guidelines for work-rest regimens, but does not replace the self-monitoring techniques described above. Work activity levels (work load) in Table 6 are based on the following ACGIH categories:

Rest includes sitting quietly in place or sitting with moderate arm movements, and is in the same environment as the work activity. Relocation to a shaded environment and/or removal of personal protective equipment to allow for more effective cool-down between work periods may be considered where possible.

Light work includes sitting with moderate arm and leg movements; standing with light work at a machine or bench while using mostly arms or with some walking about.

Moderate work includes vigorous arm movement in a standing position; walking about with moderate lifting or pushing; walking on level at 6 km/hr while carrying a 3 kg weight load.

Heavy work includes heavy arm efforts such as hand sawing carpentry; shoveling dry sand; heavy assembly work on a non-continuous basis; intermittent heavy lifting pushing or pulling (e.g., pick-and-shovel work).

Very heavy work includes strenuous work such as shoveling wet sand.

15.8. Excavation / Trenching Plan

Excavation and trenching are not activities included in this project.

Table 6: Typical Work-Rest Cycles for Warm Weather Conditions

		ACCLIMATIZED				UNACCLIMATIZED			
	Work Demands	Light	Moderate	Heavy	Very Heavy	Light	Moderate	Heavy	Very Heavy
ONE CLOTHING LAYER	100% Work	85.1°F	81.5°F	78.8°F	N/A	81.5°F	77.0°F	72.5°F	N/A
	75% Work 25% Rest	86.9°F	83.3°F	81.5°F	N/A	84.2°F	79.7°F	76.1°F	N/A
	50% Work 50% Rest	88.7°F	85.1°F	83.5°F	81.5°F	86.0°F	82.4°F	79.7°F	77.0°F
	25% Work 75% Rest	90.5°F	87.8°F	86.0°F	85.1°F	87.8°F	84.2°F	82.4°F	79.7°F
TWO LAYERS	100% Work	78.8°F	75.2°F	72.5°F	N/A	75.2°F	70.7°F	66.2°F	N/A
	75% Work 25% Rest	80.6°F	77.0°F	75.2°F	N/A	77.9°F	73.4°F	69.8°F	N/A
	50% Work 50% Rest	82.4°F	78.8°F	77.0°F	75.2□F	79.7°F	76.1°F	73.4°F	70.7°F
	25% Work 75% Rest	84.2°F	81.5°F	79.7°F	78.8°F	81.5°F	77.9°F	76.1°F	73.4°F
THREE LAYERS	100% Work	76.1°F	72.5°F	69.8°F	N/A	72.5°F	68.0°F	63.5°F	N/A
	75% Work 25% Rest	77.0°F	74.3°F	72.5°F	N/A	75.2°F	70.7°F	67.1°F	N/A
	50% Work 50% Rest	79.7°F	76.1°F	74.3°F	72.5°F	77.0°F	73.4°F	70.7°F	68.0°F
	25% Work 75% Rest	81.5°F	78.8°F	77.0°F	76.1°F	78.8°F	75.2°F	73.4°F	70.7°F

15.9. Biological Hazards

Work is scheduled for the late fall. Some of the project locations are wooded and the potential does exist for encountering poisonous plants, insects, and spiders.

15.9.1. Poisonous Plants

Persons working on the Site should be aware of the presence of poisonous plants. **Poison ivy** is a climbing plant with leaves that consist of three glossy, greenish leaflets. Poison ivy has conspicuous red foliage in the fall. Small yellowish-white flowers appear in May through July at the lower leaf axils of the plant. White berries appear from August through November. Poison ivy is typically found east of the Rockies. **Poison oak** is similar to poison ivy but its leaves are oak-like in form. Poison oak occurs mainly in the south and southwest. **Poison sumac** typically occurs as a small tree or shrub and may be 6-20 feet in height. The bark is smooth, dark and speckled with darker spots. Poison sumac is typically found in swampy areas and east of the Mississippi. The leaves have 7-13 smooth-edged leaflets and drooping clusters of ivory-white berries appear in August and last through spring.

The leaves, roots, stems and fruit of these poisonous plants contain oil called urushiol. Contact with the irritating oil causes an intensely itching skin rash and characteristic, blister-like lesions. The oil can be transmitted on soot particles when burned and may be carried on the fur of animals, equipment and apparel.

Proper identification of these plants is the key to preventing contact and subsequent dermatitis. Wear long sleeves and pants when working in wooded areas. In areas of known infestation, wear Tyvek coveralls and gloves. Oils are easily transferred from one surface to another. If you come in contact with these poisonous plants, wash all exposed areas immediately with cool water, detergent solution, or alcohol saturated wipes to remove the oils. Some commercial products such as Tecnu's Poison Oak-n-Ivy Cleanser claim to further help with the removal of oils.

15.9.2. Insects, Spiders

Insects, spiders, bees, and wasps can be prevalent during certain times of the year. Field personnel should try to avoid contact by surveying the area in which they will be walking, standing, sitting, leaning, grabbing, lifting, or reaching. Workers are encouraged to wear insect repellent when working in areas where insects are expected to be a hazard.

- Insect bites or stings - Many insects bite or sting, but few can cause serious effects by themselves, unless the person is hypersensitive to contact. Bee, wasp, and hornet hives may be present in some buildings and active during certain times of the year. The insect stings are a hazard to personnel who are hypersensitive. Site personnel with known or suspected sensitivity should carry their “bee sting kit,” which normally includes antihistamine and epinephrine to counter anaphylactic reactions.
- Although rare the only spiders dangerous spiders found in the Northeast are the black widow and brown recluse spiders - The black widow spider has a shiny black body about the size of a pea, and has a red or yellow hourglass-shaped mark on its abdomen. It weaves shapeless webs in undisturbed areas, such as woodpiles. A bite may result in severe pain, illness, or death (usually from complications and not from the bite itself). Only the female is poisonous; the male is harmless, The brown recluse is golden brown with a dark brown or black "fiddle," which is often

shiny. They range from 5-15 mm (1/4 to 3/4 inch) long. Brown recluse spiders are found primarily in the South and Midwest. They are rarely, if ever, found in New York. Brown recluse spiders live in basements and garages of houses, often hiding behind boards and boxes. Bites sometimes occur when the spiders hide in towels or clothing. They are seldom aggressive and bite only when threatened or injured. The severity of the bite of the brown recluse may vary from no symptoms at all to a reaction that is very severe. Often there is a systemic reaction within 24-36 hours of the bite characterized by restlessness, fever, chills, nausea, weakness and joint pain.

- Rocky Mountain Spotted Fever - Bites of wood ticks may result in the transmission of Rocky Mountain Spotted Fever, a serious and possibly fatal disease caused by the *Rikettsia* virus. The *Rikettsia* virus infects wood ticks, which may bite humans, thereby transferring the virus into the bloodstream. Rocky Mountain Spotted Fever occurs mostly in the late spring and early summer, and is characterized by chills, fever, severe pain in the leg muscles and joints, and a body rash.
- Lyme Disease - Female deer ticks are about 1/16 inch in length. If a tick is not removed, or if the tick is allowed to remain for days feeding on human blood, a condition known as tick paralysis can develop. This is due to a neurotoxin, which the tick apparently injects while engorging. This neurotoxin acts upon the spinal cord causing loss of coordination, weakness and paralysis. Lyme disease is most often characterized with a "bull's-eye" rash, accompanied by nonspecific symptoms such as fever, malaise, fatigue, headache, muscle aches, and joint aches. PPE offers some protection against insects, but personnel should also perform a self-search at the end of each day to check for ticks if they have been working in areas where ticks may be prevalent. To remove a tick, grasp the head, not the body, firmly with tweezers and gently pull the head out. If the bite is irritated treat it with antiseptic.

15.10. Military and Explosive Constituents

No military ordnance has been found at the Site to date, nor is ordnance expected at this site. However, if during the course of investigation ordnance is observed, team members will:

- Stop work immediately and leave the ordnance in place.
- Secure the work area with yellow hazard tape
- Leave the work area
- Contact Army Corps of Engineers Project Manager (Gregory Geopfert, 917-790-8235).

16. SITE CONTROL

16.1. Work Zones

To prevent both exposure of unprotected personnel and migration of contamination due to tracking by personnel or equipment, work areas along with personal protective equipment requirements will be clearly identified. The SSO will designate work areas or zones as suggested in the "Occupational Safety and Health Guidance Manual for Hazardous Waste Site Activities," NIOSH/OSHA/USCG/EPA, November 1985. They recommend the areas surround each of the work areas to be divided into three zones:

- Exclusion or "hot" zone
- Contamination Reduction Zone (CRZ)
- Support Zone

16.1.1. Exclusion Zone

Exclusion zones will be established around any work area where heavy equipment, such as an excavator or drill rig, is used. These zones will be demarcated with traffic cones and/or safety warning tape. All employees entering an exclusion zone or any work area must be prepared to don the required PPE for the work tasks being conducted in that area.

16.1.2. Contamination Reduction Zone

The decontamination zone will be established adjacent to the exclusion zone for the decontamination of the drill rig and tools that are in contact with the potentially contaminated soil, rock, and groundwater. Traffic cones, signs, and warning tape will be utilized as needed to restrict access to the decontamination station when not in use.

A personnel decontamination zone for personnel will be established adjacent to each immediate work area. Personnel will remove contaminated gloves and other disposable items in this area and place them in a plastic bag until they can be properly disposed of.

16.1.3. Support Zone

At this Site, the support zone will include the team member field vehicles.

16.2. Site Communication

The Site is not a remote location. Cell phones, and landlines will be used to facilitate contact between team members and the USACE on-site representative and/or local emergency response personnel in the event of an emergency.

16.3. General Field Work Rules

These rules shall be observed for all tasks conducted (as appropriate) at any project site.

- The "buddy system" shall be used at all times by field personnel in the exclusion zone.
- Visual, voice or radio communications shall be maintained at all times.

- Contact with contaminated surfaces, water, soils, and equipment shall be avoided whenever possible. Individuals shall not walk on discolored surfaces or kneel on the ground, nor lean, sit or place equipment on drums or containers.
- Eating, drinking, and smoking are prohibited in exclusion and decontamination zones. Alcoholic beverages and illegal drugs are prohibited on the job site.
- Hands and face must be thoroughly washed upon leaving a decontamination area. Further, whenever decontamination procedures for outer garments are in effect, the entire body shall be thoroughly washed as soon as possible after the protective garment is removed.
- Failure to complete a respiratory fit test shall preclude admission to any zone requiring respirator wear.
- All equipment must be decontaminated or discarded upon exiting an exclusion zone.
- Personal protective equipment shall be required for all field personnel and may include, but is not limited to, respiratory protection, hard hat, chemical resistant coveralls, boots, gloves, safety glasses, and ear protection.
- Anyone reporting to work under the influence of alcohol and/or illegal drugs will not be allowed access to the site. Any employee taking prescription medication or under a physician's care must notify the SSO prior to reporting to work.
- All persons shall listen for and yield the right-of-way to construction equipment.
- All equipment operators shall provide warning prior to movement and watch for personnel in their path.
- Employees are responsible for cleaning and maintaining the protective equipment issued to them. Any and all defects or failures of the equipment shall be reported immediately to the SSO.
- All personnel shall report all injuries and/or illnesses to the SSO regardless of the severity of injuries.
- Personnel shall avoid any potentially dangerous environmental situations such as entering a confined space without proper supervision, training and equipment.
- "Dig Safe" must be notified at least 72 hours prior to beginning excavation work.
- All personnel are required to contact the PM when conducting a field investigation or site inspection. At a minimum, they must check in and out of the Site with the PM or designee. If no other project personnel are on the site, the field person shall call the PM or Site Safety Officer at the home office prior to initiating field tasks and immediately after completing them. It is advisable to notify the PM at the time of the initial call of the estimated time required to complete the tasks.
- Personnel encountering a potentially hazardous environment (e.g., noticing strong vapor levels of unidentified substances) shall instruct all other on-site personnel to leave the Site and shall call the PM and SSO for instruction. Personnel shall not re-enter the Site without proper protective clothing, and shall not work at the Site until the unknown substance is identified and characterized.

- A NIOSH approved hard hat shall be worn at all times when overhead hazards are present.
- A shirt and long pants shall be worn at all times.
- Goggles shall be worn when handling chemicals that may damage the eyes.
- Skin abrasions must be thoroughly protected to prevent chemical exposure.
- Each employee is responsible for his or her health and safety.

17. DECONTAMINATION

17.1. Personnel Decontamination

Proper decontamination is required of all personnel before leaving the site. Decontamination will occur within the decontamination zone. Disposable PPE will be removed in the decontamination zone and placed in garbage bags. All disposable PPE will be disposed of as solid waste.

Regardless of the type of decontamination system required, potable water and liquid soap should be made available so employees can wash their hands before leaving the Site for lunch or for the day.

Potable water will be brought to the daily work location by the work vehicles. Portable sanitary facilities will be provided near work zones on the Property.

17.2. Equipment Decontamination

Hand-held equipment will be decontaminated prior to introducing it into a well, between wells, and after completion of field work at a particular well or sampling point. Decontamination will generally consist of a tap water rinse to remove gross contamination, followed by a non-phosphate detergent water wash and rinse, an isopropanol rinse, and finally a triple de-ionized or distilled water rinse.

Heavy equipment will be decontaminated as specified in the work plan. The drill rig(s), drill tools, and associated equipment shall be cleaned with a high temperature and pressure, hot water sprayer prior to commencement and after completion of drilling at each location.

The equipment decontamination shall be performed at the area designated adjacent to the work location. Portable water for decontamination will be obtained from an approved source.

17.3. Investigation-Derived Waste Handling

Development water and purge water that is pumped from the wells will be contained and analyzed for VOCs.

All soil cuttings and rock cuttings created during the drilling shall be evaluated using analytical data to determine if they are regulated waste and either temporarily stored on-site, transported to a licensed offsite disposal location, or deposited/discharged on-site. If necessary, additional analysis will be performed to fully characterize the wastes in accordance with applicable regulations.

18. EMERGENCY RESPONSE CONTINGENCY PLAN

18.1. Pre-Emergency Planning

The purpose of the emergency response contingency plan is to ensure that in the event of an emergency, personnel on the Site will have the information and understanding in place to institute a reasoned and rational response to the emergency without unnecessary delay. Potential emergencies cover a broad range of occurrences from acute (sudden onset) illness or injury, to fire, explosion, or severe weather conditions. They may be complicated by leaks or by the release of contaminants.

Minimum equipment that will be required on site includes a 20-pound ABC dry chemical fire extinguisher, a first aid kit, and a portable eye wash station. The fire extinguisher, first aid kit and portable eye wash station shall be kept in accessible locations adjacent to the work area.

18.1.1. Employee Training

Employees must be instructed in the site-specific aspects of emergency evacuation. On-site refresher or update training is required any time escape routes or procedures are modified or personnel assignments are changed.

18.1.2. Alarm Systems/Emergency Signals

An emergency communication system must be in effect at all sites. The most simple and effective emergency communication system in many situations will be direct verbal communications. Each site must be assessed at the time of initial site activity and periodically as the work progresses. Verbal communications must be supplemented any time voices cannot be clearly perceived above ambient noise levels (i.e., noise from heavy equipment; drilling rigs, backhoes, etc.) and anytime a clear line-of-sight cannot be easily maintained amongst all personnel because of distance, terrain or other obstructions.

An air horn and vehicle horns will be used in the event of an emergency. Three blasts on the horn will indicate that all on-site personnel are to immediately proceed to the primary emergency muster location (designated during the morning health and safety meeting each day based upon the work locations, wind direction, and other pertinent factors).

Verbal communications will be adequate to warn employees of hazards associated with the immediate work area. Telephone facilities are not available in the immediate work areas. Traditional cell phones or other appropriate communications equipment, such as walkie-talkies, will be used to facilitate contact between the team and the USACE on-site representative and/or the local emergency response personnel in the event of an emergency.

18.1.3. Escape Routes and Procedures

The escape route from the work area is via Avenue Y to Amsterdam Road (NY Route 5).

18.2. Personnel Responsibilities

Emergency response contingency plans can be effective only if the appropriate personnel understand their roles during the emergency, the lines of authority, the training they have received, and the necessity for clear communication.

18.2.1. General Roles and Responsibilities

The role of all site personnel in the event of an emergency is to: communicate to the SSO that an emergency exists; respond appropriately to the particular emergency signal; evacuate and secure the work area or emergency area; conduct the appropriate decontamination activities; render first aid; and call the emergency response services (e.g., the local or regional emergency medical service, fire department, or other specialized personnel or services).

Site personnel shall not engage in firefighting (beyond the use of on-site fire equipment such as extinguishers) or hazardous materials cleanup (beyond clean-up of minor spills or collection of contaminated PPE, etc.). A minor spill is defined as less than one gallon.

Each morning, the SSO will designate a primary and alternative muster location to be used in the event of an emergency. The muster locations will be described during the morning health and safety meeting based upon the work locations, wind direction, and other pertinent factors.

18.2.2. Specific Personnel Responsibilities

Individual personnel associated with site operations, and who will be responsible for maintaining and implementing the emergency response contingency plan, are summarized below.

PM: Responsible for ensuring that the features of the emergency response contingency plan are in place, personnel are appropriately trained, and required health safety, and spill response equipment are maintained on the site. In the event the SSO is not able to direct emergency responses the responsibility will fall on the PM. Under those circumstances, the PM will assume the responsibility for evacuation and other emergency actions.

Corporate Health and Safety Officer: Responsible for the overall health and safety of the personnel conducting work associated with a site, compliance with appropriate regulations, review of the emergency response contingency plan to ensure its compliance with all appropriate regulations and company policy, and to review the adequacy of any emergency response and make any corrective measures that may be necessary

Site Technicians and Contractors: Responsible for notifying the SSO immediately upon an emergency, and responding to an emergency situation as described in this plan. The initial response may be required if the SSO is not present on the site.

Site Safety Officer: The SSO is responsible for the initial response to an emergency situation. These duties include the protection of employees, evacuating and securing the work area, ensuring the protection of the public and the environment, ensuring that appropriate decontamination procedures are implemented on all personnel, determining the cause of the incident, recommending changes to prevent its reoccurrence, upgrading or downgrading the level of Personal Protective Equipment, and notification of the appropriate Federal, State and local agencies.

18.2.3. Rescue and Medical Duty Assignments

The phone numbers of the police and fire departments, ambulance service, local hospital, and Stone representatives are provided in the emergency reference sheet located at the end of this HASP/AHA (before Attachment A).

In the event an injury or illness requires more than first aid treatment, the SSO or his designee will accompany the injured person to the medical facility and will remain with the person until release or admittance is

determined. The escort will relay all appropriate medical information to the PM and the Corporate Health and Safety Officer.

18.2.4. Designation of Responsible Parties

The SSO is responsible for initiating emergency response. In the event the SSO cannot fulfill this duty, the alternate SSO will take charge. All personnel on site are responsible for knowing the escape route from the site and where to assemble after evacuation.

18.2.5. Employee Accounting Method

The SSO is responsible for identifying all team personnel on-site at all times. On small, short duration jobs such as this, this can be done informally as long as accurate accounting (i.e. via headcount) is possible.

18.3. Communication

Every effort shall be made during an emergency to communicate in clear and concise terms. Notice of an emergency shall be communicated to site personnel by a signal from a hand held compressed gas horn. The specific emergency communication signals shall be established by the Site Safety Officer, who will also be responsible for providing the means, such as a compressed gas or vehicle horn, for making this communication. Before work begins, the emergency communication signals shall be made clear to all site workers.

18.4. Emergency Procedures

Personnel shall be familiar with the emergency procedures described below in case of emergencies such as fire, explosion, spills, or leaks. Individuals shall be familiar with their responsibilities in case evacuation of the Site is necessary.

18.4.1. Fire or Explosion

In the event of a fire or explosion, call the Fire Department immediately. Upon arrival of the Fire Department, advise the commanding officer of the location, nature, and identification of hazardous materials on-site. Only trained experienced fire fighters should attempt to extinguish burning materials on site. Site personnel should not attempt to fight any fire, unless properly trained and equipped to do so.

18.4.2. Penetration of Subsurface Containers

It is possible, although highly unlikely, that a drum or similar container was buried on-site in the past, and could be penetrated during drilling. Symptoms of such an event could include:

- Sudden dropping of the drill string
- Expulsion of noxious vapors from the drill string or ground
- The presence of non-aqueous phase liquids on the drill string or in the hole.

The initial responses to one of these events will be:

- Turn off the drill rig
- Monitor air quality with a PID

- Clear the exclusion zone of all personnel and secure the area with yellow hazard tape
- Notify the FOL.

If the PID readings warrant, or if releases of vapors or liquids continue, the event should be treated similar to a spill, the release contained, and the area evacuated as necessary to protect human health and the environment (see Sections below).

After the area is secure, immediately contact Army Corps of Engineers Project Manager (Gregory Geopfert, 917-790-8235).

The State of New York requires all spills to be reported, so after informing the USACE PM, spills should be reported to the NYSDEC at 1-800-457-7362 within 2 hours of discovery. Additionally, hazardous waste spills must be reported in writing to NYSDEC within 15 days.

18.4.3. Spill or Leaks of Hazardous Materials

18.4.3.1. Scope

This section addresses the potential for spillage/accidental release of containerized liquids that potentially will be present on site. The materials susceptible to spillage that may be present on site are as follows: gasoline, diesel, hydraulic fluid, lubricants, and water in temporary holding tanks.

18.4.3.2. Preventative Measures

All containerized liquids shall be stored in a designated area, isolated from vehicular traffic and protected from the effects of weather. Measures to prevent spills or leaks include:

- all containers shall be labeled as to their contents and principal hazards, in accordance with 29 CFR 1910.120;
- a file of Material Safety Data Sheets (MSDS) shall be maintained by the SSO for all materials introduced to the site;
- flammable or combustible liquids shall be stored separately, and the area shall be placarded to warn of their presence; and
- a supply of specially designed sorbent pads and "minibooms" will be maintained on site for use in spill responses, along with drums suitable for the recovery of used sorbents and contaminated soil.

18.4.3.3. Spill Response Procedures

Training: During the initial on-site health and safety briefing, employees will be informed of the potential hazards of the materials with which they work. The following spill procedures will also be presented at that time. Only those employees trained in accordance with 29 CFR 1910.120 will assist in the clean-up procedure.

Initial response: Upon observing a spill or leakage from a container, employees will immediately step back to a safe distance and direct other employees away from the spill. Initial response actions will include the following:

- Sources of ignition within 100 feet of the spill (including vehicle engines) will be de-energized;

- Inside buildings any exhaust fans will be turned on and overhead doors opened.
- All combustible materials (such as wood, paper, oil, etc.) will be removed from the spill area.

The PM and SSO will be advised immediately of the location, size, and nature of the spill, including the identity of the spilled material (since all containers brought to the Site should be labeled, the spilling of unidentified liquids is not anticipated to be an issue);

Employees who have had contact with the spilled material will be decontaminated as needed.

Planning for Containment/Cleanup: As conditions dictate, the PM or SSO will determine the appropriate follow-up responses. These may include any combination of: initiating containment/clean-up, requesting outside assistance, and notifying affected authorities. The SSO will be notified of the occurrence of any spill in a timely manner.

The SSO will consult the MSDS for the spilled material to determine the specific approach to containing the spill, including the personal protective equipment to be worn during response activities.

The SSO will conduct air monitoring appropriate to the nature of the spilled material, to quantify exposure and detect flammable atmospheres and specify the required PPE for containment/cleanup activities.

Containment/Clean-up: The following are examples of specific measures that may be used (in combination when necessary) to respond to a spill.

- Upright or rotate containers to stop the flow of liquids. This step may be accomplished as soon as the spill or leak occurs, providing it is safe to do so;
- Sorbent pads, booms, or adjacent soil may be used to dike or berm materials subject to flow and to solidify liquids;
- Collect used/saturated sorbent pads, soil or booms in drums;
- Collect contaminated tools and equipment for subsequent cleaning or disposal.

18.4.3.4. Restoration and Salvage of Spill Control Items

The method of disposal of the spill-affected materials will depend on the identity and characteristics of the spilled material. The specific method of disposal, along with any post clean-up verification sampling or restoration procedures, will be determined by the SSO through consultation with the Health and Safety Officer.

Generally, if spill-affected materials such as PPE, sorbent pads and booms, contaminated soil or collected spillage, are determined or suspected as being hazardous waste, they will be collected and sealed in drums that will be appropriately labeled and shipped off-site as a RCRA hazardous waste.

18.4.3.5. Notification

After the area is secure, immediately contact Army Corps of Engineers Project Manager (Gregory Geopfert, 917-790-8235).

The NYSDEC requires all spills to be reported, so after informing the USACE PM, all spills meeting the criteria in Article 12 of the Navigation Law must be reported to NYSDEC within 2 hours at 1-800-457-7362. Additionally, hazardous waste spills must be reported in writing to the NYSDEC within 15 days on the form provided in Attachment H. A written short term response report documenting the measures taken to control and remediate the spill must be filed with NYSDEC within 30 days of the release.

18.4.4. Evacuation

Evacuation routes shall be determined at the beginning of each day's work. In an emergency, begin evacuation immediately. Do not delay and let the situation become more dangerous.

Notice to evacuate will be by a continuous blast on an air powered horn or other site-specific audible alarm. Stay upwind of vapors and smoke. Stay upgradient of spills.

If personnel are in an exclusion or decontamination zone at the start of an emergency, they should exit through a decontamination corridor whenever possible. If evacuation cannot be done through a decontamination corridor, site personnel should go to a safe location and remove contaminated clothing there or leave it near the exclusion zone.

19. MODIFICATION OR CHANGES TO THE HASP/AHA

19.1. Management of Change – Activity Hazard Analysis

The procedures in this HASP/AHA have been developed based on previous investigations conducted at the Site and the proposed scope of work. Every effort has been made to address the chemical hazards that may be encountered during the implementation of the proposed investigation. Similarly, this document also discusses the physical hazards associated with the proposed activities. However, unanticipated site-specific conditions or situations may occur during the implementation of this project. Also, the Stone and/or Stone contractor(s) may elect to perform certain tasks in a manner that is different from what was originally intended due to a change in field conditions. As such, this HASP/AHA must be considered a working document that is subject to change to meet the needs of this dynamic project.

Stone and its selected contractor(s) will complete a Activity Hazard Analysis (AHA) when new tasks or different investigative techniques not addressed in the HASP/AHA are proposed. The use of new techniques will be reviewed and if new hazards are associated with the proposed changes, they will be documented on the AHA form. An effective control measure must also be identified for each new hazard. AHA forms will be reviewed by the SSO prior to being implemented. Once approved, the AHAs will be reviewed with all field staff during the daily safety meeting.

Should significant information become available regarding potential on-site hazards, it may be necessary to modify this HASP/AHA. All proposed modifications to this HASP/AHA must be reviewed and approved by the Team Health and Safety Officers before such modifications are implemented. Any significant modifications must be incorporated into the written documentation as addenda and the HASP/AHA must be reissued. The PM(s) will ensure that all personnel covered by this HASP/AHA receive copies of all issued addenda. Sign-off forms will accompany each addendum and must be signed by all personnel covered by the addendum. Sign-off forms will be submitted to the PM(s). The HASP/AHA addenda should be distributed during the daily safety meeting so that they can be reviewed and discussed. Attendance forms will be collected during the meeting.

EMERGENCY REFERENCES

Ambulance/Fire/Police: 911

Medical Services:

Ellis Hospital
1101 Nott Street
Schenectady NY 12308
518-243-4000

Directions to Hospital (See Figure 2)

From the Site, depart via Y Street onto Route 5 / Amsterdam Road and turn LEFT.

In 3 miles, turn left onto Erie Boulevard.

In 0.7 mile, bear right onto Nott Street.

In 0.9 mile, arrive at 1101 Nott Street, Schenectady, NY.

On Site Telephone: Portable phones will be carried by Field Staff

Project Team Contacts:

Title	SSO	Name, Firm	Phone Number	Cell Number	Email
Program Manager, Executive Management		Seth Pitkin	802-229-2192	802-249-8700	spitkin@stone-env.com
Project Manager	X	Joseph Schmidl	802-229-5377	603-455-0946	jschmidl@stone-env.com
Site Safety Officer	X	David Linari	802-229-6434	802-578-5817	dlinari@stone-env.com
Corporate Health and Safety Officer		Kim Watson	802-299-2196	802-249-7753	kwatson@stone-env.com

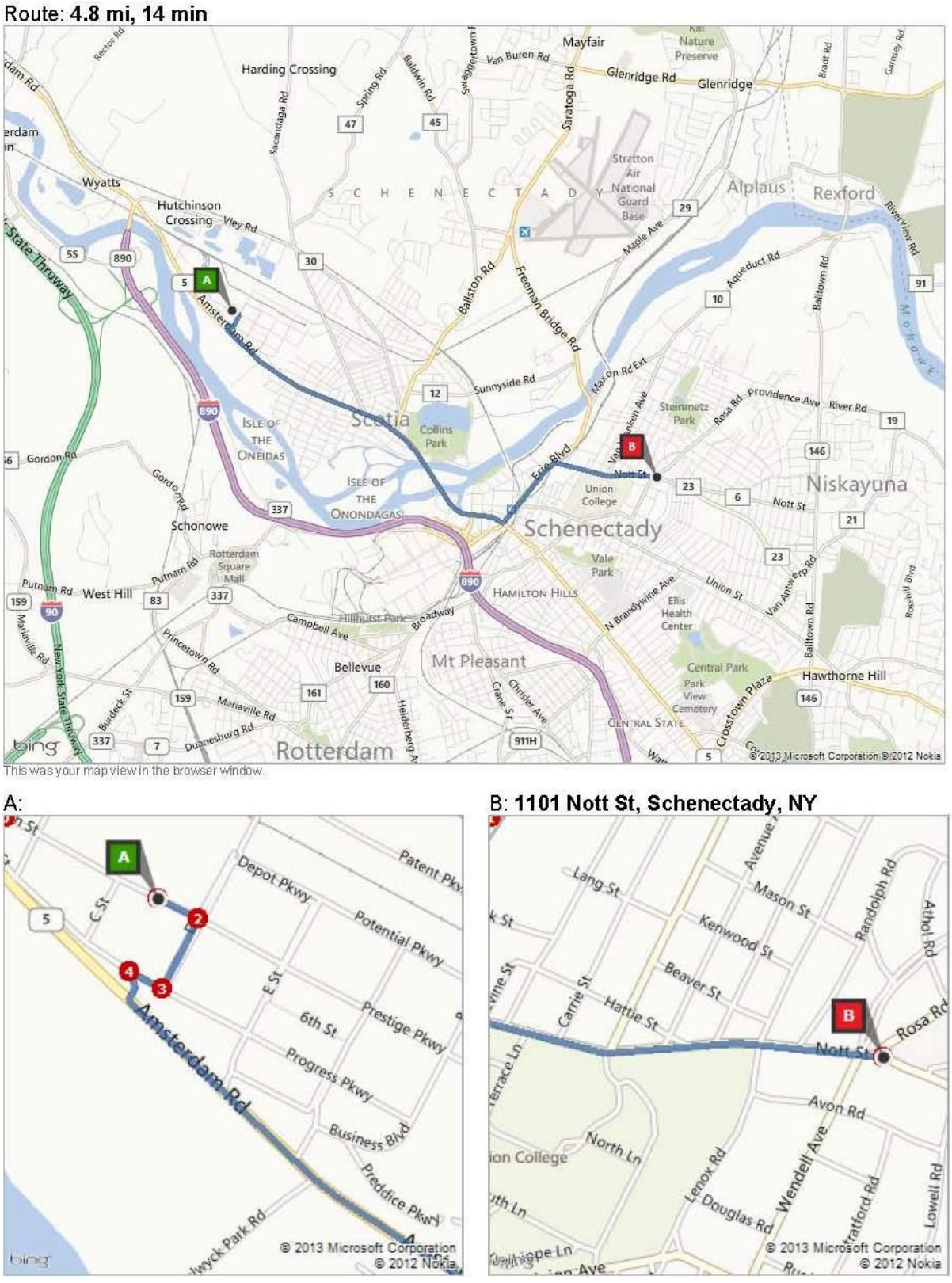


Figure 3: Hospital Route Map

ATTACHMENTS

ATTACHMENT A: HEALTH AND SAFETY PLAN RECEIPT AND ACCEPTANCE FORM

Health and Safety Plan
Receipt and Acceptance Form
DEFENSE NATIONAL STOCKPILE CENTER SCOTIA DEPOT SITE
GLENVILLE, NEW YORK

I have received a copy of the Health and Safety Plan prepared for the above referenced site, I have read and understand its content and I agree that I will abide by its requirements.

Name	Signature	Company	Date

ATTACHMENT B: ACTIVITY HAZARD ANALYSIS FORMS

Activity Hazard Analysis (AHA) – 01 Pre-Design Investigation

Task: Site Reconnaissance, Subsurface Utility Clearance, Surveying		**Stone Project Number:** 102305-R C	
Minimum Personal Protective Equipment (PPE): Level D PPE (Long pants, shirts with minimum 4" sleeve, steel toe boots, safety glasses, hard hat for overhead hazards, leather work gloves, and hearing protection, as required)		**Location:** Defense National Stockpile Center Scotia Depot Site Glenville, New York	
		Analysis Approved by: Kim B. Watson, Corporate Health and Safety	**Date:** January 2013

Activity	Potential Hazards	Recommended Controls
Perform non-intrusive site activities, including Site Reconnaissance, Subsurface Utility Clearance, and Surveying	Chemical Hazards: Potential for inhalation and/or contact exposure to chlorinated compounds from contaminated groundwater	Chemical Hazards: • Use of PPE during intrusive activities in source areas (modified Level D) to limit contact risk. Air monitoring of breathing zone with photoionization detector for the presence of volatile organic compounds (e.g., trichloroethylene (TCE) and tetrachloroethene (PCE)). Proper management of contaminated groundwater and decontamination water.
	Potential for exposure to chemicals brought on-site.	• MSDS for all chemical brought on-site shall be provided to the SSO. Conduct a utility locate to identify the location of underground utilities in boring locations and complete any required dig permits
	Physical Hazards: Slip, trip, fall hazards.	**Physical Hazards:** • Employ safe work practices and good housekeeping: keep work area free of slippery walking surfaces; keep tools and materials from underfoot; maintain three points of contact when climbing on/off drilling equipment. Apply sand to any ice covered surfaces within work areas prior to conducting field activities or utilize traction devices (e.g., ice creepers).
	Thermal Stressors (i.e. heat stress, cold stress)	• Employees will have appropriate clothing for variable weather • Use of long sleeves or application of sunscreen with a high sun protection factor (SPF) on exposed skin encouraged • Employees will take breaks and drink plenty of fluids to prevent heat stress • Warming breaks will be permitted as necessary to prevent cold stress
	Inclement weather (Thunderstorms and tornadoes)	• Halt activities immediately and take cover during thunderstorm or tornado warnings, shelter in a building if possible, stay away from windows • If outdoors, stay close to the ground • Listen to radio or television announcements for pending weather information • Do not try to outrun a tornado on foot or in a vehicle
	Biological hazards (insects, spiders, etc.)	• Workers will inspect the work area carefully and avoid placing hands and feet into concealed areas • Look in direction of travel for biological hazards to avoid

AHA – 01 Pre-Design Investigation (Concluded)

Activity	Potential Hazards	Recommended Controls
	Electrical hazards	• None
	Water hazard, working near or on water - immersion in water - drowning	• Personnel will avoid waterbodies.
Equipment Used	**Inspection Requirements**	**Training Requirements**
Vehicles and site equipment Pickup trucks or vans	Inspect all equipment prior to use; refer to manufacturers inspection requirements, if appropriate	Valid operator's license for the class of vehicle.
Geophysical instruments; Total Station or Autolevel, Stadia rod.	Calibrate and use instruments/equipment in accordance with manufacturers recommendation	Experience in groundwater quality sampling utilizing low-flow techniques and equipment. Training in related SEI SOPs.
Decontamination containment vessels/structures including containers for purge water from work in source areas wells	Prior to each use, inspect any decontamination containment vessel or structure. Inspect any container prior before depositing any soil/rock cuttings, groundwater or decontamination residuals from work in source areas.	No specific training required.
		General: Review of *Health and Safety Plan* and Activity Hazard Analyses Hazardous Sites Worker training per 29CFR 1910.120 Initial site/task specific training. Daily Safety Meetings. Review EM 385-1-1 Section 11.A – 11.D.

Activity Hazard Analysis (AHA) – 02 Pre-Design Investigation

Task: Monitoring Well Sample Collection	**Stone Project Number:** 102305-R C	
Minimum Personal Protective Equipment (PPE): Level D PPE (Long pants, shirts with minimum 4" sleeve, steel toe boots, safety glasses, hard hat for overhead hazards, leather work gloves, and hearing protection, as required)	**Location:** Defense National Stockpile Center Scotia Depot Site Glenville, New York	
	Analysis Approved by: Kim B. Watson, Corporate Health and Safety	**Date:** January 2013

Activity	Potential Hazards	Recommended Controls
Collect groundwater samples from monitoring wells	Chemical Hazards: Potential for inhalation and/or contact exposure to chlorinated compounds from contaminated groundwater	Chemical Hazards: • Use of PPE during intrusive activities in source areas (modified Level D) to limit contact risk. Air monitoring of breathing zone with photoionization detector for the presence of volatile organic compounds (e.g., trichloroethylene (TCE) and tetrachloroethene (PCE)). Proper management of contaminated groundwater and decontamination water.
	Potential for exposure to chemicals brought on-site.	• MSDS for all chemical brought on-site shall be provided to the SSO. Conduct a utility locate to identify the location of underground utilities in boring locations and complete any required dig permits
	Physical Hazards: Slip, trip, fall hazards.	**Physical Hazards:** • Employ safe work practices and good housekeeping: keep work area free of slippery walking surfaces; keep tools and materials from underfoot; maintain three points of contact when climbing on/off drilling equipment. Apply sand to any ice covered surfaces within work areas prior to conducting field activities or utilize traction devices (e.g., ice creepers).
	Thermal Stressors (i.e. heat stress, cold stress)	• Employees will have appropriate clothing for variable weather • Use of long sleeves or application of sunscreen with a high sun protection factor (SPF) on exposed skin encouraged • Employees will take breaks and drink plenty of fluids to prevent heat stress • Warming breaks will be permitted as necessary to prevent cold stress
	Inclement weather (Thunderstorms and tornadoes)	• Halt activities immediately and take cover during thunderstorm or tornado warnings, shelter in a building if possible, stay away from windows • If outdoors, stay close to the ground • Listen to radio or television announcements for pending weather information • Do not try to outrun a tornado on foot or in a vehicle

AHA – 02 Pre-Design Investigation (Continued)

Activity	Potential Hazards	Recommended Controls
	Biological hazards (insects, spiders etc.)	• Workers will inspect the work area carefully and avoid placing hands and feet into concealed areas Look in direction of travel for biological hazards to avoid
	Electrical hazards	• Sampling pumps shall be grounded by a multi-conductor cord with identified grounding conductor and multi-contact plug and receptacle. Comply with Section 11.A – 11.D of EM 385-1-1.
	Water hazard, working near or on water - immersion in water - drowning	• Sampling personnel will be accessing Davol Pond – the buddy system or pre-post sampling communication will be used. Personnel will be accessing sampling site with a canoe or skiff. The appropriate PFDs will be on board and in use.
Equipment Used	**Inspection Requirements**	**Training Requirements**
Vehicles and site equipment Pickup trucks or vans Canoe or small boat Portable generator Flexible extension cords Hand tools	Inspect all equipment prior to use; refer to manufacturers inspection requirements, if appropriate. Inspect flexible extension cords daily. Test GFIC circuits prior to use	Valid operator's license for the class of vehicle. Experience in maneuvering small watercraft.
Photoionization detector and water level indicator; sampling equipment including: downhole submersible pump, multi-parameter probe; turbidity meter; pre-preserved sample containers; and sampling tubing.	Inspect and calibrate Photoionization detector (PID) daily when in use to monitor intrusive activities. Calibrate and use instruments/equipment in accordance with manufacturers recommendation	Experience in operating and calibrating a PID. Experience in groundwater quality sampling utilizing low-flow techniques and equipment. Training in related SEI SOPs.
Decontamination containment vessels/structures including containers for purge water from work in source areas wells	Prior to each use, inspect any decontamination containment vessel or structure. Inspect any container prior before depositing any soil/rock cuttings, groundwater or decontamination residuals from work in source areas.	No specific training required.

AHA – 02 Pre-Design Investigation (Concluded)

Equipment Used	Inspection Requirements	Training Requirements
		General: Review of *Health and Safety Plan* and Activity Hazard Analyses Hazardous Sites Worker training per 29CFR 1910.120 Initial site/task specific training. Daily Safety Meetings. Review EM 385-1-1 Section 11.A – 11.D.

Activity Hazard Analysis (AHA) – 03 Pre-Design Investigation

Task: Vertical Groundwater Profiling	**Stone Project Number:** 102305-R C	
Minimum Personal Protective Equipment (PPE): Level D PPE (Long pants, shirts with minimum 4" sleeve, steel toe boots, safety glasses, hard hat for overhead hazards, leather work gloves, and hearing protection, as required)	**Location:** Defense National Stockpile Center Scotia Depot Site Glenville, New York	
	Analysis Approved by: Kim B. Watson, Corporate Health and Safety	**Date:** January 2013

Activity	Potential Hazards	Recommended Controls
Positioning and operation of drill rigs and associated tools/equipment. Obtaining/handling groundwater samples Decontamination of drill rig(s), drill tools, and associated equipment. Handling and management of soil/rock cuttings and drilling fluids.	**Chemical Hazards**: Potential for inhalation and/or contact exposure to chlorinated compounds from contaminated groundwater and/or soil/rock.	**Chemical Hazards**: • Use of PPE during intrusive activities in source areas (modified Level D) to limit contact risk. Air monitoring of breathing zone with photoionization detector for the presence of volatile organic compounds (e.g., trichloroethylene (TCE) and tetrachloroethene (PCE)). Proper management of rock/soil cuttings, contaminated groundwater and decontamination solids/water.
	Potential for exposure to chemicals brought on-site.	• MSDS for all chemicals brought on-site shall be provided to the Site Safety Officer.
	Physical Hazards: Struck by/entanglement in and/or pinched between equipment while positioning and operating drill rig, drilling tools and associated equipment.	**Physical Hazards**: • Employ safe work practices; make eye contact with operator if need to enter work area; restrict work area to essential personnel. Wear specified PPE including hard hats, eye protection and safety toed work boots. Utilize procedures outlined in the SEI Drilling Safety Program.
	Exposure to excessive noise levels. Exposure to Eye injury hazards	• Utilize hearing protection against excessive noise levels and wear eye protection.
	Contact with overhead/underground utilities	• Maintain required isolation distances from overhead power lines. Utilize Dig Safe to identify any underground utilities and to clear each drilling location. Determine that no known underground utilities exist in the drilling locations
	Thermal Stressors (i.e. heat stress, cold stress)	• Employees will have appropriate clothing for variable weather • Use of long sleeves or application of sunscreen with a high sun protection factor (SPF) on exposed skin encouraged • Employees will take breaks and drink plenty of fluids to prevent heat stress • Warming breaks will be permitted as necessary to prevent cold stress

AHA – 03 Pre-Design Investigation (Continued)

Activity	Potential Hazards	Recommended Controls
	Back Injury from Materials Handling	• Use proper lifting techniques • Loads greater than 50 pounds require assistance or mechanical equipment • Prior to lifting, check the load for jagged or sharp edges • Avoid torso twisting motions while handling or moving loads
	Slip, trip, fall hazards.	• Employ safe work practices and good housekeeping: keep work area free of slippery walking surfaces; keep tools and materials from underfoot; maintain three points of contact when climbing on/off drilling equipment. Apply sand to any ice covered surfaces within work areas prior to conducting field activities or utilize traction devices (e.g., ice creepers).
	Inclement weather (Thunderstorms and tornadoes)	• Halt activities immediately and take cover during thunderstorm or tornado warnings, shelter in a building if possible, stay away from windows • If outdoors, stay close to the ground • Listen to radio or television announcements for pending weather information • Do not try to outrun a tornado on foot or in a vehicle
	Biological hazards (insects, spiders etc.)	• Workers will inspect the work area carefully and avoid placing hands and feet into concealed areas • Look in direction of travel for biological hazards to avoid
	Contact with hot water or high-pressure sprayer	• Utilize eye protection and leather or heavy rubber (Petroflex or equivalent) gloves. Maintain a safe distance from the discharge of the spray wand. Never point the wand at another person nor use for personal decontamination.
	Compressed gas can be driven into the flesh or eyes.	• Utilize eye protection. Pneumatic impact tools and hoses should never be pointed at a person. Before hoses are disconnected, the air supply must be turned off at the control valve and the pressure bled off.
Equipment Used	**Inspection Requirements**	**Training Requirements**
Vehicle and site equipment Drill rigs Pickup trucks or vans Steam or high temperature pressure washer Air compressor Portable generator Hand tools	Inspect drill rig before initial use in accordance with EM 385-1-1 (16.M.02) and per The SEI's Drilling Safety Program or Subcontractors equivalent. Inspect all equipment prior to use; refer to manufacturers inspection requirements, if appropriate.	Valid operator's license for the class of vehicle. Experience in drill rig operation.

AHA – 03 Pre-Design Investigation (Concluded)

Equipment Used	Inspection Requirements	Training Requirements
Photoionization detector	Inspect and calibrate Photoionization detector (PID) daily when in use to monitor intrusive activities.	Experience in operating and calibrating a PID.
Decontamination containment vessels/structures: container for decontamination residuals from work in source areas	Prior to each use, inspect any decontamination containment vessel or structure. Inspect any container prior before depositing any soil/rock cuttings, groundwater or decontamination residuals from work in source areas.	No specific training required.
Air compressor or compressed inert gas.	Prior to each use, inspect all air hoses, couplings and connections to determine if leakage or other damage exists. All pipes, hoses, and fittings must have a rating of the maximum pressure of the compressor or tank, should be kept clean, and hose ends must be secured to prevent whipping if an accidental cut or break occurs.	No specific training required. Be familiar with operator's manual.
		General: Review of Site-Specific Health and Safety Plan and Activity Hazard Analyses Hazardous Sites Worker training per 29CFR 1910.120 Initial site/task specific training. Daily Safety Meetings.

Activity Hazard Analysis (AHA) – 04 Pre-Design Investigation

Task: Subsurface Soil Sampling and Well Drilling	**Stone Project Number:** 102305-R C	
Minimum Personal Protective Equipment (PPE): Level D PPE (Long pants, shirts with minimum 4" sleeve, steel toe boots, safety glasses, hard hat for overhead hazards, leather work gloves, and hearing protection, as required)	**Location:** Defense National Stockpile Center Scotia Depot Site Glenville, New York	
	Analysis Approved by: Kim B. Watson, Corporate Health and Safety	**Date:** January 2013

Activity	Potential Hazards	Recommended Controls
Positioning and operation of drill rigs and associated tools/equipment. Obtaining/handling soil and rock cutting samples Decontamination of drill rig(s), drill tools, and associated equipment. Handling and management of soil/rock cuttings and drilling fluids.	**Chemical Hazards**: Potential for inhalation and/or contact exposure to chlorinated compounds from contaminated groundwater and/or soil/rock.	**Chemical Hazards**: • Use of PPE during intrusive activities in source areas (modified Level D) to limit contact risk. Air monitoring of breathing zone with photoionization detector for the presence of volatile organic compounds (e.g., trichloroethylene (TCE) and tetrachloroethene (PCE)). Proper management of rock/soil cuttings, contaminated groundwater and decontamination solids/water.
	Potential for exposure to chemicals brought on-site.	• MSDS for all chemicals brought on-site shall be provided to the Site Safety Officer.
	Physical Hazards: Struck by/entanglement in and/or pinched between equipment while positioning and operating drill rig, drilling tools and associated equipment.	**Physical Hazards**: • Employ safe work practices; make eye contact with operator if need to enter work area; restrict work area to essential personnel. Wear specified PPE including hard hats, eye protection and safety toed work boots. Utilize procedures outlined in the SEI Drilling Safety Program.
	Exposure to excessive noise levels. Exposure to Eye injury hazards	• Utilize hearing protection against excessive noise levels and wear eye protection.
	Contact with overhead/underground utilities	• Maintain required isolation distances from overhead power lines. Utilize Dig Safe to identify any underground utilities and to clear each drilling location. Determine that no known underground utilities exist in the drilling locations
	Thermal Stressors (i.e. heat stress, cold stress)	• Employees will have appropriate clothing for variable weather • Use of long sleeves or application of sunscreen with a high sun protection factor (SPF) on exposed skin encouraged • Employees will take breaks and drink plenty of fluids to prevent heat stress • Warming breaks will be permitted as necessary to prevent cold stress

Activity	Potential Hazards	Recommended Controls
	Back Injury from Materials Handling	• Use proper lifting techniques • Loads greater than 50 pounds require assistance or mechanical equipment • Prior to lifting, check the load for jagged or sharp edges • Avoid torso twisting motions while handling or moving loads
	Slip, trip, fall hazards.	• Employ safe work practices and good housekeeping: keep work area free of slippery walking surfaces; keep tools and materials from underfoot; maintain three points of contact when climbing on/off drilling equipment. Apply sand to any ice covered surfaces within work areas prior to conducting field activities or utilize traction devices (e.g., ice creepers).
	Inclement weather (Thunderstorms and tornadoes)	• Halt activities immediately and take cover during thunderstorm or tornado warnings, shelter in a building if possible, stay away from windows • If outdoors, stay close to the ground • Listen to radio or television announcements for pending weather information • Do not try to outrun a tornado on foot or in a vehicle
	Biological hazards (insects, spiders etc.)	• Workers will inspect the work area carefully and avoid placing hands and feet into concealed areas • Look in direction of travel for biological hazards to avoid
	Contact with hot water or high-pressure sprayer	• Utilize eye protection and leather or heavy rubber (Petroflex or equivalent) gloves. Maintain a safe distance from the discharge of the spray wand. Never point the wand at another person nor use for personal decontamination.
	Compressed gas can be driven into the flesh or eyes.	• Utilize eye protection. Pneumatic impact tools and hoses should never be pointed at a person. Before hoses are disconnected, the air supply must be turned off at the control valve and the pressure bled off.
Equipment Used	**Inspection Requirements**	**Training Requirements**
Vehicle and site equipment Drill rigs Pickup trucks or vans Steam or high temperature pressure washer Air compressor Portable generator Hand tools	Inspect drill rig before initial use in accordance with EM 385-1-1 (16.M.02) and per The SEI's Drilling Safety Program or Subcontractors equivalent. Inspect all equipment prior to use; refer to manufacturers inspection requirements, if appropriate.	Valid operator's license for the class of vehicle. Experience in drill rig operation.

AHA – 04 Pre-Design Investigation (Concluded)

Equipment Used	Inspection Requirements	Training Requirements
Photoionization detector	Inspect and calibrate Photoionization detector (PID) daily when in use to monitor intrusive activities.	Experience in operating and calibrating a PID.
Decontamination containment vessels/structures: container for decontamination residuals from work in source areas.	Prior to each use, inspect any decontamination containment vessel or structure. Inspect any container prior before depositing any soil/rock cuttings, groundwater or decontamination residuals from work in source areas.	No specific training required.
Air compressor or compressed inert gas.	Prior to each use, inspect all air hoses, couplings and connections to determine if leakage or other damage exists. All pipes, hoses, and fittings must have a rating of the maximum pressure of the compressor or tank, should be kept clean, and hose ends must be secured to prevent whipping if an accidental cut or break occurs.	No specific training required. Be familiar with operator's manual.
		General: Review of Site-Specific Health and Safety Plan and Activity Hazard Analyses Hazardous Sites Worker training per 29CFR 1910.120 Initial site/task specific training. Daily Safety Meetings.

Activity Hazard Analysis (AHA) – 05 Pre-Design Investigation

Task: Chemical Analysis in Mobile Laboratory		**Stone Project Number:** 102305-R C	
Minimum Personal Protective Equipment (PPE): Level D PPE (Long pants, shirts with minimum 4" sleeve, steel toe boots, safety glasses, hard hat for overhead hazards, leather work gloves, and hearing protection, as required)		**Location:** Defense National Stockpile Center Scotia Depot Site Glenville, New York	
		Analysis Approved by: Kim B. Watson, Corporate Health and Safety	**Date:** January 2013

Activity	Potential Hazards	Recommended Controls
Preparation of VOA vials for soil sampling. Receiving, handling, storing and analysis of water and soil samples.	**Chemical Hazards**: Potential for inhalation and/or contact exposure to chlorinated compounds from contaminated groundwater	**Chemical Hazards**: • Use of PPE during intrusive activities in source areas (modified Level D) to limit contact risk. Air monitoring of breathing zone with photoionization detector for the presence of volatile organic compounds (e.g., trichloroethylene (TCE) and tetrachloroethene (PCE)). Proper management of contaminated groundwater and decontamination water.
	Potential for exposure to chemicals brought on-site.	• MSDS for all chemical brought on-site shall be provided to the SSO. Conduct a utility locate to identify the location of underground utilities in boring locations and complete any required dig permits
	Physical Hazards: Compressed gas can be driven into the flesh or eyes.	**Physical Hazards:** • Utilize eye protection. Pneumatic impact tools and hoses should never be pointed at a person. Before hoses are disconnected, the air supply must be turned off at the control valve and the pressure bled off.
Equipment Used	**Inspection Requirements**	**Training Requirements**
Gas Chromatograph (GC) with FID detector and Mass Spectrometer Detector (GC/MSD). Laboratory supplies: volumetric syringes, stirring plates, beakers. Hydrogen, helium and purified air gas cylinders.	Inspect all equipment prior to use; refer to manufacturers inspection requirements, if appropriate. Inspect and calibrate GC/GC/MS daily as required in the standard method. Inspect laboratory equipment and remove defective devices from service.	1) Corporate and Laboratory H&S training. NELAC training and training on the Laboratory Quality Manual And reference to reading and understanding the Standard Operating Procedures 2) Experience in operating and calibrating a GC/GC/MSD.
		General: Review of Site-Specific Health and Safety Plan and Activity Hazard Analyses Hazardous Sites Worker training per 29CFR 1910.120 Initial site/task specific training. Daily Safety Meetings.

Activity Hazard Analysis (AHA) – 06 Pre-Design Investigation

Task: Install and Develop Monitoring Wells	**Stone Project Number:** 102305-R C	
Minimum Personal Protective Equipment (PPE): Level D PPE (Long pants, shirts with minimum 4" sleeve, steel toe boots, safety glasses, hard hat for overhead hazards, leather work gloves, and hearing protection, as required)	**Location:** Defense National Stockpile Center Scotia Depot Site Glenville, New York	
	Analysis Approved by: Kim B. Watson, Corporate Health and Safety	**Date:** January 2013

Activity	Potential Hazards	Recommended Controls
Installation and development of monitoring wells. Decontamination of drill rig(s), drill tools, and associated equipment. Handling and management of well development water.	**Chemical Hazards**: Potential for inhalation and/or contact exposure to chlorinated compounds from contaminated groundwater and/or soil/rock.	**Chemical Hazards**: • Use of PPE during intrusive activities in source areas (modified Level D) to limit contact risk. Air monitoring of breathing zone with photoionization detector for the presence of volatile organic compounds (e.g., trichloroethylene (TCE) and tetrachloroethene (PCE)). Proper management of rock/soil cuttings, contaminated groundwater and decontamination solids/water.
	Potential for exposure to chemicals brought on-site.	• MSDS for all chemicals brought on-site shall be provided to the Site Safety Officer.
	Physical Hazards: Struck by/entanglement in and/or pinched between equipment while positioning and operating drill rig, drilling tools and associated equipment.	**Physical Hazards**: • Employ safe work practices; make eye contact with operator if need to enter work area; restrict work area to essential personnel. Wear specified PPE including hard hats, eye protection and safety toed work boots. Utilize procedures outlined in the SEI Drilling Safety Program.
	Exposure to excessive noise levels. Exposure to Eye injury hazards	• Utilize hearing protection against excessive noise levels and wear eye protection.
	Contact with overhead/underground utilities	• Maintain required isolation distances from overhead power lines. Utilize Dig Safe to identify any underground utilities and to clear each drilling location. Determine that no known underground utilities exist in the drilling locations
	Thermal Stressors (i.e. heat stress, cold stress)	• Employees will have appropriate clothing for variable weather • Use of long sleeves or application of sunscreen with a high sun protection factor (SPF) on exposed skin encouraged • Employees will take breaks and drink plenty of fluids to prevent heat stress • Warming breaks will be permitted as necessary to prevent cold stress

AHA – 06 Pre-Design Investigation (Continued)

Activity	Potential Hazards	Recommended Controls
	Back Injury from Materials Handling	• Use proper lifting techniques • Loads greater than 50 pounds require assistance or mechanical equipment • Prior to lifting, check the load for jagged or sharp edges • Avoid torso twisting motions while handling or moving loads
	Slip, trip, fall hazards.	• Employ safe work practices and good housekeeping: keep work area free of slippery walking surfaces; keep tools and materials from underfoot; maintain three points of contact when climbing on/off drilling equipment. Apply sand to any ice covered surfaces within work areas prior to conducting field activities or utilize traction devices (e.g., ice creepers).
	Inclement weather (Thunderstorms and tornadoes)	• Halt activities immediately and take cover during thunderstorm or tornado warnings, shelter in a building if possible, stay away from windows • If outdoors, stay close to the ground • Listen to radio or television announcements for pending weather information • Do not try to outrun a tornado on foot or in a vehicle
	Biological hazards (insects, spiders etc.)	• Workers will inspect the work area carefully and avoid placing hands and feet into concealed areas • Look in direction of travel for biological hazards to avoid
	Contact with hot water or high-pressure sprayer	• Utilize eye protection and leather or heavy rubber (Petroflex or equivalent) gloves. Maintain a safe distance from the discharge of the spray wand. Never point the wand at another person nor use for personal decontamination.
	Compressed gas can be driven into the flesh or eyes.	• Utilize eye protection. Pneumatic impact tools and hoses should never be pointed at a person. Before hoses are disconnected, the air supply must be turned off at the control valve and the pressure bled off.

AHA – 06 Pre-Design Investigation (Concluded)

Equipment Used	Inspection Requirements	Training Requirements
Vehicle and site equipment Drill rigs Pickup trucks or vans Steam or high temperature pressure washer Air compressor Portable generator Hand tools	Inspect drill rig before initial use in accordance with EM 385-1-1 (16.M.02) and per The SEI's Drilling Safety Program or Subcontractors equivalent. Inspect all equipment prior to use; refer to manufacturers inspection requirements, if appropriate.	Valid operator license for the class of vehicle. Experience in drill rig operation.
Photoionization detector	Inspect and calibrate Photoionization detector (PID) daily when in use to monitor intrusive activities.	Experience in operating and calibrating a PID.
Decontamination containment vessels/structures: container for decontamination residuals from work in source areas	Prior to each use, inspect any decontamination containment vessel or structure. Inspect any container prior before depositing any soil/rock cuttings, groundwater or decontamination residuals from work in source areas.	No specific training required.
Air compressor or compressed inert gas.	Prior to each use, inspect all air hoses, couplings and connections to determine if leakage or other damage exists. All pipes, hoses, and fittings must have a rating of the maximum pressure of the compressor or tank, should be kept clean, and hose ends must be secured to prevent whipping if an accidental cut or break occurs.	No specific training required. Be familiar with operator's manual.
		General: Review of Site-Specific Health and Safety Plan and Activity Hazard Analyses Hazardous Sites Worker training per 29CFR 1910.120 Initial site/task specific training. Daily Safety Meetings.

Activity Hazard Analysis (AHA) – 07 Pre-Design Investigation

Task: Contaminated water treatment and/or disposal; contaminated solids treatment and/or disposal	**Stone Project Number:** 102305-R C	
Minimum Personal Protective Equipment (PPE): Level D PPE (Long pants, shirts with minimum 4" sleeve, steel toe boots, safety glasses, hard hat for overhead hazards, leather work gloves, and hearing protection, as required)	**Location:** Defense National Stockpile Center Scotia Depot Site Glenville, New York	
	Analysis Approved by: Kim B. Watson, Corporate Health and Safety	**Date:** January 2013

Activity	Potential Hazards	Recommended Controls
Discharging water to the ground. Positioning and filling drums, containers or tanks for temporary storage or on-site transport and subsequent depositing them onto the ground surface. Handling and management of rock, soil and cuttings, samples, groundwater and decontamination residuals.	**Chemical Hazards**: Potential for inhalation and/or contact exposure to chlorinated compounds from contaminated groundwater and/or solids/soil/rock.	**Chemical Hazards**: • Use of PPE during intrusive activities in source areas (modified Level D) to limit contact risk. Air monitoring of breathing zone with photoionization detector for the presence of volatile organic compounds (e.g., trichloroethylene (TCE) and tetrachloroethene (PCE)). Proper management of rock/soil cuttings, contaminated groundwater and decontamination solids/water.
	Potential for exposure to chemicals brought on-site.	• MSDS for all chemical brought on-site shall be provided to the SSO. Conduct a utility locate to identify the location of underground utilities in boring locations and complete any required dig permits
	Physical Hazards: Struck by/entanglement and/or pinched between while positioning and filling containers.	**Physical Hazards**: • Employ safe work practices; make eye contact with operator if need to enter work area; restrict work area to essential personnel. Wear protective equipment including hard hats, eye protection and safety toed work boots.
	Exposure to excessive noise levels. Exposure to eye injury hazards	• Utilize hearing protection against excessive noise levels and wear eye protection.
	Contact with overhead/underground utilities	• Maintain required isolation distances from overhead power lines.

AHA – 07 Pre-Design Investigation (Continued)

Activity	Potential Hazard(s)	Recommended Controls
	Back Injury from Materials Handling	• Use proper lifting techniques • Loads greater than 50 pounds require assistance or mechanical equipment • Prior to lifting, check the load for jagged or sharp edges • Avoid torso twisting motions while handling or moving loads
	Slip, trip, fall hazards.	• Employ safe work practices and good housekeeping: keep work area free of slippery walking surfaces; keep tools and materials from underfoot; maintain three points of contact when climbing on/off drilling equipment. Apply sand to any ice covered surfaces within work areas prior to conducting field activities or utilize traction devices (e.g., ice creepers).
	Thermal Stressors (i.e. heat stress, cold stress)	• Employees will have appropriate clothing for variable weather • Use of long sleeves or application of sunscreen with a high sun protection factor (SPF) on exposed skin encouraged • Employees will take breaks and drink plenty of fluids to prevent heat stress • Warming breaks will be permitted as necessary to prevent cold stress
	Inclement weather (Thunderstorms and tornadoes)	• Halt activities immediately and take cover during thunderstorm or tornado warnings, shelter in a building if possible, stay away from windows • If outdoors, stay close to the ground • Listen to radio or television announcements for pending weather information • Do not try to outrun a tornado on foot or in a vehicle
	Biological hazards (insects, spiders etc.)	• Workers will inspect the work area carefully and avoid placing hands and feet into concealed areas • Look in direction of travel for biological hazards to avoid

AHA – 07 Pre-Design Investigation (Concluded)

Equipment Used	Inspection Requirements	Training Requirements
Vehicles and site equipment Excavator, skid steer Pickup trucks or vans Portable tanks, containers, hoses Hand tools	Inspect all equipment prior to use; refer to manufacturers inspection requirements, if appropriate.	Valid operators license for the class of vehicle.
Photoionization detector	Inspect and calibrate Photoionization detector (PID) daily when in use to monitor intrusive activities.	Experience in operating and calibrating a PID.
Dumpster, drums, tanks or containers for temporary storage or on-site transportation of soil/rock cuttings, groundwater and decontamination residuals from work in source areas	Prior to each use, inspect any containment vessel or structure. Inspect any container prior before depositing any soil/rock cuttings, groundwater or decontamination residuals from work in source areas.	No specific training required.
		General: Review of Site-Specific Health and Safety Plan and Activity Hazard Analyses Hazardous Sites Worker training per 29CFR 1910.120 Initial site/task specific training. Daily Safety Meetings.

Activity Hazard Analysis (AHA) – 08 Pre-Design Investigation

Task: Equipment and Personnel Decontamination		**Stone Project Number:** 102305-R C	
Minimum Personal Protective Equipment (PPE): Level D PPE (Long pants, shirts with minimum 4" sleeve, steel toe boots, safety glasses, hard hat for overhead hazards, leather work gloves, and hearing protection, as required)		**Location:** Defense National Stockpile Center Scotia Depot Site Glenville, New York	
		Analysis Approved by: Kim B. Watson, Corporate Health and Safety	**Date:** January 2013

Activity	Potential Hazards	Recommended Controls
Decontamination of equipment that comes in contact with contaminated media. Proper management and disposal of PPE. Use of hot-water pressure sprayer for equipment decontamination and the collection, handling and management of decontamination residuals.	**Chemical Hazards**: Potential for inhalation and/or contact exposure to chlorinated compounds from contaminated groundwater	**Chemical Hazards**: • Use of PPE during intrusive activities in source areas (modified Level D) to limit contact risk. Air monitoring of breathing zone with photoionization detector for the presence of volatile organic compounds (e.g., trichloroethylene (TCE) and tetrachloroethene (PCE)). Proper management of contaminated groundwater and decontamination water.
	Potential for exposure to chemicals brought on-site.	• MSDS for all chemical brought on-site shall be provided to the SSO. Conduct a utility locate to identify the location of underground utilities in boring locations and complete any required dig permits
	Physical Hazards: Slip, trip, fall hazards.	**Physical Hazards:** • Employ safe work practices and good housekeeping: keep work area free of slippery walking surfaces; keep tools and materials from underfoot; maintain three points of contact when climbing on/off drilling equipment. Apply sand to any ice covered surfaces within work areas prior to conducting field activities or utilize traction devices (e.g., ice creepers).
	Struck by/entanglement while positioning and operating drill rig, drilling tools, excavator and associated equipment for decontamination.	• Struck by/entanglement while positioning and operating drill rig, drilling tools, excavator and associated equipment for decontamination.
	Exposure to excessive noise levels	• Utilize hearing protection against excessive noise levels.
	Contact with hot water or high-pressure sprayer.	• Utilize eye protection and leather or heavy rubber (Petroflex or equivalent) gloves. Maintain a safe distance from the discharge of the spray wand. Never point the wand at another person nor use for personal decontamination.

AHA – 08 Pre-Design Investigation (Continued)

Activity	Potential Hazards	Recommended Controls
	Back Injury from Materials Handling	• Use proper lifting techniques • Loads greater than 50 pounds require assistance or mechanical equipment • Prior to lifting, check the load for jagged or sharp edges • Avoid torso twisting motions while handling or moving loads
	Thermal Stressors (i.e. heat stress, cold stress)	• Employees will have appropriate clothing for variable weather • Use of long sleeves or application of sunscreen with a high sun protection factor (SPF) on exposed skin encouraged • Employees will take breaks and drink plenty of fluids to prevent heat stress • Warming breaks will be permitted as necessary to prevent cold stress
	Inclement weather (Thunderstorms and tornadoes)	• Halt activities immediately and take cover during thunderstorm or tornado warnings, shelter in a building if possible, stay away from windows • If outdoors, stay close to the ground • Listen to radio or television announcements for pending weather information • Do not try to outrun a tornado on foot or in a vehicle
	Biological hazards (insects, spiders etc.)	• Workers will inspect the work area carefully and avoid placing hands and feet into concealed areas • Look in direction of travel for biological hazards to avoid
Equipment Used	**Inspection Requirements**	**Training Requirements**
Vehicle and site equipment • Drill rigs • Excavator • Pickup trucks or vans • Canoe or small boat • High temperature pressure washer Hand tools	Inspect all equipment prior to use; refer to manufacturers inspection requirements, if appropriate.	Valid operator's license for the class of vehicle. Experience in drill rig and heavy equipment operation to move equipment for decontamination.

AHA – 08 Pre-Design Investigation (Concluded)

Equipment Used	Inspection Requirements	Training Requirements
Photoionization detector	Inspect and calibrate Photoionization detector (PID) daily when in use to monitor intrusive activities.	Experience in operating and calibrating a PID.
Decontamination containment vessels/structures: containers for soil/rock cuttings, groundwater and decontamination residuals from work in source areas	Prior to each use, inspect any decontamination containment vessel or structure. Inspect any container prior before depositing any soil/rock cuttings, groundwater or decontamination residuals from work in source areas.	No specific training required.
		General: • Review of Site-Specific Health and Safety Plan and Activity Hazard Analyses • Hazardous Sites Worker training per 29CFR 1910.120 • Initial site/task specific training. Daily Safety Meetings.

Activity Hazard Analysis (AHA) – 09 Pre-Design Investigation

Task: Soil Vapor Investigation	**Stone Project Number:** 102305-R C	
Minimum Personal Protective Equipment (PPE): Level D PPE (Long pants, shirts with minimum 4" sleeve, steel toe boots, safety glasses, hard hat for overhead hazards, leather work gloves, and hearing protection, as required)	**Location:** Defense National Stockpile Center Scotia Depot Site Glenville, New York	
	Analysis Approved by: Kim B. Watson, Corporate Health and Safety	**Date:** January 2013

Activity	Potential Hazards	Recommended Controls
Install soil gas sample ports and collect samples	**Chemical Hazards**: Potential for inhalation and/or contact exposure to chlorinated compounds from contaminated soil gas	**Chemical Hazards**: • Use of PPE during intrusive activities in source areas (modified Level D) to limit contact risk. Air monitoring of breathing zone with photoionization detector for the presence of volatile organic compounds (e.g., trichloroethylene (TCE) and tetrachloroethene (PCE)).
	Potential for exposure to chemicals brought on-site.	• MSDS for all chemical brought on-site shall be provided to the SSO. Conduct a utility locate to identify the location of underground utilities in boring locations and complete any required dig permits
	Physical Hazards: Slip, trip, fall hazards.	**Physical Hazards:** • Employ safe work practices and good housekeeping: keep work area free of slippery walking surfaces; keep tools and materials from underfoot. Apply sand to any ice covered surfaces within work areas prior to conducting field activities or utilize traction devices (e.g., ice creepers).
	Thermal Stressors (i.e. heat stress, cold stress)	• Employees will have appropriate clothing for variable weather • Use of long sleeves or application of sunscreen with a high sun protection factor (SPF) on exposed skin encouraged • Employees will take breaks and drink plenty of fluids to prevent heat stress • Warming breaks will be permitted as necessary to prevent cold stress
	Inclement weather (Thunderstorms and tornadoes)	• Halt activities immediately and take cover during thunderstorm or tornado warnings, shelter in a building if possible, stay away from windows • If outdoors, stay close to the ground • Listen to radio or television announcements for pending weather information • Do not try to outrun a tornado on foot or in a vehicle

AHA – 09 Pre-Design Investigation (Continued)

Activity	Potential Hazards	Recommended Controls
	Biological hazards (insects, spiders etc.)	• Workers will inspect the work area carefully and avoid placing hands and feet into concealed areas • Look in direction of travel for biological hazards to avoid
	Electrical hazards	• Drill shall be grounded by a multi-conductor cord with identified grounding conductor and multi-contact plug and receptacle. Comply with Section 11.A – 11.D of EM 385-1-1.
Equipment Used	**Inspection Requirements**	**Training Requirements**
Vehicles and site equipment Pickup trucks or vans Portable generator Flexible extension cords Hand tools	Inspect all equipment prior to use; refer to manufacturers inspection requirements, if appropriate. Inspect flexible extension cords daily. Test GFIC circuits prior to use	Valid operators license for the class of vehicle. Experience in maneuvering small watercraft.
Photoionization detector; Multigas meter; Helium detector; Leak Detection Equipment; Rotary Hammer Drill	Inspect and calibrate Photoionization detector (PID) and Multigas meter daily. Bump test helium detector daily. Calibrate and use instruments/equipment in accordance with manufacturers recommendation	Experience in operating and calibrating a PID and multigas meter. Experience in soil gas sampling. Training in related SEI SOPs.
Decontamination containment vessels/structures including vacuum cleaner	Prior to each use, inspect any decontamination containment vessel or structure. Inspect any container prior before depositing any soil/concrete cuttings or decontamination residuals from work in source areas.	No specific training required.
Equipment Used	**Inspection Requirements**	**Training Requirements**
		General: Review of Health and Safety Plan and Activity Hazard Analyses Hazardous Sites Worker training per 29CFR 1910.120 Initial site/task specific training. Daily Safety Meetings. Review EM 385-1-1 Section 11.A – 11.D.

ATTACHMENT C: HEALTH AND SAFETY PLAN PRE-ENTRY BRIEFING ATTENDANCE AND DAILY SAFETY MEETING FORMS

Health and Safety Plan Pre-Entry Briefing Attendance Form

DEFENSE NATIONAL STOCKPILE CENTER SCOTIA DEPOT SITE

GLENVILLE, NEW YORK

Conducted by:		**Date Performed:**	
Topics Discussed:	1. Review of the content of the HASP/AHA (Required)		
	2.		
	3.		
	4.		

Printed Name	Signature	Time On-site	Time Off-site	Representing

STONE ENVIRONMENTAL INC

535 Stone Cutters Way
Montpelier, Vermont
05602 USA

Phone / 802.229.4541
Fax / 802.229.5417
Web Site / www.stone-env.com

Health and Safety – Daily Site Sheet

Project #: 102305-R C Date: _____

Project Name: Defense National Stockpile Center
Scotia Depot Site, Glenville, New York

Topics Covered:

_____

_____

_____

_____

_____

_____

HASP Author : Stone Environmental, Inc. ■ Other (Client) □

Person(s) Present

Signed: _____	Print: _____	Date: _____
Signed: _____	Print: _____	Date: _____
Signed: _____	Print: _____	Date: _____
Signed: _____	Print: _____	Date: _____
Signed: _____	Print: _____	Date: _____
Signed: _____	Print: _____	Date: _____
Signed: _____	Print: _____	Date: _____

ATTACHMENT D: ACCIDENT INVESTIGATION FORM

STONE ENVIRONMENTAL INC.

Incident/Accident/Injury/Illness Reporting Form

REPORTING SECTION

Form completed by: _____ Date completed: _____

Employee Name: _____ Group: _____ Reported to: _____

Location, Date and Time of Incident or Accident: _____

Description of Events Leading up to Incident or Accident: _____

_____

Equipment/Tool/Vehicle involved in the incident or accident: _____

Was Equipment/Tool/Vehicle defective? _____ If yes, describe: _____

Describe the damage or injury. _____

If necessary, please attach police report or other reporting documentation.

Was hospitalization required? _____ If yes, where? _____

Name and Address of Physician and Hospital: _____

Group Leader's Name: _____ Date of Report: _____

INVESTIGATION SECTION

Could the incident or accident have been prevented? ☐ YES ☐ NO If yes, how? _____

_____

Was Safety Equipment provided? ☐ YES ☐ NO Was it used properly? ☐ YES ☐ NO

Was any work time lost? ☐ YES ☐ NO If yes, how much time? _____

Workers Compensation Insurance Notified? ☐ YES ☐ NO

What actions have been taken to prevent similar incidents or accidents? List corrective actions, recommended changes to procedures/equipment/signs to prevent re-occurrences of this type: _____

_____

_____

Employees Signature: _____ Date: _____

Group Leaders Signature: _____ Date: _____

Safety Committee representative: _____ Date: _____

Other Comments: (if necessary attach additional sheet)

ATTACHMENT E: CURRENT TRAINING CERTIFICATIONS

DAVID J. LINARI

EDUCATION

Michigan Technological University, Houghton, MI
M.S., Geology, 2002
Design and Assessment of a Multimedia Earth Science Education Module

Michigan Technological University, Houghton, MI
B.S., Geo-Environmental Engineering, 2000

WORK EXPERIENCE

Stone Environmental, Inc.
Montpelier, Vermont, USA
Project Geologist, 03/2011 – Present

Collect data at hazardous waste site to remediate and clean up sites. Work with local, state, and federal officials to comply with applicable regulations. Trouble shoot and operate remediation systems.

Ross Environmental Associates, Inc.,
Stowe, Vermont, USA
Project Manager/Geologist, 04/2009 – 02/2011

Collect data at hazardous waste site to remediate and clean up contaminated sites. Work with local, state, and federal officials to comply with applicable regulations. Trouble shoot and operate remediation systems Managed people and brownfields projects.

State of Vermont
Waterbury, Vermont, USA
Environmental Analyst III, 01/2007 – 04/2009

Conduct initial and follow-up RCRA compliance inspections at facilities independently and in cooperation with EPA. Respond to written and telephone requests for information and regulatory interpretations. Document and investigate complaints that pose a threat to human health or the environment.

Heindel & Noyes, Inc.
Burlington, Vermont, USA
Staff Scientist III, 1/2005 – 1/2007

Collected data at hazardous waste site to remediate and clean up hazardous sites. Worked with local, and state officials to comply with state and federal laws and regulations.

Stone Environmental, Inc.
Montpelier, Vermont, USA
Staff Scientist, 4/2003 – 12/2005

Collected groundwater / contaminant plume profiling data using the Waterloo Profiling technique. Managed maintenance of profiling equipment and data acquisition truck while in the field.

Michigan Technological University
Houghton, MI, USA
Graduate Research Assistant
2001-2003

Editing of geologic and topographical maps of Utah and Colorado (ArcView/GIS). Design and building of educational software.

Michigan Technological University
Houghton, MI, USA
TA for Geology of Utah's National Parks, summers 2001 – 2002

Taught students the basics of geology in the field, worked with students in a one-on-one environment. Assisted in the planning of material taught and logistics for the field class.

Michigan Technological University
Houghton, MI, US
Instructor for Summer Youth Program, 2001 – summer

Taught students, ages 11-14, the basics of local geology.

Veritime
Troy, MI, USA
Programmer/IT Analyst, 8/2000 – 12/2000

Local Area Networking and assistance with System Administration. Worked with Linux, Windows 95/98, Unix, and some AS400 systems.

Michigan Technological University
Houghton, MI, USA
Undergraduate Research Assistant 1999-2000

Editing of geologic and topographical mapping of Utah (GIS). Graphic design and preparation for technical publications.

PUBLICATIONS AND PRESENTATIONS

Linari, David J., Huntoon, Jacqueline E., and Bluth, Gregg J.S. 2002. GSA, *Design and Assessment of a Multimedia Earth Science Education Module.* Abstracts with Programs, v.34, n.6. p. 297.

Linari, David J. 2002. *Design and Production of a Multimedia Earth Science Education Module*. Master's Thesis, Michigan Technological University.

Mankowski, L. C., Campbell, T. L., Huntoon, J. E., Gregg, W. J., and Linari, D. J. 2002. *Structural Mapping of the Uncompahgre Front near Gateway, Colorado, with emphasis on Ancestral Rocky Mountain Fabrics*. In C. Kluth, Soreghan, G.S., Snyder, W.S., Barbeau, D.L., and Lawton, T.F., (eds), Late Paleozoic Tectonics and Hydrocarbon Systems of Western North America—The Greater Ancestral Rocky Mountains. *AAPG Hedberg Research Conference.* July 21-26, Vail, CO, p.45-8.

CERTIFICATIONS

OSHA 40-Hour Hazardous Waste Operations and Emergency Response: 2003-2011

Certified in ArcView (GIS): 2001

Certified Wildland Fire Fighter S-130: 2001

OSHA Confined Space Training 2006

HONORS AND AWARDS

Eagle Scout.

TECHNICAL SKILLS

Fluent in DOS, UNIX, Mac OS 8.1-X and Windows 95-XP-7 operating systems.

Used on a daily basis: Illustrator 10, Photoshop 7.0, Fireworks 4.0, Bryce4, Director 8.5, and Origin programs.

Familiar with AutoCAD, Ideas, Canvas, Freehand, MODFlow. Contaminate modeling in EVS.

Fluent with Microsoft programs such as Works, Word, PowerPoint, and Excel.

ArcView (GIS). Working knowledge of ArcInfo, ArcMap, and Imagine.

Orgin, Labview.

Waterloo Profile operator.

GeoProbe® 6610DT direct push drill rig operator.

RELEVANT COURSES

Aerial Photography Interpretation and Geographic Information Systems
Remote Sensing and GIS in Natural Resource Management
Natural Hazards (GIS)
Hydrogeology
Groundwater Site Investigations
Ground Water Modeling (MODFLOW)
Field Geology-Utah
Field Geophysics
Environmental Regulations and Policy
Sedimentology and Stratigraphy
Soils Engineering
Aqueous Geo-Chemistry
Geophysical Fluid Mechanics

PROJECT EXPERIENCE

Contaminated soils excavation at former Kestler's Market, Alburgh, Vermont (2010-2011)
Project manager for petroleum-contaminated soil removal. Oversaw excavations, restored Site, and conducted subsequent groundwater sampling.

Lead remedial implementation at former Rhoades Salvage, Milton, Vermont (2012)
Field technician for former salvage yard site. Field analyzed soil samples to delineate extent of lead contaminated soils. Prepared report on findings. Oversaw operation of skid steer to remove contaminated soils.

Soil excavation and disposal at a residence, Randolph, Vermont (2012)
Site characterization and pre-excavation sampling of petroleum contaminated soils at a residence.

Voluntary Cleanup Site, New York
Field scientist for Waterloo Profiling and onsite laboratory project. Project involved profiling to depths of over 500 feet in a sand and gravel aquifer. Over 3 linear miles of profiles completed and over 1000 samples collected and analyzed using three rigs simultaneously with a single onsite lab.

MA DEP Site, Massachusetts
Field scientist and analytical chemist for ongoing site investigation using the Waterloo Profiler, membrane interface probe and onsite analysis via GC/MS. 55 profile locations averaging 100 feet have been completed to date.

Chlorinated Solvent Site, Connecticut
Field scientist for Triad site investigation on a multi-source chlorinated site. Over the course of 13 days in the field, 35 Waterloo Profile and 36 membrane interface probe locations were completed. All samples were analyzed using onsite GC/MS.

Chlorinated Solvent Site, South Carolina
Field scientist for Waterloo Profiling. Project involved profiling to depths over 200 feet in sand and clay. Project lasted over 11 months with over 20 locations profiled and sampled.

PCB Site, Mississippi
Field scientist for Waterloo Profiling. Project involved profiling from 50 to 120 feet in sand and clay.

Targeted Brownfield Assessment - Vermont
Task and Field Manager a Targeted Brownfield Assessment of a former AOT Railroad Garage in Moretown, Vermont. Designed and implemented a Corrective Action Plan (CAP) and implemented/oversaw the site remediation elements in close cooperation with Site interested parties, Site owners and owner representatives, VT DEC and US EPA to enable property owner limited use of the Site prior to completion of full Site investigation or remediation.

Targeted Brownfield Assessment - Vermont
Task and Field Manager for a Targeted Brownfield Assessment of a former farm located in the Intervale region in Burlington, Vermont. Designed Site investigation elements in close cooperation with Site interested parties, Site owners and owner representatives, VT DEC and US EPA to enable property owner limited use of the Site prior to completion of full Site investigation or remediation.

PCF Site - Vermont
Served as Project Manager- DJ's Corner Store-Johnson, Vermont
Managed operation of a dual-phase extraction system to treat ground water contaminated with gasoline and #2 fuel oil. Also managed the quarterly sampling and reporting to the VTDEC. Responsibilities included periodic visits to evaluate remedial progress and troubleshoot system issues, groundwater sampling, reporting and communicating with VTDEC state site managers.

Phase I/II ESA
Essex Junction, Vermont
Completed a Phase I/II ESA at a former food manufacturing plant for a prospective purchaser. During the Phase I, several areas of concern, including the historic/current use of solvents, floor drains, previously abandoned USTs, and a potential upgradient sources for contamination due to surrounding commercial actives and railroad tracks. Based on the results of the Phase I, a Phase II ESA was implemented which included a network of soil boring / sampling program, and the installation of ground water monitoring wells for sampling. The Phase II determined that site had no environmental impacts in the areas investigated. The Phase II identified the environmental risks associated with the property and enabled the prospective buyer to purchase the property.

PCF Site - Vermont
Served as Project Manager- D&C Petroleum Bulk Storage Faculty Newport, Vermont
Managed the quarterly sampling and reporting to the VTDEC. Responsibilities included, groundwater sampling, reporting and communicating with VTDEC state site managers.
SPCC Plan –
Served as Project Manager- D&C Petroleum Bulk Storage Faculty Newport, Vermont
Created and implemented a Spills Prevention, Control and Countermeasure (SPCC) document to ensure that the bulk facility was in compliance with all state and federal requirements. The plan also ensures that if a release does occur the proper procedures are in place to deal with the emergency.

SPCC Plan –
Served as Project Manager- FPH Petroleum Bulk Storage Faculty Orleans, Vermont
Created and implemented a Spills Prevention, Control and Countermeasure (SPCC) document to ensure that the bulk facility was in compliance with all state and federal requirements. The plan also ensures that if a release does occur the proper procedures are in place to deal with the emergency.

THE NATIONAL ENVIRONMENTAL TRAINERS

certify that

Dave Linari

has satisfactorily passed an exam and completed a 40 hour training course entitled

Hazardous Waste Operations and Emergency Response

meeting the requirements identified in Title 29 CFR 1910.120. This course is also eligible for 3.33 Continuance of Certification (COC) points from the Board of Certified Safety Professionals.

NATIONAL ENVIRONMENTAL TRAINERS

April 4, 2003

Signature of Instructor



Clay Bednarz, MS, CHMM (No. 3482)

CERTIFICATE OF ACHIEVEMENT

This is to certify that

Dave Linari

Has achieved recognition for completing
OSHA 8hr Annual Refresher Training for 40hr Health & Safety Class
Hazardous Waste Operations and Emergency Response (29 CFR 1910.120)
and Annual Refresher in Ethics Training.

Montpelier, Vermont on 1/12/2012

Signature : Kim B. Watson Date: 1/12/2012

STONE ENVIRONMENTAL INC

Concentra Medical Centers (VT)
654 Granger Rd Suite 1 BARRE, VT 05641
Phone: (802) 223-7499 Fax: (802) 223-4120

Service Date: 03/18/2011

Physical Exam

Name: Linari, David J. **SSN:** XXX-XX-4152 **Date:** 03/18/2011

Examination Results

_____ Able to perform essential functions as listed.

_____ Unable to perform all essential functions as listed. Please list failed essential function(s):

_____

_____

_____

__X__ No medical restrictions are indicated.

_____ The following medical restrictions are indicated:

_____

_____

_____

_____ Recommend further evaluation.

Remarks: _____

_____

_____

_____

Rebecca A Schaub PA-C
Provider Print Name Here

Rebecca A. Schaub PA-C
Provider Signature

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CONCENTRA Medical Centers

MEDICAL EXAMINER'S CERTIFICATE

I certify that I have examined DAVID J LINARI in accordance with FMCSR 49 CFR 391.41-391.49 and with knowledge of the driving duties, I find this person is qualified; and, if applicable, only when:

- [X] Wearing Corrective Lenses
- [ ] Wearing Hearing Aid
- [ ] Accompanied by a _____ waiver/exemption
- [ ] Driving within an exempt intracity zone (49 CFR 391.62)
- [ ] Accompanied by a Skill Performance Evaluation Certificate
- [ ] Qualified by operation of 49 CFR 391.64

The information I have provided regarding this physical examination is true and complete. A complete exam form with any attachment embodies my findings completely and correctly, and is on file in my office.

SIGNATURE OF MEDICAL EXAMINER: Rebecca A. Scholl PA-C	TELEPHONE NO.: 802-223-7499	DATE: 3/18/11
MEDICAL EXAMINER'S NAME (print): Rebecca Scholl	[ ] MD [ ] DO	
MEDICAL EXAMINER'S LICENSE OR CERTIFICATE NO. / ISSUING STATE: 055-0030772 VT	[ ] Chiropractor [ ] Advanced Practice Nurse [X] Physician's Assistant	
SIGNATURE OF DRIVER: [signature]	DRIVER'S LICENSE NO.: 22240960	STATE: VT
DRIVER'S ADDRESS (Street, City, State, Zip Code): 1307 JUNCTION RD BERLIN VT 05602	MED. CERT. EXPIRATION DATE: 03/18/2013	

NOTE: Driver MUST carry a copy of this certificate when operating a commercial motor vehicle in accordance with 49 CFR 391.41 (a)

CMCDOTCARD

JOSEPH SCHMIDL, P.G., C.G., C.W.S.

EDUCATION

University of Massachusetts at Lowell
M.S., Environmental Studies, College of Civil Engineering, 1995

University of Massachusetts at Amherst
B.S., Geology, 1982

WORK EXPERIENCE

Stone Environmental, Inc.
Montpelier, Vermont, USA
Senior Project Manager, 08/2012 – Present

Manage environmental programs and large project contracts for clients. Manage multidiscipline project teams and serve as senior technical resource for hydrogeology, site conceptual model development, contaminant fate and transport evaluation, risk assessment, and remedial action design and implementation.

Blackwater Environmental Design
Salisbury, New Hampshire
Designer/Scientist, 04/2003 – Present

Designed environmentally sensitive septic systems, performed soil tests, percolation tests, and septic system inspections. Performed wetland delineations and permitting for residential clients.

Weston Solutions, Inc
Concord, New Hampshire
Technical Manager, 09/1997 – 07/2012

Conducted environmental investigations for federal, state, and local government, and commercial clients under federal and state regulations. Conducted and managed geological, hydrogeological, hydrological, and wetland investigations. Lead sampling activities for groundwater, surface water, sediment, soil, and soil vapor samples, including real-time soil analysis. Developed work plans, coordinated field staff and subcontractors, regularly interacted with clients, interpreted results, reporting, technical review of deliverables, and management of project budgets. Specialized in development of multimedia conceptual site models.

Roy F. Weston, Inc.
Burlington, Massachusetts
Project Leader, 05/1995 – 09/1997

Conducted pre-remedial site assessment projects under EPA Region 1's Superfund Technical Assessment and Response Team (START). Managed six junior staff conducting site assessment projects, including technical review of deliverables, and schedule and budget tracking. Project leader for the Hazard Ranking System packages produced for EPA Region 1. Participated in evaluations of dozens of sites in New England and sites investigated under EPA's Brownfields Program.

Roy F. Weston, Inc.
Wilmington, Massachusetts
Project Geologist, 08/1994 – 05/1995

Conducted pre-remedial site assessment projects under EPA Region 1's Alternative Remedial Contract Strategy (ARCS) program. Projects included numerous site inspections (SIs), including initiation of 12 SIs conducted simultaneously in the Farmington Industrial Park in Connecticut. Supported remedial investigations at the former Pease Air Force Base in Portsmouth, New Hampshire. Duties also included a number of site investigation projects conducted to satisfy state regulations in Connecticut and Massachusetts.

TRC Companies, Inc.
Lowell, Massachusetts
Geoscientist, 07/1991 – 08/1994

Conducted pre-remedial site assessment projects under EPA Region 1's ARCS program. Projects included numerous SIs, RCRA/CERCLA Preliminary Assessments, and two Hazard Ranking System documentation records. Performed technical oversight of responsible party-led projects and a number of site investigation projects under state regulations in Connecticut, Kentucky, and Massachusetts.

NUS Corporation
Bedford, Massachusetts
Geologist, 12/1990 – 07/1991

Conducted pre-remedial site assessment projects under EPA Region 1's dedicated Field Investigation Team (FIT) contract.

Boy Scouts of America,
Dalton and Chicopee, Massachusetts
District Executive, 09/1984 – 12/1990

Served as professional representative to volunteer leaders and chartered organizations. Performed public relations, recruiting and training of volunteer leaders, fundraising, and management of summer camp operations.

ADDITIONAL EDUCATION

Preliminary Assessment and Site Assessment Training, EPA Region I, 1992.

Hazard Ranking System Training, EPA, Kansas City, Kansas, 1993.

Dangerous Goods Shipping, renewed 2010.

Bloodborne Pathogens Training—Initial, OSHA 29 CFR 1910.1030, (1995); refresher, Weston, 2012.

Confined Space Training—Entrant, Attendant, Non-Entry Rescue, OSHA 29 CFR 1910.146, Weston, 1996.

Trenching/Excavation Competent Person Training Course, OSHA 29 CFR 1026 Subpart P, Weston, 2010.

Fall Protection Competent Person Training Course—Initial, OSHA 29 CFR 1926 Subpart M, Weston, 2011.

Asbestos Inspector Title II, TSCA, Institute for Environmental Education, Inc., 2011.

Hazardous Waste Management and Shipping for Environmental Professionals, 40 CFR 265.16, Eduwhere, 2001.

Sample Shipping, Weston, 2009.

Underground Utilities Competent Person Training, Weston, 2010.

ICS-100 Introduction to Incident Command System, FEMA, 2010.

ICS-200 Basic Incident Command System, FEMA, 2010.

Lead Awareness Training, Weston, 2009.

Behavior-Based Safety Training – Phase I, Weston, 2007.

IATA/DOT Shipping Advisor Training—Initial, 2002; refresher, Weston, 2010.

DOT Hazardous Materials Training, 49 CFR 172 Subpart H, HAZMATEAM, Inc., 2008.

First Aid/CPR/AED Training, American Red Cross, 2011.

8-Hour Hazardous Waste Refresher Course, OSHA 29 CFR 1910.120(e)(8), Weston, 2011.

40-Hour Hazardous Waste Site Training Course, OSHA 29 CFR 1910.120(e)(3), NUS Corporation, 1990.

8-Hour Managers and Supervisors Course (SHSC), OSHA 29 CFR 1910.120(e)(4), Weston, 1994.

10-Hour Construction Industry Outreach Training, 29 CFR 1926, Weston, 1998.

Shipping and Transporting Dangerous Goods—Administrative / Field Personnel. Refresher, Weston Solutions, Inc., Manual of Procedures for Shipping and Transporting Dangerous Goods Training Course, 49 CFR 172, Subpart H, Weston, 2011.

Dangerous Goods Shipping Advisors Refresher, 49 CFR 172 Subpart H, HAZMATEAM, 2010.

CERTIFICATIONS

Professional Geologist, State of New Hampshire, License Number 47.

Certified Geologist, State of Maine, Certification Number GE436.

Certified Wetland Scientist, State of New Hampshire, License Number 263.

Licensed Designer of Subsurface Disposal Systems, State of New Hampshire, Number 1547.

Licensed Site Professional, State of Massachusetts, License Number 4442.

OSHA 40-hour certification

OSHA Site Supervisor certification

OSHA Construction Manager certification

Non-Entry Confined Space Entry certification

Asbestos Inspection certification.

SKILLS

Computers: fluent with Microsoft Office (Word, Excel, Powerpoint, Access); CADopia IntelliCAD; ESRI ARCView Geographic Information System; ProUCL and MAROS statistical software.

PROFESSIONAL AND COMMUNITY ACTIVITIES

Chairman of Planning Board, Town of Salisbury, New Hampshire.

PROJECT EXPERIENCE

US Environmental Protection Agency
Walton & Lonsbury Superfund Site, Attleboro, MA: Provided wetlands compliance support.

West Street Property Site, Hatfield, MA: Performed wetlands delineation and prepared a wetlands restoration plan.

Former Chlor-Alkali Facility, Berlin, NH: Lead Geologist for Remedial Investigation/Feasibility Study for this elemental mercury release site.

Eighteen Mile Creek Superfund Site, Lockport, NY: Performed wetlands delineation to support Hazard Ranking System evaluation.

St. Croix Alumina Superfund Site, St. Croix, USVI: Performed wetlands delineation and environmental groundwater, surface water and sediment sampling.

US Army Corps of Engineers
Tom Nevers Formerly Used Defense Site, Nantucket, MA: Prepared Environmental Protection Plan component of Work Plan for UXO survey and removal.

Former LO-58 Site, Caribou, ME: Project Manager for Supplemental Site Investigation involving hydrogeophysical survey and interpretation of bedrock groundwater contamination.

GE/Housatonic River Site, Pittsfield, MA: Technical review of PRP submittals for EPA via USACoE contract. Coordinated activities with Massachusetts Department of Environmental Protection staff, for this multi-jurisdictional site managed under a Consent Decree.

Air Force Center for Engineering and the Environment (AFCEE)
Former Landfill A, Westover Air Reserve Base, Chicopee, MA: Prepared Activity and Use Limitation paperwork and Response Action Outcome Statement for site closure.

Former Construction Rubble Site A, Westover Air Reserve Base, Chicopee, MA: Project Manager for Supplemental Site Investigation involving Field X-Ray Fluorescence survey and Phase II Report.

Explosive Ordinance Disposal Proficiency Range, Westover Air Reserve Base, Chicopee, MA: Project Manager for Supplemental Site Investigation involving subsurface investigation of light, non-aqueous phase liquid, and Phase II Report.

Remedial Investigation/Feasibility Study (RI/FS), Former Chlor-Alkali Facility Site, Berlin, NH, U.S. Environmental Protection Agency (EPA) Region 1, Lead Hydrogeologist

As a Team Subcontractor to Nobis Engineering, Inc., provided technical direction for the investigation of elemental mercury contamination at this former chemical plant site, where elemental mercury discharge via bedrock fractures to the Androscoggin River has occurred. Selected subsurface investigation locations (soil borings and test pits). Reviewed subsurface data collected during monitoring well installation and well development. Directed test pitting investigations, including the test pit excavated in November 2011, during which the disposal location of the former chlor-alklai cells was discovered, along with the elemental mercury they contained. Performed bench-scale testing of hypotheses regarding mercury fate and transport in groundwater. Interpreted groundwater hydrogeological and analytical results, and prepared a summary report for the site. [9-07 to present; WESTON; Proj. No. 12884.013.001 to 003]

Wetland Mapping and Sampling, St. Croix Alumina Site, St. Croix, U.S. Virgin Islands, EPA Region 2, Wetland Scientist

Conducted ground-verification of mapped forested, shrub-scrub, and emergent estustrine wetlands, as well as the confirmation of the location of the downstream surface water pathway in support of a Hazard Ranking System (HRS) documentation record. Wetland extents within the approximately 1-square-mile site were documented in 2 days. Work was accomplished ahead of schedule using only 50% of the estimated level of effort. Following the wetland identification, collected sediment and surface water samples from 30 locations in a 3-day effort [9-11; WESTON; Proj. No. 20405.012.013.1455]

Removal Assessment Support, Former Grand Union Site, Fort Edward, NY, EPA Region 2, Lead Hydrogeologist

Conducted a multimedia subsurface soil and groundwater investigation of a former grocery store property, which may have received fill material from a nearby polychlorinated biphenyl (PCB) facility. Advanced 12 soil borings to depths of 16 feet below ground surface (bgs) and 20 test pits to depths of 8 feet bgs on two potentially impacted properties. Logged soil borings and test pits, and identified buried soil horizons that allowed the extent of fill on the properties to be determined. Also installed two overburden groundwater monitoring wells, and collected soil and groundwater samples to characterize potential risks. Soil samples were analyzed by EPA Region 2's laboratory. Performed an elevation survey for the newly installed wells. Project was completed within budget, and in a

total of 7 working days. [10-11; WESTON; Proj. No. 20401.132.022.5092]

Removal Assessment Support, Jewett White Lead Company Site, Staten Island, NY, EPA Region 2, Project Manager

Conducted a multimedia subsurface soil, sediment, surface-water, and groundwater investigation of a former white lead manufacturing facility. Advanced approximately 25 soil borings and 25 test pits on two impacted properties to depths of 8 feet bgs, installed 5 overburden groundwater monitoring wells, and collected soil, sediment, surface-water, and groundwater samples to characterize potential risks. Soil samples were analyzed on-site for lead utilizing a field portable X-ray fluorescence (FPXRF) unit, with a fraction of the samples submitted to EPA Region 2's laboratory for confirmatory analysis. Performed statistical analysis of the soil lead results to determine usability of FPXRF results. Compiled the results into a Removal Actions Alternatives Report, one each for both properties. Reports included evaluation of up to five remedial options. Project was completed under budget, and within an aggressive 4-month time period. [7-10 to Present; WESTON]

Wetland Delineation and Mapping, 18-Mile Creek Site, Lockport, NY, EPA Region 2, Wetland Scientist

Conducted a delineation of forested and shrub-scrub palustrine wetlands in support of a HRS documentation record. Wetland frontage to approximately 6 stream miles of 18-Mile Creek totaling approximately 1 mile was delineated in a single day according to U.S. Army Corps of Engineers (USACE) Wetlands Delineation Manual Y-87-1 guidelines. Wetland boundary points were located using a Trimble global positioning system (GPS) unit. Work was accomplished ahead of schedule using only 70% of the estimated level of effort. [6-11; WESTON; Proj. No. 20405.012.013]

Removal Action Support, Various Locations in New Jersey and New York, EPA Region 2, Site Manager

Conducted a variety of removal action support tasks for WESTON's Removal Support Team (RST)-2 in Edison, NJ. Activities included removal support (contractor oversight, air monitoring, confirmation sampling) and Administrative Record preparation. [4-11 to 6-11; WESTON; Proj. No. 20401.122]

Wetland Delineation/Restoration Planning, West Street Site, Hatfield, MA, EPA Region 1, Wetland Scientist

Conducted a delineation of forested and shrub-scrub palustrine wetlands in advance of site remedial activities. One area of wetlands totaling approximately 4 acres was delineated in a single day according to USACE Wetlands Delineation Manual Y-87-1 guidelines. Wetland boundary flags were placed for later surveying. Plant species were inventoried to support the development of a Wetland Restoration Plan. [9-09 to Present; WESTON; Proj. No. 20114.056.998]

Disposal Site Closure, Confidential Client Sites, Wilmington, MA, EPA Region 1, Apprentice to Licensed Site Professional (LSP)

Under the authority of a WESTON LSP, conducted a series of investigations at two adjacent disposal sites to satisfy requirements of the Massachusetts Contingency Plan (MCP) (30 CMR 40.0000). The property to be investigated was formerly owned by WESTON's confidential client, which resulted in a complicated project approval process that included the current owner of the property. One disposal site was the site of a release of PCB-containing transformer oil. The other disposal site was an area of contaminated soil and groundwater resulting from contaminated fill beneath a 20-year-old building. Designed and performed or directed a series of iterative soil boring and well installation programs, and soil and groundwater sampling activities. Deliverables included Phase I Initial Site Investigations, Phase II Comprehensive Site Assessments, Phase III Remedial Action Plans, Phase IV Remedy Implementation Plans, and Response Action Outcome Statements to support site closure. In addition, prepared deliverables to satisfy EPA Region 1 Toxic Substances Control Act (TSCA) requirements pursuant to PCBs present at the sites. [2004 to Present; WESTON; Proj. No. 02181.167]

Wetland Delineation, Caryville Mill Superfund Site, Bellingham, MA, EPA Region 1, Wetland Scientist

Conducted a delineation of forested palustrine and riverine wetlands in advance of site remedial activities. Three separate areas of wetlands totaling approximately 40 acres were delineated in a single day according to USACE Wetlands Delineation Manual Y-87-1 guidelines. Wetland boundary flags were located using a sub-meter accurate GPS, and a wetlands boundary map was delivered to the client within 24 hours to expedite the initiation of remedial activities. [7-09 to Present; WESTON; Proj. No. 12884.016]

Brownfields Site Assessment – Underground Storage Tank (UST) Removal, Frank's Texaco Site, Winchester, NH, Southwest Regional Planning Commission (SWRPC), Project Manager

Directed the investigation of potential petroleum groundwater contamination at this former gasoline station site. Prepared generic and

site-specific Quality Assurance Project Plans (QAPPs) for submission to EPA Region 1 Brownfields Section for review. Determined the nature and extent of subsurface soil and groundwater contamination in accordance with New Hampshire Department of Environmental Services (NHDES) regulations (Env-Or 600 and 800). WESTON personnel performed soil sampling, groundwater sampling, and documented groundwater flow direction for the site. In addition, directed the removal of two 6,000-gallon USTs and one 1,000-gallon UST from the site, and performed post-removal confirmatory sampling. Presented hydrogeological and analytical results in a summary report for the site. [10 06 to 6-07; WESTON; Proj. No. 13877.001.002]

RIs, Westover Air Reserve Base (ARB), Chicopee, MA, Air Force Center for Engineering and the Environment (AFCEE), 4P Architect-Engineer (A-E) Contract, Project Manager

Performed environmental investigations of a number of disposal sites under contract to AFCEE pursuant to compliance with the MCP. Activities included review of previous investigations and analytical results; evaluation of existing analytical results for data usability and representativeness in compliance with Massachusetts Department of Environmental Protection (MassDEP) guidance; development of a sampling plan to eliminate data gaps; execution of the sampling program; incorporation of the new analytical results to each site data set; support to subcontracted risk assessors in performing human health and/or environmental risk characterizations; completion of revised MCP deliverables; and interaction with MassDEP personnel in addressing comments on MCP deliverables. In most cases, WESTON started work on these projects after another AFCEE contractor had recommended, and MassDEP had approved, extensive and expensive remedial actions to close these disposal sites under the MCP. By collecting additional environmental samples and eliminating data gaps, WESTON was able to perform much less conservative risk assessments (relying on data rather than conservative assumptions) and document that no remedial activities were required for disposal site closure under the MCP. This approach saved AFCEE 90% of its disposal site closure costs. [5-07 to Present; WESTON; Proj. Nos. 20077.043.093, .150, .155; 20077.048.030]

Hydrogeological Investigation, Maine Formerly Used Defense Sites (FUDSs), Caribou and Perham, ME, USACE, New England District (CENAE), Project Manager

Under contract to CENAE, performed detailed hydrogeological investigations of two FUDSs located in northern Maine with the specific objective of determining the interconnection between impacted bedrock drinking water wells. The investigation included review of previous investigations and analytical results, subcontracting of geophysical and hydrophysical surveys of the impacted drinking water wells, sampling of individual fracture zones based on the results of the geophysical and hydrophysical surveys, and the interpretation of the analytical, geophysical, and hydrophysical data to create a Conceptual Site Model (CSM) for the sites. WESTON interacted with Maine Department of Environmental Protection (MEDEP) personnel as well as CENAE in addressing comments on Work Plans and other deliverables. [3-07 to Present; WESTON; Proj. No. 03886.184.001]

Long-Term Monitoring Program Support, Woodstock, CT, EPA Region 1, Remedial Action Contract (RAC), Project Manager

Provided technical support to EPA Region 1 under its RAC (as a subcontractor to Nobis Engineering, Inc.) at the Linemaster Switch Corporation Superfund site in Woodstock, CT. Support included oversight/technical review of responsible party (RP) contractor field activities and deliverables. WESTON performed oversight of RP contractor field activities during two semiannual monitoring rounds in November 2007 and May 2008. WESTON reviewed three semiannual monitoring reports and one proposal for changes to the long-term monitoring program and provided technical comments to EPA Region 1. The project was completed significantly under budget and within schedule. [9-07 to Present; WESTON; Proj. Nos. 12884.010.001, .003, .008, .010, .011]

Emergency Response Support, Stoddard, NH, EPA Region 1, Superfund Technical Assessment and Response Team (START), Septic System Designer

Provided technical support to EPA Region 1 under its START contract at the Electrosonic Superfund site in Stoddard, NH. Support included the design of a replacement septic system for the facility following removal of the existing system due to contamination with heavy metals. The design was constrained by a small replacement area, bounded by a surface water body. WESTON also provided support in obtaining the required state and local approvals for the septic system design. [3-05 to 9-06; WESTON; Author doesn't have project number]

Hurricane Rita Response Support, Marsh Operations, Chambers County, TX, EPA Region 6 START, Wetland Scientist

Provided technical support to EPA Region 6 under its START contract at various locations in Chambers County, TX. Support included the identification of sensitive wetlands during emergency response operations, and development of alternative access plans in order to reduce environmental impacts by recovery equipment. WESTON scouted ahead of emergency response contractors, and identified routes for heavy equipment that avoided sensitive wetland environments to the greatest extent possible. WESTON then documented pre- and post-removal conditions, as well as performed removal documentation. [10-08 to 11-08; WESTON; Proj. No. 20406.012.030]Hurricane Rita Response Support, Cameron Parish, LA, EPA Region 6 START, Wetland Scientist. Provided technical support to EPA Region 6 under its START contract at various locations in Cameron Parish, LA. Support included the identification of sensitive wetlands during emergency response operations, and development of alternative access plans in order to reduce environmental impacts by recovery equipment. WESTON scouted ahead of emergency response contractors, and identified routes for heavy equipment that avoided sensitive wetland environments to the greatest extent possible. WESTON then documented pre- and post-removal conditions, as well as performed removal documentation. [11-06; WESTON; Author doesn't have project number]

RI Support, Pittsfield, MA, EPA Region 1, Project Manager

Provided technical support to regulatory agencies (EPA and MassDEP) at the General Electric (GE)/Housatonic River site in Pittsfield, MA. Support included technical review of Work Plans submitted to the agencies for compliance with approved Sampling and Analysis Plans, and the Consent Decree (CD) that promulgates the remedial actions at the site. Reviewed Work Plans for soil sampling, subsurface drilling and soil sampling, monitoring well installation, and groundwater sample collection; and provided the comments and recommendations to EPA for inclusion in approval letters. Support also included numerous research activities regarding site background information, with particular emphasis on site hydrogeology. Met regularly with EPA, MassDEP, and other WESTON Project Managers to review progress on the project. Acted as an interface with GE contractors to ensure that work was completed according to approved Work Plans, and helped develop alternatives as site conditions warranted. Provided technical support to CENAE/EPA and MassDEP through technical review of GE deliverables for compliance with the CD and Statement of Work (SOW) for Removal Actions Outside the Housatonic River, from 2001 to the present. Reviewed Removal Action Area (RAA) deliverables, including: Pre-Design Investigation Reports, Conceptual and Final Removal Design/Removal Action Work Plans, and Final Completion Reports; reviewed Groundwater Management Area (GMA) deliverables, including Baseline Groundwater Monitoring Reports, Interim Groundwater Monitoring Reports, Nonaqueous Phase Liquids Monitoring Reports, and Long-Term Groundwater Monitoring Proposals, providing the comments and recommendations to EPA for inclusion in approval letters for the subject deliverables. Support included numerous research activities regarding site background information, with particular emphasis on site hydrogeology and the distribution of nonaqueous phase liquids (NAPLs), including recommendations for supplemental groundwater investigations to address data gaps. Independently plotted groundwater GE analytical results, combining results from adjacent GMAs, to provide additional perspective to EPA regarding contaminant nature and extent. Worked closely with EPA Remedial Project Managers (RPMs) to provide background information for technical meetings. Facilitated meetings among EPA, MassDEP, GE, and their consultants to discuss topics that were incompletely described in the CD and SOW, and resolving differences of opinion. Performed independent spatial averaging calculations for RAAs to not only confirm GE's compliance with the CD, but also to test hypotheses regarding optimal soil removal results. [1998 to Present; WESTON; Proj. No. 20124.001.098]

Environmental Investigation, Former Chlor-Alkali Facility Site, Berlin, NH, NHDES, Project Manager

Directed the investigation of potential elemental mercury groundwater contamination at this former chemical plant site. Investigated the source of elemental mercury discharge via bedrock fractures to the Androscoggin River. Managed drilling subcontractor for monitoring well installation and well development. WESTON personnel performed groundwater sampling and hydraulic conductivity testing for the site. In addition, coordinated efforts with WESTON START personnel who were conducting a concurrent preliminary assessment/site inspection (PA/SI) for EPA Region 1. Interpreted groundwater hydrogeological and

analytical results, and prepared a summary report for the site. Managed three subsequent, annual mercury removals from bedrock fractures along the bank of the Androscoggin River. [6-04 to 4-07; WESTON; Proj. No. 20111.001.001 to .006]

Preparation of HRS Documentation Record, Pownal Tannery Site, Pownal, VT, EPA Region 1 START, Project Leader

Worked at several stages of the HRS evaluation of this site by EPA Region 1. Initially conducted environmental sampling at the site in 1993, and prepared the Site Inspection Prioritization (SIP) of the site under TRC Environmental Corporation's Alternative Remedial Contracting Strategy (ARCS) contract for EPA Region 1. Subsequently prepared a draft HRS Documentation Record for the site, which was shelved at the request of the Vermont Department of Environmental Conservation (VDEC). Both efforts required the review of extensive background data collected by VDEC and EPA Region 1 as part of Resource Conservation and Recovery Act (RCRA), National Pollutant Discharge Elimination System (NPDES), and Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA) regulatory activities, including analytical results collected by EPA Region 1 Technical Assistance Team (TAT) during concurrent emergency removals from the site. In 1996, directed technical staff in updating the previous draft HRS Documentation Record at the request of EPA Region 1 under WESTON's START contract. Assisted technical staff with cost tracking and scheduling, reporting monthly to the client regarding deviations from schedule and budget amendments. Duties included technical review of draft and final reports. Provided direction during field activities to ensure compliance with the assigned EPA SOW and EPA guidance documents, and the collection of HRS-quality field data. START delivered the package on time and under budget. [1996 to 1997; WESTON; Author doesn't have project number]

Environmental Investigation, Former Greene Tannery Site, Milton Mills, NH, NHDES, Project Manager

Directed the investigation of potential remnant groundwater contamination at this former tannery site, following remedial activities, in consideration of possible redevelopment. Obtained drilling subcontractor for monitoring well installation, and performed well development and groundwater sampling for the site. Required the review of extensive background data collected by NHDES and EPA Region 1 as part of RCRA, NPDES, and CERCLA regulatory activities, including analytical results collected by EPA Region 1 TAT during previous emergency removals from the site. In addition, obtained environmental data from WESTON START personnel, who were conducting a concurrent SIP for EPA Region 1. Interpreted groundwater analytical results, as well as WESTON START soil, sediment, groundwater, and surface water analytical results (prior to the SIP submission to EPA Region 1), and prepared a summary report for the site. [1997 to 1998; WESTON; Author doesn't have project number]

Preliminary Assessment-Plus Inspections, Multiple Commercial and Industrial Properties, New England, Site Leader

Conducted facility inspections to ensure compliance with EPA Region 1 regulations under CERCLA and RCRA, using National Corrective Action Prioritization System (NCAPS) criteria. Performed facility audits of hazardous materials (waste handling, storage, and disposal). Included inspection of hazardous materials storage areas, process areas, and waste accumulation or disposal areas at each facility. [1992 to 1993; TRC]

RI/Comprehensive Site Assessment Oversight, EPA Region 1, Field Team Leader

Coordinated the technical aspect of providing oversight of drilling and monitoring well installation for EPA Region 1 at its highest priority site from 1998 to the present. Developed an electronic report format to keep EPA, MEDEP, USACE, New England District (CENAE), and WESTON Project Managers abreast of daily progress on the project. Acted as an interface with potentially responsible party (PRP) contractors to ensure that work was completed according to approved Work Plans, and helped them develop alternatives as site conditions warranted. Also provided EPA Region 1 with daily summary of nonaqueous liquids noted, soil headspace screening results, and collected split samples to confirm PRP contractor laboratory results. Also conducted technical review of the reports documenting the work that was overseen, correcting errors, and reinterpreting the data presented. [1998 to 2003; WESTON; Proj. No. 20124.001.098]

Groundwater Extraction System Installation, Groveland Wells Superfund Site, Groveland, MA, Metcalf & Eddy/EPA, Project Geologist

Directed subcontractors in advancing pilot borings, installing and developing monitoring wells, and installing and developing five extraction wells (completed in overburden and/or bedrock). Assisted Project Manager with cost tracking and scheduling, and reporting costs to the client. Also assisted with the carbon filtration of wastewater

generated during the field activities, completing the project 1 week earlier than budgeted, and significantly reducing subcontractor costs. [1998; WESTON; Author doesn't have project number]

Site Assessments, New England, EPA Region 1 START, Project Leader

Managed the activities of six technical staff in conducting PAs, SIs, Mini-SIs, and HRS Documentation Records. Assisted technical staff with cost tracking and scheduling, reporting monthly to the client regarding deviations from schedule and budget amendments. Duties included technical review of deliverables such as Task Work Plans/Non-Sampling Recommendation Memoranda, trip reports, and draft and final reports. Provided direction during field activities (including site reconnaissance and facility inspections, and field sampling) to ensure compliance with the assigned EPA SOW. [1995 to 1997; WESTON; Various Project Nos.]

Preparation of HRS Documentation Record, Housatonic River, Pittsfield, MA, EPA Region 1 START, Project Leader

Managed the activities of six technical staff in preparing the HRS Documentation Record for this site. The project was EPA Region 1's highest priority from November 1996 to August 1997. Assisted technical staff with cost tracking and scheduling, reporting monthly to the client regarding deviations from schedule and budget amendments. Duties included technical review of draft and final reports. Provided direction during field activities to ensure compliance with the assigned EPA SOW and EPA guidance documents, and the collection of HRS-quality field data. As deadlines neared, anticipated workload and requisitioned additional staff to meet deadlines for deliverables. EPA Region 1 requested that START complete the project in less than the typical schedule to meet EPA Headquarters proposal deadlines. Further, the site was complicated by the overlapping CERCLA and RCRA programs, both of which had jurisdiction over the GE facility. This led to tasks and comments not typically required for an HRS Documentation Record. Despite these obstacles, START delivered the package on time in every case, receiving written congratulations from the EPA Site Assignment Manager for WESTON's efforts. [1995 to 1997; WESTON; Author doesn't have project number]

Geoprobe® Operation, Various Hazardous Waste Sites in New England, Multiple Clients, Operator

Following manufacturer's guidelines, operated the EPA Region 1 Geoprobe® hydraulic soil sampling unit at various hazardous waste site investigations in New England. Used the unit to collect deep soil samples, groundwater samples, install miniature monitoring wells, and perform continuous soil screening/logging. Performed routine maintenance on the unit, and trained geotechnical staff in its safe operation. [1992 to 1997; WESTON; Proj. Nos. various ARCS and START assignments]

Technical Skills Training, Various Locations, WESTON, Trainer

Performed training in the following areas: EPA's HRS risk assessment model, WESTON's Dangerous Goods Shipping policy supporting International Air Transport Association (IATA) shipping regulations, and operation of the Geoprobe® hydraulic soil sampling unit. Tasks included development of the training curriculum, preparation of training materials, presenting the training course, and follow-up paperwork, as required. [1998 to Present; WESTON; Proj. No. 00834.003]

HRS Documentation Record, South Weymouth Naval Air Station (NAS), MA, EPA, ARCS Contract, Task Manager

Compiled file information to document the presence of hazardous substances. Determined that analytical results of samples collected from the site by third parties did not meet EPA quality assurance/quality control (QA/QC) objectives. Employed an innovative approach to evaluate the site to develop the HRS Documentation Record. The record withstood public scrutiny, and the site was added to the National Priorities List (NPL). [1993 to 1994; TRC]

SIPs, Various Locations, EPA, ARCS Contract, Deputy Project Manager

Documented the occurrence of hazardous materials at CERCLA sites based on file information and on-site reconnaissance and sampling. Provided guidance to Task Managers in preparing sampling plans designed to meet EPA's HRS objectives. Assisted Task Managers to keep projects on schedule and within budget. Performed technical review of project deliverables. [1991 to 1994; TRC]

Site Inspection, Active Manufacturing Facility, Connecticut, EPA, ARCS Contract, Task Manager

Documented the occurrence of hazardous materials (metals and chlorinated solvents) at this Superfund site based on file information, facility inspection, and waste sample analytical results. Obtained access to four adjacent properties to collect groundwater, surface-water, and sediment samples

to document a release from on-site sources. Samples were collected to meet Level IV data quality objectives (DQOs). [1992; TRC]

RI, Drum Disposal Area, Keysburg, KY, Confidential Client, Field Geologist

Directed the installation of soil borings and monitoring wells in the vicinity of a former drum disposal area. Performed geological reconnaissance over a 20 square-mile area downgradient of the site to map the occurrence of disappearing/appearing streams, sinkholes, and other karst features. Collected groundwater samples on-site, drinking-water samples from wells located downgradient of the site, and surface-water samples that documented the release of hazardous substances (chlorinated solvents) to groundwater. [1993; TRC]

Remedial Action, Railroad Yard, Hermon, ME, Bangor-Aroostook Railroad, Field Geologist

Performed oversight of excavation of waste-oil-contaminated sediments from a wetland adjacent to a railroad yard. Performed continuous air monitoring for all personnel on the project. Collected confirmatory soil samples for the excavation to document achievement of remedial goals. Assisted with the construction of a 10 acre landfarm for on-site remediation of petroleum-contaminated sediments. [1992 to 1993; TRC]

Flood Control Study, Saugus River Estuary, Saugus, MA, USACE, Field Geologist

Project was a flood control study to determine sedimentation rates within the estuarine system. Assisted with the identification of appropriate sample locations, based on vegetation type. Collaborated on the design of sampling apparatus and establishment of sampling stations. Collected sediment cores for Cs137 dating analysis, storm event sediment and water samples, and measured river flow rates during tidal sources. [1992; TRC]

Site Assessments, Commercial and Industrial Properties, Connecticut, Confidential Mortgage Firm, Field Geologist

Assisted with environmental assessments at four separate properties in 4 days. Utilized magnetometer and electromagnetic survey equipment to document the location of underground tanks and utilities to facilitate site clearance for subsequent groundwater well installation. Collected groundwater samples from on-site monitoring wells. Prepared results for inclusion in reports within 2 day turnaround schedule. [1993; TRC]

CERTIFICATE OF ACHIEVEMENT

This is to certify that

Joseph Schmidl

Has achieved recognition for completing
OSHA 8hr Annual Refresher Training for 40hr Health & Safety Class
Hazardous Waste Operations and Emergency Response (29 CFR 1910.120)
and Annual Refresher in Ethics Training.

Montpelier, Vermont on 1/7/2013

Signature : Kim B. Watson Date: 1/7/2013

STONE ENVIRONMENTAL INC

Certificate of Training

This Document Certifies That

JOSEPH SCHMID

Has Successfully Completed The OSHA Required 8 Hour

HEALTH AND SAFETY TRAINING

Complying with OSHA STANDARD 29CFR 1910.120

MANAGER SUPERVISOR

Patrick J. Thornton

Course Director

EPT inc.
Environmental Protection Training, inc.

OCTOBER 25, 1991

Date of Completion

002278

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ROY F. WESTON, INC.

THIS CERTIFIES THAT

Joseph Schmidl

HAS COMPLETED THE

10-Hour Construction Industry Outreach Training meets 29 CFR 1926

Manchester, NH
March 30, 1998

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JOSEPH SCHMIDL

has completed the

CONFINED SPACE TRAINING FOR NON-ENTRY RESCUERS
MEETS 29 CFR 1910.146

BURLINGTON, MASSACHUSETTS
JULY 9, 1996

Training Manager

Instructor

The Continuing Education Unit

11295

Roy F. Weston, Inc.

This certifies that

Joseph Schmidl

has completed the

MANAGERS DESIGNERS/CONSULTANTS

Site Health & Safety Coordinators Course

in Accordance with 29 CFR 1910.120 (e) (4)

Wilmington, MA

George M Crawford

Corporate Health & Safety Director

December 28, 1994

Date

The Continuing Education Unit

Certificate Of Training

16-HOUR AIR TRANSPORTATION (IATA) REFRESHER

This certificate has been awarded to

JOSEPH SCHMIDL

of

WESTON SOLUTIONS, INC.

For successfully completing the HAZMATEAM, Inc. Air Transportation Training course and test and security planning on the applicable regulations of the DOT and ICAO equivalent regarding the proper Philosophy, Application, Limitations, Classification, Identification, Packing, Packaging, Marking and Labeling, and Documentation of Dangerous Goods,

as of 11/22-23/2010
Required Bi-annually
Expires 11/23/2012

HAZMATEAM, INC. HAZMAT 603-882-1112

Leo Traverse, Instructor MA TCH # BC-2009-2149 = 5 / NH CEU = 1.6

CERTIFICATE OF TRAINING

8-HR DOT GROUND TRANSPORTATION REFRESHER

This certificate has been awarded to

JOSEPH SCHMIDL

of

WESTON SOLUTIONS, INC.

For successfully completing the HAZMATEAM, Inc. hazardous materials/waste ground shipping course and security planning for the applicable regulations of the United States Department of Transportation regarding the safe and proper shipping of materials/wastes designated as hazardous,

as of 11/21/2011
Recommended Bi-Annually
Expires 11/21/2014

HAZMATEAM, INC.
HAZMAT
603-882-1112

Paul Dambek, Instructor

MA TCH # BC-2003-1223 = 8 / NH CEU = 0.8

Emergency Management Institute

FEMA

This Certificate of Achievement is to acknowledge that

JOSEPH SCHMIDL

has reaffirmed a dedication to serve in times of crisis through continued professional development and completion of the independent study course:

IS-00100.a
Introduction to the Incident Command System,
ICS-100

Issued this 15th Day of January, 2010

Cortez Lawrence, PhD
Superintendent
Emergency Management Institute

0.3 IACET CEU

Emergency Management Institute

FEMA

This Certificate of Achievement is to acknowledge that

JOSEPH SCHMIDL

has reaffirmed a dedication to serve in times of crisis through continued professional development and completion of the independent study course:

IS-00200.a
ICS for Single Resources and
Initial Action Incidents, ICS-200

Issued this 15th Day of January, 2010

0.3 IACET CEU

Cortez Lawrence, PhD
Superintendent
Emergency Management Institute

American Red Cross

This recognizes that

JOSEPH SCHMIDT

has completed the requirements for

Adult FA/CPR/AED

conducted by

American Red Cross

Date completed: **01/07/2013**

Valid for **2** year(s)

CERTIFICATE OF ACHIEVEMENT

This is to certify that

Lee Rosberg

Has achieved recognition for completing
OSHA 8hr Annual Refresher Training for 40hr Health & Safety Class
Hazardous Waste Operations and Emergency Response (29 CFR 1910.120)
and Annual Refresher in Ethics Training.

Montpelier, Vermont on 1/7/2013

Signature : Kim B. Watson Date: 1/7/2013

STONE ENVIRONMENTAL INC

Chairman, American Red Cross

Instructor's Signature

Carol E Sheldon

Chapter

CNTRL VT/NH VALLEY CHAPTER

Holder's Signature

Lee Rosberg

Cert. 653998 (Rev. Oct. 2001)

American Red Cross

Together, we can save a life

This recognizes that

LEE ROSBERG

has completed the requirements for

FIRST AID

conducted by

CNTRL VT/NH VALLEY CHAPTER

Date completed JUN 16, 2008

The American Red Cross recognizes this certificate as valid for 3 year(s) from completion date.

Chairman, American Red Cross

Instructor's Signature

Carol E Sheldon

Chapter

CNTRL VT/NH VALLEY CHAPTER

Holder's Signature

Lee Rosberg

Cert. 653998 (Rev. Oct. 2001)

American Red Cross

Together, we can save a life

This recognizes that

LEE ROSBERG

has completed the requirements for

ADULT CPR/AED

conducted by

CNTRL VT/NH VALLEY CHAPTER

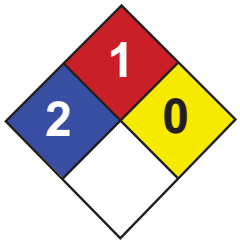
Date completed JUN 16, 2008

The American Red Cross recognizes this certificate as valid for 1 year(s) from completion date.

ATTACHMENT F: MATERIAL SAFETY DATA SHEETS (MSDS)

1. 1,1,1-trichloroethane
2. 1,1-dichloroethene
3. 100 ppm isobutylene
4. Alconox
5. Carbon Tetrachloride
6. Chloroform
7. Cis-1,2-dichloroethene
8. Conductivity Standard Solutions
9. Diesel Fuel
10. Regular Unleaded Gasoline
11. Hydrochloric Acid
12. Methanol
13. AMCO Clear® 1.0 NTU Turbidity Calibration Standard
14. AMCO Clear® 10 NTU Turbidity Calibration Standard
15. AMCO Clear® 100 NTU Turbidity Calibration Standard
16. Buffer Standard pH 7.00
17. Buffer Standard pH 10.00
18. Buffer Standard pH 4.00
19. Tetrachloroethylene
20. Trichloroethylene
21. Helium (compressed)







Material Safety Data Sheet
1,1,1-Trichloroethane MSDS

Section 1: Chemical Product and Company Identification

Product Name: 1,1,1-Trichloroethane

Catalog Codes: SLT4180, SLT2167, SLT3460

CAS#: 71-55-6

RTECS: KJ2975000

TSCA: TSCA 8(b) inventory: 1,1,1-Trichloroethane

CI#: Not available.

Synonym:

Chemical Formula: CH3CCl3

Contact Information:

Sciencelab.com, Inc.
14025 Smith Rd.
Houston, Texas 77396

US Sales: **1-800-901-7247**
International Sales: **1-281-441-4400**

Order Online: ScienceLab.com

CHEMTREC (24HR Emergency Telephone), call:
1-800-424-9300

International CHEMTREC, call: 1-703-527-3887

For non-emergency assistance, call: 1-281-441-4400

Section 2: Composition and Information on Ingredients

Composition:

Name	CAS #	% by Weight
{1,1,1-}Trichloroethane	71-55-6	100

Toxicological Data on Ingredients: 1,1,1-Trichloroethane: ORAL (LD50): Acute: 9600 mg/kg [Rat]. 6000 mg/kg [Mouse]. DERMAL (LD50): Acute: 15800 mg/kg [Rabbit]. VAPOR (LC50): Acute: 18000 ppm 4 hour(s) [Rat].

Section 3: Hazards Identification

Potential Acute Health Effects:
Very hazardous in case of eye contact (irritant), of ingestion. Hazardous in case of skin contact (irritant, permeator), of inhalation. Inflammation of the eye is characterized by redness, watering, and itching.

Potential Chronic Health Effects:
CARCINOGENIC EFFECTS: Not available. MUTAGENIC EFFECTS: Not available. TERATOGENIC EFFECTS: Not available. DEVELOPMENTAL TOXICITY: Not available. The substance is toxic to lungs, the nervous system, liver, mucous membranes. Repeated or prolonged exposure to the substance can produce target organs damage.

Section 4: First Aid Measures

Eye Contact:

Check for and remove any contact lenses. Immediately flush eyes with running water for at least 15 minutes, keeping eyelids open. Cold water may be used. Do not use an eye ointment. Seek medical attention.

Skin Contact:
After contact with skin, wash immediately with plenty of water. Gently and thoroughly wash the contaminated skin with running water and non-abrasive soap. Be particularly careful to clean folds, crevices, creases and groin. Cover the irritated skin with an emollient. If irritation persists, seek medical attention. Wash contaminated clothing before reusing.

Serious Skin Contact:
Wash with a disinfectant soap and cover the contaminated skin with an anti-bacterial cream. Seek medical attention.

Inhalation: Allow the victim to rest in a well ventilated area. Seek immediate medical attention.

Serious Inhalation:
Evacuate the victim to a safe area as soon as possible. Loosen tight clothing such as a collar, tie, belt or waistband. If breathing is difficult, administer oxygen. If the victim is not breathing, perform mouth-to-mouth resuscitation. Seek medical attention.

Ingestion:
Do not induce vomiting. Loosen tight clothing such as a collar, tie, belt or waistband. If the victim is not breathing, perform mouth-to-mouth resuscitation. Seek immediate medical attention.

Serious Ingestion: Not available.

Section 5: Fire and Explosion Data

Flammability of the Product: May be combustible at high temperature.

Auto-Ignition Temperature: 537°C (998.6°F)

Flash Points: Not available.

Flammable Limits: LOWER: 7.5% UPPER: 12.5%

Products of Combustion: These products are carbon oxides (CO, CO2), halogenated compounds.

Fire Hazards in Presence of Various Substances: Slightly flammable to flammable in presence of oxidizing materials, of acids, of alkalis.

Explosion Hazards in Presence of Various Substances:
Risks of explosion of the product in presence of mechanical impact: Not available. Risks of explosion of the product in presence of static discharge: Not available. Slightly explosive to explosive in presence of oxidizing materials, of acids, of alkalis.

Fire Fighting Media and Instructions:
SMALL FIRE: Use DRY chemical powder. LARGE FIRE: Use water spray, fog or foam. Do not use water jet.

Special Remarks on Fire Hazards: Not available.

Special Remarks on Explosion Hazards: Not available.

Section 6: Accidental Release Measures

Small Spill: Absorb with an inert material and put the spilled material in an appropriate waste disposal.

Large Spill:
Absorb with an inert material and put the spilled material in an appropriate waste disposal. Be careful that the product is not present at a concentration level above TLV. Check TLV on the MSDS and with local authorities.

Section 7: Handling and Storage

Precautions:

Keep away from heat. Keep away from sources of ignition. Empty containers pose a fire risk, evaporate the residue under a fume hood. Ground all equipment containing material. Do not ingest. Do not breathe gas/fumes/ vapour/spray. In case of insufficient ventilation, wear suitable respiratory equipment If ingested, seek medical advice immediately and show the container or the label. Avoid contact with skin and eyes

Storage:
Keep container dry. Keep in a cool place. Ground all equipment containing material. Keep container tightly closed. Keep in a cool, well-ventilated place. Combustible materials should be stored away from extreme heat and away from strong oxidizing agents.

Section 8: Exposure Controls/Personal Protection

Engineering Controls:
Provide exhaust ventilation or other engineering controls to keep the airborne concentrations of vapors below their respective threshold limit value. Ensure that eyewash stations and safety showers are proximal to the work-station location.

Personal Protection:
Splash goggles. Lab coat. Vapor respirator. Be sure to use an approved/certified respirator or equivalent. Gloves.

Personal Protection in Case of a Large Spill:
Splash goggles. Full suit. Vapor respirator. Boots. Gloves. A self contained breathing apparatus should be used to avoid inhalation of the product. Suggested protective clothing might not be sufficient; consult a specialist BEFORE handling this product.

Exposure Limits:
TWA: 350 STEL: 440 CEIL: 440 (ppm) from ACGIH (TLV) [1995] TWA: 1900 STEL: 2460 CEIL: 2380 (mg/m3) from ACGIH [1995]Consult local authorities for acceptable exposure limits.

Section 9: Physical and Chemical Properties

Physical state and appearance: Liquid.

Odor: Not available.

Taste: Not available.

Molecular Weight: 133.41 g/mole

Color: Not available.

pH (1% soln/water): Not available.

Boiling Point: 74.1°C (165.4°F)

Melting Point: -32.5°C (-26.5°F)

Critical Temperature: Not available.

Specific Gravity: 1.3376 (Water = 1)

Vapor Pressure: 100 mm of Hg (@ 20°C)

Vapor Density: 4.6 (Air = 1)

Volatility: Not available.

Odor Threshold: 400 ppm

Water/Oil Dist. Coeff.: The product is equally soluble in oil and water; log(oil/water) = 0

Ionicity (in Water): Not available.

Dispersion Properties: Not available.

Solubility: Very slightly soluble in cold water.

Section 10: Stability and Reactivity Data

Stability: The product is stable.

Instability Temperature: Not available.

Conditions of Instability: Not available.

Incompatibility with various substances: Not available.

Corrosivity: Non-corrosive in presence of glass.

Special Remarks on Reactivity: Not available.

Special Remarks on Corrosivity: Not available.

Polymerization: No.

Section 11: Toxicological Information

Routes of Entry: Dermal contact. Eye contact. Inhalation. Ingestion.

Toxicity to Animals:
WARNING: THE LC50 VALUES HEREUNDER ARE ESTIMATED ON THE BASIS OF A 4-HOUR EXPOSURE. Acute oral toxicity (LD50): 6000 mg/kg [Mouse]. Acute dermal toxicity (LD50): 15800 mg/kg [Rabbit]. Acute toxicity of the vapor (LC50): 18000 ppm 4 hour(s) [Rat].

Chronic Effects on Humans: The substance is toxic to lungs, the nervous system, liver, mucous membranes.

Other Toxic Effects on Humans:
Very hazardous in case of ingestion. Hazardous in case of skin contact (irritant, permeator), of inhalation.

Special Remarks on Toxicity to Animals: Not available.

Special Remarks on Chronic Effects on Humans: Detected in maternal milk in human.

Special Remarks on other Toxic Effects on Humans: Not available.

Section 12: Ecological Information

Ecotoxicity: Not available.

BOD5 and COD: Not available.

Products of Biodegradation:
Possibly hazardous short term degradation products are not likely. However, long term degradation products may arise.

Toxicity of the Products of Biodegradation: The products of degradation are more toxic.

Special Remarks on the Products of Biodegradation: Not available.

Section 13: Disposal Considerations

Waste Disposal:

Section 14: Transport Information

DOT Classification: CLASS 6.1: Poisonous material.

Identification: : 1,1,1-Trichloroethane : UN2831 PG: III

Special Provisions for Transport: Not available.

Section 15: Other Regulatory Information

Federal and State Regulations:
Pennsylvania RTK: 1,1,1-Trichloroethane Massachusetts RTK: 1,1,1-Trichloroethane TSCA 8(b) inventory: 1,1,1-Trichloroethane SARA 313 toxic chemical notification and release reporting: 1,1,1-Trichloroethane CERCLA: Hazardous substances.: 1,1,1-Trichloroethane

Other Regulations: OSHA: Hazardous by definition of Hazard Communication Standard (29 CFR 1910.1200).

Other Classifications:

WHMIS (Canada): CLASS D-1B: Material causing immediate and serious toxic effects (TOXIC).

DSCL (EEC):
R38- Irritating to skin. R41- Risk of serious damage to eyes.

HMIS (U.S.A.):

Health Hazard: 2

Fire Hazard: 1

Reactivity: 0

Personal Protection: h

National Fire Protection Association (U.S.A.):

Health: 2

Flammability: 1

Reactivity: 0

Specific hazard:

Protective Equipment:
Gloves. Lab coat. Vapor respirator. Be sure to use an approved/certified respirator or equivalent. Wear appropriate respirator when ventilation is inadequate. Splash goggles.

Section 16: Other Information

References: Not available.

Other Special Considerations: Not available.

Created: 10/10/2005 08:31 PM

Last Updated: 11/01/2010 12:00 PM

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Material Safety Data Sheet

Version 4.3
Revision Date 01/19/2012
Print Date 07/10/2012

1. PRODUCT AND COMPANY IDENTIFICATION

Product name	:	1,1-Dichloroethene
Product Number	:	48526
Brand	:	Supelco
Supplier	:	Sigma-Aldrich 3050 Spruce Street SAINT LOUIS MO 63103 USA
Telephone	:	+1 800-325-5832
Fax	:	+1 800-325-5052
Emergency Phone # (For both supplier and manufacturer)	:	(314) 776-6555
Preparation Information	:	Sigma-Aldrich Corporation Product Safety - Americas Region 1-800-521-8956

2. HAZARDS IDENTIFICATION

Emergency Overview

OSHA Hazards
Flammable liquid, Target Organ Effect, Toxic by ingestion, Irritant, Carcinogen

Target Organs

Liver, Kidney, Central nervous system

GHS Classification
Flammable liquids (Category 1)
Acute toxicity, Oral (Category 3)
Skin irritation (Category 2)
Eye irritation (Category 2A)
Carcinogenicity (Category 2)

GHS Label elements, including precautionary statements

Pictogram



Signal word Danger

Hazard statement(s)

H224	Extremely flammable liquid and vapour.
H301	Toxic if swallowed.
H315	Causes skin irritation.
H319	Causes serious eye irritation.
H351	Suspected of causing cancer.

Precautionary statement(s)

P210	Keep away from heat/sparks/open flames/hot surfaces. - No smoking.
P281	Use personal protective equipment as required.
P301 + P310	IF SWALLOWED: Immediately call a POISON CENTER or doctor/ physician.
P305 + P351 + P338	IF IN EYES: Rinse cautiously with water for several minutes. Remove contact lenses, if present and easy to do. Continue rinsing.

HMIS Classification

Health hazard:	2
Chronic Health Hazard:	*
Flammability:	4
Physical hazards:	2

NFPA Rating

Health hazard:	2
Fire:	4
Reactivity Hazard:	0

Potential Health Effects

Inhalation	May be harmful if inhaled. Causes respiratory tract irritation.
Skin	May be harmful if absorbed through skin. Causes skin irritation.
Eyes	Causes eye irritation.
Ingestion	Toxic if swallowed.

3. COMPOSITION/INFORMATION ON INGREDIENTS

Synonyms : 1,1-Dichloroethylene
Vinylidene chloride

Formula : $C_2H_2Cl_2$

Molecular Weight : 96.94 g/mol

Component		Concentration
Vinylidene chloride		
CAS-No.	75-35-4	-
EC-No.	200-864-0	
Index-No.	602-025-00-8	

4. FIRST AID MEASURES

General advice
Consult a physician. Show this safety data sheet to the doctor in attendance.Move out of dangerous area.

If inhaled
If breathed in, move person into fresh air. If not breathing, give artificial respiration. Consult a physician.

In case of skin contact
Wash off with soap and plenty of water. Take victim immediately to hospital. Consult a physician.

In case of eye contact
Rinse thoroughly with plenty of water for at least 15 minutes and consult a physician.

If swallowed
Do NOT induce vomiting. Never give anything by mouth to an unconscious person. Rinse mouth with water. Consult a physician.

5. FIREFIGHTING MEASURES

Conditions of flammability
Flammable in the presence of a source of ignition when the temperature is above the flash point. Keep away from heat/sparks/open flame/hot surface. No smoking.

Suitable extinguishing media
Use water spray, alcohol-resistant foam, dry chemical or carbon dioxide.

Special protective equipment for firefighters
Wear self contained breathing apparatus for fire fighting if necessary.

Hazardous combustion products
Hazardous decomposition products formed under fire conditions. - Carbon oxides, Hydrogen chloride gas

Further information
Use water spray to cool unopened containers.

6. ACCIDENTAL RELEASE MEASURES

Personal precautions
Wear respiratory protection. Avoid breathing vapors, mist or gas. Ensure adequate ventilation. Remove all sources of ignition. Evacuate personnel to safe areas. Beware of vapours accumulating to form explosive concentrations. Vapours can accumulate in low areas.

Environmental precautions
Prevent further leakage or spillage if safe to do so. Do not let product enter drains.

Methods and materials for containment and cleaning up
Contain spillage, and then collect with an electrically protected vacuum cleaner or by wet-brushing and place in container for disposal according to local regulations (see section 13).

7. HANDLING AND STORAGE

Precautions for safe handling
Avoid contact with skin and eyes. Avoid inhalation of vapour or mist.
Use explosion-proof equipment. Keep away from sources of ignition - No smoking. Take measures to prevent the build up of electrostatic charge.

Conditions for safe storage
Keep container tightly closed in a dry and well-ventilated place. Containers which are opened must be carefully resealed and kept upright to prevent leakage.

Air and moisture sensitive. Store under inert gas.

8. EXPOSURE CONTROLS/PERSONAL PROTECTION

Components with workplace control parameters

Components	CAS-No.	Value	Control parameters	Basis
Remarks	Potential Occupational Carcinogen See Appendix A			
Vinylidene chloride	75-35-4	TWA	5 ppm	USA. ACGIH Threshold Limit Values (TLV)
	Liver & kidney damage Not classifiable as a human carcinogen			
		TWA	1 ppm 4 mg/m3	USA. OSHA - TABLE Z-1 Limits for Air Contaminants - 1910.1000

Personal protective equipment

Respiratory protection
Where risk assessment shows air-purifying respirators are appropriate use a full-face respirator with multi-purpose combination (US) or type AXBEK (EN 14387) respirator cartridges as a backup to engineering controls. If the respirator is the sole means of protection, use a full-face supplied air respirator. Use respirators and components tested and approved under appropriate government standards such as NIOSH (US) or CEN (EU).

Hand protection
Handle with gloves. Gloves must be inspected prior to use. Use proper glove removal technique (without touching glove's outer surface) to avoid skin contact with this product. Dispose of contaminated gloves after use in accordance with applicable laws and good laboratory practices. Wash and dry hands.

Eye protection
Face shield and safety glasses Use equipment for eye protection tested and approved under appropriate government standards such as NIOSH (US) or EN 166(EU).

Skin and body protection

Complete suit protecting against chemicals, Flame retardant antistatic protective clothing, The type of protective equipment must be selected according to the concentration and amount of the dangerous substance at the specific workplace.

Hygiene measures
Avoid contact with skin, eyes and clothing. Wash hands before breaks and immediately after handling the product.

9. PHYSICAL AND CHEMICAL PROPERTIES

Appearance

Form	liquid, clear
Colour	colourless

Safety data

pH	no data available
Melting point/freezing point	Melting point/range: -122 °C (-188 °F) - lit.
Boiling point	30 - 32 °C (86 - 90 °F) - lit.
Flash point	-25.0 °C (-13.0 °F) - closed cup
Ignition temperature	520 °C (968 °F)
Autoignition temperature	520.0 °C (968.0 °F) 580.0 °C (1,076.0 °F)
Lower explosion limit	6.5 %(V)
Upper explosion limit	15.5 %(V)
Vapour pressure	658.6 hPa (494.0 mmHg) 667.3 hPa (500.5 mmHg) at 20.0 °C (68.0 °F) 2,137.4 hPa (1,603.2 mmHg) at 55.0 °C (131.0 °F)
Density	1.213 g/cm3 at 20 °C (68 °F)
Water solubility	0.2 g/l at 20 °C (68 °F)
Partition coefficient: n-octanol/water	no data available
Relative vapour density	no data available
Odour	no data available
Odour Threshold	no data available
Evaporation rate	no data available

10. STABILITY AND REACTIVITY

Chemical stability
Stable under recommended storage conditions.

Possibility of hazardous reactions

Vapours may form explosive mixture with air.

Conditions to avoid
Heat, flames and sparks. Extremes of temperature and direct sunlight.

Materials to avoid
Oxidizing agents, Copper, Aluminum, and its alloys, Peroxides, Strong bases, Oxygen

Hazardous decomposition products
Hazardous decomposition products formed under fire conditions. - Carbon oxides, Hydrogen chloride gas
Other decomposition products - no data available

11. TOXICOLOGICAL INFORMATION

Acute toxicity

Oral LD50
LD50 Oral - rat - 200.0 mg/kg

Inhalation LC50
Lung irritation

Dermal LD50
no data available

Other information on acute toxicity
no data available

Skin corrosion/irritation
no data available

Serious eye damage/eye irritation
no data available

Respiratory or skin sensitization
no data available

Germ cell mutagenicity
Laboratory experiments have shown mutagenic effects.

Carcinogenicity

This product is or contains a component that has been reported to be possibly carcinogenic based on its IARC, ACGIH, NTP, or EPA classification.

Limited evidence of carcinogenicity in animal studies

IARC:	3 - Group 3: Not classifiable as to its carcinogenicity to humans (Vinylidene chloride)
NTP:	No component of this product present at levels greater than or equal to 0.1% is identified as a known or anticipated carcinogen by NTP.
OSHA:	No component of this product present at levels greater than or equal to 0.1% is identified as a carcinogen or potential carcinogen by OSHA.

Reproductive toxicity

no data available

Teratogenicity

no data available

Specific target organ toxicity - single exposure (Globally Harmonized System)

no data available

Specific target organ toxicity - repeated exposure (Globally Harmonized System)
no data available

Aspiration hazard
no data available

Potential health effects

Inhalation	May be harmful if inhaled. Causes respiratory tract irritation.
Ingestion	Toxic if swallowed.
Skin	May be harmful if absorbed through skin. Causes skin irritation.
Eyes	Causes eye irritation.

Signs and Symptoms of Exposure
Nausea, Headache, Vomiting, Dizziness, Drowsiness, Confusion., Incoordination., Central nervous system depression, To the best of our knowledge, the chemical, physical, and toxicological properties have not been thoroughly investigated.

Synergistic effects
no data available

Additional Information
RTECS: KV9275000

12. ECOLOGICAL INFORMATION

Toxicity

Toxicity to fish	LC50 - Daphnia magna (Water flea) - 11.60 - 11.79 mg/l
	LC50 - Pimephales promelas (fathead minnow) - 108.00 - 169.00 mg/l
	LC50 - Lepomis macrochirus (Bluegill) - 74.00 - 220.00 mg/l
	LC50 - Cyprinodon variegatus (sheepshead minnow) - 249.00 mg/l
	LC50 - other fish - 250.00 mg/l
	LC50 - other fish - 224.00 mg/l
	LC50 - Pimephales promelas (fathead minnow) - 108 mg/l - 96 h
	NOEC - Cyprinodon variegatus (sheepshead minnow) - 80 mg/l - 96 h
Toxicity to daphnia and other aquatic invertebrates	LC50 - Daphnia magna (Water flea) - 11.6 mg/l - 48 h

Persistence and degradability
no data available

Bioaccumulative potential
no data available

Mobility in soil
no data available

PBT and vPvB assessment
no data available

Other adverse effects

no data available

13. DISPOSAL CONSIDERATIONS

Product
Burn in a chemical incinerator equipped with an afterburner and scrubber but exert extra care in igniting as this material is highly flammable. Offer surplus and non-recyclable solutions to a licensed disposal company. Contact a licensed professional waste disposal service to dispose of this material.

Contaminated packaging
Dispose of as unused product.

14. TRANSPORT INFORMATION

DOT (US)
UN number: 1303 Class: 3 Packing group: I
Proper shipping name: Vinylidene chloride, stabilized
Reportable Quantity (RQ): 100 lbs
Marine pollutant: No
Poison Inhalation Hazard: No

IMDG
UN number: 1303 Class: 3 Packing group: I EMS-No: F-E, S-D
Proper shipping name: VINYLIDENE CHLORIDE, STABILIZED
Marine pollutant: Marine pollutant

IATA
UN number: 1303 Class: 3 Packing group: I
Proper shipping name: Vinylidene chloride, stabilized

15. REGULATORY INFORMATION

OSHA Hazards
Flammable liquid, Target Organ Effect, Toxic by ingestion, Irritant, Carcinogen

SARA 302 Components
SARA 302: No chemicals in this material are subject to the reporting requirements of SARA Title III, Section 302.

SARA 313 Components
The following components are subject to reporting levels established by SARA Title III, Section 313:

	CAS-No.	Revision Date
Vinylidene chloride	75-35-4	2007-07-01

SARA 311/312 Hazards
Fire Hazard, Acute Health Hazard, Chronic Health Hazard

Massachusetts Right To Know Components

	CAS-No.	Revision Date
Vinylidene chloride	75-35-4	2007-07-01

Pennsylvania Right To Know Components

	CAS-No.	Revision Date
Vinylidene chloride	75-35-4	2007-07-01

New Jersey Right To Know Components

	CAS-No.	Revision Date
Vinylidene chloride	75-35-4	2007-07-01

California Prop. 65 Components
This product does not contain any chemicals known to State of California to cause cancer, birth defects, or any other reproductive harm.

16. OTHER INFORMATION

Further information
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The above information is believed to be correct but does not purport to be all inclusive and shall be used only as a guide. The information in this document is based on the present state of our knowledge and is applicable to the product with regard to appropriate safety precautions. It does not represent any guarantee of the properties of the product. Sigma-Aldrich Corporation and its Affiliates shall not be held liable for any damage resulting from handling or from contact with the above product. See www.sigma-aldrich.com and/or the reverse side of invoice or packing slip for additional terms and conditions of sale.

MATERIAL SAFETY DATA SHEET
29 CFR 1910.1200 OSHA Hazard
Communication Rule Format
Chem-Tel 24 Hour Emergency # 1-800-255-3924

MINE SAFETY APPLIANCES COMPANY
P.O. Box 426
Pittsburgh, PA 15230
PHONE (412) 967-3000

This product contains isobutene, oxygen and nitrogen, substances subject to the Pennsylvania Worker and Community Right-To-Know Act.

PRODUCT IDENTITY

LABEL IDENTITY - MSA P/N 815704 Calibration Check Gas, 100 ppm isobutylene in air

CHEMICAL NAME - Isobutylene, Oxygen, Nitrogen Mixture

ADDITIONAL IDENTITIES - MSA P/N 815704 calibration gas, MSA P/N 815704 Squirt Gas

FORMULA - C_4H_8 in Air

APPLICABLE CHEMICAL CONTENTS

	ppm	TWA
Isobutene (CAS 115-11-7)	100	None
Air	Balance	None

NOTE: Gas under pressure, 155 PSIG at 70°F, Approx. 11 liters gas at atmospheric pressure.

PHYSICAL AND CHEMICAL PROPERTIES

APPEARANCE AND ODOR - Colorless gas, faint odor

BOILING POINT - N/A

SPECIFIC GRAVITY (H_2O = 1) - N/A

VAPOR PRESSURE - N/A

PERCENT VOLATILE BY VOLUME - N/A

VAPOR DENSITY (AIR = 1) - > 1

SOLUBILITY IN WATER - Isobutylene - Insoluble
Oxygen - 3.2 cm^3/100 ml (25°C)
Nitrogen - 2.3 cm^3/100 ml (0°C)

N/A - Not Applicable

PHYSICAL HAZARD INFORMATION

PHYSICAL HAZARD - Compressed Gas, 155 PSIG at 70°F

CONDITIONS OR MATERIALS TO AVOID - None

FLASH POINT - N/A LEL - N/A UEL - N/A

EXTINGUISHING MEDIA - This calibration gas mixture is not flammable.

SPECIAL FIRE FIGHTING PROCEDURES - See next item.

UNUSUAL FIRE AND EXPLOSION HAZARDS - Gas under pressure, 155 PSIG at 70°F. Do not exceed 120°F.

HEALTH HAZARDS

HEALTH HAZARDS - None Known for 100 ppm Isobutylene in Air. Isobutylene Inhalation Rat LC_{50}: 620 $Gm/M^3/4H$. Isobutylene Inhalation Mouse LC_{50}: 415 $Gm/M^3/2H$.

SIGNS AND SYMPTOMS OF EXPOSURE - N/A to this gas mixture.

PRIMARY ROUTES OF ENTRY - Inhalation

TARGET ORGANS - No information. Isobutylene is an asphyxiant and a weak anesthetic.

MEDICAL CONDITIONS GENERALLY RECOGNIZED AS BEING AGGRAVATED BY EXPOSURE - No information

EXPOSURE LIMITS - None (ACGIH 2005)

CARCINOGENICITY DATA - Component gases are not listed by NIOSH RTECS, OSHA, NTP or IARC.

EMERGENCY AND FIRST AID PROCEDURES - None

SAFE HANDLING AND USE

HYGIENIC PRACTICES - Avoid breathing gas

PROTECTIVE MEASURES DURING REPAIR AND MAINTENANCE OF CONTAMINATED EQUIPMENT - N/A

PROCEDURES FOR SPILL OR LEAK CLEANUP - Ventilate area

WASTE DISPOSAL - Do not puncture or incinerate cylinder. Before discarding cylinder, slowly release contents to a safe exhaust. Dispose of cylinder in accordance with local, state and federal regulations.

STORAGE - Store in a cool, dry, well-ventilated area. Do not exceed 120°F.

CONTROL MEASURES

PERSONAL PROTECTIVE EQUIPMENT - Due to the limited amount of gas in the cylinder, and the low release rate employed in instrument calibration, respiratory protection is not indicated under conditions of intended use.

ENGINEERING CONTROLS - None

WORK PRACTICES - Use in well-ventilated areas. Follow the calibration procedure detailed in the MSA instruction manual provided with the instrument under calibration.

DATE OF PREPARATION - Rev. 5, June 2005.

WARNING: This is a hazardous chemical product. By following the directions and warnings provided with this product, the hazards associated with the use of this product can be greatly reduced but never entirely eliminated. Mine Safety Appliances Company makes no warranties, expressed or implied, with respect to this product and EXPRESSLY DISCLAIMS THE WARRANTY OF MERCHANTABILITY AND ANY WARRANTY OF FITNESS FOR A PARTICULAR PURPOSE. Users assume all risks in handling, using or storing this product.

MATERIAL SAFETY DATA SHEET

ALCONOX®

Prepared to U.S. OSHA, CMA, ANSI, Canadian WHMIS, Australian WorkSafe, Japanese Industrial Standard JIS Z 7250:2000, and European Union REACH Regulations

SECTION 1 - PRODUCT AND COMPANY IDENTIFICATION

PRODUCT NAME:	**ALCONOX®**
CHEMICAL FAMILY NAME:	Detergent.
PRODUCT USE:	Critical-cleaning detergent for laboratory, healthcare and industrial applications
U.N. NUMBER:	Not Applicable
U.N. DANGEROUS GOODS CLASS:	Non-Regulated Material
SUPPLIER/MANUFACTURER'S NAME:	Alconox, Inc.
ADDRESS:	30 Glenn St., Suite 309, White Plains, NY 10603. USA
EMERGENCY PHONE:	**TOLL-FREE in USA/Canada** 800-255-3924
	International calls 813-248-0585
BUSINESS PHONE:	914-948-4040
DATE OF PREPARATION:	May 2011
DATE OF LAST REVISION:	February 2008

SECTION 2 - HAZARDS IDENTIFICATION

EMERGENCY OVERVIEW: This product is a white granular powder with little or no odor. Exposure can be irritating to eyes, respiratory system and skin. It is a non-flammable solid. The Environmental effects of this product have not been investigated.

US DOT SYMBOLS

Non-Regulated

CANADA (WHMIS) SYMBOLS



EUROPEAN and (GHS) Hazard Symbols



Signal Word: **Warning!**

EU LABELING AND CLASSIFICATION:

Classification of the substance or mixture according to Regulation (EC) No1272/2008 Annex 1
EC# 205-633-8 This substance is not classified in the Annex I of Directive 67/548/EEC
EC# 268-356-1 This substance is not classified in the Annex I of Directive 67/548/EEC
EC# 231-838-7 This substance is not classified in the Annex I of Directive 67/548/EEC
EC# 231-767-1 This substance is not classified in the Annex I of Directive 67/548/EEC
EC# 207-638-8 Index# 011-005-00-2
EC# 205-788-1 This substance is not classified in the Annex I of Directive 67/548/EEC

GHS Hazard Classification(s):

Eye Irritant Category 2A

Hazard Statement(s):

H319: Causes serious eye irritation

Precautionary Statement(s):

P260: Do not breath dust/fume/gas/mist/vapors/spray
P264: Wash hands thoroughly after handling
P271: Use only in well ventilated area.
P280: Wear protective gloves/protective clothing/eye protection/face protection/

Hazard Symbol(s):

[Xi] Irritant

Risk Phrases:
R20: Harmful by inhalation
R36/37/38: Irritating to eyes, respiratory system and skin

Safety Phrases:
S8: Keep container dry
S22: Do not breath dust
S24/25: Avoid contact with skin and eyes

HEALTH HAZARDS OR RISKS FROM EXPOSURE:

ACUTE: Exposure to this product may cause irritation of the eyes, respiratory system and skin. Ingestion may cause gastrointestinal irritation including pain, vomiting or diarrhea.

CHRONIC: This product contains an ingredient which may be corrosive.

TARGET ORGANS: ACUTE: Eye, respiratory System, Skin CHRONIC: None Known

SECTION 3 - COMPOSITION and INFORMATION ON INGREDIENTS

HAZARDOUS INGREDIENTS:	CAS #	EINECS #	ICSC #	WT %	HAZARD CLASSIFICATION; RISK PHRASES
Sodium Bicarbonate	144-55-8	205-633-8	1044	33 - 43%	HAZARD CLASSIFICATION: None RISK PHRASES: None
Sodium (C10 – C16) Alkylbenzene Sulfonate	68081-81-2	268-356-1	Not Listed	10 – 20%	HAZARD CLASSIFICATION: None RISK PHRASES: None
Sodium Tripolyphosphate	7758-29-4	231-838-7	1469	5 - 15%	HAZARD CLASSIFICATION: None RISK PHRASES: None
Tetrasodium Pyrophosphate	7722-88-5	231-767-1	1140	5 - 15%	HAZARD CLASSIFICATION: None RISK PHRASES: None
Sodium Carbonate	497-19-8	207-638-8	1135	1 - 10%	HAZARD CLASSIFICATION: [Xi] Irritant RISK PHRASES: R36
Sodium Alcohol Sulfate	151-21-3	205-788-1	0502	1 – 5%	HAZARD CLASSIFICATION: None RISK PHRASES: None
Balance of other ingredients are non-hazardous or less than 1% in concentration (or 0.1% for carcinogens, reproductive toxins, or respiratory sensitizers).					

NOTE: ALL WHMIS required information is included in appropriate sections based on the ANSI Z400.1-2004 format. This product has been classified in accordance with the hazard criteria of the CPR and the MSDS contains all the information required by the CPR, EU Directives and the Japanese Industrial Standard *JIS Z 7250: 2000*.

SECTION 4 - FIRST-AID MEASURES

Contaminated individuals of chemical exposure must be taken for medical attention if any adverse effect occurs. Rescuers should be taken for medical attention, if necessary. Take copy of label and MSDS to health professional with contaminated individual.

EYE CONTACT: If product enters the eyes, open eyes while under gentle running water for at least 15 minutes. Seek medical attention if irritation persists.

SKIN CONTACT: Wash skin thoroughly after handling. Seek medical attention if irritation develops and persists. Remove contaminated clothing. Launder before re-use.

INHALATION: If breathing becomes difficult, remove victim to fresh air. If necessary, use artificial respiration to support vital functions. Seek medical attention if breathing dificulty continues.

INGESTION: If product is swallowed, call physician or poison control center for most current information. If professional advice is not available, do not induce vomiting. Never induce vomiting or give diluents (milk or water) to someone who is unconscious, having convulsions, or who cannot swallow. Seek medical advice. Take a copy of the label and/or MSDS with the victim to the health professional.

MEDICAL CONDITIONS AGGRAVATED BY EXPOSURE: Pre-existing skin, or eye problems may be aggravated by prolonged contact.

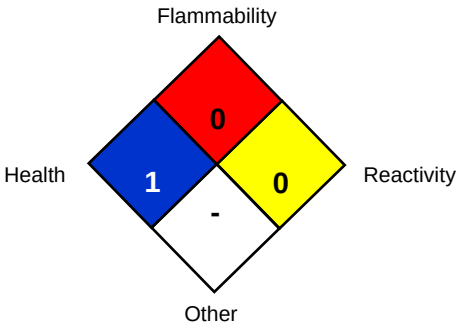
RECOMMENDATIONS TO PHYSICIANS: Treat symptoms and reduce over-exposure.

SECTION 5 - FIRE-FIGHTING MEASURES

FLASH POINT: Not Flammable
AUTOIGNITION TEMPERATURE: Not Applicable
FLAMMABLE LIMITS (in air by volume, %): Lower (LEL): NA Upper (UEL): NA
FIRE EXTINGUISHING MATERIALS: As appropriate for surrounding fire. Carbon dioxide, foam, dry chemical, halon, or water spray.

UNUSUAL FIRE AND EXPLOSION HAZARDS: This product is non-flammable and has no known explosion hazards.
Explosion Sensitivity to Mechanical Impact: Not Sensitive.
Explosion Sensitivity to Static Discharge: Not Sensitive
SPECIAL FIRE-FIGHTING PROCEDURES: Incipient fire responders should wear eye protection. Structural firefighters must wear Self-Contained Breathing Apparatus and full protective equipment. Isolate materials not yet involved in the fire and protect personnel. Move containers from fire area if this can be done without risk; otherwise, cool with carefully applied water spray. If possible, prevent runoff water from entering storm drains, bodies of water, or other environmentally sensitive areas.

NFPA RATING SYSTEM



HMIS RATING SYSTEM

HAZARDOUS MATERIAL IDENTIFICATION SYSTEM

HEALTH HAZARD (BLUE)	1
FLAMMABILITY HAZARD (RED)	0
PHYSICAL HAZARD (YELLOW)	0

PROTECTIVE EQUIPMENT			
EYES	RESPIRATORY	HANDS	BODY
	See Sect 8		See Sect 8

For Routine Industrial Use and Handling Applications

Hazard Scale: **0** = Minimal **1** = Slight **2** = Moderate **3** = Serious **4** = Severe * = Chronic hazard

SECTION 6 - ACCIDENTAL RELEASE MEASURES

SPILL AND LEAK RESPONSE: Personnel should be trained for spill response operations.
SPILLS: Contain spill if safe to do so. Prevent entry into drains, sewers, and other waterways. Sweep, shovel or vacuum spilled material and place in an appropriate container for re-use or disposal. Avoid dust generation if possible. Dispose of in accordance with applicable Federal, State, and local procedures (see Section 13, Disposal Considerations).

SECTION 7 - HANDLING and STORAGE

WORK PRACTICES AND HYGIENE PRACTICES: As with all chemicals, avoid getting this product ON YOU or IN YOU. Wash thoroughly after handling this product. Do not eat, drink, smoke, or apply cosmetics while handling this product. Avoid breathing dusts generated by this product. Use in a well-ventilated location. Remove contaminated clothing immediately.

STORAGE AND HANDLING PRACTICES: Containers of this product must be properly labeled. Store containers in a cool, dry location. Keep container tightly closed when not in use. Store away from strong acids or oxidizers.

SECTION 8 - EXPOSURE CONTROLS - PERSONAL PROTECTION

EXPOSURE LIMITS/GUIDELINES:

Chemical Name	CAS#	ACGIH TWA	OSHA TWA	SWA
Sodium Bicarbonate	144-55-8	10 mg/m³ Total Dust	15 mg/m³ Total Dust	10 mg/m³ Total Dust
Sodium (C10 – C16) Alkylbenzene Sulfonate	68081-81-2	10 mg/m³ Total Dust	15 mg/m³ Total Dust	10 mg/m³ Total Dust
Sodium Tripolyphosphate	7758-29-4	10 mg/m³ Total Dust	15 mg/m³ Total Dust	10 mg/m³ Total Dust
Tetrasodium Pyrophosphate	7722-88-5	5 mg/m³	5 mg/m³	5 mg/m³
Sodium Carbonate	497-19-8	10 mg/m³ Total Dust	15 mg/m³ Total Dust	10 mg/m³ Total Dust
Sodium Alcohol Sulfate	151-21-3	10 mg/m³ Total Dust	15 mg/m³ Total Dust	10 mg/m³ Total Dust

Currently, International exposure limits are not established for the components of this product. Please check with competent authority in each country for the most recent limits in place.

VENTILATION AND ENGINEERING CONTROLS: Use with adequate ventilation to ensure exposure levels are maintained below the limits provided below. Use local exhaust ventilation to control airborne dust. Ensure eyewash/safety shower stations are available near areas where this product is used.

The following information on appropriate Personal Protective Equipment is provided to assist employers in complying with OSHA regulations found in 29 CFR Subpart I (beginning at 1910.132) or equivalent standard of Canada, or standards of EU member states (including EN 149 for respiratory PPE, and EN 166 for face/eye protection), and those of Japan. Please reference applicable regulations and standards for relevant details.

RESPIRATORY PROTECTION: Based on test data, exposure limits should not be exceeded under normal use conditions when using Alconox Detergent. Maintain airborne contaminant concentrations below guidelines listed above, if applicable. If necessary, use only respiratory protection authorized in the U.S. Federal OSHA Respiratory Protection Standard (29 CFR 1910.134), equivalent U.S. State standards, Canadian CSA Standard Z94.4-93, the European Standard EN149, or EU member states.

EYE PROTECTION: Safety glasses. If necessary, refer to U.S. OSHA 29 CFR 1910.133 or appropriate Canadian Standards.

HAND PROTECTION: Use chemical resistant gloves to prevent skin contact.. If necessary, refer to U.S. OSHA 29 CFR 1910.138 or appropriate Standards of Canada.

BODY PROTECTION: Use body protection appropriate to prevent contact (e.g. lab coat, overalls). If necessary, refer to appropriate Standards of Canada, or appropriate Standards of the EU, Australian Standards, or relevant Japanese Standards.

SECTION 9 - PHYSICAL and CHEMICAL PROPERTIES

PHYSICAL STATE:	Solid
APPEARANCE & ODOR:	White granular powder with little or no odor.
ODOR THRESHOLD (PPM):	Not Available
VAPOR PRESSURE (mmHg):	Not Applicable
VAPOR DENSITY (AIR=1):	Not Applicable.
BY WEIGHT:	Not Available
EVAPORATION RATE (nBuAc = 1):	Not Applicable.
BOILING POINT (C°):	Not Applicable.
FREEZING POINT (C°):	Not Applicable.
pH:	9.5 (1% aqueous solution)
SPECIFIC GRAVITY 20°C: (WATER =1)	0.85 – 1.1
SOLUBILITY IN WATER (%)	>10% w/w
COEFFICIENT OF WATER/OIL DIST.:	Not Available
VOC:	None
CHEMICAL FAMILY:	Detergent

SECTION 10 - STABILITY and REACTIVITY

STABILITY: Product is stable
DECOMPOSITION PRODUCTS: When heated to decomposition this product produces Oxides of carbon (COx)
MATERIALS WITH WHICH SUBSTANCE IS INCOMPATIBLE: Strong acids and strong oxidizing agents.
HAZARDOUS POLYMERIZATION: Will not occur.
CONDITIONS TO AVOID: Contact with incompatible materials and dust generation.

SECTION 11 - TOXICOLOGICAL INFORMATION

TOXICITY DATA: Toxicity data is available for mixture:

CAS# 497-19-8 LD50 Oral (Rat)	4090 mg/kg
CAS# 497-19-8 LD50 Oral (Mouse)	6600 mg/kg
CAS# 497-19-8 LC50 Inhalation (Rat)	2300 mg/m³ 2H
CAS# 497-19-8 LC50 Inhalation (Mouse)	1200 mg/m³ 2H
CAS# 7758-29-4 LD50 Oral (Rat)	3120 mg/kg
CAS# 7758-29-4 LD50 Oral (Mouse)	3100 mg/kg
CAS# 7722-88-5 LD50 Oral (Rat)	4000 mg/kg

SUSPECTED CANCER AGENT: None of the ingredients are found on the following lists: FEDERAL OSHA Z LIST, NTP, CAL/OSHA, IARC and therefore is not considered to be, nor suspected to be a cancer-causing agent by these agencies.
IRRITANCY OF PRODUCT: Contact with this product can be irritating to exposed skin, eyes and respiratory system.
SENSITIZATION OF PRODUCT: This product is not considered a sensitizer.
REPRODUCTIVE TOXICITY INFORMATION: No information concerning the effects of this product and its components on the human reproductive system.

SECTION 12 - ECOLOGICAL INFORMATION

ALL WORK PRACTICES MUST BE AIMED AT ELIMINATING ENVIRONMENTAL CONTAMINATION.
ENVIRONMENTAL STABILITY: No Data available at this time.
EFFECT OF MATERIAL ON PLANTS or ANIMALS: No evidence is currently available on this product's effects on plants or animals.

EFFECT OF CHEMICAL ON AQUATIC LIFE: No evidence is currently available on this product's effects on aquatic life.

SECTION 13 - DISPOSAL CONSIDERATIONS

PREPARING WASTES FOR DISPOSAL: Waste disposal must be in accordance with appropriate Federal, State, and local regulations, those of Canada, Australia, EU Member States and Japan.

SECTION 14 - TRANSPORTATION INFORMATION

US DOT; IATA; IMO; ADR:

THIS PRODUCT IS NOT HAZARDOUS AS DEFINED BY 49 CFR 172.101 BY THE U.S. DEPARTMENT OF TRANSPORTATION.
PROPER SHIPPING NAME: Non-Regulated Material
HAZARD CLASS NUMBER and DESCRIPTION: Not Applicable
UN IDENTIFICATION NUMBER: Not Applicable
PACKING GROUP: Not Applicable.
DOT LABEL(S) REQUIRED: Not Applicable
NORTH AMERICAN EMERGENCY RESPONSE GUIDEBOOK NUMBER (2004): Not Applicable
MARINE POLLUTANT: None of the ingredients are classified by the DOT as a Marine Pollutant (as defined by 49 CFR 172.101, Appendix B)

U.S. DEPARTMENT OF TRANSPORTATION (DOT) SHIPPING REGULATIONS:
This product is not classified as dangerous goods, per U.S. DOT regulations, under 49 CFR 172.101.

TRANSPORT CANADA, TRANSPORTATION OF DANGEROUS GOODS REGULATIONS:
This product is not classified as Dangerous Goods, per regulations of Transport Canada.

INTERNATIONAL AIR TRANSPORT ASSOCIATION (IATA):
This product is not classified as Dangerous Goods, by rules of IATA:

INTERNATIONAL MARITIME ORGANIZATION (IMO) DESIGNATION:
This product is not classified as Dangerous Goods by the International Maritime Organization.

EUROPEAN AGREEMENT CONCERNING THE INTERNATIONAL CARRIAGE OF DANGEROUS GOODS BY ROAD (ADR):

This product is not classified by the United Nations Economic Commission for Europe to be dangerous goods.

SECTION 15 - REGULATORY INFORMATION

UNITED STATES REGULATIONS

SARA REPORTING REQUIREMENTS: This product is not subject to the reporting requirements of Sections 302, 304 and 313 of Title III of the Superfund Amendments and Reauthorization Act., as follows: None

TSCA: All components in this product are listed on the US Toxic Substances Control Act (TSCA) inventory of chemicals.

SARA 311/312:

Acute Health: Yes Chronic Health: No Fire: No Reactivity: No

U.S. SARA THRESHOLD PLANNING QUANTITY: There are no specific Threshold Planning Quantities for this product. The default Federal MSDS submission and inventory requirement filing threshold of 10,000 lb (4,540 kg) may apply, per 40 CFR 370.20.

U.S. CERCLA REPORTABLE QUANTITY (RQ): None

CALIFORNIA SAFE DRINKING WATER AND TOXIC ENFORCEMENT ACT (PROPOSITION 65): None of the ingredients are on the California Proposition 65 lists.

CANADIAN REGULATIONS:

CANADIAN DSL/NDSL INVENTORY STATUS: All of the components of this product are on the DSL Inventory

CANADIAN ENVIRONMENTAL PROTECTION ACT (CEPA) PRIORITIES SUBSTANCES LISTS: No component of this product is on the CEPA First Priorities Substance Lists.

CANADIAN WHMIS CLASSIFICATION and SYMBOLS: This product is categorized as a Controlled Product, Hazard Class D2B as per the Controlled Product Regulations

EUROPEAN ECONOMIC COMMUNITY INFORMATION:

EU LABELING AND CLASSIFICATION:

Classification of the mixture according to Regulation (EC) No1272/2008. See section 2 for details.

AUSTRALIAN INFORMATION FOR PRODUCT:

AUSTRALIAN INVENTORY OF CHEMICAL SUBSTANCES (AICS) STATUS: All components of this product are listed on the AICS.

STANDARD FOR THE UNIFORM SCHEDULING OF DRUGS AND POISONS: Not applicable.

JAPANESE INFORMATION FOR PRODUCT:

JAPANESE MINISTER OF INTERNATIONAL TRADE AND INDUSTRY (MITI) STATUS: The components of this product are not listed as Class I Specified Chemical Substances, Class II Specified Chemical Substances, or Designated Chemical Substances by the Japanese MITI.

INTERNATIONAL CHEMICAL INVENTORIES:

Listing of the components on individual country Chemical Inventories is as follows:

Asia-Pac:	Listed
Australian Inventory of Chemical Substances (AICS):	Listed
Korean Existing Chemicals List (ECL):	Listed
Japanese Existing National Inventory of Chemical Substances (ENCS):	Listed
Philippines Inventory if Chemicals and Chemical Substances (PICCS):	Listed
Swiss Giftliste List of Toxic Substances:	Listed
U.S. TSCA:	Listed

SECTION 16 - OTHER INFORMATION

PREPARED BY: Paul Eigbrett Global Safety Management, 10006 Cross Creek Blvd. Suite 440, Tampa, FL 33647

Disclaimer: To the best of Alconox, Inc. knowledge, the information contained herein is reliable and accurate as of this date; however, accuracy, suitability or completeness is not guaranteed and no warranties of any type either express or implied are provided. The information contained herein relates only to this specific product.

ANNEX:

IDENTIFIED USES OF ALCONOX® AND DIRECTIONS FOR USE

Used to clean: Healthcare instruments, laboratory ware, vacuum equipment, tissue culture ware, personal protective equipment, sampling apparatus, catheters, tubing, pipes, radioactive contaminated articles, optical parts, electronic components, pharmaceutical apparatus, cosmetics manufacturing equipment, metal castings, forgings and stampings, industrial parts, tanks and reactors. Authorized by USDA for use in federally inspected meat and poultry plants. Passes inhibitory residue test for water analysis. FDA certified.

Used to remove: Soil, grit, grime, buffing compound, slime, grease, oils, blood, tissue, salts, deposits, particulates, solvents, chemicals, radioisotopes, radioactive contaminations, silicon oils, mold release agents.

Surfaces cleaned: Corrosion inhibited formulation recommended for glass, metal, stainless steel, porcelain, ceramic, plastic, rubber and fiberglass. Can be used on soft metals such as copper, aluminum, zinc and magnesium if rinsed promptly. Corrosion testing may be advisable.

Cleaning method: Soak, brush, sponge, cloth, ultrasonic, flow through clean-inplace. Will foam—not for spray or machine use.

Directions: Make a fresh 1% solution (2 1/2 Tbsp. per gal., 1 1/4 oz. per gal. or 10 grams per liter) in cold, warm, or hot water. If available use warm water. Use cold water for blood stains. For difficult soils, raise water temperature and use more detergent. Clean by soak, circulate, wipe, or ultrasonic method. Not for spray machines, will foam. For nonabrasive scouring, make paste. Use 2% solution to soak frozen stopcocks. To remove silver tarnish, soak in 1% solution in aluminum container. RINSE THOROUGHLY—preferably with running water. For critical cleaning, do final or all rinsing in distilled, deionized, or purified water. For food contact surfaces, rinse with potable water. Used on a wide range of glass, ceramic, plastic, and metal surfaces. Corrosion testing may be advisable.

Material Safety Data Sheet

1. Product and Company Identification

Product name : **Carbon Tetrachloride**

Chemical formula : CCl4

Synonyms : Tetrachloromethane; Perchloromethane; Carbon Chloride (CCl4); Benzinoform; R 10 (Refrigerant); UN 1846

Company : Specialty Gases of America, Inc
6055 Brent Dr.
Toledo, OH 43611

Telephone : 419-729-7732

Emergency : 800-424-9300

2. Composition/Information on Ingredients

Components	CAS Number	% Volume
Carbon Tetrachloride	56-23-5	100%

3. Hazards Identification

Emergency Overview

May cause central nervous system depression, suspect cancer hazard (in animals)

Potential Health Effects

Inhalation : Irritation, digestive disorders, headache, drowsiness, dizziness, loss of coordination, lung congestion, effects on the brain, convulsions, coma. May cause irritation, digestive disorders, headache, drowsiness, dizziness, loss of coordination, visual disturbances, lung congestion, kidney damage, liver damage, reproductive effects, effects on the brain, convulsions, coma, cancer in long term exposure.

Eye contact : Irritation.

Skin contact : Irritation, rash, absorption may occur, digestive disorders, headache, drowsiness, dizziness, loss of coordination, lung congestion, effects on the brain, convulsions, coma. May cause visual disturbances, kidney damage, liver damage, reproductive effects, cancer in long term exposure.

Ingestion : Irritation, digestive disorders, headache, drowsiness, dizziness, loss of coordination, lung congestion, effects on the brain, convulsions, coma. May cause kidney damage, liver damage, cancer in long term exposure.

Chronic Health Hazard : None.

4. First Aid Measures

General advice : None.

Eye contact : Immediately flush eyes with plenty of water for at least 15 minutes. Then get immediate medical attention.

Skin contact	:	Wash skin with soap and water for at least 15 minutes while removing contaminated clothing and shoes. Get immediate medical attention. Thoroughly clean and dry contaminated clothing and shoes before reuse.
Ingestion	:	If swallowed, drink plenty of water, do NOT induce vomiting. Get immediate medical attention. Induce vomiting only at the instructions of a physician. Do not give anything by mouth to unconscious or convulsive person.
Inhalation	:	If adverse effects occur, remove to uncontaminated area. Give artificial respiration if not breathing. If breathing is difficult, oxygen should be administered by qualified personnel. Get immediate medical attention.
Note to physicians	:	For ingestion, consider gastric lavage.

5. Fire-Fighting Measures

Suitable extinguishing media	:	Regular dry chemical, regular foam, water. Large fires: Use regular foam or flood with fine water spray.
Specific hazards	:	Slight fire hazard.
Fire fighting	:	Move container from fire area if it can be done without risk. Fight large fires from a protected location or safe distance. Stay away from the ends of tanks. Dike for later disposal. Do not scatter spilled material with high-pressure water streams. Do not attempt to extinguish fire unless flow of material can be stopped first. Use extinguishing agents appropriate for surrounding fire. Flood with fine water spray. Cool containers with water spray until well after the fire is out. Apply water from a protected location or from a safe distance. Avoid inhalation of material or combustion by-products. Stay upwind and keep out of low areas. Consider downward evacuation if material is leaking.

6. Accidental Release Measures

Air release	:	Reduce vapors with water spray. Stay upwind and keep out of low areas.
Soil release	:	Trap spilled material at bottom in deep water pockets, excavated holding areas or within sand bag barriers. Dike for later disposal. Absorb with sand or other non-combustible material. Collect with absorbent into suitable container.
Water release	:	Subject to California Safe Drinking Water and Toxic Enforcement Act of 1986 (Proposition 65). Keep out of water supplies and sewers. Trap spilled material at bottom in deep water pockets, excavated holding areas or within sand bag barriers. Remove trapped material with suction hoses. Absorb with activated carbon. Collect spilled material using mechanical equipment.
Occupational spill/release	:	Do not touch spilled material. Stop leak if possible without personal risk. Reduce vapors with water spray. Small spills: Absorb with sand or other non-combustible material. Collect spilled material in appropriate container for disposal. Small dry spills: Move containers away from spill to a safe area. Large spills: Dike for later disposal. Keep unnecessary people away. Isolate hazard area and deny entry. Ventilate closed spaces before entering. Notify Local Emergency Planning Committee and State Emergency Response Commission for release greater than or equal to RQ (U.S. SARA Section 304). If release occurs in the U.S. and is reportable under CERCLA Section 103, notify the National Response Center at (800)424-8802 (USA) or (202)426-2675 (USA).
Additional advice	:	None.

7. Handling and Storage

Handling

Subject to handling regulations: U.S. OSHA 29 CFR 1910.119.

Storage

Store in accordance with all current regulations and standards. Protect from physical damage. Store in a cool, dry place. Store in a well-ventilated area. Avoid heat, flames, sparks and other sources of ignition. Keep separated from incompatible substances.

8. Exposure Controls / Personal Protection

Exposure limits

ACGIH	:	5 ppm TWA 10 ppm STEL Skin – potential significant contribution to overall exposure by the cutaneous route
OSHA (final)	:	10 ppm TWA 25 ppm Ceiling
OSHA (vacated)	:	2 ppm TWA; 12.6 mg/m3 TWA
NIOSH	:	2 ppm STEL 60 min; 12.6 mg/m3 STEL 60 min

IDLH

200 ppm

Engineering measures/Ventilation

Provide local exhaust or process enclosure ventilation system. Ensure compliance with applicable exposure limits.

Personal protective equipment

Respiratory protection	:	The following respirators and maximum use concentrations are drawn from NIOSH and/or OSHA. At any detectable concentration – Any self-contained breathing apparatus that has a full facepiece and is operated in a pressure-demand or other positive-pressure mode. Any supplied-air respirator with a full facepiece that is operated in a pressure-demand or other positive-pressure mode in combination with an auxiliary self-contained breathing apparatus operated in pressure-demand or other positive-pressure mode. Escape – Any air-purifying full-face respirator (gas mask) with a chin-style, front-mounted or back-mounted canister providing protection against the compound of concern. Any appropriate escape-type, self-contained breathing apparatus. For unknown concentrations or immediately dangerous to life or death – Any supplied-air respirator with a full facepiece that is operated in a pressure-demand or other positive-pressure mode in combination with an auxiliary self-contained breathing apparatus operated in pressure-demand or other positive-pressure mode. Any self-contained breathing apparatus that has a full facepiece and is operated in a pressure-demand or other positive-pressure mode.
Hand protection	:	Wear appropriate chemical resistant gloves.
Eye protection	:	Wear splash resistant safety goggles. Provide an emergency eye wash fountain and quick drench shower in the immediate work area.
Skin and body protection	:	Wear appropriate chemical resistant clothing.

9. Physical and Chemical Properties

Form	:	Liquid.
Color	:	Colorless.
Odor	:	Distinct odor.
Molecular weight	:	153.82

Vapor pressure	:	91.3 mmHg @ 20°C
Vapor density	:	5.32 (air = 1)
Boiling point	:	77°C
Melting point	:	-23°C
Water solubility	:	0.08% @ 20°C
Specific gravity	:	1.5940 (water = 1)
Evaporation rate	:	12.8 (Butyl acetate = 1)
Solvent solubility	:	Soluble: alcohol, benzene, chloroform, ether, carbon disulfide, petroleum ether, naphtha, acetone, fixed & volatile oils

10. Stability and Reactivity

Stability	:	Stable under normal conditions.
Conditions to avoid	:	Avoid heat, flames, sparks or other sources of ignition. Containers may rupture or explode if exposed to heat.
Materials to avoid	:	Combustible materials, material salts, peroxides, halogens, oxidizing materials, metals, bases, amines.
Hazardous decomposition products	:	Thermal decomposition products: phosgene, halogenated compounds, oxides of carbon.

11. Toxicological Information

The components of this material have been reviewed in various sources and the following endpoints are published:

CARBON TETRACHLORIDE (56-23-5)	:	Inhalation LC50 Rat: 8000 ppm/4H; Oral LD50 Rat: 2350 mg/kg; Dermal LD50 Rat: 5070 mg/kg

Acute Toxicity Level

CARBON TETRACHLORIDE (56-23-5)	:	Moderately toxic: Ingestion Slightly toxic: Inhalation, dermal absorption

Component Carcinogenicity

ACGIH	:	A2 – Suspected Human Carcinogen
IARC	:	Monograph 71 [1999]; Supplement 7 [1987]; Monograph 20 [1979] (Group 2B (possibly carcinogenic to humans))
DFG	:	Category 4 (no significant contribution to human cancer) Present Reasonably Anticipated To Be A Human Carcinogen

Target Organs

CARBON TETRACHLORIDE (56-23-5)	:	Central nervous system, liver, kidneys.

Medical Conditions Aggravated by Exposure

History of alcoholism, alcoholism.

Additional Data

May cross the placenta. May be excreted in breast milk. Alcohol may enhance the toxic effects. Stimulants such as epinephrine may induce ventricular fibrillation.

12. Ecological Information

Aquatic Toxicity

CARBON TETRACHLORIDE (56-23-5)	:	Fish: 96 Hr LC50 Pimephales promelas: 36.3 – 47.3 mg/L [flow-through]; 96 Hr LC50 Pimephales promelas: 9.68 – 11.3 mg/L [static]; 96 Hr LC50 Lepomis macrochirus: 23 – 33 mg/L [static] Algae: 24 Hr EC50 Tetrahymena pyriformis: 830 mg/L Invertebrate: 24 Hr EC50 Daphnia magna: 28 mg/L; 48 Hr EC50 Daphnia magna: 29 mg/L

13. Disposal Considerations

Waste from residues / unused products	:	Dispose in accordance with all applicable regulations. Subject to disposal regulations: U.S. EPA 40 CFR 262. Hazardous Waste Number(s): U211. Hazardous Waste Number(s): D019. Dispose in accordance with U.S. EPA 40 CFR 262 for concentrations at or above the Regulatory level. Regulatory level – 0.5 mg/L.
Contaminated packaging	:	Return cylinder to supplier.
Component Waste Numbers	:	RCRA: waste_number U211 0.5 mg/L regulatory level

14. Transport Information

DOT (US only)

Proper shipping name	:	Carbon tetrachloride
Class	:	6.1, Packing Group II
UN/ID No.	:	UN1846
Labeling	:	Poison

Component Marine Pollutants

This material contains one of more of the following chemicals required by U.S. DOT to be identified as marine pollutants.

Component	CAS	
CARBON TETRACHLORIDE	56-23-5	DOT regulated marine pollutant

15. Regulatory Information

U.S. Federal Regulations

This material contains one or more of the following chemicals required under SARA Section 302/304 (40 CFR 355 Appendix A), SARA Section 313 (40 CFR 372.65), CERCLA (40 CFR 302.4), TSCA 12(b), and/or require an OSHA process safety plan.

CARBON TETRACHLORIDE (56-23-5)	:	10 lb final RQ; 4.54 kg final RQ SARA 313: 0.1% de minimis concentration CERCLA: 10 lb final RQ; 4.54 kg final RQ

SARA 311/312

Acute: Yes
Chronic: Yes
Fire: No
Reactive: No
Pressure: No

U.S. State Regulations

The following components appear on one or more of the following state hazardous substances lists:

Component	CAS	CA	MA	MN	NJ	PA	RI
CARBON TETRACHLORIDE	56-23-5	Yes	Yes	Yes	Yes	Yes	Yes

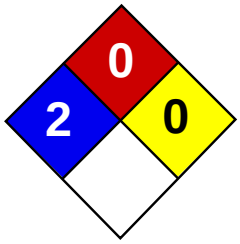
The following statement(s) are provided under the California Safe Drinking Water and Toxic Enforcement Act of 1986 (Proposition 65):

WARNING! This product contains a chemical known to the state of California to cause cancer.

16. Other Information

Prepared by : Specialty Gases of America, Inc.
For additional information, please visit our website at www.americangasgroup.com.







Material Safety Data Sheet
Chloroform MSDS

Section 1: Chemical Product and Company Identification

Product Name: Chloroform

Catalog Codes: SLC1888, SLC5044

CAS#: 67-66-3

RTECS: FS9100000

TSCA: TSCA 8(b) inventory: Chloroform

CI#: Not available.

Synonym: Trichloromethane; Methane, trichlor-

Chemical Name: Chloroform

Chemical Formula: CHCl3

Contact Information:

Sciencelab.com, Inc.
14025 Smith Rd.
Houston, Texas 77396

US Sales: **1-800-901-7247**
International Sales: **1-281-441-4400**

Order Online: ScienceLab.com

CHEMTREC (24HR Emergency Telephone), call:
1-800-424-9300

International CHEMTREC, call: 1-703-527-3887

For non-emergency assistance, call: 1-281-441-4400

Section 2: Composition and Information on Ingredients

Composition:

Name	CAS #	% by Weight
Chloroform	67-66-3	100

Toxicological Data on Ingredients: Chloroform: ORAL (LD50): Acute: 695 mg/kg [Rat]. 36 mg/kg [Mouse]. 820 mg/kg [Guinea pig]. DERMAL (LD50): Acute: >20000 mg/kg [Rabbit]. VAPOR (LC50): Acute: 47702 mg/m 4 hours [Rat].

Section 3: Hazards Identification

Potential Acute Health Effects: Hazardous in case of skin contact (irritant), of eye contact (irritant), of ingestion, of inhalation. Slightly hazardous in case of skin contact (permeator).

Potential Chronic Health Effects: CARCINOGENIC EFFECTS: Classified + (Proven.) by NIOSH. Classified A3 (Proven for animal.) by ACGIH, 2B (Possible for human.) by IARC. Classified 2 (Some evidence.) by NTP. MUTAGENIC EFFECTS: Mutagenic for mammalian somatic cells. Mutagenic for bacteria and/or yeast. TERATOGENIC EFFECTS: Not available. DEVELOPMENTAL TOXICITY: Not available. The substance may be toxic to kidneys, liver, heart. Repeated or prolonged exposure to the substance can produce target organs damage.

Section 4: First Aid Measures

Eye Contact: Check for and remove any contact lenses. In case of contact, immediately flush eyes with plenty of water for at least 15 minutes. Cold water may be used. WARM water MUST be used. Get medical attention.

Skin Contact: In case of contact, immediately flush skin with plenty of water. Cover the irritated skin with an emollient. Remove contaminated clothing and shoes. Wash clothing before reuse. Thoroughly clean shoes before reuse. Get medical attention.

Serious Skin Contact: Wash with a disinfectant soap and cover the contaminated skin with an anti-bacterial cream. Seek immediate medical attention.

Inhalation: If inhaled, remove to fresh air. If not breathing, give artificial respiration. If breathing is difficult, give oxygen. Get medical attention.

Serious Inhalation: Evacuate the victim to a safe area as soon as possible. Loosen tight clothing such as a collar, tie, belt or waistband. If breathing is difficult, administer oxygen. If the victim is not breathing, perform mouth-to-mouth resuscitation. WARNING: It may be hazardous to the person providing aid to give mouth-to-mouth resuscitation when the inhaled material is toxic, infectious or corrosive. Seek medical attention.

Ingestion: Do NOT induce vomiting unless directed to do so by medical personnel. Never give anything by mouth to an unconscious person. If large quantities of this material are swallowed, call a physician immediately. Loosen tight clothing such as a collar, tie, belt or waistband.

Serious Ingestion: Not available.

Section 5: Fire and Explosion Data

Flammability of the Product: Non-flammable.

Auto-Ignition Temperature: Not applicable.

Flash Points: Not applicable.

Flammable Limits: Not applicable.

Products of Combustion: Not available.

Fire Hazards in Presence of Various Substances: Not applicable.

Explosion Hazards in Presence of Various Substances: Risks of explosion of the product in presence of mechanical impact: Not available. Risks of explosion of the product in presence of static discharge: Not available.

Fire Fighting Media and Instructions: Not applicable.

Special Remarks on Fire Hazards: Not available.

Special Remarks on Explosion Hazards: May explode if it comes in contact with aluminum powder, lithium, perchlorate, pentoxide, bis(dimethylamino)dimethylstannane, potassium, potassium-sodium alloy, sodium (or sodium hydroxide or sodium methoxide), and methanol

Section 6: Accidental Release Measures

Small Spill: Absorb with an inert material and put the spilled material in an appropriate waste disposal.

Large Spill: Absorb with an inert material and put the spilled material in an appropriate waste disposal. Be careful that the product is not present at a concentration level above TLV. Check TLV on the MSDS and with local authorities.

Section 7: Handling and Storage

Precautions: Do not ingest. Do not breathe gas/fumes/ vapor/spray. Wear suitable protective clothing. In case of insufficient ventilation, wear suitable respiratory equipment. If ingested, seek medical advice immediately and show the container or the label. Avoid contact with skin and eyes. Keep away from incompatibles such as metals, alkalis.

Storage: Keep container tightly closed. Keep container in a cool, well-ventilated area. Sensitive to light. Store in light-resistant containers.

Section 8: Exposure Controls/Personal Protection

Engineering Controls: Provide exhaust ventilation or other engineering controls to keep the airborne concentrations of vapors below their respective threshold limit value. Ensure that eyewash stations and safety showers are proximal to the work-station location.

Personal Protection: Splash goggles. Lab coat. Vapor respirator. Be sure to use an approved/certified respirator or equivalent. Gloves.

Personal Protection in Case of a Large Spill: Splash goggles. Full suit. Vapor respirator. Boots. Gloves. A self contained breathing apparatus should be used to avoid inhalation of the product. Suggested protective clothing might not be sufficient; consult a specialist BEFORE handling this product.

Exposure Limits: TWA: 10 (ppm) [Australia] Inhalation TWA: 2 (ppm) from OSHA (PEL) [United States] Inhalation STEL: 9.78 (mg/m3) from NIOSH Inhalation STEL: 2 (ppm) from NIOSH Inhalation TWA: 9.78 (mg/m3) from OSHA (PEL) [United States] Inhalation TWA: 10 (ppm) from ACGIH (TLV) [United States] [1999] Inhalation TWA: 2 (ppm) [United Kingdom (UK)] Inhalation TWA: 9.9 (mg/m3) [United Kingdom (UK)] InhalationConsult local authorities for acceptable exposure limits.

Section 9: Physical and Chemical Properties

Physical state and appearance: Liquid.

Odor: Pleasant. Sweetish. Etheric. Non-irritating

Taste: Burning. Sweet.

Molecular Weight: 119.38 g/mole

Color: Colorless. Clear

pH (1% soln/water): Not available.

Boiling Point: 61°C (141.8°F)

Melting Point: -63.5°C (-82.3°F)

Critical Temperature: 263.33°C (506°F)

Specific Gravity: 1.484 (Water = 1)

Vapor Pressure: 21.1 kPa (@ 20°C)

Vapor Density: 4.36 (Air = 1)

Volatility: Not available.

Odor Threshold: 85 ppm

Water/Oil Dist. Coeff.: The product is more soluble in oil; log(oil/water) = 2

Ionicity (in Water): Not available.

Dispersion Properties: Not available.

Solubility: Very slightly soluble in cold water.

Section 10: Stability and Reactivity Data

Stability: The product is stable.

Instability Temperature: Not available.

Conditions of Instability: Incompatible materials, Light

Incompatibility with various substances: Reactive with metals, alkalis.

Corrosivity: Non-corrosive in presence of glass.

Special Remarks on Reactivity: Light Sensitive. Incompatible with triisopropyl phosphine, acetone, disilane, fluorine, strong bases and reactive metals (aluminum, magnesium in powdered form), light.

Special Remarks on Corrosivity: It will attack some forms of plastics, rubber, and coatings.

Polymerization: Will not occur.

Section 11: Toxicological Information

Routes of Entry: Absorbed through skin. Eye contact. Inhalation.

Toxicity to Animals: WARNING: THE LC50 VALUES HEREUNDER ARE ESTIMATED ON THE BASIS OF A 4-HOUR EXPOSURE. Acute oral toxicity (LD50): 36 mg/kg [Mouse]. Acute dermal toxicity (LD50): >20000 mg/kg [Rabbit]. Acute toxicity of the vapor (LC50): 47702 mg/m 4 hours [Rat]. 3

Chronic Effects on Humans: CARCINOGENIC EFFECTS: Classified + (Proven.) by NIOSH. Classified A3 (Proven for animal.) by ACGIH, 2B (Possible for human.) by IARC. Classified 2 (Some evidence.) by NTP. MUTAGENIC EFFECTS: Mutagenic for mammalian somatic cells. Mutagenic for bacteria and/or yeast. May cause damage to the following organs: kidneys, liver, heart.

Other Toxic Effects on Humans: Hazardous in case of skin contact (irritant), of ingestion, of inhalation. Slightly hazardous in case of skin contact (permeator).

Special Remarks on Toxicity to Animals: Not available.

Special Remarks on Chronic Effects on Humans: May affect genetic material (possible mutangen) and cause adverse reproductive effects(embryotoxicity and fetotoxicity) Suspected carcinogen (tumorigenic) and teratogen based on animal data. Human: passes the placental barrier, detected in maternal milk.

Special Remarks on other Toxic Effects on Humans: Acute Potential Health Effects: Skin: Causes skin irritation and may cause chemical burns. Eye: Causes eye irritation, burning pain and reversible injury to corneal epithelium. Inhalation: Causes irritation of the respiratory system (mucous membranes). May affect behavior/Nervous system (CNS depressant, fatigue, dizziness, nervousness, giddiness, euphoria, loss of coordination and judgement, weakness, hallucinations, muscle contraction/spasticity, general anesthetic, spastic paralysis, headache), anorexia (neurological and gastrointestinal symtoms resembling chronic alcoholism), and possibly coma and death. May affect the liver, kidneys and gastrointestinal tract (nausea, vomiting). Ingestion: Causes gastrointestinal tract irritation (nausea, vomiting). May affect the liver, urinary system (kidneys), respiration, behavior/nervous system (symptoms similar to inhalation),and heart. Chronic Potential Health Effects: Inhalation: Prolonged or repeated inhalation may affect the liver (hepatitis, jaundice, hepatocellular necrosis), metabolism (weight loss), respiration (fibrosis, pneumoconoisis), behavior/central nervous system (symptoms similar to acute inhalation), blood, musculoskeletal system, and kidneys. Ingestion: Prolonged or repeated ingestion may affect the liver, kidneys, metabolism (weight loss), endocrine system (spleen), blood (changes in cell count).

Section 12: Ecological Information

Ecotoxicity: Ecotoxicity in water (LC50): 43.8 mg/l 96 hours [Trout].

BOD5 and COD: Not available.

Products of Biodegradation: Possibly hazardous short term degradation products are not likely. However, long term degradation products may arise.

Toxicity of the Products of Biodegradation: The products of degradation are as toxic as the product itself.

Special Remarks on the Products of Biodegradation: Not available.

Section 13: Disposal Considerations

Waste Disposal: Waste must be disposed of in accordance with federal, state and local environmental control regulations.

Section 14: Transport Information

DOT Classification: CLASS 6.1: Poisonous material.

Identification: : Chloroform UNNA: UN1888 PG: III

Special Provisions for Transport: Not available.

Section 15: Other Regulatory Information

Federal and State Regulations: California prop. 65: This product contains the following ingredients for which the State of California has found to cause cancer, birth defects or other reproductive harm, which would require a warning under the statute: Chloroform California prop. 65 (no significant risk level): Chloroform: 0.02 mg/day (value) California prop. 65: This product contains the following ingredients for which the State of California has found to cause cancer which would require a warning under the statute: Chloroform New York release reporting list: Chloroform Rhode Island RTK hazardous substances: Chloroform Pennsylvania RTK: Chloroform Massachusetts RTK: Chloroform New Jersey: Chloroform California Director's List of Hazardous Subtances (8 CCR 339): Chloroform Tennessee: Chloroform TSCA 8(b) inventory: Chloroform TSCA 8(d) H and S data reporting: Chloroform: effective: 6/1/87; sunset: 6/1/97 SARA 302/304/311/312 extremely hazardous substances: Chloroform SARA 313 toxic chemical notification and release reporting: Chloroform CERCLA: Hazardous substances.: Chloroform: 10 lbs. (4.536 kg)

Other Regulations: OSHA: Hazardous by definition of Hazard Communication Standard (29 CFR 1910.1200). EINECS: This product is on the European Inventory of Existing Commercial Chemical Substances.

Other Classifications:

WHMIS (Canada): CLASS D-1A: Material causing immediate and serious toxic effects (VERY TOXIC). CLASS D-2A: Material causing other toxic effects (VERY TOXIC).

DSCL (EEC): R20/22- Harmful by inhalation and if swallowed. R38- Irritating to skin. R40- Possible risks of irreversible effects. S36/37- Wear suitable protective clothing and gloves.

HMIS (U.S.A.):

Health Hazard: 2

Fire Hazard: 0

Reactivity: 0

Personal Protection: h

National Fire Protection Association (U.S.A.):

Health: 2

Flammability: 0

Reactivity: 0

Specific hazard:

Protective Equipment: Gloves. Lab coat. Vapor respirator. Be sure to use an approved/certified respirator or equivalent. Wear appropriate respirator when ventilation is inadequate. Splash goggles.

Section 16: Other Information

References: Not available.

Other Special Considerations: Not available.

Created: 10/10/2005 08:16 PM

Last Updated: 11/01/2010 12:00 PM

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Material Safety Data Sheet
cis-1,2-Dichloroethylene, 97%

ACC# 97773

Section 1 - Chemical Product and Company Identification

MSDS Name: cis-1,2-Dichloroethylene, 97%
Catalog Numbers: AC113380000, AC113380025, AC113380100
Synonyms: cis-Acetylene dichloride.
Company Identification:

Acros Organics N.V.
One Reagent Lane
Fair Lawn, NJ 07410

For information in North America, call: 800-ACROS-01
For emergencies in the US, call CHEMTREC: 800-424-9300

Section 2 - Composition, Information on Ingredients

CAS#	Chemical Name	Percent	EINECS/ELINCS
156-59-2	cis-1,2-Dichloroethylene	97	205-859-7

Section 3 - Hazards Identification

EMERGENCY OVERVIEW

Appearance: Clear liquid. Flash Point: 6 deg C.
Warning! Flammable liquid and vapor. Harmful if inhaled. Unstabilized substance may polymerize. Causes eye and skin irritation. May be harmful if swallowed. May cause respiratory tract irritation.
Target Organs: Central nervous system, respiratory system, eyes, skin.

Potential Health Effects

Eye: Causes moderate eye irritation.
Skin: Causes moderate skin irritation. May cause dermatitis.
Ingestion: May cause gastrointestinal irritation with nausea, vomiting and diarrhea. May be harmful if swallowed. May cause central nervous system depression.
Inhalation: May cause respiratory tract irritation. May cause narcotic effects in high concentration. Eye irritation, vertigo, and nausea were reported in humans exposed at 2200 ppm.

Chronic: Not available. Some German investigators reported fatty degeneration of the liver upon repeated narcotic doses in rats and

Section 4 - First Aid Measures

Eyes: In case of contact, immediately flush eyes with plenty of water for a t least 15 minutes. Get medical aid.

Skin: In case of contact, flush skin with plenty of water. Remove contaminated clothing and shoes. Get medical aid if irritation develops and persists. Wash clothing before reuse.
Ingestion: If swallowed, do not induce vomiting unless directed to do so by medical personnel. Never give anything by mouth to an unconscious person. Get medical aid.
Inhalation: If inhaled, remove to fresh air. If not breathing, give artificial respiration. If breathing is difficult, give oxygen. Get medical aid.
Notes to Physician: Treat symptomatically and supportively.

Section 5 - Fire Fighting Measures

General Information: As in any fire, wear a self-contained breathing apparatus in pressure-demand, MSHA/NIOSH (approved or equivalent), and full protective gear. Vapors may form an explosive mixture with air. Use water spray to keep fire-exposed containers cool. Flammable liquid and vapor. Fire or excessive heat may result in violent rupture of the container due to bulk polymerization. Vapors are heavier than air and may travel to a source of ignition and flash back. Vapors can spread along the ground and collect in low or confined areas. Hazardous polymerization may occur under fire conditions.
Extinguishing Media: Use water fog, dry chemical, carbon dioxide, or regular foam.
Flash Point: 6 deg C (42.80 deg F)
Autoignition Temperature: 440 deg C (824.00 deg F)
Explosion Limits, Lower:9.70 vol %
Upper: 12.80 vol %
NFPA Rating: (estimated) Health: 2; Flammability: 3; Instability: 2

Section 6 - Accidental Release Measures

General Information: Use proper personal protective equipment as indicated in Section 8.
Spills/Leaks: Absorb spill with inert material (e.g. vermiculite, sand or earth), then place in suitable container. Remove all sources of ignition. Use a spark-proof tool. Provide ventilation.

Section 7 - Handling and Storage

Handling: Wash thoroughly after handling. Remove contaminated clothing and wash before reuse. Ground and bond containers when transferring material. Use spark-proof tools and explosion proof equipment. Avoid contact with eyes, skin, and clothing. Empty containers retain product residue, (liquid and/or vapor), and can be dangerous. Avoid ingestion and inhalation. Do not pressurize, cut, weld, braze, solder, drill, grind, or expose empty containers to heat, sparks or open flames. Use only with adequate ventilation. Pure vapor will be uninhibited and may polymerize in vents or other confined spaces.
Storage: Keep away from sources of ignition. Store in a tightly closed container. Flammables-area. Store protected from light and air.

Section 8 - Exposure Controls, Personal Protection

Engineering Controls: Use process enclosure, local exhaust ventilation, or other engineering controls to control airborne levels below recommended exposure limits. Facilities storing or utilizing this material should be equipped with an eyewash facility and a safety shower.

Exposure Limits

Chemical Name	ACGIH	NIOSH	OSHA - Final PELs
cis-1,2-Dichloroethylene	200 ppm TWA	none listed	none listed

OSHA Vacated PELs: cis-1,2-Dichloroethylene: No OSHA Vacated PELs are listed for this chemical.

Personal Protective Equipment

Eyes: Wear chemical splash goggles.
Skin: Wear appropriate protective gloves to prevent skin exposure.
Clothing: Wear appropriate protective clothing to prevent skin exposure.
Respirators: Follow the OSHA respirator regulations found in 29 CFR 1910.134 or European Standard EN 149. Use a NIOSH/MSHA or European Standard EN 149 approved respirator if exposure limits are exceeded or if irritation or other symptoms are experienced.

Section 9 - Physical and Chemical Properties

Physical State: Liquid
Appearance: Clear
Odor: Pleasant odor
pH: Not available.
Vapor Pressure: 201 mm Hg @ 25 deg C
Vapor Density: 3.34 (air=1)
Evaporation Rate: Not available.
Viscosity: Not available.
Boiling Point: 60 deg C @ 760 mm Hg
Freezing/Melting Point:-80 deg C
Decomposition Temperature: Not available.
Solubility: Insoluble.
Specific Gravity/Density:1.2800
Molecular Formula:C2H2Cl2
Molecular Weight:96.94

Section 10 - Stability and Reactivity

Chemical Stability: Stable under normal temperatures and pressures. This material is a monomer and may polymerize under certain conditions if the stabilizer is lost.
Conditions to Avoid: Light, ignition sources, exposure to air, excess heat.
Incompatibilities with Other Materials: Strong oxidizing agents, strong bases, copper.
Hazardous Decomposition Products: Hydrogen chloride, phosgene, carbon monoxide, carbon dioxide.
Hazardous Polymerization: May occur.

Section 11 - Toxicological Information

RTECS#:
CAS# 156-59-2: KV9420000
LD50/LC50:
CAS# 156-59-2:
Inhalation, rat: LC50 = 13700 ppm;

.

Carcinogenicity:
CAS# 156-59-2: Not listed by ACGIH, IARC, NTP, or CA Prop 65.

Epidemiology: No data available.
Teratogenicity: No data available.
Reproductive Effects: No data available.
Mutagenicity: No data available.
Neurotoxicity: No data available.
Other Studies:

Section 12 - Ecological Information

No information available.

Section 13 - Disposal Considerations

Chemical waste generators must determine whether a discarded chemical is classified as a hazardous waste. US EPA guidelines for the classification determination are listed in 40 CFR Parts 261.3. Additionally, waste generators must consult state and local hazardous waste regulations to ensure complete and accurate classification.
RCRA P-Series: None listed.
RCRA U-Series: None listed.

Section 14 - Transport Information

	US DOT	Canada TDG
Shipping Name:	DOT regulated - small quantity provisions apply (see 49CFR173.4)	1,2-DICHLOROETHYLENE
Hazard Class:		3
UN Number:		UN1150
Packing Group:		II

Section 15 - Regulatory Information

US FEDERAL

TSCA
CAS# 156-59-2 is listed on the TSCA inventory.
Health & Safety Reporting List
None of the chemicals are on the Health & Safety Reporting List.
Chemical Test Rules
None of the chemicals in this product are under a Chemical Test Rule.
Section 12b
None of the chemicals are listed under TSCA Section 12b.
TSCA Significant New Use Rule
None of the chemicals in this material have a SNUR under TSCA.
CERCLA Hazardous Substances and corresponding RQs

None of the chemicals in this material have an RQ.

SARA Section 302 Extremely Hazardous Substances

None of the chemicals in this product have a TPQ.

Section 313 No chemicals are reportable under Section 313.

Clean Air Act:

This material does not contain any hazardous air pollutants.
This material does not contain any Class 1 Ozone depletors.
This material does not contain any Class 2 Ozone depletors.

Clean Water Act:

None of the chemicals in this product are listed as Hazardous Substances under the CWA.
None of the chemicals in this product are listed as Priority Pollutants under the CWA.
None of the chemicals in this product are listed as Toxic Pollutants under the CWA.

OSHA:

None of the chemicals in this product are considered highly hazardous by OSHA.

STATE

CAS# 156-59-2 can be found on the following state right to know lists: Pennsylvania, Massachusetts.

California Prop 65

California No Significant Risk Level: None of the chemicals in this product are listed.

European / International Regulations

European Labeling in Accordance with EC Directives

Hazard Symbols:

XN F

Risk Phrases:

R 11 Highly flammable.
R 20 Harmful by inhalation.
R 52/53 Harmful to aquatic organisms, may cause long-term adverse effects in the aquatic environment.

Safety Phrases:

S 16 Keep away from sources of ignition - No smoking.
S 29 Do not empty into drains.
S 7 Keep container tightly closed.
S 61 Avoid release to the environment. Refer to special instructions /safety data sheets.

WGK (Water Danger / Protection)

CAS# 156-59-2: No information available.

Canada - DSL / NDSL

CAS# 156-59-2 is listed on Canada's NDSL List.

Canada - WHMIS

WHMIS: Not available.

This product has been classified in accordance with the hazard criteria of the Controlled Products Regulations and the MSDS contains all of the information required by those regulations.

Canadian Ingredient Disclosure List

Section 16 - Additional Information

MSDS Creation Date: 2/09/1998
Revision #5 Date: 3/16/2007

The information above is believed to be accurate and represents the best information currently available to us.

However, we make no warranty of merchantability or any other warranty, express or implied, with respect to such information, and we assume no liability resulting from its use. Users should make their own investigations to determine the suitability of the information for their particular purposes. In no event shall Fisher be liable for any claims, losses, or damages of any third party or for lost profits or any special, indirect, incidental, consequential or exemplary damages, howsoever arising, even if Fisher has been advised of the possibility of such damages.

OAKTON® MATERIAL SAFETY DATA SHEET

Section 1. Chemical Product and Company Identification

Catalog Number(s)

00606-10, 00653-15, 00653-16, 00653-18, 00653-20, 00653-23, 00653-27, 00653-32, 00653-47, 00653-50, 00653-89, 35653-09, 35653-10, 35653-11, 35653-12, 35653-13, 35656-18, 35656-47

Product Identity

CONDUCTIVITY STANDARD SOLUTIONS, < 90 mS

Manufacturer's Name	Emergency Telephone Number (24 hr)
RICCA CHEMICAL COMPANY	CHEMTREC®: 800-424-9300
Address (Number, Street, City, State, and ZIP Code)	Telephone Number For Information
P.O. Box 13090	817-461-5601
	Date Prepared
Arlington, Texas 76094	3-17-2000

Section 2. Composition / Information on Ingredients

Component	CAS Registry #	Percent Concentration	Exposure Limits ACGIH TLV	Exposure Limits OSHA PEL
Potassium Chloride	7447-40-7	< 6	N/A	N/A
Water, Deionized	7732-18-5	Balance	N/A	N/A

Section 3. Hazards Identification

☆☆☆

INHALATION: Remove to fresh air. Give artificial respiration if necessary.

SKIN CONTACT: Flush with plenty of water for at least 15 minutes.

INGESTION: Dilute with water or milk. Call a physician if necessary.

Section 5. Fire Fighting Measures

FLAMMABLE PROPERTIES:

FLASH POINT: N/A METHOD USED: N/A

FLAMMABLE LIMITS

LFL: N/A UFL: N/A

EXTINGUISHING MEDIA: Use any means suitable for extinguishing surrounding fire.

FIRE & EXPLOSION HAZARDS: Not considered to be a fire or explosion hazard.

FIRE FIGHTING INSTRUCTIONS: Use normal procedures/instructions.

FIRE FIGHTING EQUIPMENT: Use protective clothing and breathing equipment appropriate for the surrounding fire.

Section 6. Accidental Release Measures

Absorb with suitable material (paper towels, etc.) and dispose of in accordance with local regulations. Small amounts may be flushed to the sewer with plenty of water.

Section 7. Handling and Storage

As with all chemicals, wash hands thoroughly after handling. Avoid contact with eyes and skin. Protect from freezing and physical damage. SAFETY STORAGE CODE: GENERAL

Section 8. Exposure Controls / Personal Protection

ENGINEERING CONTROLS: No specific controls are needed. Normal room ventilation is adequate.

RESPIRATORY PROTECTION: Normal room ventilation is adequate.

SKIN PROTECTION: Chemical resistant gloves are recommended.

EYE PROTECTION: Safety glasses or goggles.

Section 9. Physical and chemical Properties

APPEARANCE:	Clear, colorless liquid	**pH:**	approximately 7
ODOR:	Odorless	**BOILING POINT (^{o}C):**	approximately 100
SOLUBILITY IN WATER:	Infinite	**MELTING POINT (^{o}C):**	approximately 0
SPECIFIC GRAVITY:	approximately 1.0 – 1.04	**VAPOR PRESSURE:**	N/A

Section 10. Stability and Reactivity

CHEMICAL STABILITY: Stable under normal conditions of use and storage.

INCOMPATIBILITY: Bromine Trifluoride, Potassium Permanganate plus Sulfuric Acid.

HAZARDOUS DECOMPOSITION PRODUCTS: Oxides of Potassium.

HAZARDOUS POLYMERIZATION: Will not occur.

Section 11. Toxicological Information

LD_{50}, Oral, Rat: 2600 mg/kg (Potassium Chloride), details of toxic effects not reported other than lethal dose value. Irritation: eye, rabbit (500mg/24 hr mild).

Section 12. Ecological Information

ECOTOXICOLOGICAL INFORMATION: No information found.

CHEMICAL FATE INFORMATION: No information found.

Section 13. Disposal Considerations

Dilute with water and flush to sewer if local regulations allow. If not allowed, save for recovery or recycling in an approved waste disposal facility. Always dispose of in accordance with local, state and federal regulations.

Section 14. Transport Information (Not meant to be all inclusive)

D.O.T. SHIPPING NAME:	Not regulated
D.O.T. HAZARD CLASS:	None
U.N. / N.A. NUMBER:	None
PACKING GROUP:	None
D.O.T. LABEL:	None

Section 15. Regulatory Information (Not meant to be all inclusive - selected regulation represented)

OSHA STATUS: The above items either do not contain any specifically hazardous material or the potentially hazardous material is present in such low concentration that the items do not present any immediate threat to health and safety. These items do not meet the OSHA Hazard Communication Standard (29 CFR 1910.1200) definition of a hazardous material.

TSCA STATUS: All components of this solution are listed on the TSCA Inventory.

CERCLA REPORTABLE QUANTITY: Not reportable

SARA TITLE III:

SECTION 302 EXTREMELY HAZARDOUS SUBSTANCES: No

SECTION 311/312 HAZARDOUS CATEGORIES: No

SECTION 313 TOXIC CHEMICALS: No

RCRA STATUS: No

CALIFORNIA PROPOSITION 65: Not listed

Section 16. Other Information

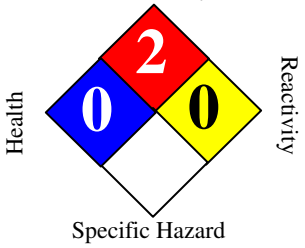
NFPA® Ratings:	**Health: 0**	**Flammability: 0**	**Reactivity: 0**	**Special Notice Key: None**
HMIS® Ratings:	**Health: 0**	**Flammability: 0**	**Reactivity: 0**	**Protective Equipment: B (Protective eyewear, gloves)**

Rev 1, 10-16-2000: (Section 1) added catalog numbers 35653-10, 35653-11, 35653-12, and 35653-13.
Rev 2, 12-06-2001: (Section 1) added catalog number 35653-09; revised description from 23µ - 80 mS.
Rev 3, 03-25-2003: Reviewed and approved, (Section 3) added mutation statement, (Section 11) added irritation data.
Rev 4, 03-20-2006: Reviewed and approved.

When handled properly by qualified personnel, the product described herein does not present a significant health or safety hazard. Alteration of its characteristics by concentration, evaporation, addition of other substances, or other means may present hazards not specifically addressed herein and which must be evaluated by the user. The information furnished herein is believed to be accurate and represents the best data currently available to us. No warranty, expressed or implied, is made and RICCA CHEMICAL COMPANY assumes no legal responsibility or liability whatsoever resulting from its use.

Material Safety Data Sheet
Diesel Low Sulfur (LSD) and Ultra Low Sulfur Diesel (ULSD)

NFPA:



TESORO

HMIS III:

HEALTH	1
FLAMMABILITY	2
PHYSICAL	0

0 = Insignificant, 1 = Slight, 2 = Moderate, 3 = High, 4 = Extreme

SECTION 1. PRODUCT AND COMPANY IDENTIFICATION

Product name	:	Diesel Low Sulfur (LSD) and Ultra Low Sulfur Diesel (ULSD)		
Synonyms	:	CARB Diesel, 888100004478		
MSDS Number	:	888100004478	**Version**	: 2.19
Product Use Description	:	Fuel		
Company	:	For: Tesoro Refining & Marketing Co. 19100 Ridgewood Parkway, San Antonio, TX 78259		
Tesoro Call Center	:	(877) 783-7676	**Chemtrec (Emergency Contact)**	: (800) 424-9300

SECTION 2. HAZARDS IDENTIFICATION

Emergency Overview

Regulatory status : This material is considered hazardous by the Occupational Safety and Health Administration (OSHA) Hazard Communication Standard (29 CFR 1910.1200).

Signal Word : WARNING

Hazard Summary : Toxic. Combustible Liquid

Potential Health Effects

Eyes : Eye irritation may result from contact with liquid, mists, and/or vapors.

Inhalation : Vapors or mists from this material can irritate the nose, throat, and lungs, and can cause signs and symptoms of central nervous system depression, depending on the concentration and duration of exposure.

Skin : Skin irritation leading to dermatitis may occur upon prolonged or repeated contact. Liquid may be absorbed through the skin in toxic amounts if large areas of skin are repeatedly exposed. Long-term, repeated skin contact may cause skin cancer

Ingestion : Harmful or fatal if swallowed. Do NOT induce vomiting. This material can irritate the mouth, throat, stomach, and cause nausea, vomiting, diarrhea and restlessness Aspiration hazard if liquid is inhaled into lungs, particularly from vomiting after ingestion. Aspiration may result in chemical pneumonia, severe lung damage, respiratory failure and even death.

Target Organs : Central nervous system, Eyes, Skin, Kidney, Liver

SECTION 3. COMPOSITION/INFORMATION ON INGREDIENTS

Component	CAS-No.	Weight %
Fuels, diesel, No 2; Gasoil - unspecified	68476-34-6	100%
Nonane	111-84-2	0 - 5%
Naphthalene	91-20-3	0 - 1%
1,2,4-Trimethylbenzene	95-63-6	0 - 2%
Xylene	1330-20-7	0 - 2%
Sulfur	7704-34-9	15 ppm maximum

SECTION 4. FIRST AID MEASURES

Inhalation : Move to fresh air. Give oxygen. If breathing is irregular or stopped, administer artificial respiration. Seek medical attention immediately.

Skin contact : Take off all contaminated clothing immediately. Wash off immediately with soap and plenty of water. Wash contaminated clothing before re-use. If skin irritation persists, seek medical attention immediately.

Eye contact : Remove contact lenses. Rinse thoroughly with plenty of water for at least 15 minutes. If symptoms persist, seek medical attention.

Ingestion : Do not induce vomiting without medical advice. If a person vomits when lying on his back, place him in the recovery position. Seek medical attention immediately.

Notes to physician : Symptoms: Dizziness, Discomfort, Headache, Nausea, Disorder, Vomiting, Lung edema, Aspiration may cause pulmonary edema and pneumonitis, Liver disorders, Kidney disorders.

SECTION 5. FIRE-FIGHTING MEASURES

Form : Liquid

Flash point : 38℃ Minimum for #1 Diesel, 52℃ Minimum for #2 Diesel

Auto Ignition temperature : 257 ℃ (495 ℉)

Lower explosive limit : 0.6 %(V)

Upper explosive limit : 4.7 %(V)

Suitable extinguishing media : Carbon dioxide (CO2), Water spray, Dry chemical, Foam, Keep containers and surroundings cool with water spray.

Specific hazards during fire fighting : Fire Hazard Do not use a solid water stream as it may scatter and spread fire. Cool closed containers exposed to fire with water spray.

Special protective equipment for fire-fighters	:	Wear self-contained breathing apparatus and protective suit. Use personal protective equipment.
Further information	:	Exposure to decomposition products may be a hazard to health. Isolate area around container involved in fire. Cool tanks, shells, and containers exposed to fire and excessive heat with water. For massive fires the use of unmanned hose holders or monitor nozzles may be advantageous to further minimize personnel exposure. Major fires may require withdrawal, allowing the tank to burn. Large storage tank fires typically require specially trained personnel and equipment to extinguish the fire, often including the need for properly applied fire fighting foam.

SECTION 6. ACCIDENTAL RELEASE MEASURES

Personal precautions	:	Evacuate nonessential personnel and remove or secure all ignition sources. Consider wind direction; stay upwind and uphill, if possible. Evaluate the direction of product travel, diking, sewers, etc. to contain spill areas. Spills may infiltrate subsurface soil and groundwater; professional assistance may be necessary to determine the extent of subsurface impact. Ensure adequate ventilation. Use personal protective equipment.
Environmental precautions	:	Carefully contain and stop the source of the spill, if safe to do so. Protect bodies of water by diking, absorbents, or absorbent boom, if possible. Do not flush down sewer or drainage systems, unless system is designed and permitted to handle such material. The use of fire fighting foam may be useful in certain situations to reduce vapors. The proper use of water spray may effectively disperse product vapors or the liquid itself, preventing contact with ignition sources or areas/equipment that require protection. Discharge into the environment must be avoided. If the product contaminates rivers and lakes or drains inform respective authorities.
Methods for cleaning up	:	Take up with sand or oil absorbing materials. Carefully shovel, scoop or sweep up into a waste container for reclamation or disposal - caution, flammable vapors may accumulate in closed containers. Response and clean-up crews must be properly trained and must utilize proper protective equipment (see Section 8).

SECTION 7. HANDLING AND STORAGE

Handling	:	Keep away from fire, sparks and heated surfaces. No smoking near areas where material is stored or handled. The product should only be stored and handled in areas with intrinsically safe electrical classification.
Advice on protection against fire and explosion	:	Hydrocarbon liquids including this product can act as a non-conductive flammable liquid (or static accumulators), and may form ignitable vapor-air mixtures in storage tanks or other containers. Precautions to prevent static-initated fire or explosion during transfer, storage or handling, include but are not limited to these examples: (1) Ground and bond containers during product transfers. Grounding and bonding may not be adequate protection to prevent ignition or explosion of hydrocarbon liquids and vapors that are static accumulators. (2) Special slow load procedures for "switch loading" must be followed to avoid the static ignition hazard that can exist when higher flash point material (such as fuel oil or diesel) is loaded into tanks previously containing low flash point products (such gasoline or naphtha). (3) Storage tank level floats must be effectively bonded. For more information on precautions to prevent static-initated fire or explosion, see NFPA 77, Recommended Practice on Static Electricity (2007), and API

Recommended Practice 2003, Protection Against Ignitions Arising Out of Static, Lightning, and Stray Currents (2008).

Dust explosion class : Not applicable

Requirements for storage areas and containers : Keep away from flame, sparks, excessive temperatures and open flame. Use approved containers. Keep containers closed and clearly labeled. Empty or partially full product containers or vessels may contain explosive vapors. Do not pressurize, cut, heat, weld or expose containers to sources of ignition. Store in a well-ventilated area. The storage area should comply with NFPA 30 "Flammable and Combustible Liquid Code". The cleaning of tanks previously containing this product should follow API Recommended Practice (RP) 2013 "Cleaning Mobile Tanks In Flammable and Combustible Liquid Service" and API RP 2015 "Cleaning Petroleum Storage Tanks".

Other data : Emergency eye wash capability should be available in the near proximity to operations presenting a potential splash exposure.

Advice on common storage Keep away from food, drink and animal feed. Incompatible with oxidizing agents. Incompatible with acids.

SECTION 8. EXPOSURE CONTROLS / PERSONAL PROTECTION

Exposure Guidelines

List	Components	CAS-No.	Type:	Value
OSHA Z1	Xylene	1330-20-7	PEL	100 ppm 435 mg/m3
	Naphthalene	91-20-3	PEL	10 ppm 50 mg/m3
ACGIH	Diesel Fuel	68476-30-2	TWA	100 mg/m3
	Xylene	1330-20-7	TWA	100 ppm
		1330-20-7	STEL	150 ppm
	Naphthalene	91-20-3	TWA	10 ppm
		91-20-3	STEL	15 ppm
	Nonane	111-84-2	TWA	200 ppm

Engineering measures : Use adequate ventilation to keep gas and vapor concentrations of this product below occupational exposure and flammability limits, particularly in confined spaces. Use only intrinsically safe electrical equipment approved for use in classified areas.

Eye protection : Safety glasses or goggles are recommended where there is a possibility of splashing or spraying.

Hand protection : Gloves constructed of nitrile, neoprene, or PVC are recommended. Consult manufacturer specifications for further information.

Skin and body protection : If needed to prevent skin contact, chemical protective clothing such as of DuPont

		TyChem®, Saranex or equivalent recommended based on degree of exposure. The resistance of specific material may vary from product to product as well as with degree of exposure.
Respiratory protection	:	A NIOSH/ MSHA-approved air-purifying respirator with organic vapor cartridges or canister may be permissible under certain circumstances where airborne concentrations are or may be expected to exceed exposure limits or for odor or irritation. Protection provided by air-purifying respirators is limited. Refer to OSHA 29 CFR 1910.134, ANSI Z88.2-1992, NIOSH Respirator Decision Logic, and the manufacturer for additional guidance on respiratory protection selection. Use a NIOSH/ MSHA-approved positive-pressure supplied-air respirator if there is a potential for uncontrolled release, exposure levels are not known, in oxygen-deficient atmospheres, or any other circumstance where an air-purifying respirator may not provide adequate protection.
Work / Hygiene practices	:	Emergency eye wash capability should be available in the near proximity to operations presenting a potential splash exposure. Use good personal hygiene practices. Avoid repeated and/or prolonged skin exposure. Wash hands before eating, drinking, smoking, or using toilet facilities. Do not use as a cleaning solvent on the skin. Do not use solvents or harsh abrasive skin cleaners for washing this product from exposed skin areas. Waterless hand cleaners are effective. Promptly remove contaminated clothing and launder before reuse. Use care when laundering to prevent the formation of flammable vapors which could ignite via washer or dryer. Consider the need to discard contaminated leather shoes and gloves.

SECTION 9. PHYSICAL AND CHEMICAL PROPERTIES

Form	:	Liquid
Appearance	:	Clear, straw colored
Odor	:	Characteristic petroleum (kerosene) odor
Flash point - typical	:	38°C Minimum for #1 Diesel, 52°C Minimum for #2 Diesel
Auto Ignition temperature	:	257 °C (495 °F)
Thermal decomposition	:	No decomposition if stored and applied as directed.
Lower explosive limit	:	0.6 %(V)
Upper explosive limit	:	4.7 %(V)
pH	:	Not applicable
Freezing point	:	No data available
Boiling point	:	154 - 372 °C(310° - 702 °F)
Vapor Pressure	:	< 2 mm Hg at 20 °C
Density	:	**0.86** g/cm3
Water solubility	:	Negligible
Viscosity, dynamic	:	1.7 - 40 mPa.s at 37.8 °C (100.0 °F)

Percent Volatiles	:	100 %
Conductivity (conductivity can be reduced by environmental factors such as a decrease in temperature		Diesel Fuel Oils at terminal load rack: At least 25 pS/m Ultra Low Sulfur Diesel (ULSD) without conductivity additive: 0 pS/m to 5 pS/m ULSD at terminal load rack with conductivity additive: At least 50 pS/m but conductivity may decrease from environmental factors such as temperature drop. JP-8 at terminal load rack: 150 pS/m to 600 pS/m

SECTION 10. STABILITY AND REACTIVITY

Conditions to avoid	:	Avoid high temperatures, open flames, sparks, welding, smoking and other ignition sources. Keep away from strong oxidizers. Viton ® ; Fluorel ®
Materials to avoid	:	Strong oxidizing agents. Peroxides
Hazardous decomposition products	:	Carbon monoxide, carbon dioxide and noncombusted hydrocarbons (smoke). Diesel exhaust particulates may be a lung hazard - see Section 11.
Thermal decomposition	:	No decomposition if stored and applied as directed.
Hazardous reactions	:	Keep away from oxidizing agents, and acidic or alkaline products.

SECTION 11. TOXICOLOGICAL INFORMATION

Carcinogenicity

NTP	:	Naphthalene (CAS-No.: 91-20-3)
IARC	:	Naphthalene (CAS-No.: 91-20-3)
OSHA	:	No component of this product which is present at levels greater than or equal to 0.1 % is identified as a carcinogen or potential carcinogen by OSHA.
CA Prop 65	:	WARNING! This product contains a chemical known to the State of California to cause cancer. naphthalene (CAS-No.: 91-20-3)
Skin irritation	:	Irritating to skin.
Eye irritation	:	Irritating to eyes.
Further information	:	Studies have shown that similar products produce skin cancer or skin tumors in laboratory animals following repeated applications without washing or removal. The significance of this finding to human exposure has not been determined. Other studies with active skin carcinogens have shown that washing the animal's skin with soap and water between applications reduced tumor formation. Positive mutagenicity results have been reported. Repeated over-exposure may cause liver and kidney injury IARC classifies whole diesel fuel exhaust particulates as probably carcinogenic to humans (Group 2A). NIOSH regards whole diesel fuel exhaust particulates as a potential cause of occupational lung cancer based on animal studies and limited evidence in humans.

Component:

Fuels, diesel, No 2; Gasoil - unspecified	68476-34-6	Acute oral toxicity: LD50 rat Dose: 5,001 mg/kg Acute dermal toxicity: LD50 rabbit

		Dose: 2,001 mg/kg Acute inhalation toxicity: LC50 rat Dose: 7.64 mg/l Exposure time: 4 h Skin irritation: Classification: Irritating to skin. Result: Severe skin irritation Eye irritation: Classification: Irritating to eyes. Result: Mild eye irritation
Nonane	111-84-2	Acute oral toxicity: LD50 mouse Dose: 218 mg/kg Acute inhalation toxicity: LC50 rat Exposure time: 4 h
Naphthalene	91-20-3	Acute oral toxicity: LD50 rat Dose: 2,001 mg/kg Acute dermal toxicity: LD50 rat Dose: 2,501 mg/kg Acute inhalation toxicity: LC50 rat Dose: 101 mg/l Exposure time: 4 h Skin irritation: Classification: Irritating to skin. Result: Mild skin irritation Eye irritation: Classification: Irritating to eyes. Result: Mild eye irritation Carcinogenicity: N11.00422130
1,2,4-Trimethylbenzene	95-63-6	Acute inhalation toxicity: LC50 rat Dose: 18 mg/l Exposure time: 4 h Skin irritation: Classification: Irritating to skin. Result: Skin irritation Eye irritation: Classification: Irritating to eyes. Result: Eye irritation
Xylene	1330-20-7	Acute oral toxicity: LD50 rat Dose: 2,840 mg/kg Acute dermal toxicity: LD50 rabbit Dose: ca. 4,500 mg/kg Acute inhalation toxicity: LC50 rat Dose: 6,350 mg/l Exposure time: 4 h Skin irritation: Classification: Irritating to skin. Result: Mild skin irritation Repeated or prolonged exposure may cause skin irritation and dermatitis, due to degreasing properties of the product. Eye irritation: Classification: Irritating to eyes. Result: Mild eye irritation

SECTION 12. ECOLOGICAL INFORMATION

Additional ecological : Keep out of sewers, drainage areas, and waterways. Report spills and releases, as

information applicable, under Federal and State regulations.

Component:

Naphthalene	91-20-3	Toxicity to algae: EC50 Species: Dose: 33 mg/l Exposure time: 24 h
1,2,4-Trimethylbenzene	95-63-6	Toxicity to fish: LC50 Species: Pimephales promelas (fathead minnow) Dose: 7.72 mg/l Exposure time: 96 h Acute and prolonged toxicity for aquatic invertebrates: EC50 Species: Daphnia Dose: 3.6 mg/l Exposure time: 48 h

SECTION 13. DISPOSAL CONSIDERATIONS

Disposal : In accordance with local and national regulations.

SECTION 14. TRANSPORT INFORMATION

CFR

Proper shipping name	: DIESEL FUEL
UN-No.	: UN1202 (NA 1993)
Class	: 3
Packing group	: III

TDG

Proper shipping name	: DIESEL FUEL
UN-No.	: UN1202 (NA 1993)
Class	: 3
Packing group	: III

IATA Cargo Transport

UN UN-No.	: UN1202 (NA 1993)
Description of the goods	: DIESEL FUEL
Class	: 3
Packaging group	: III
ICAO-Labels	: 3
Packing instruction (cargo aircraft)	: 366
Packing instruction (cargo aircraft)	: Y344

IATA Passenger Transport

UN UN-No.	: UN1202 (NA 1993)
Description of the goods	: DIESEL FUEL
Class	: 3
Packaging group	: III

	ICAO-Labels	: 3
	Packing instruction (passenger aircraft)	: 355
	Packing instruction (passenger aircraft)	: Y344
IMDG-Code		
	UN-No.	: UN 1202 (NA 1993)
	Description of the goods	: DIESEL FUEL
	Class	: 3
	Packaging group	: III
	IMDG-Labels	: 3
	EmS Number	: F-E S-E
	Marine pollutant	: No

SECTION 15. REGULATORY INFORMATION

OSHA Hazards : Combustible Liquid
Moderate skin irritant
Moderate eye irritant
Toxic by ingestion
POSSIBLE CANCER HAZARD

CERCLA SECTION 103 and SARA SECTION 304 (RELEASE TO THE ENVIROMENT)
The CERCLA definition of hazardous substances contains a "petroleum exclusion" clause which exempts crude oil. Fractions of crude oil, and products (both finished and intermediate) from the crude oil refining process and any indigenous components of such from the CERCLA Section 103 reporting requirements. However, other federal reporting requirements, including SARA Section 304, as well as the Clean Water Act may still apply.

TSCA Status : On TSCA Inventory

DSL Status : All components of this product are on the Canadian DSL list.

SARA 311/312 Hazards : Fire Hazard
Acute Health Hazard
Chronic Health Hazard

SARA III US. EPA Emergency Planning and Community Right-To-Know Act (EPCRA) SARA Title III Section 313 Toxic Chemicals (40 CFR 372.65) - Supplier Notification Required

Components	CAS-No.
Xylene	1330-20-7
1,2,4-Trimethylbenzene	95-63-6
Naphthalene	91-20-3

PENN RTK US. Pennsylvania Worker and Community Right-to-Know Law (34 Pa. Code Chap. 301-323)

Components	CAS-No.
Nonane	111-84-2

Naphthalene	91-20-3
1,2,4-Trimethylbenzene	95-63-6
xylene	1330-20-7
Fuels, diesel, No 2; Gasoil - unspecified	68476-34-6

MASS RTK — US. Massachusetts Commonwealth's Right-to-Know Law (Appendix A to 105 Code of Massachusetts Regulations Section 670.000)

Components	CAS-No.
Xylene	1330-20-7
1,2,4-Trimethylbenzene	95-63-6
Naphthalene	91-20-3
Nonane	111-84-2

NJ RTK — US. New Jersey Worker and Community Right-to-Know Act (New Jersey Statute Annotated Section 34:5A-5)

Components	CAS-No.
Nonane	111-84-2
Naphthalene	91-20-3
1,2,4-Trimethylbenzene	95-63-6
Xylene	1330-20-7
Fuels, diesel, No 2; Gasoil - unspecified	68476-34-6

California Prop. 65 : WARNING! This product contains a chemical known to the State of California to cause cancer.

Naphthalene 91-20-3

SECTION 16. OTHER INFORMATION

Further information

The information provided in this Safety Data Sheet is correct to the best of our knowledge, information and belief at the date of its publication. The information given is designed only as guidance for safe handling, use, processing, storage, transportation, disposal and release and is not to be considered a warranty or quality specification. The information relates only to the specific material designated and may not be valid for such material used in combination with any other materials or in any process, unless specified in the text.

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12/01/2011

1153, 1250, 1443, 1454, 1814, 1815, 1866, 1925

Material Safety Data Sheet

SECTION 1 PRODUCT AND COMPANY IDENTIFICATION

CHEVRON REGULAR UNLEADED GASOLINE

Product Number(s): CPS201000 [See Section 16 for Additional Product Numbers]
Synonyms: Calco Regular Unleaded Gasoline

Company Identification
Chevron Products Company
Marketing, MSDS Coordinator
6001 Bollinger Canyon Road
San Ramon, CA 94583
United States of America

Transportation Emergency Response
CHEMTREC: (800) 424-9300 or (703) 527-3887
Health Emergency
ChevronTexaco Emergency Information Center: Located in the USA. International collect calls accepted. (800) 231-0623 or (510) 231-0623
Product Information
Technical Information: (510) 242-5357

SPECIAL NOTES: This MSDS applies to: Federal Reformulated Gasoline, California Reformulated Gasoline, Wintertime Oxygenated Gasoline, Low RVP Gasoline and Conventional Gasoline.

SECTION 2 COMPOSITION/ INFORMATION ON INGREDIENTS

COMPONENTS	CAS NUMBER	AMOUNT
Gasoline	86290-81-5	100 %volume
Benzene	71-43-2	0.1 - 4.9 %volume
Ethyl benzene	100-41-4	0.1 - 3 %volume
Naphthalene	91-20-3	0.1 - 2 %volume
Ethanol	64-17-5	0 - 10 %volume
Methyl tert-butyl ether (MTBE)	1634-04-4	0 - 15 %volume
Tertiary amyl methyl ether (TAME)	994-05-8	0 - 17 %volume
Ethyl tert-butyl ether (ETBE)	637-92-3	0 - 18 %volume

Motor gasoline is considered a mixture by EPA under the Toxic Substances Control Act (TSCA). The refinery streams used to blend motor gasoline are all on the TSCA Chemical Substances Inventory. The appropriate CAS number for refinery blended motor gasoline is 86290-81-5. The product specifications of motor gasoline sold in your area will depend on applicable Federal and State regulations.

SECTION 3 HAZARDS IDENTIFICATION

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EMERGENCY OVERVIEW

- EXTREMELY FLAMMABLE LIQUID AND VAPOR. VAPOR MAY CAUSE FLASH FIRE
- HARMFUL OR FATAL IF SWALLOWED - MAY CAUSE LUNG DAMAGE IF SWALLOWED
- VAPOR HARMFUL
- CAUSES SKIN IRRITATION
- CAUSES EYE IRRITATION
- LONG-TERM EXPOSURE TO VAPOR HAS CAUSED CANCER IN LABORATORY ANIMALS
- KEEP OUT OF REACH OF CHILDREN
- TOXIC TO AQUATIC ORGANISMS

****

IMMEDIATE HEALTH EFFECTS
Eye: Contact with the eyes causes irritation. Symptoms may include pain, tearing, reddening, swelling and impaired vision.
Skin: Contact with the skin causes irritation. Skin contact may cause drying or defatting of the skin. Symptoms may include pain, itching, discoloration, swelling, and blistering. Contact with the skin is not expected to cause an allergic skin response. Not expected to be harmful to internal organs if absorbed through the skin.
Ingestion: Because of its low viscosity, this material can directly enter the lungs, if swallowed, or if subsequently vomited. Once in the lungs it is very difficult to remove and can cause severe injury or death.
Inhalation: The vapor or fumes from this material may cause respiratory irritation. Symptoms of respiratory irritation may include coughing and difficulty breathing. Breathing this material at concentrations above the recommended exposure limits may cause central nervous system effects. Central nervous system effects may include headache, dizziness, nausea, vomiting, weakness, loss of coordination, blurred vision, drowsiness, confusion, or disorientation. At extreme exposures, central nervous system effects may include respiratory depression, tremors or convulsions, loss of consciousness, coma or death.

DELAYED OR OTHER HEALTH EFFECTS:
Reproduction and Birth Defects: This material is not expected to cause birth defects or other harm to the developing fetus based on animal data.
Cancer: Prolonged or repeated exposure to this material may cause cancer. Gasoline has been classified as a Group 2B carcinogen (possibly carcinogenic to humans) by the International Agency for Research on Cancer (IARC).

Contains benzene, which has been classified as a carcinogen by the National Toxicology Program (NTP) and a Group 1 carcinogen (carcinogenic to humans) by the International Agency for Research on Cancer (IARC).
Contains ethylbenzene which has been classified as a Group 2B carcinogen (possibly carcinogenic to humans) by the International Agency for Research on Cancer (IARC).
Contains naphthalene, which has been classified as a Group 2B carcinogen (possibly carcinogenic to humans) by the International Agency for Research on Cancer (IARC).

Whole gasoline exhaust has been classified as a Group 2B carcinogen (possibly carcinogenic to humans) by the International Agency for Research on Cancer (IARC).

Risk depends on duration and level of exposure. See Section 11 for additional information.

SECTION 4 FIRST AID MEASURES

Eye: Flush eyes with water immediately while holding the eyelids open. Remove contact lenses, if worn, after initial flushing, and continue flushing for at least 15 minutes. Get medical attention if irritation persists.
Skin: Wash skin with water immediately and remove contaminated clothing and shoes. Get medical attention if any symptoms develop. To remove the material from skin, use soap and water. Discard contaminated clothing and shoes or thoroughly clean before reuse.
Ingestion: If swallowed, get immediate medical attention. Do not induce vomiting. Never give anything by mouth to an unconscious person.
Inhalation: Move the exposed person to fresh air. If not breathing, give artificial respiration. If breathing is difficult, give oxygen. Get medical attention if breathing difficulties continue.
Note to Physicians: Ingestion of this product or subsequent vomiting may result in aspiration of light hydrocarbon liquid, which may cause pneumonitis.

SECTION 5 FIRE FIGHTING MEASURES

See Section 7 for proper handling and storage.

FIRE CLASSIFICATION:
OSHA Classification (29 CFR 1910.1200): Flammable liquid.

NFPA RATINGS: Health: 1 Flammability: 3 Reactivity: 0

FLAMMABLE PROPERTIES:
Flashpoint: (Tagliabue Closed Cup) < -45 °C (< -49 °F)
Autoignition: > 280 °C (> 536 °F)
Flammability (Explosive) Limits (% by volume in air): Lower: 1.4 Upper: 7.6

EXTINGUISHING MEDIA: Dry Chemical, CO2, AFFF Foam or alcohol resistant foam if >15% volume polar solvents (oxygenates).

PROTECTION OF FIRE FIGHTERS:
Fire Fighting Instructions: Use water spray to cool fire-exposed containers and to protect personnel. For fires involving this material, do not enter any enclosed or confined fire space without proper protective equipment, including self-contained breathing apparatus.
Combustion Products: Highly dependent on combustion conditions. A complex mixture of airborne solids, liquids, and gases including carbon monoxide, carbon dioxide, and unidentified organic compounds will be evolved when this material undergoes combustion.

SECTION 6 ACCIDENTAL RELEASE MEASURES

Protective Measures: Eliminate all sources of ignition in the vicinity of the spill or released vapor. If this material is released into the work area, evacuate the area immediately. Monitor area with combustible gas indicator.
Spill Management: Stop the source of the release if you can do it without risk. Contain release to prevent further contamination of soil, surface water or groundwater. Clean up spill as soon as possible, observing precautions in Exposure Controls/Personal Protection. Use appropriate techniques such as applying non-combustible absorbent materials or pumping. All equipment used when handling the product must be grounded. A vapor suppressing foam may be used to reduce vapors. Use clean non-sparking tools to collect absorbed material. Where feasible and appropriate, remove contaminated soil. Place contaminated materials in disposable containers and dispose of in a manner consistent with applicable regulations.
Reporting: Report spills to local authorities and/or the U.S. Coast Guard's National Response Center at (800) 424-8802 as appropriate or required. This material is covered by EPA's Comprehensive Environmental Response, Compensation and Liability Act (CERCLA) Petroleum Exclusion. Therefore, releases to the environment may not be reportable under CERCLA.

SECTION 7 HANDLING AND STORAGE

Precautionary Measures: READ AND OBSERVE ALL PRECAUTIONS ON PRODUCT LABEL. This product presents an extreme fire hazard. Liquid very quickly evaporates, even at low temperatures, and forms vapor (fumes) which can catch fire and burn with explosive violence. Invisible vapor spreads easily and can be set on fire by many sources such as pilot lights, welding equipment, and electrical motors and switches. Never siphon gasoline by mouth.
Use only as a motor fuel. Do not use for cleaning, pressure appliance fuel, or any other such use. Do not store in open or unlabeled containers. Do not get in eyes, on skin, or on clothing. Do not taste or swallow. Do not breathe vapor or fumes. Wash thoroughly after handling. Keep out of the reach of children.
Unusual Handling Hazards: WARNING! Do not use as portable heater or appliance fuel. Toxic fumes may accumulate and cause death.
General Handling Information: Avoid contaminating soil or releasing this material into sewage and drainage systems and bodies of water.
Static Hazard: Electrostatic charge may accumulate and create a hazardous condition when handling this material. To minimize this hazard, bonding and grounding may be necessary but may not, by themselves, be sufficient. Review all operations which have the potential of generating an accumulation of electrostatic charge and/or a flammable atmosphere (including tank and container filling, splash filling, tank cleaning, sampling, gauging, switch loading, filtering, mixing, agitation, and vacuum truck operations) and use appropriate mitigating procedures. For more information, refer to OSHA Standard 29 CFR 1910.106, 'Flammable and Combustible Liquids', National Fire Protection Association (NFPA 77, 'Recommended Practice on Static Electricity', and/or the American Petroleum Institute (API) Recommended Practice 2003, 'Protection Against Ignitions Arising Out of Static, Lightning, and Stray Currents'. Improper filling of portable gasoline containers creates danger of fire. Only dispense gasoline into approved and properly labeled gasoline containers. Always place portable containers on the ground. Be sure pump nozzle is in contact with the container while filling. Do not use a nozzle's lock-open device. Do not fill portable containers that are inside a vehicle or truck/trailer bed.

General Storage Information: DO NOT USE OR STORE near heat, sparks or open flames. USE AND STORE ONLY IN WELL VENTILATED AREA. Keep container closed when not in use.
Container Warnings: Container is not designed to contain pressure. Do not use pressure to empty container or it may rupture with explosive force. Empty containers retain product residue (solid, liquid, and/or vapor) and can be dangerous. Do not pressurize, cut, weld, braze, solder, drill, grind, or expose such containers to heat, flame, sparks, static electricity, or other sources of ignition. They may explode and cause injury or death. Empty containers should be completely drained, properly closed, and promptly returned to a drum reconditioner or disposed of properly.

SECTION 8 EXPOSURE CONTROLS/PERSONAL PROTECTION

GENERAL CONSIDERATIONS:
Consider the potential hazards of this material (see Section 3), applicable exposure limits, job activities, and other substances in the work place when designing engineering controls and selecting personal protective equipment. If engineering controls or work practices are not adequate to

prevent exposure to harmful levels of this material, the personal protective equipment listed below is recommended. The user should read and understand all instructions and limitations supplied with the equipment since protection is usually provided for a limited time or under certain circumstances.

ENGINEERING CONTROLS:
Use process enclosures, local exhaust ventilation, or other engineering controls to control airborne levels below the recommended exposure limits.

PERSONAL PROTECTIVE EQUIPMENT
Eye/Face Protection: No special eye protection is normally required. Where splashing is possible, wear safety glasses with side shields as a good safety practice.
Skin Protection: No special protective clothing is normally required. Where splashing is possible, select protective clothing depending on operations conducted, physical requirements and other substances in the workplace. Suggested materials for protective gloves include: Chlorinated Polyethylene (or Chlorosulfonated Polyethylene), Nitrile Rubber, Polyurethane, Viton.
Respiratory Protection: Determine if airborne concentrations are below the recommended exposure limits. If not, wear an approved respirator that provides adequate protection from measured concentrations of this material, such as: Air-Purifying Respirator for Organic Vapors. When used as a fuel, this material can produce carbon monoxide in the exhaust. Determine if airborne concentrations are below the occupational exposure limit for carbon monoxide. If not, wear an approved positive-pressure air-supplying respirator.
Use a positive pressure air-supplying respirator in circumstances where air-purifying respirators may not provide adequate protection.

Occupational Exposure Limits:

Component	Limit	TWA	STEL	Ceiling	Notation
Benzene	ACGIH_TLV	.5 ppm	2.5 ppm		Skin A1
Benzene	OSHA_PEL	1 ppm	5 ppm		
Benzene	OSHA_Z2	10 ppm		25 ppm	
Ethanol	ACGIH_TLV	1000 ppm			A4
Ethanol	OSHA_PEL	1000 ppm			
Ethyl benzene	ACGIH_TLV	100 ppm	125 ppm		A3
Ethyl benzene	OSHA_PEL	100 ppm	125 ppm		
Ethyl tert-butyl ether (ETBE)	ACGIH_TLV	5 ppm			
Gasoline	ACGIH_TLV	300 ppm	500 ppm		A3
Gasoline	OSHA_PEL	300 ppm	500 ppm		
Methyl tert-butyl ether (MTBE)	ACGIH_TLV	50 ppm			A3
Naphthalene	ACGIH_TLV	10 ppm	15 ppm		Skin A4
Naphthalene	OSHA_PEL	10 ppm	15 ppm		
Tertiary amyl methyl ether (TAME)	CHEVRON		50 ppm		

Refer to the OSHA Benzene Standard (29 CFR 1910.1028) and Table Z-2 for detailed training, exposure monitoring, respiratory protection and medical surveillance requirements before using this product.

SECTION 9 PHYSICAL AND CHEMICAL PROPERTIES

Attention: the data below are typical values and do not constitute a specification.

Color: Colorless to yellow
Physical State: Liquid
Odor: Petroleum odor
pH: NA
Vapor Pressure: 5 psi - 15 psi (Typical) @ 37.8°C (100°F)
Vapor Density (Air = 1): 3 - 4 (Typical)
Boiling Point: 37.8°C (100°F) - 204.4°C (400°F) (Typical)
Solubility: Insoluble in water; miscible with most organic solvents.
Freezing Point: NA
Melting Point: NA
Specific Gravity: 0.7 g/ml - 0.8 g/ml @ 15.6°C (60.1°F)
Viscosity: <1 SUS @ 37.8°C (100°F)

SECTION 10 STABILITY AND REACTIVITY

Chemical Stability: This material is considered stable under normal ambient and anticipated storage and handling conditions of temperature and pressure.
Incompatibility With Other Materials: May react with strong oxidizing agents, such as chlorates, nitrates, peroxides, etc.
Hazardous Decomposition Products: None known (None expected)
Hazardous Polymerization: Hazardous polymerization will not occur.

SECTION 11 TOXICOLOGICAL INFORMATION

IMMEDIATE HEALTH EFFECTS
Eye Irritation: The Draize eye irritation mean score in rabbits for a 24-hour exposure was: 0/110.
Skin Irritation: For a 4-hour exposure, the Primary Irritation Index (PII) in rabbits is: 4.8/8.0.
Skin Sensitization: This material did not cause sensitization reactions in a Modified Buehler guinea pig test.
Acute Dermal Toxicity: 24 hour(s) LD50: >3.75g/kg (rabbit).
Acute Oral Toxicity: LD50: >5 ml/kg (rat)
Acute Inhalation Toxicity: 4 hour(s) LD50: >2000ppm (rat).

ADDITIONAL TOXICOLOGY INFORMATION:
Gasolines are highly volatile and can produce significant concentrations of vapor at ambient temperatures. Gasoline vapor is heavier than air and at high concentrations may accumulate in confined spaces to present both safety and heath hazards. When vapor exposures are low, or short duration and infrequent, such as during refuelling and tanker loading/unloading, neither total hydrocarbon nor components such as benzene are likely to result in any adverse health effects. In situations such as accidents or spills where exposure to gasoline vapor is potentially high, attention should be paid to potential toxic effects of specific components. Information about specific components in gasoline can be found in Sections 2, 8 and 15 of this MSDS. More detailed information on the health hazard of specific gasoline components can be obtained calling the Chevron Emergency Information Center (see Section 1 for phone numbers).

NEUROTOXICITY: Pathological misuse of solvents and gasoline, involving repeated and prolonged exposure to high concentrations of vapor is a significant exposure on which there are many reports in the medical literature. As with other solvents, persistent abuse involving repeated and prolonged exposures to high concentrations of vapor has been reported to result in central nervous system damage and eventually, death. In a study in which ten human volunteers were exposed for 30 minutes to approximately 200, 500 or 1000 ppm concentrations of gasoline vapor, irritation of the eyes was the only significant effect observed, based on both subjective and objective assessments. In an inhalation study, groups of 6 Fischer rats (3 male. 3 female) were exposed to 2056 ppm of wholy vaporized unleaded gasoline for 6 hours perday, 5 day per week for up to 18 months. Histopathology of the peripheral nervous system and spinal cord revealed no distal axonal neuropthy of the type associated with exposure to n-hexane even though gasoline contained 1.9% n-hexane. The authors concluded that gasoline treatment may have amplified the incidence and prominence of some naturally occurring age-related (subclinical) in the nervous system. BIRTH DEFECTS AND REPRODUCTIVE TOXICITY: An inhalation study with rats exposed to 0, 400 and 1600 ppm of wholly vaporized unleaded gasoline, 6 hours per day on day 6 through 16 of gestation, showed no teratogenic effects nor indication of toxicity to either the mother or the fetus. Another inhalation study in rats exposed to 3000, 6000, or 9000 ppm of gasoline vapor, 6 hours per day on day 6 through 20 of gestation, also showed no teratogenic effects nor indications of toxicity to either the mother or the fetus.

CHRONIC TOXICITY/CANCER: Wholly vaporized unleaded gasoline was used in a 3 month inhalation study. Groups of 40 rats (20 males, 20 female) and 8 squirrel monkeys (4 male, 4 female) were exposed 6 hours per day and 5 days per week for 13 weeks to 384 or 1552 ppm gasoline. One group of each species served as unexposed controls. The initial conclusion of this study was that inhalation of gasoline at airborne concentrations of up to 1522 ppm caused no toxicity in rats or monkeys. However, further histopathological examination of male rat kidneys on the highest dose group revealed an increased incidence and severity of regenerative epithelium and dilated tubules containing proteinaceous deposits. Lifetime inhalation of wholly vaporized unleaded gasoline at 2056 ppm has caused increased liver tumors in female mice. The mechanism of this response is still being investigated but it is thought to be an epigenetic process unique to the female mouse.

This exposure also caused kidney damage and eventually kidney cancer in male rats. No other animal model studied has shown these adverse

kidney effects and there is no physiological reason to believe that they would occur in man. EPA has concluded that mechanism by which wholly vaporzied unleaded gasoline causes kidney damage is unque to the male rat. The effects in that species (kidney damage and cancer) should not be used in human risk assesment. In their 1988 review of carcinogenic risk from gasoline, The International Agency for Research on Cancer (IARC) noted that, because published epidemiology studies did not include any exposure data, only occupations where gasoline exposure may have ocurred were reviewed. These included gasoline service station attendants and automobile mechanics. IARC also noted that there was no opportunity to separate effects of combustion products from those of gasoline itself. Although IARC allocated gasoline a final overall classification of Group 2B, i.e. possibly carcinogenic to humans, this was based on limited evidence in experimental animals plus supporting evidence including the presence in gasoline of benzene and 1, 3-butadiene. The actual evidence for carcinogenicity in humans was considered inadequate.

MUTAGENICITY: Gasoline was not mutagenic, with or without activation, in the Ames assay (Salmonella typhimurium), Saccharamyces cerevisesae, or mouse lymphoma assays. In addition, point mutations were not induced in human lymphocytes. Gasoline was not mutagenic when tested in the mouse dominant lethal assay. Administration of gasoline to rats did not cause chomosomal aberrations in their bone marrow cells. EPIDEMIOLOGY: To explore the health effects of workers potentially exposed to gasoline vapors in the marketing and distribution sectors of the petroleum industry, the American Petroleum Institute sponsored a cohort mortality study (Publication 4555), a nested case-control study (Publication 4551), and an exposure assessment study (Publication 4552). Histories of exposure to gasoline were reconstructed for cohort of more than 18,000 employees from four companies for the time period between 1946 and 1985. The results of the cohort mortality study indicated that there was no increased mortality from either kidney cancer or leukemia among marketing and marine distribution employees who were exposed to gasoline in the petroleum industry, when compared to the general population. More importantly, based on internal comparisons, there was no association between mortality from kidney cancer or leukemia and various indices of gasoline exposure. In particular, neither duration of employment, duration of exposure, age at first exposure, year of first exposure, job category, cumulative exposure, frequency of peak exposure, nor average intensity of exposure had any effect on kidney cancer or leukemia mortality. The results of the nested case-control study confirmed the findings of the original cohort study. That is, exposure to gasoline at the levels experienced by this cohort of distribution workers is not a significant risk factor for leukemia (all cell types), acute myeloid leukemia, kidney cancer or multiple myeloma.

SECTION 12 ECOLOGICAL INFORMATION

ECOTOXICITY

The 96 hour(s) LC50 for rainbow trout (Oncorhynchus mykiss) is 2.7 mg/l.
The 48 hour(s) LC50 for water flea (Daphnia magna) is 3.0 mg/l.
The 96 hour(s) LC50 for sheepshead minnow (Cyprinodon variegatus) is 8.3 mg/l.
The 96 hour(s) LC50 for mysid shrimp (Mysidopsis bahia) is 1.8 mg/l.
This material is expected to be toxic to aquatic organisms. Gasoline studies have been conducted in the laboratory under a variety of test conditions with a range of fish and invertebrate species. An even more extensive database is available on the aquatic toxicity of individual aromatic constituents. The majority of published studies do not identify the type of gasoline evaluated, or even provide distinguishing characteristics such as aromatic content or presence of lead alkyls. As a result, comparison of results among studies using open and closed vessels, different ages and species of test animals and different gasoline types, is difficult.

The bulk of the available literature on gasoline relates to the environmental impact of monoaromatic (BTEX) and diaromatic (naphthalene, methylnaphthalenes) constituents. In general, non-oxygenated gasoline exhibits some short-term toxicity to freshwater and marine organisms, especially under closed vessel or flow-through exposure conditions in the laboratory. The components which are the most prominent in the water soluble fraction and cause aquatic toxicity, are also highly volatile and can be readily biodegraded by microorganisms.

ENVIRONMENTAL FATE

This material is expected to be readily biodegradable. Following spillage, the more volatile components of gasoline will be rapidly lost, with concurrent dissolution of these and other constituents into the water. Factors such as local environmental conditions (temperature, wind, mixing or wave action, soil type, etc), photo-oxidation, biodegradation and adsorption onto suspended sediments, can contribute to the weathering of spilled gasoline.

The aqueous solubility of non-oxygenated unleaded gasoline, based on analysis of benzene, toluene, ethylbenzene+xylenes and naphthalene, is reported to be 112 mg/l. Solubility data on individual gasoline constituents also available.

SECTION 13 DISPOSAL CONSIDERATIONS

Use material for its intended purpose or recycle if possible. This material, if it must be discarded, may meet the criteria of a hazardous waste as defined by US EPA under RCRA (40 CFR 261) or other State and local regulations. Measurement of certain physical properties and analysis for regulated components may be necessary to make a correct determination. If this material is classified as a hazardous waste, federal law requires disposal at a licensed hazardous waste disposal facility.

SECTION 14 TRANSPORT INFORMATION

The description shown may not apply to all shipping situations. Consult 49CFR, or appropriate Dangerous Goods Regulations, for additional description requirements (e.g., technical name) and mode-specific or quantity-specific shipping requirements.

DOT Shipping Name: GASOLINE
DOT Hazard Class: 3 (Flammable Liquid)
DOT Identification Number: UN1203
DOT Packing Group: II

SECTION 15 REGULATORY INFORMATION

SARA 311/312 CATEGORIES:
1. Immediate (Acute) Health Effects: YES
2. Delayed (Chronic) Health Effects: YES
3. Fire Hazard: YES
4. Sudden Release of Pressure Hazard: NO
5. Reactivity Hazard: NO

REGULATORY LISTS SEARCHED:

4_I1=IARC Group 1	15=SARA Section 313
4_I2A=IARC Group 2A	16=CA Proposition 65
4_I2B=IARC Group 2B	17=MA RTK
05=NTP Carcinogen	18=NJ RTK
06=OSHA Carcinogen	19=DOT Marine Pollutant
09=TSCA 12(b)	20=PA RTK

The following components of this material are found on the regulatory lists indicated.

Benzene	15, 16, 17, 18, 20, 4_I1, 5, 6
Ethanol	17, 18, 20
Ethyl benzene	15, 17, 18, 20, 4_I2B
Gasoline	17, 18, 20
Methyl tert-butyl ether (MTBE)	15, 17, 18, 20, 9
Naphthalene	15, 16, 17, 18, 20, 4_I2B
Tertiary amyl methyl ether (TAME)	9

CERCLA REPORTABLE QUANTITIES(RQ)/SARA 302 THRESHOLD PLANNING QUANTITIES(TPQ):

Component	Component RQ	Component TPQ	Product RQ
Benzene	10 lbs	None	186 lbs
Ethanol	100 lbs	None	1961 lbs

Ethyl benzene	1000 lbs	None	34964 lbs
Methyl tert-butyl ether (MTBE)	1000 lbs	None	7513 lbs
Naphthalene	100 lbs	None	4000 lbs

CHEMICAL INVENTORIES:
CANADA: All the components of this material are on the Canadian DSL or have been notified under the New Substance Notification Regulations, but have not yet been published in the Canada Gazette.
UNITED STATES: All of the components of this material are on the Toxic Substances Control Act (TSCA) Chemical Inventory.

WHMIS CLASSIFICATION:
Class B, Division 2: Flammable Liquids
Class D, Division 2, Subdivision A: Very Toxic Material - Carcinogenicity
Class D, Division 2, Subdivision B: Toxic Material - Skin or Eye Irritation

SECTION 16 OTHER INFORMATION

NFPA RATINGS: Health: 1 Flammability: 3 Reactivity: 0

(0-Least, 1-Slight, 2-Moderate, 3-High, 4-Extreme, PPE:- Personal Protection Equipment Index recommendation, *- Chronic Effect Indicator). These values are obtained using the guidelines or published evaluations prepared by the National Fire Protection Association (NFPA) or the National Paint and Coating Association (for HMIS ratings).

Additional Product Number(s): CPS201023, CPS201054, CPS201055, CPS201075, CPS201090, CPS201105, CPS201106, CPS201120, CPS201121, CPS201122, CPS201126, CPS201128, CPS201131, CPS201136, CPS201141, CPS201142, CPS201148, CPS201153, CPS201158, CPS201161, CPS201162, CPS201168, CPS201181, CPS201185, CPS201186, CPS201188, CPS201216, CPS201217, CPS201218, CPS201236, CPS201237, CPS201238, CPS201266, CPS201267, CPS201268, CPS201277, CPS201278, CPS201279, CPS201286, CPS201287, CPS201289, CPS201296, CPS201297, CPS201298, CPS201849, CPS201850, CPS201855, CPS201856, CPS201857, CPS204000, CPS204001, CPS204002, CPS204003, CPS204010, CPS204011, CPS204022, CPS204023, CPS204046, CPS204047, CPS204070, CPS204071, CPS204088, CPS204089, CPS204104, CPS204105, CPS204116, CPS204117, CPS204140, CPS204141, CPS204164, CPS204165, CPS204188, CPS204189, CPS204200, CPS204201, CPS204212, CPS204213, CPS204224, CPS204225, CPS204248, CPS204249, CPS204272, CPS204273, CPS204290, CPS204291, CPS204322, CPS204323, CPS204324, CPS204350, CPS204352, CPS204354, CPS204356, CPS204358, CPS204359, CPS204364, CPS204365, CPS204370, CPS204371, CPS204376, CPS204377, CPS204382, CPS204383, CPS204388, CPS204389, CPS204394, CPS204395, CPS204400, CPS204401, CPS204406, CPS204407, CPS204412, CPS204413, CPS204418, CPS204419, CPS204424, CPS204425, CPS204430, CPS204431, CPS204436, CPS204437, CPS204442, CPS204446, CPS204450, CPS204454, CPS204458, CPS204462, CPS204466, CPS204467, CPS204484, CPS204485, CPS204502, CPS204503, CPS204520, CPS204521, CPS204538, CPS204539, CPS204556, CPS204557, CPS204574, CPS204575, CPS204592, CPS204593, CPS204610, CPS204611, CPS204628, CPS204629, CPS204646, CPS204647, CPS204664, CPS204665, CPS204682, CPS204690, CPS204691, CPS204696, CPS204697, CPS204702, CPS204703, CPS204708, CPS204709, CPS204721, CPS204722, CPS204727, CPS204728, CPS241765

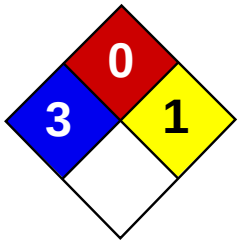
REVISION STATEMENT: This revision updates the following sections of this Material Safety Data Sheet: Section 1 (Product Codes). This Material Safety Data Sheet has been prepared using the ProSteward MSDS system.

ABBREVIATIONS THAT MAY HAVE BEEN USED IN THIS DOCUMENT:

TLV	-	Threshold Limit Value	TWA	-	Time Weighted Average
STEL	-	Short-term Exposure Limit	PEL	-	Permissible Exposure Limit
			CAS	-	Chemical Abstract Service Number
NDA	-	No Data Available	NA	-	Not Applicable
<=	-	Less Than or Equal To	>=	-	Greater Than or Equal To

Prepared according to the OSHA Hazard Communication Standard (29 CFR 1910.1200) and the ANSI MSDS Standard (Z400.1) by the ChevronTexaco Energy Research & Technology Company, 100 Chevron Way, Richmond, California 94802.

The above information is based on the data of which we are aware and is believed to be correct as of the date hereof. Since this information may be applied under conditions beyond our control and with which we may be unfamiliar and since data made available subsequent to the date hereof may suggest modifications of the information, we do not assume any responsibility for the results of its use. This information is furnished upon condition that the person receiving it shall make his own determination of the suitability of the material for his particular purpose.





Material Safety Data Sheet
Hydrochloric acid MSDS

Section 1: Chemical Product and Company Identification

Product Name: Hydrochloric acid

Catalog Codes: SLH1462, SLH3154

CAS#: Mixture.

RTECS: MW4025000

TSCA: TSCA 8(b) inventory: Hydrochloric acid

CI#: Not applicable.

Synonym: Hydrochloric Acid; Muriatic Acid

Chemical Name: Not applicable.

Chemical Formula: Not applicable.

Contact Information:

Sciencelab.com, Inc.
14025 Smith Rd.
Houston, Texas 77396

US Sales: **1-800-901-7247**
International Sales: **1-281-441-4400**

Order Online: ScienceLab.com

CHEMTREC (24HR Emergency Telephone), call:
1-800-424-9300

International CHEMTREC, call: 1-703-527-3887

For non-emergency assistance, call: 1-281-441-4400

Section 2: Composition and Information on Ingredients

Composition:

Name	CAS #	% by Weight
Hydrogen chloride	7647-01-0	20-38
Water	7732-18-5	62-80

Toxicological Data on Ingredients: Hydrogen chloride: GAS (LC50): Acute: 4701 ppm 0.5 hours [Rat].

Section 3: Hazards Identification

Potential Acute Health Effects:
Very hazardous in case of skin contact (corrosive, irritant, permeator), of eye contact (irritant, corrosive), of ingestion, . Slightly hazardous in case of inhalation (lung sensitizer). Non-corrosive for lungs. Liquid or spray mist may produce tissue damage particularly on mucous membranes of eyes, mouth and respiratory tract. Skin contact may produce burns. Inhalation of the spray mist may produce severe irritation of respiratory tract, characterized by coughing, choking, or shortness of breath. Severe over-exposure can result in death. Inflammation of the eye is characterized by redness, watering, and itching. Skin inflammation is characterized by itching, scaling, reddening, or, occasionally, blistering.

Potential Chronic Health Effects:
Slightly hazardous in case of skin contact (sensitizer). CARCINOGENIC EFFECTS: Classified 3 (Not classifiable for human.) by IARC [Hydrochloric acid]. MUTAGENIC EFFECTS: Not available. TERATOGENIC EFFECTS: Not available. DEVELOPMENTAL TOXICITY: Not available. The substance may be toxic to kidneys, liver, mucous membranes, upper respiratory tract, skin, eyes, Circulatory System, teeth. Repeated or prolonged exposure to the substance can produce target

organs damage. Repeated or prolonged contact with spray mist may produce chronic eye irritation and severe skin irritation. Repeated or prolonged exposure to spray mist may produce respiratory tract irritation leading to frequent attacks of bronchial infection. Repeated exposure to a highly toxic material may produce general deterioration of health by an accumulation in one or many human organs.

Section 4: First Aid Measures

Eye Contact:
Check for and remove any contact lenses. In case of contact, immediately flush eyes with plenty of water for at least 15 minutes. Cold water may be used. Get medical attention immediately.

Skin Contact:
In case of contact, immediately flush skin with plenty of water for at least 15 minutes while removing contaminated clothing and shoes. Cover the irritated skin with an emollient. Cold water may be used.Wash clothing before reuse. Thoroughly clean shoes before reuse. Get medical attention immediately.

Serious Skin Contact:
Wash with a disinfectant soap and cover the contaminated skin with an anti-bacterial cream. Seek immediate medical attention.

Inhalation:
If inhaled, remove to fresh air. If not breathing, give artificial respiration. If breathing is difficult, give oxygen. Get medical attention immediately.

Serious Inhalation:
Evacuate the victim to a safe area as soon as possible. Loosen tight clothing such as a collar, tie, belt or waistband. If breathing is difficult, administer oxygen. If the victim is not breathing, perform mouth-to-mouth resuscitation. WARNING: It may be hazardous to the person providing aid to give mouth-to-mouth resuscitation when the inhaled material is toxic, infectious or corrosive. Seek immediate medical attention.

Ingestion:
If swallowed, do not induce vomiting unless directed to do so by medical personnel. Never give anything by mouth to an unconscious person. Loosen tight clothing such as a collar, tie, belt or waistband. Get medical attention immediately.

Serious Ingestion: Not available.

Section 5: Fire and Explosion Data

Flammability of the Product: Non-flammable.

Auto-Ignition Temperature: Not applicable.

Flash Points: Not applicable.

Flammable Limits: Not applicable.

Products of Combustion: Not available.

Fire Hazards in Presence of Various Substances: of metals

Explosion Hazards in Presence of Various Substances: Non-explosive in presence of open flames and sparks, of shocks.

Fire Fighting Media and Instructions: Not applicable.

Special Remarks on Fire Hazards:
Non combustible. Calcium carbide reacts with hydrogen chloride gas with incandescence. Uranium phosphide reacts with hydrochloric acid to release spontaneously flammable phosphine. Rubidium acetylene carbides burns with slightly warm hydrochloric acid. Lithium silicide in contact with hydrogen chloride becomes incandescent. When dilute hydrochloric acid is used, gas spontaneously flammable in air is evolved. Magnesium boride treated with concentrated hydrochloric acid produces spontaneously flammble gas. Cesium acetylene carbide burns hydrogen chloride gas. Cesium carbide ignites in contact with hydrochloric acid unless acid is dilute. Reacts with most metals to produce flammable Hydrodgen gas.

Special Remarks on Explosion Hazards:

Hydrogen chloride in contact with the following can cause an explosion, ignition on contact, or other violent/vigorous reaction: Acetic anhydride AgClO + CCl4 Alcohols + hydrogen cyanide, Aluminum Aluminum-titanium alloys (with HCl vapor), 2-Amino ethanol, Ammonium hydroxide, Calcium carbide Ca3P2 Chlorine + dinitroanilines (evolves gas), Chlorosulfonic acid Cesium carbide Cesium acetylene carbide, 1,1-Difluoroethylene Ethylene diamine Ethylene imine, Fluorine, HClO4 Hexalithium disilicide H2SO4 Metal acetylides or carbides, Magnesium boride, Mercuric sulfate, Oleum, Potassium permanganate, beta-Propiolactone Propylene oxide Rubidium carbide, Rubidium, acetylene carbide Sodium (with aqueous HCl), Sodium hydroxide Sodium tetraselenium, Sulfonic acid, Tetraselenium tetranitride, U3P4 , Vinyl acetate. Silver perchlorate with carbon tetrachloride in the presence of hydrochloric acid produces trichloromethyl perchlorate which detonates at 40 deg. C.

Section 6: Accidental Release Measures

Small Spill:
Dilute with water and mop up, or absorb with an inert dry material and place in an appropriate waste disposal container. If necessary: Neutralize the residue with a dilute solution of sodium carbonate.

Large Spill:
Corrosive liquid. Poisonous liquid. Stop leak if without risk. Absorb with DRY earth, sand or other non-combustible material. Do not get water inside container. Do not touch spilled material. Use water spray curtain to divert vapor drift. Use water spray to reduce vapors. Prevent entry into sewers, basements or confined areas; dike if needed. Call for assistance on disposal. Neutralize the residue with a dilute solution of sodium carbonate. Be careful that the product is not present at a concentration level above TLV. Check TLV on the MSDS and with local authorities.

Section 7: Handling and Storage

Precautions:
Keep locked up.. Keep container dry. Do not ingest. Do not breathe gas/fumes/ vapor/spray. Never add water to this product. In case of insufficient ventilation, wear suitable respiratory equipment. If ingested, seek medical advice immediately and show the container or the label. Avoid contact with skin and eyes. Keep away from incompatibles such as oxidizing agents, organic materials, metals, alkalis, moisture. May corrode metallic surfaces. Store in a metallic or coated fiberboard drum using a strong polyethylene inner package.

Storage: Keep container tightly closed. Keep container in a cool, well-ventilated area.

Section 8: Exposure Controls/Personal Protection

Engineering Controls:
Provide exhaust ventilation or other engineering controls to keep the airborne concentrations of vapors below their respective threshold limit value. Ensure that eyewash stations and safety showers are proximal to the work-station location.

Personal Protection:
Face shield. Full suit. Vapor respirator. Be sure to use an approved/certified respirator or equivalent. Gloves. Boots.

Personal Protection in Case of a Large Spill:
Splash goggles. Full suit. Vapor respirator. Boots. Gloves. A self contained breathing apparatus should be used to avoid inhalation of the product. Suggested protective clothing might not be sufficient; consult a specialist BEFORE handling this product.

Exposure Limits:
CEIL: 5 (ppm) from OSHA (PEL) [United States] CEIL: 7 (mg/m3) from OSHA (PEL) [United States] CEIL: 5 from NIOSH CEIL: 7 (mg/m3) from NIOSH TWA: 1 STEL: 5 (ppm) [United Kingdom (UK)] TWA: 2 STEL: 8 (mg/m3) [United Kingdom (UK)]Consult local authorities for acceptable exposure limits.

Section 9: Physical and Chemical Properties

Physical state and appearance: Liquid.

Odor: Pungent. Irritating (Strong.)

Taste: Not available.

Molecular Weight: Not applicable.

Color: Colorless to light yellow.

pH (1% soln/water): Acidic.

Boiling Point:
108.58 C @ 760 mm Hg (for 20.22% HCl in water) 83 C @ 760 mm Hg (for 31% HCl in water) 50.5 C (for 37% HCl in water)

Melting Point:
-62.25°C (-80°F) (20.69% HCl in water) -46.2 C (31.24% HCl in water) -25.4 C (39.17% HCl in water)

Critical Temperature: Not available.

Specific Gravity:
1.1- 1.19 (Water = 1) 1.10 (20%and 22% HCl solutions) 1.12 (24% HCl solution) 1.15 (29.57% HCl solution) 1.16 (32% HCl solution) 1.19 (37% and 38%HCl solutions)

Vapor Pressure: 16 kPa (@ 20°C) average

Vapor Density: 1.267 (Air = 1)

Volatility: Not available.

Odor Threshold: 0.25 to 10 ppm

Water/Oil Dist. Coeff.: Not available.

Ionicity (in Water): Not available.

Dispersion Properties: See solubility in water, diethyl ether.

Solubility: Soluble in cold water, hot water, diethyl ether.

Section 10: Stability and Reactivity Data

Stability: The product is stable.

Instability Temperature: Not available.

Conditions of Instability: Incompatible materials, water

Incompatibility with various substances:
Highly reactive with metals. Reactive with oxidizing agents, organic materials, alkalis, water.

Corrosivity:
Extremely corrosive in presence of aluminum, of copper, of stainless steel(304), of stainless steel(316). Non-corrosive in presence of glass.

Special Remarks on Reactivity:
Reacts with water especially when water is added to the product. Absorption of gaseous hydrogen chloride on mercuric sulfate becomes violent @ 125 deg. C. Sodium reacts very violently with gaseous hydrogen chloride. Calcium phosphide and hydrochloric acid undergo very energetic reaction. It reacts with oxidizers releasing chlorine gas. Incompatible with, alkali metals, carbides, borides, metal oxides, vinyl acetate, acetylides, sulphides, phosphides, cyanides, carbonates. Reacts with most metals to produce flammable Hydrogen gas. Reacts violently (moderate reaction with heat of evolution) with water especially when water is added to the product. Isolate hydrogen chloride from heat, direct sunlight, alkalies (reacts vigorously), organic materials, and oxidizers (especially nitric acid and chlorates), amines, metals, copper and alloys (e.g. brass), hydroxides, zinc (galvanized materials), lithium silicide (incandescence), sulfuric acid(increase in temperature and pressure) Hydrogen chloride gas is emitted when this product is in contact with sulfuric acid. Adsorption of Hydrochloric Acid onto silicon dioxide results in exothmeric reaction. Hydrogen chloride causes aldehydes and epoxides to violently polymerize. Hydrogen chloride or Hydrochloric Acid in contact with the folloiwng can cause explosion or ignition on contact or

Special Remarks on Corrosivity:

Highly corrosive. Incompatible with copper and copper alloys. It attacks nearly all metals (mercury, gold, platinium, tantalum, silver, and certain alloys are exceptions). It is one of the most corrosive of the nonoxidizing acids in contact with copper alloys. No corrosivity data on zinc, steel. Severe Corrosive effect on brass and bronze

Polymerization: Will not occur.

Section 11: Toxicological Information

Routes of Entry: Absorbed through skin. Dermal contact. Eye contact. Inhalation.

Toxicity to Animals:
Acute oral toxicity (LD50): 900 mg/kg [Rabbit]. Acute toxicity of the vapor (LC50): 1108 ppm, 1 hours [Mouse]. Acute toxicity of the vapor (LC50): 3124 ppm, 1 hours [Rat].

Chronic Effects on Humans:
CARCINOGENIC EFFECTS: Classified 3 (Not classifiable for human.) by IARC [Hydrochloric acid]. May cause damage to the following organs: kidneys, liver, mucous membranes, upper respiratory tract, skin, eyes, Circulatory System, teeth.

Other Toxic Effects on Humans:
Very hazardous in case of skin contact (corrosive, irritant, permeator), of ingestion, . Hazardous in case of eye contact (corrosive), of inhalation (lung corrosive).

Special Remarks on Toxicity to Animals:
Lowest Published Lethal Doses (LDL/LCL) LDL [Man] -Route: Oral; 2857 ug/kg LCL [Human] - Route: Inhalation; Dose: 1300 ppm/30M LCL [Rabbit] - Route: Inhalation; Dose: 4413 ppm/30M

Special Remarks on Chronic Effects on Humans:
May cause adverse reproductive effects (fetoxicity). May affect genetic material.

Special Remarks on other Toxic Effects on Humans:
Acute Potential Health Effects: Skin: Corrosive. Causes severe skin irritation and burns. Eyes: Corrosive. Causes severe eye irritation/conjuntivitis, burns, corneal necrosis. Inhalation: May be fatal if inhaled. Material is extremely destructive to tissue of the mucous membranes and upper respiratory tract. Inhalation of hydrochloric acid fumes produces nose, throat, and larryngeal burning, and irritation, pain and inflammation, coughing, sneezing, choking sensation, hoarseness, laryngeal spasms, upper respiratory tract edema, chest pains, as well has headache, and palpitations. Inhalation of high concentrations can result in corrosive burns, necrosis of bronchial epithelium, constriction of the larynx and bronchi, nasospetal perforation, glottal closure, occur, particularly if exposure is prolonged. May affect the liver. Ingestion: May be fatal if swallowed. Causes irritation and burning, ulceration, or perforation of the gastrointestinal tract and resultant peritonitis, gastric hemorrhage and infection. Can also cause nausea, vomitting (with "coffee ground" emesis), diarrhea, thirst, difficulty swallowing, salivation, chills, fever, uneasiness, shock, strictures and stenosis (esophogeal, gastric, pyloric). May affect behavior (excitement), the cardiovascular system (weak rapid pulse, tachycardia), respiration (shallow respiration), and urinary system (kidneys- renal failure, nephritis). Acute exposure via inhalation or ingestion can also cause erosion of tooth enamel. Chronic Potential Health Effects: dyspnea, bronchitis. Chemical pneumonitis and pulmonary edema can also

Section 12: Ecological Information

Ecotoxicity: Not available.

BOD5 and COD: Not available.

Products of Biodegradation:
Possibly hazardous short term degradation products are not likely. However, long term degradation products may arise.

Toxicity of the Products of Biodegradation: The products of degradation are less toxic than the product itself.

Special Remarks on the Products of Biodegradation: Not available.

Section 13: Disposal Considerations

Waste Disposal:

Waste must be disposed of in accordance with federal, state and local environmental control regulations.

Section 14: Transport Information

DOT Classification: Class 8: Corrosive material

Identification: : Hydrochloric acid, solution UNNA: 1789 PG: II

Special Provisions for Transport: Not available.

Section 15: Other Regulatory Information

Federal and State Regulations:
Connecticut hazardous material survey.: Hydrochloric acid Illinois toxic substances disclosure to employee act: Hydrochloric acid Illinois chemical safety act: Hydrochloric acid New York release reporting list: Hydrochloric acid Rhode Island RTK hazardous substances: Hydrochloric acid Pennsylvania RTK: Hydrochloric acid Minnesota: Hydrochloric acid Massachusetts RTK: Hydrochloric acid Massachusetts spill list: Hydrochloric acid New Jersey: Hydrochloric acid New Jersey spill list: Hydrochloric acid Louisiana RTK reporting list: Hydrochloric acid Louisiana spill reporting: Hydrochloric acid California Director's List of Hazardous Substances: Hydrochloric acid TSCA 8(b) inventory: Hydrochloric acid TSCA 4(a) proposed test rules: Hydrochloric acid SARA 302/304/311/312 extremely hazardous substances: Hydrochloric acid SARA 313 toxic chemical notification and release reporting: Hydrochloric acid CERCLA: Hazardous substances.: Hydrochloric acid: 5000 lbs. (2268 kg)

Other Regulations:
OSHA: Hazardous by definition of Hazard Communication Standard (29 CFR 1910.1200). EINECS: This product is on the European Inventory of Existing Commercial Chemical Substances.

Other Classifications:

WHMIS (Canada):
CLASS D-2A: Material causing other toxic effects (VERY TOXIC). CLASS E: Corrosive liquid.

DSCL (EEC):
R34- Causes burns. R37- Irritating to respiratory system. S26- In case of contact with eyes, rinse immediately with plenty of water and seek medical advice. S45- In case of accident or if you feel unwell, seek medical advice immediately (show the label where possible).

HMIS (U.S.A.):

Health Hazard: 3

Fire Hazard: 0

Reactivity: 1

Personal Protection:

National Fire Protection Association (U.S.A.):

Health: 3

Flammability: 0

Reactivity: 1

Specific hazard:

Protective Equipment:
Gloves. Full suit. Vapor respirator. Be sure to use an approved/certified respirator or equivalent. Wear appropriate respirator when ventilation is inadequate. Face shield.

Section 16: Other Information

References:
-Hawley, G.G.. The Condensed Chemical Dictionary, 11e ed., New York N.Y., Van Nostrand Reinold, 1987. -SAX, N.I. Dangerous Properties of Indutrial Materials. Toronto, Van Nostrand Reinold, 6e ed. 1984. -The Sigma-Aldrich Library of Chemical Safety Data, Edition II. -Guide de la loi et du règlement sur le transport des marchandises dangeureuses au canada. Centre de conformité internatinal Ltée. 1986.

Other Special Considerations: Not available.

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MATERIAL SAFETY DATA SHEET

This Material Safety Data Sheet (MSDS) complies with the United Nations Globally Harmonized System (GHS) of Classification and Labeling, Second revised Edition and meets or exceeds the Canadian and United States Requirements for Hazard Communication

1. Product and Supplier Identification

Product Name: Methanol
GHS Product Identifier: Methanol

Recommended Use: Solvent, fuel, feedstock

Restrictions on Use: Do not use in a confined area without proper ventilation. Contact lenses may cause further damage in case of splash into eye. Avoid use near heat, flames, sparks, and other sources of ignition.

Product:	**Methanol (CH_3OH)**	**Methanex Tel. #:** (604) 661-2600
Synonyms:	Methanol, methyl hydrate, wood spirit, methyl hydroxide	**Emergency Tel. #:** 1-800-262-8200 **(CHEMTREC)** (Canada and USA)
Company Identification:	**Methanex Corporation 1800 Waterfront Centre 200 Burrard Street Vancouver, B.C. V6C 3M1**	
Importer:	**Methanex Methanol Company 15301 Dallas Parkway, Suite 900 Addison, Texas 75001 Telephone: (972) 702-0909**	

2. Hazards Identification

Classification: Flammable Liquid, Category 1, Acute Toxicity Category 1*, Reproductive Toxicity 1B, Specific Target Organ Toxicity (Repeated Exposure)

Label:



Hazard Communication: DANGER! Extremely flammable liquid and vapour. Fatal if swallowed.
May damage fertility or the unborn child (fetotoxic and teratogenic effects).
May cause damage to eyes and central nervous system if ingested or inhaled.

***Note:** Assigned to classification based on human experience rather than the strict application of classification criteria set out in the Recommendations on the Transport of Dangerous Goods, Model Regulations Special Provision 279.

Hazards: Colourless liquid, with a mild, characteristic alcohol odour when pure. Crude methanol may have a repulsive, pungent odour. Hygroscopic (moisture absorbing).

***FLAMMABLE LIQUID AND VAPOUR*:** Burns with a clean, clear flame, which is almost invisible in daylight, or a light blue flame. Can decompose at high temperatures forming carbon monoxide and formaldehyde.

Confined space toxicity hazard. Mild central nervous system depressant following inhalation, skin absorption or ingestion. May cause headache, nausea, dizziness, drowsiness, and un-coordination. Severe vision effects, including increased sensitivity to light, blurred vision, and blindness may develop following an 8-24 hour symptom-free period. Coma and death may result.

IRRITANT: Causes eye irritation. Aspiration hazard. Swallowing or vomiting of the liquid may result in aspiration (breathing) into the lungs.

POSSIBLE REPRODUCTIVE HAZARD: May cause fetotoxic (toxic to the fetus during the latter stages of pregnancy, often through the placenta) and teratogenic effects (causing malformations of the fetus), based on animal information.

NFPA Ratings: (Health, Fire, Reactivity): 1, 3, 0

3. Composition

Component	% (w/w)	Exposure Limits (ACGIH)*	LD_{50}	LC_{50}
Methanol (CAS 67-56-1)	99-100	ACGIH* TLV-TWA: 200 ppm, skin; TLV-STEL: 250 ppm, skin PEL-TWA: 200 ppm, skin PEL-STEL: 250 ppm, skin IDLH: 6000 ppm, acute inhalation toxicity to animals TLV Basis, critical effects: neuropathy, vision, central nervous system(CNS)	5628 mg/kg (oral/rat) 15800 mg/kg (dermal/ rabbit)	64000 ppm (inhalation/rat)

* Exposure limits may vary from time to time and from one jurisdiction to another. Check with local regulatory agency for the exposure limits in your area. ACGIH, American Conference of Governmental Industrial Hygienists.

4. First Aid Measures

Note: *Emergency assistance may also be available from the local poison control centre.*

Eye Contact: Remove contact lenses if worn. In case of contact, immediately flush eyes with plenty of clean running water for at least 15 minutes, lifting the upper and lower eyelids occasionally. Obtain medical attention.

Skin Contact: In case of contact, remove contaminated clothing. In a shower, wash affected areas with soap and water for at least 15 minutes. Seek medical attention if irritation occurs or persists. Wash clothing before reuse. Prolonged contact with methanol may defat skin tissue, resulting in drying and cracking.

Inhalation: Remove to fresh air, restore or assist breathing if necessary. Obtain medical attention.

Ingestion: Swallowing methanol is potentially life threatening. Onset of symptoms may be delayed for 18 to 24 hours after digestion. If conscious and medical aid is not immediately available, do not induce vomiting. In actual or suspected cases of ingestion, transport to medical facility immediately.

NOTE TO PHYSICIAN: Acute exposure to methanol, either through ingestion or breathing high airborne concentrations can result in symptoms appearing between 40 minutes and 72 hours after exposure. Symptoms and signs are usually limited to the Central Nervous System (CNS), eyes and gastrointestinal tract. Because of the initial CNS's effects of headache, vertigo, lethargy and confusion, there may be an impression of ethanol intoxication. Blurred vision, decreased acuity and photophobia are common complaints. Treatment with ipecac or lavage is indicated in any patient presenting within two hours of ingestion. A profound metabolic acidosis occurs in severe poisoning and serum bicarbonate levels are a

more accurate measure of severity than serum methanol levels. Treatment protocols are available from most major hospitals and early collaboration with appropriate hospitals is recommended.

Ethanol significantly decreases the toxicity of methanol because it competes for the same metabolic enzymes, and has been used to treat methanol poisoning.

5. Fire Fighting Measures

Suitable Extinguishing Media: Extinguishing Media: Small fires: Dry chemical, CO_2, water spray
Large fires: Water spray(see note in Unsuitable Extinguishing Media), AFFF(R) (Aqueous Film Forming Foam (alcohol resistant)) type with either a 3% or 6% foam proportioning system.

Unsuitable Extinguishing Media: General purpose synthetic foams or protein foams may work, but much less effectively. Water may be effective for cooling, but may not be effective for extinguishing a fire because it may not cool methanol below its flash point.

Specific Hazards: Methanol vapours may burn with an invisible flame. During a fire, carbon monoxide, carbon dioxide and irritation and toxic gases such as formaldehyde may be generated. Vapours can accumulate in confined spaces resulting in a toxicity and flammability hazard. Closed containers may rupture violently and suddenly release large quantities of methanol when exposed to fire or excessive heat for a sufficient period of time. Vapours are slightly heavier than air and may travel long distances toward sources of ignition.

Hazardous Combustion Products: Toxic gases and vapours; oxides of carbon and formaldehyde.

Fire Fighting Instructions: Methanol burns with a clean clear flame that is almost invisible in daylight. Stay upwind! Isolate and restrict area access. Concentrations of greater that 25% methanol in water can be ignited. Use fine water spray or fog to control fire spread and cool adjacent structures or containers. Contain fire control water for later disposal. Fire fighters must wear full face, positive pressure, self-contained breathing apparatus or airline and appropriate protective fire fighting clothing as per NFPA. Note that methanol fires may require proximity suits. Take care not to walk through any spilled chemical.

Special Information: Vapours can flow along surfaces to distant ignition sources and flash back.

6. Accidental Release Measures

Overview: Flammable liquid! Can burn without a visible flame. Release can cause an immediate risk of fire and explosion. Eliminate all ignition sources, stop leak and use absorbent materials. If necessary, contain spill by diking. Fluorocarbon alcohol resistant foams may be applied to spill to diminish vapour and fire hazard. Maximize methanol recovery for recycling or re-use. Restrict access to area until completion of cleanup. Ensure cleanup is conducted by trained personnel only. Wear adequate personal protection and remove all sources of ignition. Notify all governmental agencies as required by law.

Personal Protection: Full face, positive pressure self-contained breathing apparatus or airline, and fire resistant protective clothing with chemical resistant splash suit must be worn. If product ignites, approach and fire fighting must be done with appropriate fire fighting clothing.

Environmental Precautions: Biodegrades easily in water. Methanol in fresh or salt water may have serious effects on aquatic life. A study on methanol's toxic efffects on sewage sludge bacteria reported little effect on digestion at 0.1% while 0.5% methanol retarded digestion. Methanol will be broken down to carbon dioxide and water.

Remedial Measures: Flammable liquid. Release can cause an immediate fire/explosion hazard. Eliminate all sources of ignition, stop leak and use absorbent materials. Collect liquid with explosion proof pumps. Do not walk through spill product as it may be on fire and not visible.

Small Spills: Soak up spill with non-combustible absorbent material. Recover methanol and dilute with water to reduce fire hazard. Prevent spilled methanol from entering sewers, confined spaces, drains, or waterways. Restict access to unprotected personnel. Put material in suitable, covered, labeled containers. Flush area with water.

Large Spills: If necessary, contain spill by diking. Fluorocarbon alcohol resistant foams may be applied to spill to diminish vapour and fire hazard. Maximize methanol recovery for recycling or reuse. Collect liquid with explosion proof pumps.

7. Handling and Storage

Precautions for Handling: No smoking or open flame in storage, use or handling areas. Use explosion proof electrical equipment. Ensure proper electrical grounding procedures are in place.

Storage: Store in totally enclosed equipment, designed to avoid ignition and human contact. Tanks must be grounded, vented, and should have vapour emission controls. Tanks must be diked as per NFPA or API Standards. A flammable mixture of methanol vapour and air is possible inside a storage tank or transportation tank, and handlers should take appropriate precautions to reduce the risk of ignition. Handlers must eliminate ignition sources or purge the tank with an inert gas such as nitrogen. All equipment must be grounded - bonded when transferring product in order to avoid static discharge from the equipment, and subsequent possible fire. Avoid storage with incompatible materials. Anhydrous methanol is non-corrosive to most metals at ambient temperatures except for lead, nickel, monel, cast iron and high silicon iron. Coatings of copper (or copper alloys), zinc (including galvanized steel), or aluminum are unsuitable for storage. These materials may be attacked slowly by the methanol. Storage tanks of welded construction are normally satisfactory. They should be designed and built in conformance with good engineering practice for the material being stored. While plastics can be used for short term storage, they are generally not recommended for long-term storage due to deterioration effects and the subsequent risk of contamination.

Corrosion rates for several construction materials:

<0.508 mm/year:	Cast iron, monel, lead, nickel
<0.051 mm/year:	High silicon iron
Some attack:	Polyethylene
Satisfactory:	Neoprene, phenolic resins, polyesters, natural rubber, butyl rubber
Resistant:	Polyvinyl chloride, unplasticized

8. Exposure Controls, Personal Protection

Occupational Controls: ACGIH* TLV-TWA: 200 ppm, skin (262 mg/m^3);
TLV-STEL: 250 ppm, skin (328 mg/m^3);
PEL-TWA: 200 ppm, skin
PEL-STEL: 250 ppm, skin
TLV Basis: critical effects: neuropathy, vision, central nervous system(CNS)
IDLH: 6000 ppm, acute inhalation toxicity to animals

Engineering Controls: In confined areas, local and general ventilation should be provided to maintain airborne concentrations below permissable exposure limits. Ventilation systems must be designed according to approved engineering standards.

Respiratory Protection: NIOSH/OSHA recommendations for methanol concentrations in air:

Up to 2000 ppm: supplied air respirator
Up to 5000 ppm: supplied air respirator operated in a continuous-flow mode.
Up to 6000 ppm: supplied air respirator with a tight-fitting facepiece operated in a continuous- flow mode; or Full-facepiece self-contained breathing apparatus or Full-facepiece supplied air respirator.

Cartridge type respirators are NOT recommended.

Emergency or Planned entry into unkown concentrations or IDLH (immediately dangerous to life or health) conditions:

Respirator selection must be done by a qualified person and be based upon a risk assessment of the work activities and exposure levels. Respirators must be fit tested and users must be clean shaven where the respirator seals to the face. Exposure must be kept at or below the applicable exposure limits and the maximum use concentration of the respirator must not be exceeded.

Positive pressure, full-facepiece self-contained breathing apparatus; or Positive pressure, full-facepiece supplied air respirator with an auxiliary positive pressure self-contained breathing apparatus.

Skin Protection: Butyl and nitrile rubbers are recommended for gloves. Check with manufacturer. Wear chemical resistant pants and jackets, preferably of butyl or nitrile rubber. Check with manufacturer.

Eye and Face Protection: Face shield and chemical splash goggles when transferring is taking place. Contact lenses should not be worn when working with methanol.

Footwear: Chemical resistant and as specified by the workplace.

Other: Eyewash and showers should be located near work areas. NOTE: PPE must not be considered a long-term solution to exposure control. PPE usage must be accompanied by employer programs to properly select, maintain, clean, fit and use. Consult a competent industrial hygiene resource to determine hazard potential and/or the PPE manufacturers to ensure adequate protection.

Careful consideration must be made of the added danger of the concenentration being in the LEL/UEL range and so there may be a fire/explosion hazard.

9. Physical and Chemical Properties

Appearance: Liquid, clear, colourless
Odour: Mild characteristic alcohol odour
Odour Threshold: detection: 4.2 - 5960 ppm
(geometric mean) 160 ppm
recognition: 53 – 8940 ppm
(geometric mean) 690 ppm
pH: Not applicable
Freezing Point: -97.8°C
Boiling Point: 64.7°C
Boiling Range: Not determined
Flash Point: 11.0°C
Solubility: Completely soluble
Partial Coefficient: Log P (oct) = -0.82
Vapour Pressure: 12.8 kPa @ 20°C
Upper Explosive Limit (UEL): 36.5 %
Lower Explosive Limit (LEL): 6%
Auto Ignition Temperature: 464°C
Solvent Solubility: Soluble in all proportions in ethanol, benzene, other alcohols, chloroform, diethyl ether, other ethers, esters, ketones and most organic solvents
Critical Temperature: 239.4°C
Specific Gravity: 0.791 @ 20°C
Evaporation Rate: 4.1 (n-butyl acetate =1)
Vapour Density: 1.105 @ 15°C (air = 1)
Decomposition Temperature: Not determined
Sensitivity to Impact: No
Sensitivity to Static Charge: Low

10. Stability and Reactivity

Chemical Stability: Stable as supplied.

Hazardous Reactions: Yes. Avoid contact with strong oxidizers, strong mineral or organic acids, and strong bases. Contact with these materials may cause a violent or explosive reaction. May be corrosive to lead, aluminum, magnesium, and platinum.

Conditions to Avoid: Avoid contact with sparks, heat, open flame, or ignition sources.

Incompatibility: Yes. Avoid contact with strong oxidizers, strong mineral or organic acids, and strong bases. Contact with these materials may cause a violent or explosive reaction. May be corrosive to lead, aluminum, magnesium, and platinum. May react with metallic aluminum or magnesium and generate hydrogen gas. May attack some forms of plastic, rubber, and coatings.

Hazardous Decomposition Products: Formaldehyde, carbon dioxide, and carbon monoxide.

Hazardous Polymerization: Will not occur.

11. Toxicological Information



Signal Word/Label: DANGER! Extremely flammable liquid and vapour. Fatal if swallowed.
May damage fertility or the unborn child (fetotoxic and teratogenic effects).
May cause damage to eyes and central nervous system if ingested or inhaled.

Primary Routes of Entry:

Skin Contact:	Yes
Skin Absorption:	Yes
Eye Contact:	Yes
Ingestion:	Yes
Inhalation:	Yes

Emergency Overview: Colourless liquid, with a mild, characteristic alcohol odour when pure. Crude methanol may have a repulsive, pungent odour. Hygroscopic. Can decompose at high temperatures forming carbon monoxide and formaldehyde. Confined space toxicity hazard. Mild central nervous system depressant following inhalation, skin absorption or ingestion. May cause headache, nausea, dizziness, drowsiness, and incoordination. Severe vision effects, including increased sensitivity to light, blurred vision, and blindness may develop following an 8-24 hour symptom-free period. Coma and death may result. Causes eye irritation. Aspiration hazard. Swallowing or vomiting of the liquid may result in aspiration (breathing) into the lungs. May cause fetotoxic (toxic to the fetus during the latter stages of pregnancy, often through the placenta) and teratogenic effects (causing malformations of the fetus), based on animal information.

Acute Exposure:

Inhalation: Inhalation of high airborne concentrations can also irriate mucous membranes, cause headaches, sleepiness, nausea, confusion, loss of consciousness, digestive and visual disturbances and even death. NOTE: Odour threshhold of methanol is several times higher than the TLV-TWA. Depending upon severity of poisoning and the promptness of treatment, survivors may recover completely or may have permanent blindness, vision disturbances and/or nervous system effects. Concentrations in air exceeding 1000 ppm may cause irritation of the mucous membranes.

Skin Contact: Methanol is moderately irritating to the skin. Methanol can be absorbed through the skin and harmful effects have been reported by this route of entry. Effects are similar to those described in “Inhalation”.

Eye Contact: Methanol is a mild to moderate eye irritant. High vapour concentration or liquid contact with eyes causes irritation, tearing and burning.

Ingestion: Swallowing even small amounts of methanol could potentially cause blindness or death. Effects of sub lethal doses may be nausea, headache, abdominal pain, vomiting and visual disturbances ranging from blurred vision to light sensitivity.

Chronic Exposure:

Irritancy: Prolonged contact with skin may defat tissue causing dermititis or aggravate existing skin problems.

Sensitization: None reported.

Carcinogenicity: Not listed by IARC, NTP, ACGIH, or OSHA as a carcinogen.

Teratogenicity: Methanol has produced fetotoxicity in rats and teratogenicity in mice exposed by inhalation to high concentrations of methanol vapours **.**

Reproductive Toxicity: Information available does not suggest that methanol is a reproductive toxin.

Mutagenicity: There is insufficient information available to conclude that methanol is mutagenic.

Synergistic Products: In animals, high concentrations of methanol can increase the toxicity of other chemicals, particularly liver toxins like carbon tetrachloride. Ethanol significantly reduces the toxicity of methanol because it competes for the same metabolic enzymes, and has been usd to treat methanol poisoning.

Potential for Accumulation: Methanol is readily absorbed into the body following inhalation and ingestion. Skin absorption may occur if the skin is broken or exposure is prolonged. Once absorbed, methanol is rapidly distributed to body tissues. A small amount is excreted unchanged in exhaled air and the urine. The rest is first metabolized to formaldehyde, which is then metabolized to formic acid and/or formate. The formic acid and formate are eventually converted to carbon dioxide and water. In humans, methanol clears from the body, after inhalation or oral exposure, with a half-life of 1 day or more for high doses (greater than 1000 mg/kg) or about 1.5-3 hours for low doses (less than 100 mg/kg or 76.5-230 ppm (100-300 mg/m^3)).

Medical Conditions Aggravated By Exposure: Persons with pre-existing skin disorders, eye problems, respiratory conditions, or impaired liver or kidney functions may be more susceptable to the effects of this substance.

12. Ecological Information

Environmental toxicity: DO NOT discharge into sewer or waterways.

Methanol:

LC_{50} Pimephales promelas (fathead minnows) 29.4 g/L/96 hr, (28-29 days old), confidence limit= 28.5-30.4; Test conditions: Water temp= 25°C, dissolved oxygen= 7.3 mg/L, water hardness= 43.5 mg/l $CaCO_3$, alkalinity= 46.6 $CaCO_3$, tank volume= 6.3 L, additions= 5.71 V/D, pH= 7.66
LC_{50} Pimephales promelas (Fathead minnow, 28-32 day old, 0.126 g) 29,700 mg/L/24 hr; flow-through, 23.3+/-1.7°C, hardness 46.4 mg/L $CaCO_3$, pH 7.0-8.0
LC_{50} Pimephales promelas (Fathead minnow, 30 day old 0.12 g) 28,100 mg/L/96 hr; flow-through, 24-26°C, hardness 45.5 mg/L $CaCO_3$, pH 7.5
LC_{50} Daphnia pulex (Water flea, <24 hr old) 19,500 mg/L/18 hr; static, 22°C, hardness 23+/-2 mg/L $CaCO_3$
EC_{50} Daphnia obtusa (Water flea, <24 hr old; immobilization) 23,500 mg/L/24 hr; static, 20+/-2°C, hardness 250 mg/L $CaCO_3$, pH 7.8+/-0.2
EC_{50} Daphnia obtusa (Water flea, <24 hr old; immobilization) 22,200 mg/L/48 hr; static, 20+/-2°C, hardness 250 mg/L $CaCO_3$, pH 7.8+/-0.2

log K_{ow}: -0.82 – -0.66
Half-life (hr) air: 427
Half-life (hr) H_2O surface water: 5.3 – 64
Henry's Law constant (atm m^3/mol): $4.55X10^{-6}$
BOD 5 if unstated: 0.76 – 1.12
COD: 1.05 – 1.50, 99%
ThOD: 1.05
BCF: 0.2 – 10
TLm(48 hr): 8000mg/L (trout)
Toxicity Arthropoda: NOEL 10 g/L/48 hr (Daphnia)
HSNO Classification: 9.3C – Harmful to terrestrial vertebrates

Methanol in fresh or salt water may have serious effects on aquatic life. A study on methanol's toxic effects on sewage sludge bacteria reported little effect on digestion at 0.1% while 0.5% methanol retarded digestion. Methanol will be broken down into carbon dioxide and water.

Environmental Fate:

Biodegradability: Biodegrades easily in water and soil.

Bioaccumulation:

- **TERRESTRIAL FATE:** Based on a classification scheme, an estimated Koc value of 1 determined from a structure estimation method indicates that methanol is expected to have very high mobility in soil. Volatilization of methanol from moist soil surfaces is expected to be an important fate process given a Henry's Law constant of $4.55X10^{-6}$ (atm m^3/mol). The potential for volatilization of methanol from dry soil surfaces may exist based upon a vapor pressure of 127 mm Hg. Biodegradation is expected to be an important fate process for methanol.

- **AQUATIC FATE:** Based on a classification scheme, an estimated Koc value of 1, determined from a structure estimation method, indicates that methanol is not expected to adsorb to suspended solids and sediment. Volatilization from water surfaces is expected based upon a Henry's Law constant of $4.55X10^{-6}$ (atm m^3/mol). Using this Henry's Law constant and an estimation method, volatilization half-lives for a model river and model lake are three and 35 days, respectively. According to a classification scheme, a BCF of less than 10 measured in fish, suggests bioconcentration in aquatic organisms is low. Hydrolysis and photolysis in sunlit surface waters is not expected to be an important environmental fate process for methanol since this compound lacks functional groups that hydrolyze or absorb light under environmentally relevant conditions. Methanol has been shown to undergo rapid biodegradation in a variety of screening studies using sewage seed and activated sludge inoculum, which suggests that biodegradation will occur in aquatic environments.
- **ATMOSPHERIC FATE:** According to a model of gas/particle partitioning of semi volatile organic compounds in the atmosphere, methanol, which has a vapor pressure of 127 mm Hg at 25°C, is expected to exist solely as a vapor in the ambient atmosphere. Vapor-phase methanol is degraded in the atmosphere by reaction with photo chemically-produced hydroxyl radicals; the half-life for this reaction in air is estimated to be 17 days, calculated from its rate constant of $9.4X10^{-13}$ cu cm/molecule-sec at 25°C

13. Disposal Considerations

Review federal, provincial or state, and local government requirements prior to disposal. Store material for disposal as indicated in Section #7, ***Handling and Storage***. Disposal by controlled incineration or by secure land fill may be acceptable.

Recycle wherever possible. Large volumes may be suitable for re-distillation or, if contaminated, incinerated. Can be disposed of in a sewage treatment facility. Methanol levels of up to 0.1% act as a food source for bacteria; above this level may be toxic to bacteria. When pumping through sewage collection systems, the level of methanol should be kept below the flammable range (a 25% methanol/water mixture is non-flammable at temperatures below 39**°C**). 1 ppm of methanol is equivalent to 1.5 ppm BOD loading in the sewage plant.

Container disposal:

Empty containers may contain hazardous residue. Return to supplier for reuse if possible. Never weld, cut or grind empty containers. If disposing of containers, ensure they are well rinsed with water, then disposed of at an authorised landfill. After cleaning, all existing labels should be removed.

14. Transport Information

Canada Transportation of Dangerous Goods (TDG):	UN 1230, Methanol, Class 3(6.1), P.G. II Limited Quantity: ≤ 1 litres ERG Guide Number: 131
United States Department of Transport (49CFR): (Domestic Only)	UN 1230, Methanol, Class 3, P.G. II, (RQ 5000 lbs/2270 kg) Limited Quantity: ≤ 1 litres ERG Guide Number: 131
International Air Transport Association (IATA):	UN 1230, Methanol, Class 3(6.1), P.G. II Packaging Instruction (passenger aircraft): 305, 1 litre maximum per package,
International Maritime Organization (IMO):	UN 1230, Methanol, Class 3(6.1), P.G.II, Flash Point = 11°C EmS No. F-E, S-D Stowage Category "B", Clear of living quarters
Marine Pollutant:	No

15. Regulatory Information

CANADIAN FEDERAL REGULATIONS:

CEPA, DOMESTIC SUBSTANCES LIST: Listed (Canadian Environmental Protection Act (CEPA) Schedule I)

WHMIS CLASSIFICATION: B2, D1B, D2A, D2B

UNITED STATES REGULATIONS:

29CFR 1910.1200 (OSHA): Hazardous

40CFR 116-117 (EPA): Hazardous

40CFR 355, Appendices A and B: Subject to Emergency Planning and Notification

40CFR 372 (SARA Title III): Listed

40CFR 302 (CERCLA): Listed

TOXIC SUBSTANCES CONTROL ACT (TSCA): Listed in the inventory.

16. Other Information

References:

1. International Programme on Chemical Safety, Methanol, Environmental Health Criteria, World Health Organization 1997.
2. Patty's Industrial Hygiene and Toxicology, 5th Edition.
3. Fire Protection Guide to Hazardous Materials, 13th Edition.
4. Lanigan, S., Final report on the Safety Assessment of Methyl Alcohol, International Journal of Toxicology., Volume 20, Supplement 1 (2001).
5. Forsberg, K., Quick Selection Guide to Chemical Protective Clothing.
6. Nelson, B.K., Teratological assessment of Methanol and Ethanol at high inhalation levels in rats, Fundamental and Applied Toxicology, Volume 5.
7. NIOSH Guide to Chemical Hazards
8. Hazardous Substance Data Base (HSDB).
9. Cheminfo.

Original Preparation Date: September 22, 2005

Prepared by: Kel-Ex Agencies Ltd., P.O. Box 52201, Lynnmour RPO, North Vancouver, B.C., Canada, V7J 3V5

Disclaimer: The information above is believed to be accurate and represents the best information currently available to us. Users should make their own investigations to determine the suitability of the information for their particular purposes. This document is intended as a guide to the appropriate precautionary handling of the material by a properly trained person using this product.

Methanex Corporation and its subsidiaries make no representations or warranties, either express or implied, including without limitation any warranties of merchantability, fitness for a particular purpose with respect to the information set forth herein or the product to which the information refers. Accordingly, Methanex Corp. will not be responsible for damages resulting from use of or reliance upon this information.

This Material Safety Data Sheet may not be changed, or altered in any way without the expressed knowledge and permission of Methanex Corporation

Revisions: Revised and re-issued in GHS Format September 22, 2008

GFS CHEMICALS, INC.
P.O. Box 245 Powell, OH 43065
740-881-5501(Tel.) 740-881-5989(Fax)
1-800-424-9300(Chemtrec 24Hr. Info.)

MATERIAL SAFETY DATA SHEET

AmcoClear

AMCO CLEAR® TURBIDITY STANDARD

CHEMICAL NAME & SYNONYMS	**DOT CLASS**	**SARA TITLE 313**
AMCO CLEAR® Turbidity Standard	NR	No
	TSCA listed - Yes	

FORMULA	**REPORTABLE QUANTITY**	**F.W.**	**CAS#**
Styrene Divinyl Benzene Copolymer Beads <1%	N/A	N/A	9003-70-7
H_2O >99%	N/A	18.02	7732-18-5

PHYSICAL DATA
Boiling point 100°C; Density 1.0; melting point 0°C; pH 6.7

APPEARANCE & ODOR
White powder suspended in clear, colorless liquid. Depending on concentration, solution may be clear, hazy or opaque. Odorless.

REACTIVITY & CONDITIONS TO AVOID
Stable. Incompatible with organic matter (no hazardous reaction). Hazardous polymerization will not occur. Keep from freezing (once frozen, polymer will not remain completely suspended).

FIRE HAZARDS
None. NFPA # 0-0-0.

EXTINGUISHER	**FLASHPOINT**	**LEL**	**UEL**
Fight surrounding fire.	N/A	N/A	N/A

HEALTH HAZARDS
No health hazards by normal means of exposure. LD_{50} (oral-rabbit) 368 g (water)/kg. OSHA PEL/ACGIH TLV not established. No evidence of carcinogenicity.

SPECIAL PRECAUTIONS
Always use good laboratory practices. Keep from freezing, avoid contaminating solution.

FIRST AID
Flush eyes with water. Seek medical attention if irritation develops. Wash contacted skin with water. Ingestion is not hazardous. Inhalation is not an expected route of exposure.

SPILLS & LEAKS
Wash up with water. Flush to drain with plenty of water or general trash.

CATALOG #	**PREPARED BY**	**DATE**
Amco Clear	MDM	April 22, 2009

GFS CHEMICALS, INC.
P.O. Box 245 Powell, OH 43065
740-881-5501(Tel.) 740-881-5989(Fax)
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MATERIAL SAFETY DATA SHEET

AmcoClear

AMCO CLEAR® TURBIDITY STANDARD

CHEMICAL NAME & SYNONYMS	**DOT CLASS**	**SARA TITLE 313**
AMCO CLEAR® Turbidity Standard	NR	No
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EXTINGUISHER	**FLASHPOINT**	**LEL**	**UEL**
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CATALOG #	**PREPARED BY**	**DATE**
Amco Clear	MDM	April 22, 2009

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MATERIAL SAFETY DATA SHEET

AmcoClear

AMCO CLEAR® TURBIDITY STANDARD

CHEMICAL NAME & SYNONYMS	**DOT CLASS**	**SARA TITLE 313**
AMCO CLEAR® Turbidity Standard	NR	No
	TSCA listed - Yes	

FORMULA	**REPORTABLE QUANTITY**	**F.W.**	**CAS#**
Styrene Divinyl Benzene Copolymer Beads <1%	N/A	N/A	9003-70-7
H_2O >99%	N/A	18.02	7732-18-5

PHYSICAL DATA
Boiling point 100°C; Density 1.0; melting point 0°C; pH 6.7

APPEARANCE & ODOR
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REACTIVITY & CONDITIONS TO AVOID
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FIRE HAZARDS
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EXTINGUISHER	**FLASHPOINT**	**LEL**	**UEL**
Fight surrounding fire.	N/A	N/A	N/A

HEALTH HAZARDS
No health hazards by normal means of exposure. LD_{50} (oral-rabbit) 368 g (water)/kg. OSHA PEL/ACGIH TLV not established. No evidence of carcinogenicity.

SPECIAL PRECAUTIONS
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FIRST AID
Flush eyes with water. Seek medical attention if irritation develops. Wash contacted skin with water. Ingestion is not hazardous. Inhalation is not an expected route of exposure.

SPILLS & LEAKS
Wash up with water. Flush to drain with plenty of water or general trash.

CATALOG #	**PREPARED BY**	**DATE**
Amco Clear	MDM	April 22, 2009

OAKTON® MATERIAL SAFETY DATA SHEET

Section 1. Chemical Product and Company Identification

Catalog Number(s)

00654-00, 05942-21, 05942-22, 05942-24, 05942-25, 05942-26, 05942-27, 35653-01, 35654-00

Product Identity

BUFFER, Standard, pH 4.01; BUFFER, High Accuracy, pH 4.000 (Color Coded Red)

Manufacturer's Name	Emergency Telephone Number (24 hr)
RICCA CHEMICAL COMPANY	CHEMTREC®: 800-424-9300
Address (Number, Street, City, State, and ZIP Code)	Telephone Number For Information
P.O. Box 13090	817-461-5601
	Date Prepared
Arlington, Texas 76094	3-7-2000

Section 2. Composition / Information on Ingredients

Component	CAS Registry #	Percent Concentration	Exposure Limits ACGIH TLV	Exposure Limits OSHA PEL
Potassium Acid Phthalate	877-24-7	0.95 – 1.05	N/A	N/A
Preservative* *(No Mercury compounds or Formaldehyde)	proprietary	<0.5	N/A	N/A
Inert Dye	proprietary	<0.1	N/A	N/A
Water, Deionized	7732-18-5	Balance	N/A	N/A

Section 3. Hazards Identification

☆☆☆

Section 4. First Aid Measures – In all cases, seek qualified evaluation.

EYE CONTACT: Irrigate immediately with large quantity of water for at least 15 minutes. Call a physician if irritation develops.

INHALATION: Remove to fresh air. Give artificial respiration if necessary. If breathing is difficult, give oxygen.

SKIN CONTACT: Flush with plenty of water for at least 15 minutes. Call a physician if irritation develops.

INGESTION: Dilute with water or milk. Call a physician if necessary.

Section 5. Fire Fighting Measures

FLAMMABLE PROPERTIES:

FLASH POINT: N/A METHOD USED: N/A

FLAMMABLE LIMITS

LFL: N/A UFL: N/A

EXTINGUISHING MEDIA: Use any means suitable for extinguishing surrounding fire.

FIRE & EXPLOSION HAZARDS: Not considered to be a fire or explosion hazard.

FIRE FIGHTING INSTRUCTIONS: Use normal procedures/instructions.

FIRE FIGHTING EQUIPMENT: Use protective clothing and breathing equipment appropriate for the surrounding fire.

Section 6. Accidental Release Measures

Absorb with suitable material and dispose of in accordance with local regulations.

Section 7. Handling and Storage

As with all chemicals, wash hands thoroughly after handling. Avoid contact with eyes and skin. Protect from freezing and physical damage. SAFETY STORAGE CODE: GENERAL

Section 8. Exposure Controls / Personal Protection

ENGINEERING CONTROLS: No specific controls are needed. Normal room ventilation is adequate.

RESPIRATORY PROTECTION: Normal room ventilation is adequate.

SKIN PROTECTION: Chemical resistant gloves.

EYE PROTECTION: Safety glasses or goggles.

Section 9. Physical and chemical Properties

APPEARANCE:	Clear, red colored liquid	**pH:**	4
ODOR:	odorless	**BOILING POINT (^{o}C):**	approximately 100
SOLUBILITY IN WATER:	infinite	**MELTING POINT (^{o}C):**	approximately 0
SPECIFIC GRAVITY:	approximately 1	**VAPOR PRESSURE:**	N/A

Section 10. Stability and Reactivity

CHEMICAL STABILITY: Stable under normal conditions of use and storage.

INCOMPATIBILITY: Nitric Acid

HAZARDOUS DECOMPOSITION PRODUCTS: Oxides of Carbon and Potassium.

HAZARDOUS POLYMERIZATION: Will not occur.

Section 11. Toxicological Information

LD50, Oral, Rat: >3200 mg/kg (Potassium Acid Phthalate), details of toxic effects not reported other than lethal dose value.

Section 12. Ecological Information

ECOTOXICOLOGICAL INFORMATION: No information found.

CHEMICAL FATE INFORMATION: No information found.

Section 13. Disposal Considerations

Dilute with water, neutralize with weak sodium hydroxide solution, and then flush to sewer if local regulations allow. If not allowed, save for recovery or recycling in an approved waste disposal facility. Always dispose of in accordance with local, state and federal regulations.

Section 14. Transport Information (Not meant to be all inclusive)

D.O.T. SHIPPING NAME:	Not regulated
D.O.T. HAZARD CLASS:	None
U.N. / N.A. NUMBER:	None
PACKING GROUP:	None
D.O.T. LABEL:	None

Section 15. Regulatory Information (Not meant to be all inclusive - selected regulation represented)

OSHA STATUS: The above items either do not contain any specifically hazardous material or the potentially hazardous material is present in such low concentration that the items do not present any immediate threat to health and safety. These items do not meet the OSHA Hazard Communication Standard (29 CFR 1910.1200) definition of a hazardous material.

TSCA STATUS: All components of this solution are listed on the TSCA Inventory.
CERCLA REPORTABLE QUANTITY: Not reportable
SARA TITLE III:

SECTION 302 EXTREMELY HAZARDOUS SUBSTANCES: No
SECTION 311/312 HAZARDOUS CATEGORIES: No
SECTION 313 TOXIC CHEMICALS: No

RCRA STATUS: No

CALIFORNIA PROPOSITION 65: Not listed

Section 16. Other Information

NFPA® Ratings:	**Health: 1**	**Flammability: 0**	**Reactivity: 0**	**Special Notice Key: None**
HMIS® Ratings:	**Health: 1**	**Flammability: 0**	**Reactivity: 0**	**Protective Equipment: B (Protective eyewear, gloves)**

Rev 1, 10-16-2000: (Section 1) added catalog number 35653-01.
Rev 2, 03-25-2003: Reviewed and approved.
Rev 3, 03-20-2006: Reviewed and approved.

When handled properly by qualified personnel, the product described herein does not present a significant health or safety hazard. Alteration of its characteristics by concentration, evaporation, addition of other substances, or other means may present hazards not specifically addressed herein and which must be evaluated by the user. The information furnished herein is believed to be accurate and represents the best data currently available to us. No warranty, expressed or implied, is made and RICCA CHEMICAL COMPANY assumes no legal responsibility or liability whatsoever resulting from its use.

OAKTON® MATERIAL SAFETY DATA SHEET

Section 1. Chemical Product and Company Identification

Catalog Number(s)

00654-04, 35654-04, 05942-41, 05942-42, 05942-44, 05942-45, 35653-02

Product Identity

BUFFER, Standard, pH 7.00 (Color Coded Green)

Manufacturer's Name	Emergency Telephone Number (24 hr)
RICCA CHEMICAL COMPANY	CHEMTREC®: 800-424-9300
Address (Number, Street, City, State, and ZIP Code)	Telephone Number For Information
P.O. Box 13090	817-461-5601
	Date Prepared
Arlington, Texas 76094	3-8-2000

Section 2. Composition / Information on Ingredients

Component	CAS Registry #	Percent Concentration	Exposure Limits ACGIH TLV	Exposure Limits OSHA PEL
Sodium Phosphate, Dibasic	7558-79-4	< 1	N/A	N/A
Potassium Phosphate, Monobasic	7778-77-0	< 1	N/A	N/A
Preservative* *(No Mercury Compounds or Formaldehyde)	Proprietary	< 0.1	N/A	N/A
Inert Dye	Proprietary	< 0.1	N/A	N/A
Water, Deionized	7732-18-5	Balance	N/A	N/A

Section 3. Hazards Identification

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Section 4. First Aid Measures – In all cases, seek qualified evaluation.

EYE CONTACT: Irrigate immediately with large quantity of water for at least 15 minutes. Call a physician if irritation develops.

INHALATION: Remove to fresh air. Give artificial respiration if necessary. If breathing is difficult, give oxygen.

SKIN CONTACT: Flush with plenty of water for at least 15 minutes. Call a physician if irritation develops.

INGESTION: Dilute with water or milk. Call a physician if necessary.

Section 5. Fire Fighting Measures

FLAMMABLE PROPERTIES:

FLASH POINT: N/A METHOD USED: N/A

FLAMMABLE LIMITS

LFL: N/A UFL: N/A

EXTINGUISHING MEDIA: Use any means suitable for extinguishing surrounding fire.

FIRE & EXPLOSION HAZARDS: Not considered to be a fire or explosion hazard.

FIRE FIGHTING INSTRUCTIONS: Use normal procedures/instructions.

FIRE FIGHTING EQUIPMENT: Use protective clothing and breathing equipment appropriate for the surrounding fire.

Section 6. Accidental Release Measures

Absorb with suitable material (vermiculite, clay, etc.) and dispose of in accordance with local regulations. Check with local agencies for the proper disposal of phosphate containing solutions.

Section 7. Handling and Storage

As with all chemicals, wash hands thoroughly after handling. Avoid contact with eyes and skin. Protect from freezing and physical damage. SAFETY STORAGE CODE: GENERAL

Section 8. Exposure Controls / Personal Protection

ENGINEERING CONTROLS: No specific controls are needed. Normal room ventilation is adequate.

RESPIRATORY PROTECTION: Normal room ventilation is adequate.

SKIN PROTECTION: Chemical resistant gloves.

EYE PROTECTION: Safety glasses or goggles.

Section 9. Physical and chemical Properties

APPEARANCE:	Clear, green liquid	**pH:**	7
ODOR:	Odorless	**BOILING POINT (^{o}C):**	approximately 100
SOLUBILITY IN WATER:	Infinite	**MELTING POINT (^{o}C):**	approximately 0
SPECIFIC GRAVITY:	approximately 1	**VAPOR PRESSURE:**	N/A

Section 10. Stability and Reactivity

CHEMICAL STABILITY: Stable under normal conditions of use and storage.

INCOMPATIBILITY: None identified.

HAZARDOUS DECOMPOSITION PRODUCTS: Phosphorus oxides may form when heated to decomposition.

HAZARDOUS POLYMERIZATION: Will not occur.

Section 11. Toxicological Information

LD50, Oral, Rat: (Sodium Phosphate Dibasic) 17 gm/kg; LD50, Dermal, Rabbit: (Potassium Phosphate Monobasic) >4640 mg/kg; details of toxic effects not reported other than lethal dose value.

Section 12. Ecological Information

ECOTOXICOLOGICAL INFORMATION: No information found.

CHEMICAL FATE INFORMATION: No information found.

Section 13. Disposal Considerations

Dilute with water, then flush to sewer if local regulations allow for the flushing of phosphate containing solutions. If not allowed, save for recovery or recycling in an approved waste disposal facility. Always dispose of in accordance with local, state and federal regulations.

Section 14. Transport Information (Not meant to be all inclusive)

D.O.T. SHIPPING NAME:	Not regulated
D.O.T. HAZARD CLASS:	None
U.N. / N.A. NUMBER:	None
PACKING GROUP:	None
D.O.T. LABEL:	None

Section 15. Regulatory Information (Not meant to be all inclusive - selected regulation represented)

OSHA STATUS: The above items either do not contain any specifically hazardous material or the potentially hazardous material is present in such low concentration that the items do not present any immediate threat to health and safety. These items do not meet the OSHA Hazard Communication Standard (29 CFR 1910.1200) definition of a hazardous material.

TSCA STATUS: All components of this solution are listed on the TSCA Inventory or are mixtures (hydrates) of items listed on the TSCA Inventory.

CERCLA REPORTABLE QUANTITY: Sodium Phosphate, Dibasic - 5,000 pounds.

SARA TITLE III:

SECTION 302 EXTREMELY HAZARDOUS SUBSTANCES: No

SECTION 311/312 HAZARDOUS CATEGORIES: No

SECTION 313 TOXIC CHEMICALS: No

RCRA STATUS: No

CALIFORNIA PROPOSITION 65: Not listed.

PENNSYLVANIA: Sodium Phosphate Dibasic is listed as an environmental hazard on the state Hazardous Substance list.

Section 16. Other Information

NFPA Ratings:	**Health: 1**	**Flammability: 0**	**Reactivity: 0**	**Special Notice Key: None**
HMIS® Ratings:	**Health: 1**	**Flammability: 0**	**Reactivity: 0**	**Protective Equipment: B (Protective eyewear, gloves)**

Rev 1, 8-25-2000: (Section 2) corrected concentration of preservative from 1 – 2 to < 0.1%.
Rev 2, 03-25-2003: Reviewed and approved, (Section 15) added CERCLA reportable quantity.
Rev 3, 03-20-2006: Reviewed and approved.

When handled properly by qualified personnel, the product described herein does not present a significant health or safety hazard. Alteration of its characteristics by concentration, evaporation, addition of other substances, or other means may present hazards not specifically addressed herein and which must be evaluated by the user. The information furnished herein is believed to be accurate and represents the best data currently available to us. No warranty, expressed or implied, is made and RICCA CHEMICAL COMPANY assumes no legal responsibility or liability whatsoever resulting from its use.

OAKTON® MATERIAL SAFETY DATA SHEET

Section 1. Chemical Product and Company Identification

Catalog Number(s)

00654-08, 35654-08, 05942-61, 05942-62, 05942-64, 05942-65, 05942-66, 05942-67, 35653-03

Product Identity

BUFFER, Standard, pH 10.00; BUFFER, High Accuracy, pH 10.000 (Color Coded Blue)

Manufacturer's Name	Emergency Telephone Number (24 hr)
RICCA CHEMICAL COMPANY	CHEMTREC®: 800-424-9300
Address (Number, Street, City, State, and ZIP Code)	Telephone Number For Information
P.O. Box 13090	817-461-5601
	Date Prepared
Arlington, Texas 76094	3-8-2000

Section 2. Composition / Information on Ingredients

Component	CAS Registry #	Percent Concentration	Exposure Limits ACGIH TLV	Exposure Limits OSHA PEL
Sodium Carbonate	497-19-8	< 1	N/A	N/A
Sodium Bicarbonate	144-55-8	< 1	N/A	N/A
Preservative* *(No Mercury compounds or Formaldehyde)	proprietary	< 0.1	N/A	N/A
Inert Dye	proprietary	< 0.1	N/A	N/A
Water, Deionized	7732-18-5	Balance	N/A	N/A

Section 3. Hazards Identification

☆☆☆

REPRODUCTION INFORMATION:

Reproductive data cited in "Registry of Toxic Effects of Chemical Substances" for Sodium Bicarbonate and Sodium Carbonate in mice.

Section 4. First Aid Measures – In all cases, seek qualified evaluation.

EYE CONTACT: Irrigate immediately with large quantity of water for at least 15 minutes. Call a physician if irritation develops.

INHALATION: Remove to fresh air. Give artificial respiration if necessary. If breathing is difficult, give oxygen.

SKIN CONTACT: Flush with plenty of water for at least 15 minutes. Call a physician if irritation develops.

INGESTION: Dilute with water or milk. Call a physician if necessary.

Section 5. Fire Fighting Measures

FLAMMABLE PROPERTIES:

FLASH POINT: N/A METHOD USED: N/A

FLAMMABLE LIMITS

LFL: N/A UFL: N/A

EXTINGUISHING MEDIA: Use any means suitable for extinguishing surrounding fire.

FIRE & EXPLOSION HAZARDS: Not considered to be a fire or explosion hazard.

FIRE FIGHTING INSTRUCTIONS: Use normal procedures/instructions.

FIRE FIGHTING EQUIPMENT: Use protective clothing and breathing equipment appropriate for the surrounding fire.

Section 6. Accidental Release Measures

Absorb with suitable material and treat as normal refuse. Small amounts of the liquid may be flushed to the drain with excess water. Always dispose of in accordance with local regulations.

Section 7. Handling and Storage

As with all chemicals, wash hands thoroughly after handling. Avoid contact with eyes and skin. Protect from freezing and physical damage. SAFETY STORAGE CODE: GENERAL

Section 8. Exposure Controls / Personal Protection

ENGINEERING CONTROLS: No specific controls are needed. Normal room ventilation is adequate.

RESPIRATORY PROTECTION: Normal room ventilation is adequate.

SKIN PROTECTION: Chemical resistant gloves.

EYE PROTECTION: Safety glasses or goggles.

Section 9. Physical and chemical Properties

APPEARANCE:	Clear, blue colored liquid	**pH:**	10
ODOR:	Odorless	**BOILING POINT (^{o}C):**	approximately 100
SOLUBILITY IN WATER:	Infinite	**MELTING POINT (^{o}C):**	approximately 0
SPECIFIC GRAVITY:	approximately 1	**VAPOR PRESSURE:**	N/A

Section 10. Stability and Reactivity

CHEMICAL STABILITY: Stable under normal conditions of use and storage.

INCOMPATIBILITY: Acids

HAZARDOUS DECOMPOSITION PRODUCTS: Oxides of Sodium.

HAZARDOUS POLYMERIZATION: Will not occur.

Section 11. Toxicological Information

LD50, Oral, Rat: 4090 mg/kg (Sodium Carbonate), 4220 mg/kg (Sodium Bicarbonate), details of toxic effects not reported other than lethal dose value.

Section 12. Ecological Information

ECOTOXICOLOGICAL INFORMATION: No information found.

CHEMICAL FATE INFORMATION: No information found.

Section 13. Disposal Considerations

Dilute with water, then flush to sewer if local regulations allow. If not allowed, save for recovery or recycling in an approved waste disposal facility. Always dispose of in accordance with local, state and federal regulations.

Section 14. Transport Information (Not meant to be all inclusive)

D.O.T. SHIPPING NAME:	Not regulated
D.O.T. HAZARD CLASS:	None
U.N. / N.A. NUMBER:	None
PACKING GROUP:	None
D.O.T. LABEL:	None

Section 15. Regulatory Information (Not meant to be all inclusive - selected regulation represented)

OSHA STATUS: The above items either do not contain any specifically hazardous material or the potentially hazardous material is present in such low concentration that the items do not present any immediate threat to health and safety. These items do not meet the OSHA Hazard Communication Standard (29 CFR 1910.1200) definition of a hazardous material.

TSCA STATUS: All components of this solution are listed on the TSCA Inventory.

CERCLA REPORTABLE QUANTITY: Not reportable

SARA TITLE III:

SECTION 302 EXTREMELY HAZARDOUS SUBSTANCES: No

SECTION 311/312 HAZARDOUS CATEGORIES: No

SECTION 313 TOXIC CHEMICALS: No

RCRA STATUS: No

CALIFORNIA PROPOSITION 65: Not listed.

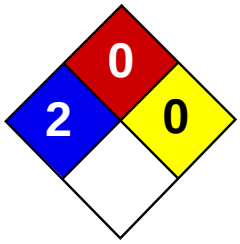
Section 16. Other Information

NFPA® Ratings:	**Health: 1**	**Flammability: 0**	**Reactivity: 0**	**Special Notice Key: None**
HMIS® Ratings:	**Health: 1**	**Flammability: 0**	**Reactivity: 0**	**Protective Equipment: B (Protective eyewear, gloves)**

Rev 1, 01-15-2003: added catalog number 35653-03.
Rev 2, 03-25-2003: Reviewed and approved.
Rev 3, 03-20-2006: Reviewed and approved.

When handled properly by qualified personnel, the product described herein does not present a significant health or safety hazard. Alteration of its characteristics by concentration, evaporation, addition of other substances, or other means may present hazards not specifically addressed herein and

which must be evaluated by the user. The information furnished herein is believed to be accurate and represents the best data currently available to us. No warranty, expressed or implied, is made and RICCA CHEMICAL COMPANY assumes no legal responsibility or liability whatsoever resulting from its use.



Health	2
Fire	0
Reactivity	0
Personal Protection	G

Material Safety Data Sheet
Tetrachloroethylene MSDS

Section 1: Chemical Product and Company Identification

Product Name: Tetrachloroethylene

Catalog Codes: SLT3220

CAS#: 127-18-4

RTECS: KX3850000

TSCA: TSCA 8(b) inventory: Tetrachloroethylene

CI#: Not available.

Synonym: Perchloroethylene; 1,1,2,2-Tetrachloroethylene; Carbon bichloride; Carbon dichloride; Ankilostin; Didakene; Dilatin PT; Ethene, tetrachloro-; Ethylene tetrachloride; Perawin; Perchlor; Perclene; Perclene D; Percosolvel; Tetrachloroethene; Tetraleno; Tetralex; Tetravec; Tetroguer; Tetropil

Chemical Name: Ethylene, tetrachloro-

Chemical Formula: C2-Cl4

Contact Information:

Sciencelab.com, Inc.
14025 Smith Rd.
Houston, Texas 77396

US Sales: **1-800-901-7247**
International Sales: **1-281-441-4400**

Order Online: ScienceLab.com

CHEMTREC (24HR Emergency Telephone), call: 1-800-424-9300

International CHEMTREC, call: 1-703-527-3887

For non-emergency assistance, call: 1-281-441-4400

Section 2: Composition and Information on Ingredients

Composition:

Name	CAS #	% by Weight
Tetrachloroethylene	127-18-4	100

Toxicological Data on Ingredients: Tetrachloroethylene: ORAL (LD50): Acute: 2629 mg/kg [Rat]. DERMAL (LD): Acute: >3228 mg/kg [Rabbit]. MIST(LC50): Acute: 34200 mg/m 8 hours [Rat]. VAPOR (LC50): Acute: 5200 ppm 4 hours [Mouse].

Section 3: Hazards Identification

Potential Acute Health Effects:
Hazardous in case of skin contact (irritant), of inhalation. Slightly hazardous in case of skin contact (permeator), of eye contact (irritant), of ingestion.

Potential Chronic Health Effects:
CARCINOGENIC EFFECTS: Classified A3 (Proven for animal.) by ACGIH. Classified 2A (Probable for human.) by IARC, 2 (anticipated carcinogen) by NTP. MUTAGENIC EFFECTS: Mutagenic for bacteria and/or yeast. TERATOGENIC EFFECTS: Not available. DEVELOPMENTAL TOXICITY: Not available. The substance may be toxic to kidneys, liver, peripheral nervous system, respiratory tract, skin, central nervous system (CNS). Repeated or prolonged exposure to the substance can produce target organs damage.

Section 4: First Aid Measures

Eye Contact:
Check for and remove any contact lenses. In case of contact, immediately flush eyes with plenty of water for at least 15 minutes. Get medical attention if irritation occurs.

Skin Contact:
In case of contact, immediately flush skin with plenty of water. Cover the irritated skin with an emollient. Remove contaminated clothing and shoes. Wash clothing before reuse. Thoroughly clean shoes before reuse. Get medical attention.

Serious Skin Contact:
Wash with a disinfectant soap and cover the contaminated skin with an anti-bacterial cream. Seek medical attention.

Inhalation:
If inhaled, remove to fresh air. If not breathing, give artificial respiration. If breathing is difficult, give oxygen. Get medical attention if symptoms appear.

Serious Inhalation:
Evacuate the victim to a safe area as soon as possible. Loosen tight clothing such as a collar, tie, belt or waistband. If breathing is difficult, administer oxygen. If the victim is not breathing, perform mouth-to-mouth resuscitation. Seek medical attention.

Ingestion:
Do NOT induce vomiting unless directed to do so by medical personnel. Never give anything by mouth to an unconscious person. Loosen tight clothing such as a collar, tie, belt or waistband. Get medical attention if symptoms appear.

Serious Ingestion: Not available.

Section 5: Fire and Explosion Data

Flammability of the Product: Non-flammable.

Auto-Ignition Temperature: Not applicable.

Flash Points: Not applicable.

Flammable Limits: Not applicable.

Products of Combustion: Not available.

Fire Hazards in Presence of Various Substances: Not applicable.

Explosion Hazards in Presence of Various Substances:
Risks of explosion of the product in presence of mechanical impact: Not available. Risks of explosion of the product in presence of static discharge: Not available.

Fire Fighting Media and Instructions: Not applicable.

Special Remarks on Fire Hazards: Not available.

Special Remarks on Explosion Hazards: Not available.

Section 6: Accidental Release Measures

Small Spill: Absorb with an inert material and put the spilled material in an appropriate waste disposal.

Large Spill:
Absorb with an inert material and put the spilled material in an appropriate waste disposal. Be careful that the product is not present at a concentration level above TLV. Check TLV on the MSDS and with local authorities.

Section 7: Handling and Storage

Precautions:
Do not ingest. Do not breathe gas/fumes/ vapor/spray. Avoid contact with skin. Wear suitable protective clothing. In case of insufficient ventilation, wear suitable respiratory equipment. If ingested, seek medical advice immediately and show the container or the label. Keep away from incompatibles such as oxidizing agents, metals, acids, alkalis.

Storage: Keep container tightly closed. Keep container in a cool, well-ventilated area.

Section 8: Exposure Controls/Personal Protection

Engineering Controls:
Provide exhaust ventilation or other engineering controls to keep the airborne concentrations of vapors below their respective threshold limit value.

Personal Protection:
Safety glasses. Lab coat. Vapor respirator. Be sure to use an approved/certified respirator or equivalent. Gloves.

Personal Protection in Case of a Large Spill:
Splash goggles. Full suit. Vapor respirator. Boots. Gloves. A self contained breathing apparatus should be used to avoid inhalation of the product. Suggested protective clothing might not be sufficient; consult a specialist BEFORE handling this product.

Exposure Limits:
TWA: 25 (ppm) from OSHA (PEL) [United States] TWA: 25 STEL: 100 (ppm) from ACGIH (TLV) [United States] TWA: 170 (mg/m3) from OSHA (PEL) [United States] Consult local authorities for acceptable exposure limits.

Section 9: Physical and Chemical Properties

Physical state and appearance: Liquid.

Odor: Ethereal.

Taste: Not available.

Molecular Weight: 165.83 g/mole

Color: Clear Colorless.

pH (1% soln/water): Not available.

Boiling Point: 121.3°C (250.3°F)

Melting Point: -22.3°C (-8.1°F)

Critical Temperature: 347.1°C (656.8°F)

Specific Gravity: 1.6227 (Water = 1)

Vapor Pressure: 1.7 kPa (@ 20°C)

Vapor Density: 5.7 (Air = 1)

Volatility: Not available.

Odor Threshold: 5 - 50 ppm

Water/Oil Dist. Coeff.: The product is more soluble in oil; log(oil/water) = 3.4

Ionicity (in Water): Not available.

Dispersion Properties: Not available.

Solubility:
Miscible with alcohol, ether, chloroform, benzene, hexane. It dissolves in most of the fixed and volatile oils. Solubility in water: 0.015 g/100 ml @ 25 deg. C It slowly decomposes in water to yield Trichloroacetic and Hydrochloric acids.

Section 10: Stability and Reactivity Data

Stability: The product is stable.

Instability Temperature: Not available.

Conditions of Instability: Incompatible materials

Incompatibility with various substances: Reactive with oxidizing agents, metals, acids, alkalis.

Corrosivity: Non-corrosive in presence of glass.

Special Remarks on Reactivity:
Oxidized by strong oxidizing agents. Incompatible with sodium hydroxide, finely divided or powdered metals such as zinc, aluminum, magnesium, potassium, chemically active metals such as lithium, beryllium , barium. Protect from light.

Special Remarks on Corrosivity: Slowly corrodes aluminum, iron, and zinc.

Polymerization: Will not occur.

Section 11: Toxicological Information

Routes of Entry: Absorbed through skin. Eye contact. Inhalation. Ingestion.

Toxicity to Animals:
WARNING: THE LC50 VALUES HEREUNDER ARE ESTIMATED ON THE BASIS OF A 4-HOUR EXPOSURE. Acute oral toxicity (LD50): 2629 mg/kg [Rat]. Acute dermal toxicity (LD50): >3228 mg/kg [Rabbit]. Acute toxicity of the vapor (LC50): 5200 4 hours [Mouse].

Chronic Effects on Humans:
CARCINOGENIC EFFECTS: Classified A3 (Proven for animal.) by ACGIH. Classified 2A (Probable for human.) by IARC, 2 (Some evidence.) by NTP. MUTAGENIC EFFECTS: Mutagenic for bacteria and/or yeast. May cause damage to the following organs: kidneys, liver, peripheral nervous system, upper respiratory tract, skin, central nervous system (CNS).

Other Toxic Effects on Humans:
Hazardous in case of skin contact (irritant), of inhalation. Slightly hazardous in case of skin contact (permeator), of ingestion.

Special Remarks on Toxicity to Animals:
Lowest Publishe Lethal Dose/Conc: LDL [Rabbit] - Route: Oral; Dose: 5000 mg/kg LDL [Dog] - Route: Oral; Dose: 4000 mg/kg LDL [Cat] - Route: Oral; Dose: 4000 mg/kg

Special Remarks on Chronic Effects on Humans:
May cause adverse reproductive effects and birth defects(teratogenic). May affect genetic material (mutagenic). May cause cancer.

Special Remarks on other Toxic Effects on Humans:
Acute Potential Health Effects: Skin: Causes skin irritation with possible dermal blistering or burns. Symtoms may include redness, itching, pain, and possible dermal blistering or burns. It may be absorbed through the skin with possible systemic effects. A single prolonged skin exposure is not likely to result in the material being absorbed in harmful amounts. Eyes: Contact causes transient eye irritation, lacrimation. Vapors cause eye/conjunctival irritation. Symptoms may include redness and pain. Inhalation: The main route to occupational exposure is by inhalation since it is readily absorbed through the lungs. It causes respiratory tract irritation, . It can affect behavior/central nervous system (CNS depressant and anesthesia ranging from slight inebriation to death, vertigo, somnolence, anxiety, headache, excitement, hallucinations, muscle incoordination, dizziness, lightheadness, disorentiation, seizures, enotional instability, stupor, coma). It may cause pulmonary edema Ingestion: It can cause nausea, vomiting, anorexia, diarrhea, bloody stool. It may affect the liver, urinary system (proteinuria, hematuria, renal failure, renal tubular disorder), heart (arrhythmias). It may affect behavior/central nervous system with symptoms similar to that of inhalation. Chronic Potential Health Effects: Skin: Prolonged or repeated skin contact may result in excessive drying of the skin, and irritation. Ingestion/Inhalation: Chronic exposure can affect the liver(hepatitis,fatty liver degeneration), kidneys, spleen, and heart (irregular heartbeat/arrhythmias, cardiomyopathy, abnormal EEG), brain, behavior/ central nervous system/peripheral nervous system (impaired memory, numbness of extremeties, peripheral neuropathy and other

Section 12: Ecological Information

Ecotoxicity:
Ecotoxicity in water (LC50): 18.4 mg/l 96 hours [Fish (Fatthead Minnow)]. 18 mg/l 48 hours [Daphnia (daphnia)]. 5 mg/l 96 hours [Fish (Rainbow Trout)]. 13 mg/l 96 hours [Fish (Bluegill sunfish)].

BOD5 and COD: Not available.

Products of Biodegradation:
Possibly hazardous short term degradation products are not likely. However, long term degradation products may arise.

Toxicity of the Products of Biodegradation: The product itself and its products of degradation are not toxic.

Special Remarks on the Products of Biodegradation: Not available.

Section 13: Disposal Considerations

Waste Disposal:
Waste must be disposed of in accordance with federal, state and local environmental control regulations.

Section 14: Transport Information

DOT Classification: CLASS 6.1: Poisonous material.

Identification: : Tetrachloroethylene UNNA: 1897 PG: III

Special Provisions for Transport: Marine Pollutant

Section 15: Other Regulatory Information

Federal and State Regulations:
California prop. 65: This product contains the following ingredients for which the State of California has found to cause cancer, birth defects or other reproductive harm, which would require a warning under the statute: Tetrachloroethylene California prop. 65: This product contains the following ingredients for which the State of California has found to cause cancer which would require a warning under the statute: Tetrachloroethylene Connecticut hazardous material survey.: Tetrachloroethylene Illinois toxic substances disclosure to employee act: Tetrachloroethylene Illinois chemical safety act: Tetrachloroethylene New York release reporting list: Tetrachloroethylene Rhode Island RTK hazardous substances: Tetrachloroethylene Pennsylvania RTK: Tetrachloroethylene Minnesota: Tetrachloroethylene Michigan critical material: Tetrachloroethylene Massachusetts RTK: Tetrachloroethylene Massachusetts spill list: Tetrachloroethylene New Jersey: Tetrachloroethylene New Jersey spill list: Tetrachloroethylene Louisiana spill reporting: Tetrachloroethylene California Director's List of Hazardous Substances: Tetrachloroethylene TSCA 8(b) inventory: Tetrachloroethylene TSCA 8(d) H and S data reporting: Tetrachloroethylene: Effective date: 6/1/87; Sunset date: 6/1/97 SARA 313 toxic chemical notification and release reporting: Tetrachloroethylene CERCLA: Hazardous substances.: Tetrachloroethylene: 100 lbs. (45.36 kg)

Other Regulations:
OSHA: Hazardous by definition of Hazard Communication Standard (29 CFR 1910.1200). EINECS: This product is on the European Inventory of Existing Commercial Chemical Substances.

Other Classifications:

WHMIS (Canada):
CLASS D-1B: Material causing immediate and serious toxic effects (TOXIC). CLASS D-2A: Material causing other toxic effects (VERY TOXIC).

DSCL (EEC):
R40- Possible risks of irreversible effects. R51/53- Toxic to aquatic organisms, may cause long-term adverse effects in the aquatic environment. S23- Do not breathe gas/fumes/vapour/spray S26- In case of contact with eyes, rinse immediately with plenty of water and seek medical advice. S37- Wear suitable gloves. S61- Avoid release to the environment. Refer to special instructions/Safety data sheets.

HMIS (U.S.A.):

Health Hazard: 2

Fire Hazard: 0

Reactivity: 0

Personal Protection: g

National Fire Protection Association (U.S.A.):

Health: 2

Flammability: 0

Reactivity: 0

Specific hazard:

Protective Equipment:
Gloves. Lab coat. Vapor respirator. Be sure to use an approved/certified respirator or equivalent. Wear appropriate respirator when ventilation is inadequate. Safety glasses.

Section 16: Other Information

References: Not available.

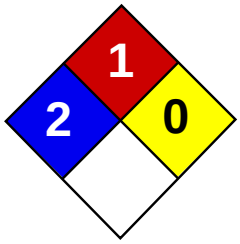
Other Special Considerations: Not available.

Created: 10/10/2005 08:29 PM

Last Updated: 11/01/2010 12:00 PM

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Material Safety Data Sheet
Trichloroethylene MSDS

Section 1: Chemical Product and Company Identification

Product Name: Trichloroethylene

Catalog Codes: SLT3310, SLT2590

CAS#: 79-01-6

RTECS: KX4560000

TSCA: TSCA 8(b) inventory: Trichloroethylene

CI#: Not available.

Synonym:

Chemical Formula: C2HCl3

Contact Information:

Sciencelab.com, Inc.
14025 Smith Rd.
Houston, Texas 77396

US Sales: **1-800-901-7247**
International Sales: **1-281-441-4400**

Order Online: ScienceLab.com

CHEMTREC (24HR Emergency Telephone), call:
1-800-424-9300

International CHEMTREC, call: 1-703-527-3887

For non-emergency assistance, call: 1-281-441-4400

Section 2: Composition and Information on Ingredients

Composition:

Name	CAS #	% by Weight
Trichloroethylene	79-01-6	100

Toxicological Data on Ingredients: Trichloroethylene: ORAL (LD50): Acute: 5650 mg/kg [Rat]. 2402 mg/kg [Mouse]. DERMAL (LD50): Acute: 20001 mg/kg [Rabbit].

Section 3: Hazards Identification

Potential Acute Health Effects: Hazardous in case of skin contact (irritant, permeator), of eye contact (irritant), of ingestion, of inhalation.

Potential Chronic Health Effects:
CARCINOGENIC EFFECTS: Classified + (PROVEN) by OSHA. Classified A5 (Not suspected for human.) by ACGIH. MUTAGENIC EFFECTS: Not available. TERATOGENIC EFFECTS: Not available. DEVELOPMENTAL TOXICITY: Not available. The substance is toxic to kidneys, the nervous system, liver, heart, upper respiratory tract. Repeated or prolonged exposure to the substance can produce target organs damage.

Section 4: First Aid Measures

Eye Contact:

Check for and remove any contact lenses. Immediately flush eyes with running water for at least 15 minutes, keeping eyelids open. Cold water may be used. Do not use an eye ointment. Seek medical attention.

Skin Contact:
After contact with skin, wash immediately with plenty of water. Gently and thoroughly wash the contaminated skin with running water and non-abrasive soap. Be particularly careful to clean folds, crevices, creases and groin. Cover the irritated skin with an emollient. If irritation persists, seek medical attention. Wash contaminated clothing before reusing.

Serious Skin Contact:
Wash with a disinfectant soap and cover the contaminated skin with an anti-bacterial cream. Seek medical attention.

Inhalation: Allow the victim to rest in a well ventilated area. Seek immediate medical attention.

Serious Inhalation:
Evacuate the victim to a safe area as soon as possible. Loosen tight clothing such as a collar, tie, belt or waistband. If breathing is difficult, administer oxygen. If the victim is not breathing, perform mouth-to-mouth resuscitation. Seek medical attention.

Ingestion:
Do not induce vomiting. Loosen tight clothing such as a collar, tie, belt or waistband. If the victim is not breathing, perform mouth-to-mouth resuscitation. Seek immediate medical attention.

Serious Ingestion: Not available.

Section 5: Fire and Explosion Data

Flammability of the Product: May be combustible at high temperature.

Auto-Ignition Temperature: 420°C (788°F)

Flash Points: Not available.

Flammable Limits: LOWER: 8% UPPER: 10.5%

Products of Combustion: These products are carbon oxides (CO, CO2), halogenated compounds.

Fire Hazards in Presence of Various Substances: Not available.

Explosion Hazards in Presence of Various Substances:
Risks of explosion of the product in presence of mechanical impact: Not available. Risks of explosion of the product in presence of static discharge: Not available.

Fire Fighting Media and Instructions:
SMALL FIRE: Use DRY chemical powder. LARGE FIRE: Use water spray, fog or foam. Do not use water jet.

Special Remarks on Fire Hazards: Not available.

Special Remarks on Explosion Hazards: Not available.

Section 6: Accidental Release Measures

Small Spill: Absorb with an inert material and put the spilled material in an appropriate waste disposal.

Large Spill:
Absorb with an inert material and put the spilled material in an appropriate waste disposal. Be careful that the product is not present at a concentration level above TLV. Check TLV on the MSDS and with local authorities.

Section 7: Handling and Storage

Precautions:
Keep locked up Keep away from heat. Keep away from sources of ignition. Empty containers pose a fire risk, evaporate the residue under a fume hood. Ground all equipment containing material. Do not ingest. Do not breathe gas/fumes/ vapour/

spray. Wear suitable protective clothing In case of insufficient ventilation, wear suitable respiratory equipment If ingested, seek medical advice immediately and show the container or the label. Avoid contact with skin and eyes

Storage:
Keep container dry. Keep in a cool place. Ground all equipment containing material. Carcinogenic, teratogenic or mutagenic materials should be stored in a separate locked safety storage cabinet or room.

Section 8: Exposure Controls/Personal Protection

Engineering Controls:
Provide exhaust ventilation or other engineering controls to keep the airborne concentrations of vapors below their respective threshold limit value. Ensure that eyewash stations and safety showers are proximal to the work-station location.

Personal Protection:
Splash goggles. Lab coat. Vapor respirator. Be sure to use an approved/certified respirator or equivalent. Gloves.

Personal Protection in Case of a Large Spill:
Splash goggles. Full suit. Vapor respirator. Boots. Gloves. A self contained breathing apparatus should be used to avoid inhalation of the product. Suggested protective clothing might not be sufficient; consult a specialist BEFORE handling this product.

Exposure Limits:
TWA: 50 STEL: 200 (ppm) from ACGIH (TLV) TWA: 269 STEL: 1070 (mg/m3) from ACGIH Consult local authorities for acceptable exposure limits.

Section 9: Physical and Chemical Properties

Physical state and appearance: Liquid.

Odor: Not available.

Taste: Not available.

Molecular Weight: 131.39 g/mole

Color: Clear Colorless.

pH (1% soln/water): Not available.

Boiling Point: 86.7°C (188.1°F)

Melting Point: -87.1°C (-124.8°F)

Critical Temperature: Not available.

Specific Gravity: 1.4649 (Water = 1)

Vapor Pressure: 58 mm of Hg (@ 20°C)

Vapor Density: 4.53 (Air = 1)

Volatility: Not available.

Odor Threshold: 20 ppm

Water/Oil Dist. Coeff.: The product is equally soluble in oil and water; log(oil/water) = 0

Ionicity (in Water): Not available.

Dispersion Properties: See solubility in water, methanol, diethyl ether, acetone.

Solubility:
Easily soluble in methanol, diethyl ether, acetone. Very slightly soluble in cold water.

Section 10: Stability and Reactivity Data

Stability: The product is stable.

Instability Temperature: Not available.

Conditions of Instability: Not available.

Incompatibility with various substances: Not available.

Corrosivity:
Extremely corrosive in presence of aluminum. Non-corrosive in presence of glass.

Special Remarks on Reactivity: Not available.

Special Remarks on Corrosivity: Not available.

Polymerization: No.

Section 11: Toxicological Information

Routes of Entry: Dermal contact. Eye contact. Inhalation. Ingestion.

Toxicity to Animals:
Acute oral toxicity (LD50): 2402 mg/kg [Mouse]. Acute dermal toxicity (LD50): 20001 mg/kg [Rabbit].

Chronic Effects on Humans:
CARCINOGENIC EFFECTS: Classified + (PROVEN) by OSHA. Classified A5 (Not suspected for human.) by ACGIH. The substance is toxic to kidneys, the nervous system, liver, heart, upper respiratory tract.

Other Toxic Effects on Humans: Hazardous in case of skin contact (irritant, permeator), of ingestion, of inhalation.

Special Remarks on Toxicity to Animals: Not available.

Special Remarks on Chronic Effects on Humans: Passes through the placental barrier in human. Detected in maternal milk in human.

Special Remarks on other Toxic Effects on Humans: Not available.

Section 12: Ecological Information

Ecotoxicity: Not available.

BOD5 and COD: Not available.

Products of Biodegradation:
Possibly hazardous short term degradation products are not likely. However, long term degradation products may arise.

Toxicity of the Products of Biodegradation: The products of degradation are more toxic.

Special Remarks on the Products of Biodegradation: Not available.

Section 13: Disposal Considerations

Waste Disposal:

Section 14: Transport Information

DOT Classification: CLASS 6.1: Poisonous material.

Identification: : Trichloroethylene : UN1710 PG: III

Special Provisions for Transport: Not available.

Section 15: Other Regulatory Information

Federal and State Regulations:
California prop. 65: This product contains the following ingredients for which the State of California has found to cause cancer, birth defects or other reproductive harm, which would require a warning under the statute: Trichloroethylene California prop. 65: This product contains the following ingredients for which the State of California has found to cause cancer which would require a warning under the statute: Trichloroethylene Pennsylvania RTK: Trichloroethylene Florida: Trichloroethylene Minnesota: Trichloroethylene Massachusetts RTK: Trichloroethylene New Jersey: Trichloroethylene TSCA 8(b) inventory: Trichloroethylene CERCLA: Hazardous substances.: Trichloroethylene

Other Regulations: OSHA: Hazardous by definition of Hazard Communication Standard (29 CFR 1910.1200).

Other Classifications:

WHMIS (Canada):
CLASS D-1B: Material causing immediate and serious toxic effects (TOXIC). CLASS D-2B: Material causing other toxic effects (TOXIC).

DSCL (EEC):
R36/38- Irritating to eyes and skin. R45- May cause cancer.

HMIS (U.S.A.):

Health Hazard: 2

Fire Hazard: 1

Reactivity: 0

Personal Protection: h

National Fire Protection Association (U.S.A.):

Health: 2

Flammability: 1

Reactivity: 0

Specific hazard:

Protective Equipment:
Gloves. Lab coat. Vapor respirator. Be sure to use an approved/certified respirator or equivalent. Wear appropriate respirator when ventilation is inadequate. Splash goggles.

Section 16: Other Information

References: Not available.

Other Special Considerations: Not available.

Created: 10/10/2005 08:54 PM

Last Updated: 11/01/2010 12:00 PM

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Material Safety Data Sheet

Helium

Section 1. Chemical product and company identification

Product name : Helium

Supplier : AIRGAS INC., on behalf of its subsidiaries
259 North Radnor-Chester Road
Suite 100
Radnor, PA 19087-5283
1-610-687-5253

Product use : Synthetic/Analytical chemistry.

Synonym : helium (dot); Helium-4; He; o-Helium; UN 1046; UN 1963; Liquid Helium; Helium, Refrigerated Liquid

MSDS # : 001025

Date of Preparation/Revision : **8/27/2010.**

In case of emergency : 1-866-734-3438

Section 2. Hazards identification

Physical state : Gas. [Colorless, Odorless Gas, Cryogenic liquid and gas]

Emergency overview : WARNING!

GAS:
CONTENTS UNDER PRESURE.
Do not puncture or incinerate container.
Can cause rapid suffocation.
May cause severe frostbite.
LIQUID:
Extremely cold liquid and gas under pressure.
Can cause rapid suffocation.
May cause severe frostbite.

Do not puncture or incinerate container.

Contact with rapidly expanding gases or liquids can cause frostbite.

Routes of entry : Inhalation

Potential acute health effects

Eyes : Contact with rapidly expanding gas may cause burns or frostbite. Contact with cryogenic liquid can cause frostbite and cryogenic burns.

Skin : Contact with rapidly expanding gas may cause burns or frostbite. Contact with cryogenic liquid can cause frostbite and cryogenic burns.

Inhalation : Acts as a simple asphyxiant.

Ingestion : Ingestion is not a normal route of exposure for gases. Contact with cryogenic liquid can cause frostbite and cryogenic burns.

Potential chronic health effects : **CARCINOGENIC EFFECTS**: Not available.
MUTAGENIC EFFECTS: Not available.
TERATOGENIC EFFECTS: Not available.

Medical conditions aggravated by over-exposure : Acute or chronic respiratory conditions may be aggravated by overexposure to this gas.

See toxicological information (section 11)

Section 3. Composition, Information on Ingredients

Name	CAS number	% Volume	Exposure limits
Helium	7440-59-7	100	Oxygen Depletion [Asphyxiant]

Section 4. First aid measures

No action shall be taken involving any personal risk or without suitable training.If it is suspected that fumes are still present, the rescuer should wear an appropriate mask or self-contained breathing apparatus.It may be dangerous to the person providing aid to give mouth-to-mouth resuscitation.

Eye contact : Check for and remove any contact lenses. Immediately flush eyes with plenty of water for at least 15 minutes, occasionally lifting the upper and lower eyelids. Get medical attention immediately.

Skin contact : None expected.

Frostbite : Try to warm up the frozen tissues and seek medical attention.

Inhalation : Move exposed person to fresh air. If not breathing, if breathing is irregular or if respiratory arrest occurs, provide artificial respiration or oxygen by trained personnel. Loosen tight clothing such as a collar, tie, belt or waistband. Get medical attention immediately.

Ingestion : As this product is a gas, refer to the inhalation section.

Section 5. Fire-fighting measures

Flammability of the product : Non-flammable.

Products of combustion : No specific data.

Fire-fighting media and instructions : Use an extinguishing agent suitable for the surrounding fire.

Apply water from a safe distance to cool container and protect surrounding area. If involved in fire, shut off flow immediately if it can be done without risk.

Contains gas under pressure. In a fire or if heated, a pressure increase will occur and the container may burst or explode.

Special protective equipment for fire-fighters : Fire-fighters should wear appropriate protective equipment and self-contained breathing apparatus (SCBA) with a full face-piece operated in positive pressure mode.

Section 6. Accidental release measures

Personal precautions : Immediately contact emergency personnel. Keep unnecessary personnel away. Use suitable protective equipment (section 8). Shut off gas supply if this can be done safely. Isolate area until gas has dispersed.

Environmental precautions : Avoid dispersal of spilled material and runoff and contact with soil, waterways, drains and sewers.

Methods for cleaning up : Immediately contact emergency personnel. Stop leak if without risk. Note: see section 1 for emergency contact information and section 13 for waste disposal.

Section 7. Handling and storage

Handling : High pressure gas. Do not puncture or incinerate container. Use equipment rated for cylinder pressure. Close valve after each use and when empty. Protect cylinders from physical damage; do not drag, roll, slide, or drop. Use a suitable hand truck for cylinder movement.

Never allow any unprotected part of the body to touch uninsulated pipes or vessels that contain cryogenic liquids. Prevent entrapment of liquid in closed systems or piping without pressure relief devices. Some materials may become brittle at low temperatures and will easily fracture.

Storage : Cylinders should be stored upright, with valve protection cap in place, and firmly secured to prevent falling or being knocked over. Cylinder temperatures should not exceed 52 °C (125 °F).
For additional information concerning storage and handling refer to Compressed Gas Association pamphlets P-1 Safe Handling of Compressed Gases in Containers and P-12 Safe Handling of Cryogenic Liquids available from the Compressed Gas Association, Inc.

Section 8. Exposure controls/personal protection

Engineering controls : Use only with adequate ventilation. Use process enclosures, local exhaust ventilation or other engineering controls to keep worker exposure to airborne contaminants below any recommended or statutory limits.

Personal protection

Eyes : Safety eyewear complying with an approved standard should be used when a risk assessment indicates this is necessary to avoid exposure to liquid splashes, mists or dusts.

When working with cryogenic liquids, wear a full face shield.

Skin : Personal protective equipment for the body should be selected based on the task being performed and the risks involved and should be approved by a specialist before handling this product.

Respiratory : Use a properly fitted, air-purifying or air-fed respirator complying with an approved standard if a risk assessment indicates this is necessary. Respirator selection must be based on known or anticipated exposure levels, the hazards of the product and the safe working limits of the selected respirator.

The applicable standards are (US) 29 CFR 1910.134 and (Canada) Z94.4-93

Hands : Chemical-resistant, impervious gloves complying with an approved standard should be worn at all times when handling chemical products if a risk assessment indicates this is necessary.

Insulated gloves suitable for low temperatures

Personal protection in case of a large spill : Self-contained breathing apparatus (SCBA) should be used to avoid inhalation of the product.

Product name

helium	Oxygen Depletion [Asphyxiant]

Consult local authorities for acceptable exposure limits.

Section 9. Physical and chemical properties

Molecular weight : 4 g/mole

Molecular formula : He

Boiling/condensation point : -268.9°C (-452°F)

Melting/freezing point : -272.2°C (-458°F)

Critical temperature : -267.9°C (-450.2°F)

Vapor density : 0.14 (Air = 1) Liquid Density@BP: 7.8 lb/ft3 (125 kg/m3)

Specific Volume (ft ^{3}/lb) : 96.1538

Gas Density (lb/ft ^{3}) : 0.0104

Section 10. Stability and reactivity

Stability and reactivity : The product is stable.

Hazardous decomposition products : Under normal conditions of storage and use, hazardous decomposition products should not be produced.

Hazardous polymerization : Under normal conditions of storage and use, hazardous polymerization will not occur.

Section 11. Toxicological information

Toxicity data

Other toxic effects on humans : No specific information is available in our database regarding the other toxic effects of this material to humans.

Specific effects

Carcinogenic effects : No known significant effects or critical hazards.

Mutagenic effects : No known significant effects or critical hazards.

Reproduction toxicity : No known significant effects or critical hazards.

Section 12. Ecological information

Aquatic ecotoxicity

Not available.

Environmental fate : Not available.

Environmental hazards : No known significant effects or critical hazards.

Toxicity to the environment : Not available.

Section 13. Disposal considerations

Product removed from the cylinder must be disposed of in accordance with appropriate Federal, State, local regulation.Return cylinders with residual product to Airgas, Inc.Do not dispose of locally.

Section 14. Transport information

Regulatory information	UN number	Proper shipping name	Class	Packing group	Label	Additional information
DOT Classification	UN1046 UN1963	HELIUM, COMPRESSED Helium, refrigerated liquid	2.2	Not applicable (gas).		**Limited quantity** Yes. **Packaging instruction** **Passenger aircraft** Quantity limitation: 75 kg **Cargo aircraft** Quantity limitation: 150 kg
TDG Classification	UN1046 UN1963	HELIUM, COMPRESSED Helium, refrigerated liquid	2.2	Not applicable (gas).		**Explosive Limit and Limited Quantity Index** 0.125 **Passenger Carrying Road or Rail Index** 75

Helium						
Mexico Classification	UN1046 UN1963	HELIUM, COMPRESSED Helium, refrigerated liquid	2.2	Not applicable (gas).		-

"Refer to CFR 49 (or authority having jurisdiction) to determine the information required for shipment of the product."

Section 15. Regulatory information

United States

U.S. Federal regulations : **United States inventory (TSCA 8b):** This material is listed or exempted.

SARA 302/304/311/312 extremely hazardous substances: No products were found.
SARA 302/304 emergency planning and notification: No products were found.
SARA 302/304/311/312 hazardous chemicals: helium
SARA 311/312 MSDS distribution - chemical inventory - hazard identification: helium: Sudden release of pressure

Clean Water Act (CWA) 307: No products were found.

Clean Water Act (CWA) 311: No products were found.

Clean Air Act (CAA) 112 accidental release prevention: No products were found.

Clean Air Act (CAA) 112 regulated flammable substances: No products were found.

Clean Air Act (CAA) 112 regulated toxic substances: No products were found.

State regulations : **Connecticut Carcinogen Reporting:** This material is not listed.
Connecticut Hazardous Material Survey: This material is not listed.
Florida substances: This material is not listed.
Illinois Chemical Safety Act: This material is not listed.
Illinois Toxic Substances Disclosure to Employee Act: This material is not listed.
Louisiana Reporting: This material is not listed.
Louisiana Spill: This material is not listed.
Massachusetts Spill: This material is not listed.
Massachusetts Substances: This material is listed.
Michigan Critical Material: This material is not listed.
Minnesota Hazardous Substances: This material is not listed.
New Jersey Hazardous Substances: This material is listed.
New Jersey Spill: This material is not listed.
New Jersey Toxic Catastrophe Prevention Act: This material is not listed.
New York Acutely Hazardous Substances: This material is not listed.
New York Toxic Chemical Release Reporting: This material is not listed.
Pennsylvania RTK Hazardous Substances: This material is listed.
Rhode Island Hazardous Substances: This material is not listed.

Canada

WHMIS (Canada) : Class A: Compressed gas.

CEPA Toxic substances: This material is not listed.
Canadian ARET: This material is not listed.
Canadian NPRI: This material is not listed.
Alberta Designated Substances: This material is not listed.
Ontario Designated Substances: This material is not listed.
Quebec Designated Substances: This material is not listed.

Section 16. Other information

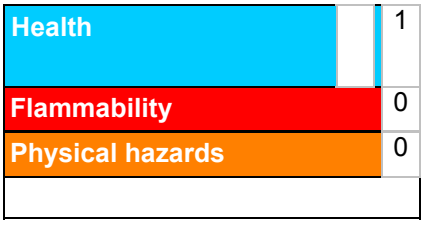
United States

Label requirements : GAS:
CONTENTS UNDER PRESURE.
Do not puncture or incinerate container.
Can cause rapid suffocation.
May cause severe frostbite.
LIQUID:
Extremely cold liquid and gas under pressure.
Can cause rapid suffocation.
May cause severe frostbite.

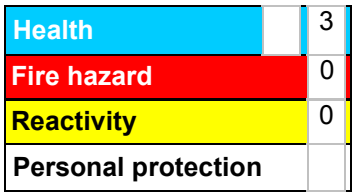
Canada

Label requirements : Class A: Compressed gas.

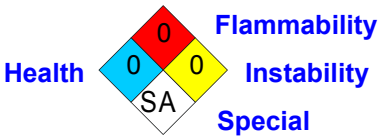
Hazardous Material Information System (U.S.A.) :



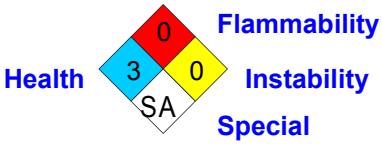
liquid:



National Fire Protection Association (U.S.A.) :



liquid:



Notice to reader

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Final determination of suitability of any material is the sole responsibility of the user. All materials may present unknown hazards and should be used with caution. Although certain hazards are described herein, we cannot guarantee that these are the only hazards that exist.